

**UNIVERSIDAD NACIONAL DE INGENIERÍA**  
**FACULTAD DE INGENIERÍA QUÍMICA Y TEXTIL**



**“ESTUDIO EXPERIMENTAL DEL USO DE TIERRAS  
DIATOMACEAS EN LA PRODUCCION DE CONCRTEOS  
DE ALTA RESISTENCIA”**

**TESIS**

**PARA OPTAR EL TITULO PROFESIONAL DE :  
INGENIERO QUIMICO**

**PRESENTADO POR:  
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**LIMA-PERU**

**2007**

A mis padres, José y Guillermina

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## RESUMEN

La construcción de edificios de gran altura, como también las obras públicas de gran envergadura, impulsó el desarrollo tecnológico de la industria del concreto para mejorar sus diferentes características técnicas, principalmente elevar la resistencia a la compresión a un nivel por encima del concreto convencional.

Investigaciones, tanto básicas como experimentales a fin de obtener los denominados Concretos de Alta Resistencia (CAR), concluyeron en la necesidad de adicionar al concreto convencional otros minerales, entre las cuales destacan las rocas puzolánicas por su importante actividad química. Actualmente el Concreto de Alta Resistencia, principalmente a base de Microsilíce importada, es un desarrollo tecnológico que la industria concretera nacional hace uso en las obras de ingeniería civil que lo requieran.

La diatomita es una roca silíceas de origen orgánico del cual el Perú posee yacimientos en explotación. El propósito principal del presente trabajo es determinar, mediante actividades experimentales, cual sería el rango de proporcionamiento de la diatomita nacional que permita sustituir a la Microsilíce en los diferentes diseños de CAR utilizados por la industria nacional y que igualen o mejoren sus características mecánicas.

Este trabajo consta de cinco partes, la primera Estado del Arte de la Ciencia y la Técnica, dividido en cuatro títulos, expone los conceptos generales acerca del concreto y los resultados de la exhaustiva investigación bibliográfica realizada acerca de las diatomeas enfocándose principalmente en su actividad química a fin de comprender y otorgar sustento científico a las actividades experimentales por desarrollar, la segunda Ensayos Experimentales Preliminares en la cual se realiza interpretación y análisis del comportamiento de algunas variables en prueba, previo a los Ensayos Experimentales Finales, que representa la tercera parte, en la cual se compara propiedades del concreto tanto en estado fresco como endurecido. En la cuarta parte se evalúa el Costo-Beneficio del proyecto y finalmente las Conclusiones y Recomendaciones.

El presente proyecto-tesis demuestra que la diatomita puede ser utilizada como adición mineral, en reemplazo de la Microsilíce, para producir CAR de una

resistencia a la compresión que varía desde 40 a 80 MPa empleando una proporción de 5 a 10% de reemplazo de cemento.

El apoyo recibido por los profesionales de la empresa UNICON SA, propulsora del presente proyecto experimental, ha permitido completar y entender los conocimientos propios a la especialidad de la industria de producción de concretos CAR.

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# CAPÍTULO I: ESTADO DEL ARTE DE LA CIENCIA Y LA TECNICA

## 1.1. EL CONCRETO

### 1.1.1. Generalidades

El concreto es el material constituido por la mezcla en ciertas proporciones de cemento, agua, agregados y opcionalmente aditivos. En su elaboración, el concreto se presenta inicialmente como una mezcla fluida para luego pasar a ser una estructura plástica y moldeable, industrialmente denominado concreto fresco, para finalmente adquirir una consistencia rígida con propiedades aislantes y resistentes, que lo hace un concreto endurecido ideal para la construcción (Fig. 1.1).

La pasta, compuesta de cemento y agua, cubre la superficie del agregado fino y grueso. El concreto es un material híbrido que fusiona las características de sus componentes. En la Fig. 1.2 se muestra las proporciones generales para un concreto normal.

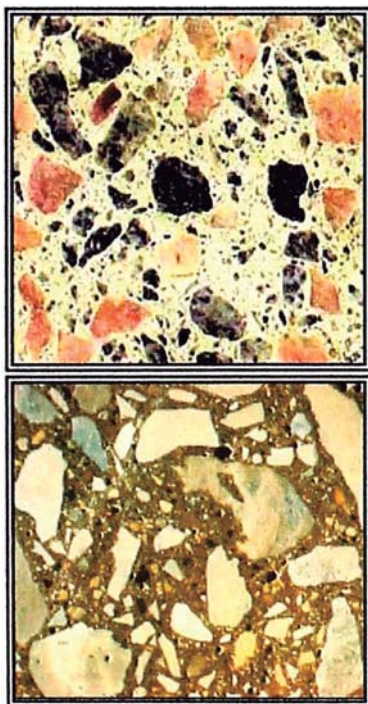


Fig. 1.1. Rocas de Concreto

<b>Aditivos= 0.1 a 0.2%</b>
<b>Aire = 1 a 3 %</b>
<b>Cemento = 7 a 15 %</b>
<b>Agua = 15 a 22 %</b>
<b>Agregados 60 % a 75 %</b>

Fig. 1.2. Proporciones típicas en volumen absoluto de los componentes del Concreto.

Como cualquier material, el concreto se contrae al bajar la temperatura, se dilata si esta aumenta, se ve afectado por sustancias agresivas y se rompe si es sometido a esfuerzos mecánicos que superan sus posibilidades, por lo que responde perfectamente a las leyes físicas y químicas.

### 1.1.2. Tipos de Concreto

El desarrollo tecnológico de la industria del concreto ha determinado que en la actualidad exista una gran variedad de tipos de concreto. Por ejemplo tenemos:

- De acuerdo a su Resistencia a la Compresión:
  - De Baja Resistencia:  
Menos de 20 MPa (204 Kgf/cm<sup>2</sup>)
  - De Resistencia Moderada o Concreto Normal (Convencional):  
De 20 a 40 MPa (204 a 408 Kgf/cm<sup>2</sup>)
  - De Alta Resistencia:  
Mas de 40 MPa (408 Kgf/cm<sup>2</sup>)
  
- De acuerdo al tipo de Agregado:
  - De Peso Normal (2 300 a 2 400 Kg/m<sup>3</sup>)
  - Concreto Ligero (agregados de baja densidad como el polipropileno)
  - Concreto Pesado (agregados de alta densidad)
  
- Otros tipos de concreto:

Actualmente se esta utilizando el Concreto Superfluidificado para colocaciones masivas, que bajo condiciones de clima caliente, no requieren compactación mecánica (Vibrado). En los últimos 25 años, se ha utilizado con éxito el Concreto de Contracción Controlada que contiene cementos y aditivos expansivos para contrarrestar el problema de la retracción del concreto al secarse produciendo agrietamiento, así mismo, se han desarrollado concretos que contienen polímeros, los cuales muestran muy baja permeabilidad y una excelente resistencia química.

## **1.2. CONCRETO DE ALTA RESISTENCIA (CAR)**

### **1.2.1. Generalidades**

El concreto de alta resistencia es algunas veces considerado como un nuevo material, su desarrollo ha sido gradual. Por muchos años, concreto con resistencias a la compresión por encima de los 418 Kg/cm<sup>2</sup> (41 MPa) solo se alcanzaba en países tecnológicamente desarrollados. Sin embargo, en años recientes, las aplicaciones de Concreto de Alta Resistencia (CAR) se han incrementado, y ahora son usados en muchas partes del mundo.

El crecimiento ha sido posible como resultado del reciente desarrollo en la tecnología de los aditivos reductores de agua y a la demanda de concretos de resistencias más altas. [4]

En los años 70 se produjo un desarrollo significativo de la industria del concreto debido a las investigaciones realizadas en Japón y Alemania sobre nuevos aditivos basados en moléculas orgánicas que permitieron una reducción drástica en el contenido de agua, y en Canadá, a la necesidad de encontrarle una aplicación al subproducto principal de la industria de las ferro-aleaciones, la microsilice.

Estudios experimentales [19] muestran que, en muchos aspectos, la microestructura y las propiedades del concreto con resistencias a la compresión por encima de 42 MPa, son considerablemente diferentes de aquellas del concreto convencional, así como también una mejor resistencia al ataque de agentes agresivos y una baja porosidad así como menor permeabilidad.

En los últimos 15 años, el CAR ha evolucionado gradualmente produciendo concreto de resistencia cada vez más alta. [23]

### **1.2.2. Diferencia entre un CAR y un Concreto Convencional**

Los componentes de un concreto de alta resistencia son similares a un concreto convencional hasta un cierto punto.

Tenemos:

Los CAR contienen adiciones minerales como la microsilice, mientras que, los concretos convencionales no lo contienen.

- En un CAR es imprescindible tener la menor cantidad de agua necesaria para que se realice la hidratación completa del cementante.
- Lo dicho anteriormente conllevaría a la necesidad de la utilización de superplastificantes lo que un concreto normal no requiere.
- El agregado tiene que ser cuidadosamente seleccionado debido a que en CAR se utilizan agregados más pequeños que en un concreto normal, minimizando la generación de microgrietas.

### **1.2.3. Materias Primas, Insumos y Aditivos en el CAR**

El avance de la tecnología del concreto ha otorgado como resultado se desarrollen y utilicen una gran variedad de insumos dependiendo de la aplicación del concreto. Entre los principales materiales tenemos:

- Cemento Pórtland
- Adiciones Minerales
- Agregados (Áridos)
- Aditivos
- Agua

#### **1.2.3.1. Cemento Pórtland**

El cemento Pórtland es un producto artificial, que se obtiene por la transformación de una mezcla de calizas, arcillas y otros minerales extraída de canteras. Esta materia prima finamente molida y homogenizada, es llevada a altas temperaturas (1,000°C – 1,500°C), a través de un horno (rotativo o vertical), de donde se obtiene un producto intermedio denominado CLINKER, el cual, después de molerse finamente con alrededor de 5% en peso de yeso ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) se obtiene el cemento. Para un mejor entendimiento de las características de este insumo, componente principal del concreto, se resume:

- La composición química y mineralógica de un cemento,
- Tipos de cemento
- Calidad en el cemento y;
- Determinación del tipo de cemento a utilizar en los CAR:



**Composición Química y Mineralógica:**

En el Cuadro 1.1 se hace un resumen de las principales características de los compuestos mineralógicos del Clínter del cemento.

**Cuadro 1.1. Composición Mineralógica del CLINKER. Características.**

Nombre	Silicato Tricálcico	Silicato Dicálcico	Aluminato Tricálcico	Ferro-Aluminato Tetracálcico
Composición aproximada	$3\text{CaO} \cdot \text{SiO}_2$	$\beta$ $2\text{CaO} \cdot \text{SiO}_2$	$3\text{CaO} \cdot \text{Al}_2\text{O}_3$	$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$
Formula abreviada	$\text{C}_3\text{S}$	$\beta \text{C}_2\text{S}$	$\text{C}_3\text{A}$	$\text{C}_4\text{AF}$
Nombre común	Alita	Belita	-	Fase ferrita
<b>Proporciones de los compuestos presentes (%)</b>				
Variación	35-65	10-40	0-15	5-15
Promedio en el cemento ordinario	50	25	8	8
Velocidad de Hidratación	Media	Lenta	Rápida	Media
<b>Contribución a la resistencia</b>				
Edad temprana	Buena	Pobre	Buena	Buena
Ultima	Buena	Excelente	Media	Media
Calor de hidratación	Medio	Bajo	Alto	Medio
Típico (cal/g)	120	60	320	100

Generalmente en la industria cementera, los resultados de los análisis químicos del cemento Pórtland son reportados en términos de óxidos de los componentes mineralógicos presentes. Los principales óxidos son los siguientes:

**Cuadro 1.2.** Óxidos de los Componentes del Cemento

Nombre	Óxido	Abreviatura
Cal	CaO	C
Silice	SiO <sub>2</sub>	S
Alúmina	Al <sub>2</sub> O <sub>3</sub>	A
Óxido férrico	Fe <sub>2</sub> O <sub>3</sub>	F
Trióxido de azufre	SO <sub>3</sub>	Ŝ
Magnesio	MgO	M
Óxido de sodio	Na <sub>2</sub> O	N
Óxido de potasio	K <sub>2</sub> O	K

Las abreviaturas de los óxidos, han sido aceptadas en la industria mundial del cemento. El SO<sub>3</sub> nunca se encuentra como tal, sino combinado, formando por ejemplo el yeso, el cual es añadido como regulador del fraguado ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O} = \text{C}\hat{\text{S}}\text{H}_2$ )

#### Tipos de Cemento Pórtland:

De acuerdo a las Normas Técnicas Peruanas (basadas en las normas internacionales ASTM), los cementos están clasificados:

1. Cementos Pórtland Comunes.
2. Cementos Pórtland con Inclusión de Aire
3. Cementos Pórtland Adicionados

Existen además otros tipos de cemento, que no son de uso común, solo para trabajo muy específicos, tales como: Cementos Expansivos, Cementos de Alta Alúmina, Cementos para Pozos de petróleo, Cementos Hidrofóbicos, Cementos Impermeables, Cementos Blancos, Cementos de Fraguado Regulado.

Se hará una descripción breve de los Cementos Pórtland Comunes pues son los más utilizados. Tenemos varios tipos especificados en la Norma ASTM C150, Cuadro 1.3., dependiendo cada uno de su uso

especifico y se diferencian entre ellos en las exigencias de ciertos requerimientos físicos y químicos, tanto máximos como mínimos.

**Cuadro 1.3.** Tipos de Cemento Pórtland

<b>TIPO</b>	<b>DESCRIPCION</b>
TIPO I	De uso general, cuando no se requiere ninguna propiedad especial.
TIPO II	De resistencia moderada a los sulfatos o calor moderado de hidratación. Contenido máximo de $C_3A$ es 8% y límite opcional máximo de 58% en la suma de $C_3A$ y $C_3S$ .
TIPO III	De alta resistencia inicial, contenido máximo de $C_3A$ de 15%, superficie específica mas alta aprox. 500 $m^2/Kg$ Blaine, en lugar de 330-400 $m^2/Kg$ del Cemento Pórtland Tipo I.
TIPO IV	De bajo calor de hidratación, límites máximos de 35% y 7% de $C_3S$ y $C_3A$ respectivamente y requiere un mínimo de 40% de $C_2S$ .
TIPO V	De alta resistencia a los sulfatos. Límite máximo de 5% de $C_3A$ .

Las diferentes marcas existentes en el mundo tienen distintas características de resistencia debido a las variaciones en su composición y en su finura, pero tienen que estar dentro de los límites que permite la Norma ASTM C150 para el tipo de cemento especificado.

Algunas empresas colocan algún sufijo al tipo de cemento para indicar alguna característica especial, por ejemplo la compañía Cementos Lima emplea el sufijo BA (Cemento Tipo I BA), para indicar que es Cemento Pórtland Tipo I bajo en álcalis.

### Calidad del Cemento Pórtland:

Las exigencias químicas y físicas que tiene que cumplir cada tipo de cemento Pórtland están señalizados en la Norma ASTM C150.

Los principales puntos de interés por verificar en un cemento manufacturado son los siguientes:

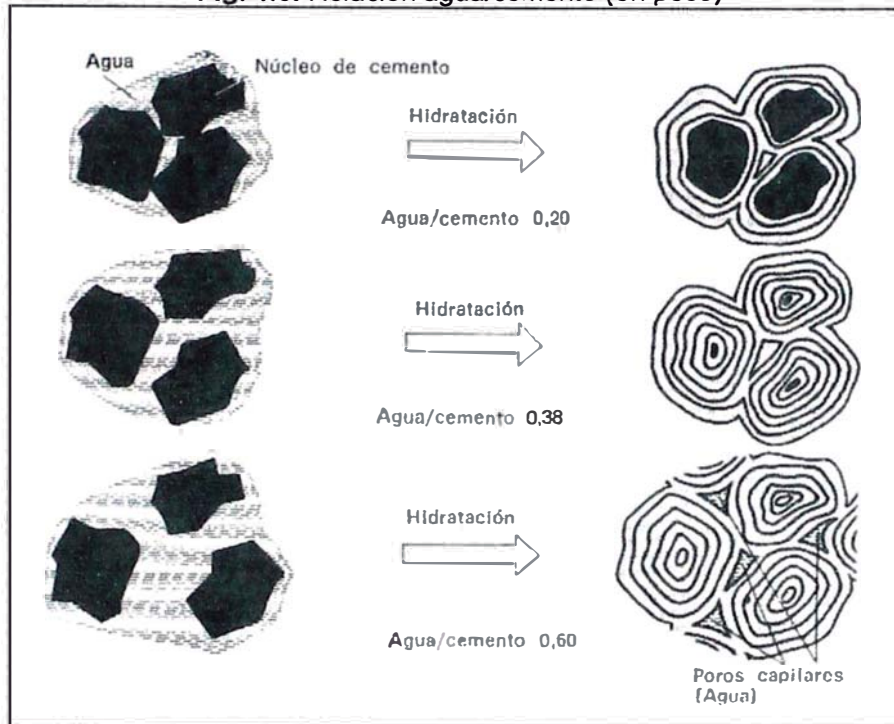
- Finura
- Composición Mineralógica del Clínter
- Presencia del  $C_3A$
- Densidad
- Residuos Insolubles (RI)
- Pérdida al fuego
- Contenido de MgO (periclusa)
- Contenido de CaO libre
- Contenido de  $SO_3$

### Tipo Cemento Pórtland a utilizar para producir CAR:

Para la producción de CAR no hay un criterio específico para determinar el tipo de cemento a emplear. Existen características deseables en un CAR tales como resistencia a la compresión, durabilidad y comportamiento reológico para lo cual algunos autores consideran que el Cemento Pórtland Tipo V es el más adecuado por su bajo contenido en  $C_3A$  (AÍTCIN, 2000). Investigaciones han demostrado que se pueden obtener un CAR con características aceptables empleando Cemento Pórtland Tipo I, siempre y cuando la relación agua/cemento ( $a/c$ ) sea baja, entre 0.35 y 0.38 (ver Fig. 1.3.), con la finalidad de conseguir el máximo de resistencia y sea lo suficientemente impermeable que le permita tener una buena durabilidad.

Para CAR se recomienda un alto contenido de cemento, generalmente por encima de 400 kg de Cemento/ $m^3$  de concreto. [19] El contenido de cemento de aproximadamente 600 kg de cemento /  $m^3$  de concreto y niveles más altos se ha encontrado que resultan inadecuados.

**Fig. 1.3.** Relación agua/cemento (en peso)



### 1.2.3.2. Adiciones Minerales

Son minerales de origen natural o de subproductos industriales, que se incorporan al cemento en diferentes porcentajes, generalmente en molienda conjunta o directamente en el concreto, con el fin de mejorar sus propiedades.

El empleo de adiciones minerales en los aglomerantes hidráulicos es de antigua data. Los romanos las utilizaron perennizando sus estructuras en base a morteros de cal y puzolana. Su progresión ha sido notoria, a partir de la primera crisis del petróleo de 1974 y posteriormente con las regulaciones ambientales. En la actualidad es común el uso de cementos adicionados (puzolanas, escorias, materiales calizos). Una gran mayoría de países han introducido en las Normas de Cemento diferentes tipos de adiciones minerales. En Perú las adiciones puzolánicas y de hidraulicidad latente datan desde los años 1966 y 1967 cuando se fabricó cemento Pórtland de escorias.

Ventajas:

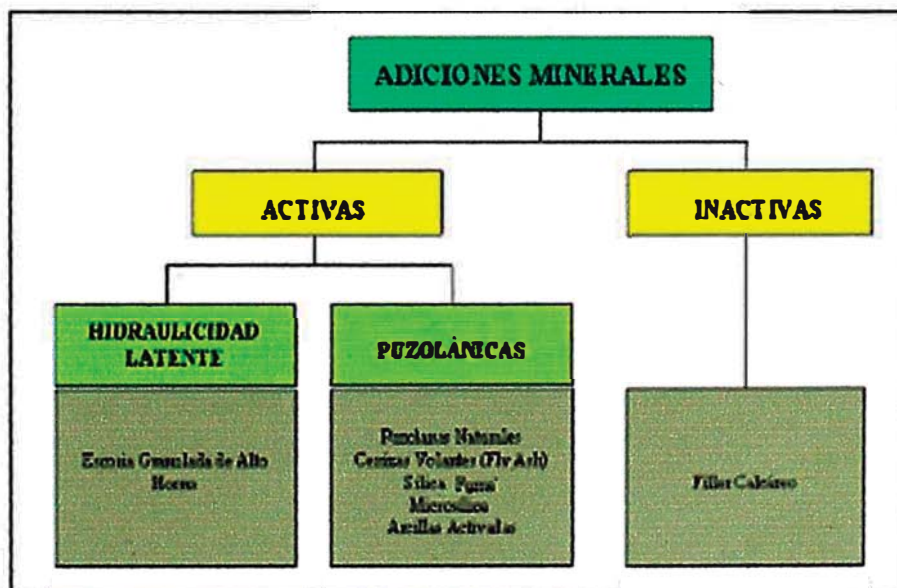
- Mejoran la resistencia y otorgan mayor durabilidad al concreto
- Reducen el agrietamiento térmico debido al menor calor de hidratación.
- Mejoran la impermeabilidad debido al refinamiento de los poros.
- Permite el ahorro de energía no renovable.
- Reducción de la emisión de CO<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub> y material particulado.
- Permite el aprovechamiento de materiales contaminantes (subproductos industriales) como sucede con las escorias.

Clasificación:

## a. De acuerdo a su actividad Química:

La Figura 1.4 muestra la clasificación de las adiciones minerales, de acuerdo a su actividad química en el concreto. Mas adelante solo se describirán las adiciones minerales puzolánicas, por ser el material por adicionar objeto del proyecto-tesis

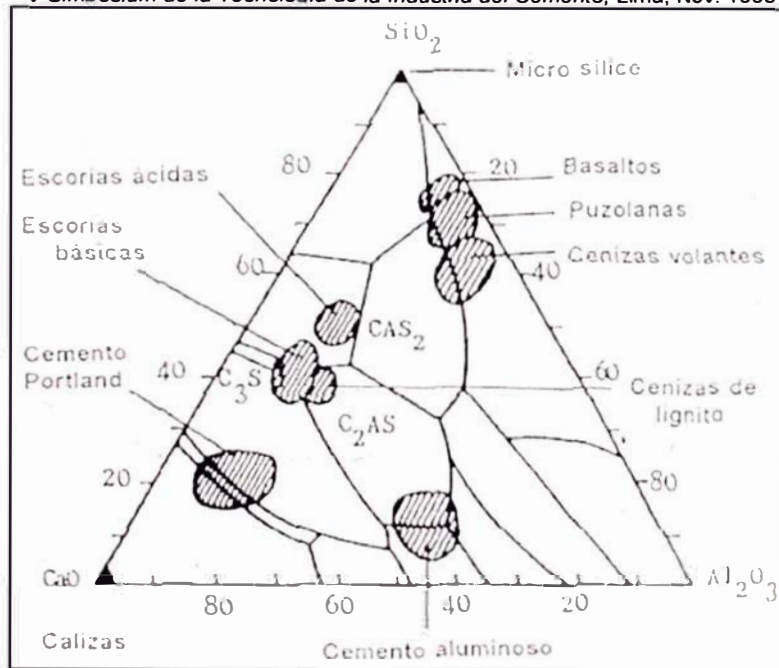
Fig. 1.4. Clasificación de la Adiciones Minerales de acuerdo a su actividad química



b. De acuerdo a su composición Química:

El siguiente diagrama de fases  $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ , muestra las principales adiciones minerales, las cuales se diferencian por su composición química.

**Fig. 1.5.** Principales Componentes Químicos de las Adiciones minerales  
"Cementos Pórtland con adiciones minerales" ASOCEM;  
V Simposium de la Tecnología de la Industria del Cemento; Lima; Nov. 1993



#### Adiciones Minerales Puzolánicas

Llamada también puzolanas, son materiales naturales o artificiales (estos últimos como sub-productos industriales), de material silíceo o sílico-aluminoso. Estas adiciones pueden tener poca o ninguna actividad hidráulica pero, presentan actividad puzolánica, cuya reacción se describe en el punto 1.2.4.2. En el Cuadro 1.4, se presenta una breve descripción de los diferentes tipos de puzolana. Para la presente tesis las adiciones minerales que se utilizarán son la diatomita y la microsilice.

**Cuadro 1.4. Características de los minerales con actividad Puzolánica [19]**

<b>Puzolana</b>	<b>Naturaleza</b>	<b>Composición Química y Mineralógica</b>
Ceniza Volante Alta en Calcio	Subproducto Industrial de centrales termoeléctricas, utilizan carbón pulverizado como combustible.	Silicatos (Ca, Mg, Al, y álcalis), Cuarzo, C <sub>3</sub> A, Carbón y cal libre. Esferas sólidas de diámetros menores que 20µm. La superficie de las partículas es lisa
Microsilíce	Subproducto Industrial de reducción del cuarzo con carbón para la producción de silicio o aleaciones de ferrosilicio	Silíce amorfa, con 90% a 96% de SiO <sub>2</sub> . Polvo extremadamente fino: esferas sólidas de 0.1µm de diámetro promedio.
Ceniza de Cáscara de Arroz	Subproducto Industrial Producida por la calcinación controlada de la cáscara de arroz	Silíce puro en forma no cristalina. Las partículas son generalmente menores que 45µm
Ceniza volante Baja en calcio	Subproducto Industrial	Silicatos (Al, Fe y álcalis), cuarzo, mullita, silimanita, hematita y magnetita. Esferas de 20µm de diámetro promedio.
Vidrios Volcánicos	Natural: Minerales Volcánicos	Formado por erupciones de carácter explosivo, en pequeñas partículas que son templadas a temperatura ambiente, originando estado vítreo.
Turbas volcánicas	Natural	Producto de la acción hidrotermal sobre las cenizas volcánicas y de su posterior cementación diagenética.
Arcillas Calcinadas O Lulitas	Natural	Estructura cristalina destruida mediante tratamiento térmico a 600 y 900 °C.
Tierras Diatomáceas	Natural: Origen Orgánico	Depósitos de caparzones silíceos de microscópicas algas acuáticas unicelulares (diatomeas).

Las Normas ASTM especifican requerimientos químicos y físicos de las puzolanas cuando serán parte de la composición del cemento o cuando van a ser adicionados directamente al concreto. Para el presente trabajo es importante lo segundo.



Las especificaciones técnicas de las puzolanas naturales y cenizas volantes como adición mineral al concreto, están descritas en la siguiente norma:

*ASTM C618: Standard Specification for fly-ash and raw or calcined Natural Pozzolan for use as a Mineral Admixture in Portland Cement Concrete.*

Dicha Norma clasifica a las puzolanas en las siguientes clases:

- Clase N: Puzolanas calcinadas o naturales tales como las tierras diatomáceas, los horstemos opalinos, turbas y cenizas volcánicas y materiales calcinados como las arcillas o lulitas.
- Clase F: Ceniza volante generalmente producida por la quema de carbón antracita o bituminoso
- Clase C: Ceniza volante generalmente producida por la quema de lignito o de carbón sub-bituminoso (además de ser Puzolánica esta ceniza también es cementante)

Los Cuadros 1.5 y 1.6 muestran los requisitos químicos y físicos, de acuerdo a la norma ASTM C618.

**Cuadro 1.5. Puzolanas. Requisitos Químicos**

Requisitos ASTM C618	Clase de Adición Mineral		
	N	F	C
Dióxido de silicio + óxido de aluminio + óxido de hierro, min., %	70.0	70.0	50.0
Trióxido de azufre (SO <sub>3</sub> ), máx., %	4.0	5.0	5.0
Contenido de Humedad, máx., %	3.0	3.0	3.0
Perdida por Calcinación, máx., %	10.0	6.0	6.0

**Cuadro 1.6. Puzolanas. Requisitos Físicos**

Requisitos ASTM C618	Clase de Adición Mineral		
	N	F	C
Fineza: Cantidad retenida en el tamizado vía húmeda en la malla de 45 µm (N° 325), máx., %	34	34	34
Índice de actividad resistente: (i) Con cemento Pórtland, a 7 días, min., % Con cemento Pórtland, a 28 días, min., %	75 75	75 75	75 75
Demanda de agua, máx., % del control	115	105	105
Estabilidad: (ii) Expansión, contracción en autoclave, máx., %	0.8	0.8	0.8
Requisitos de uniformidad: Densidad, máxima variación del promedio, % Porcentaje retenido en 45 µm (N° 325), variación máx., puntos de % del promedio	5 5	5 5	5 5

(i) El índice de actividad de resistencia con cemento Pórtland no debe ser considerado como una medida de la resistencia a la compresión del concreto

(ii) Si la adición mineral constituye más del 20% en peso del material cementante en el diseño de mezcla proyectado.

### Adiciones Minerales en el CAR

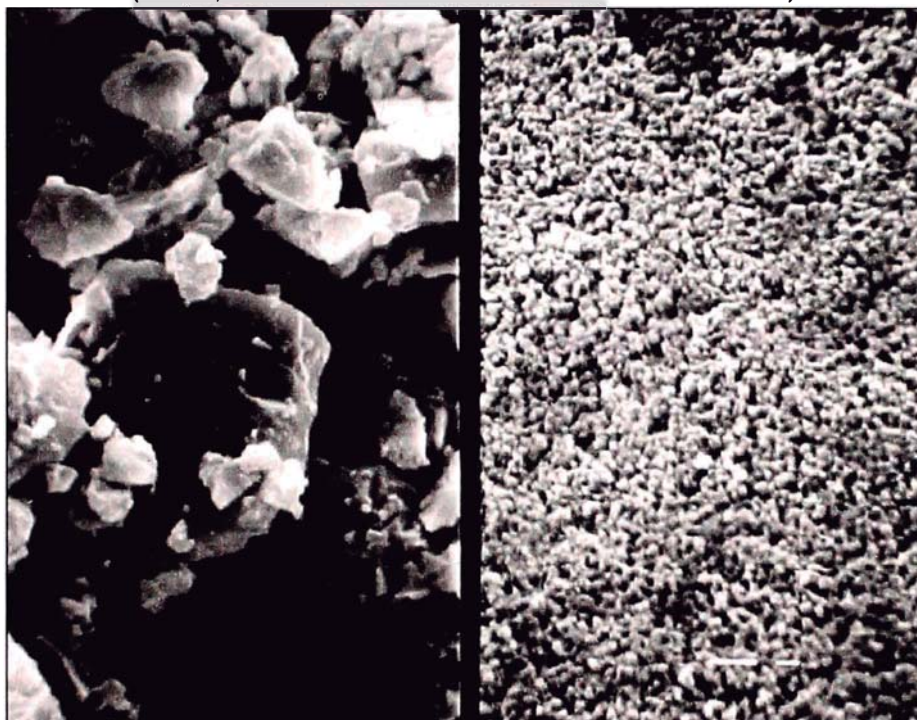
Como adiciones minerales para obtener CAR se tienen la Microsilice, el fly ash y las escorias sub producto de la industria metalúrgica. A nivel mundial es preferido el uso de la microsilice por sus reconocidas propiedades físicas y químicas. Se recomienda que el contenido de microsilice debe estar entre 5 y 15% [23].

En el Perú la Microsilice es relativamente costosa, lo cual llevó a buscar un producto alternativo. La Diatomita, que es una puzolana natural con alto contenido de sílice, resulta ser una buena alternativa.

La Microsilice es un subproducto de la reducción de cuarzo de alta pureza con carbón en hornos de arco eléctrico a temperaturas mayores a los 2000 °C para la producción de silicio o aleaciones de ferrosilicio. A esta temperatura se producen vapores de SiO, los que oxidados y condensados en zonas de baja temperatura se forman partículas esféricas muy pequeñas de sílice no cristalizada. El material retenido por filtración de los gases de escape del horno, en

filtros de mangas, poseen un diámetro promedio de  $0.1 \mu\text{m}$  y áreas de superficie que oscilan en un rango de 20 a  $25 \text{ m}^2/\text{g}$ .

**Fig. 1.6.** Fotografía de granos de Cemento Pórtland (izq) y partículas de Microsilice (der). La línea blanca más grande en el lado izquierdo representa una micra. (Holland, Silica Fume User's Manual-2005-Silica Fume Association)



**Cuadro 1.7.** Comparación del Tamaño de la Microsilice con otros componentes del concreto (Holland, Silica Fume User's Manual-2005-Silica Fume Association)

Material	Tamaño Nominal	Tamaño SI
Partícula de Microsilice	N/A	$0.5 \mu\text{m}$
Grano de Cemento	N° 325	$45 \mu\text{m}$
Grano de Arena	N° 8	$2.36 \text{ mm}$
Partícula de Agregado	$\frac{3}{4}$ "	$19.0 \text{ mm}$

La microsilice tiene cualidades propias de una puzolana al ser altamente reactiva, su velocidad de reacción es superior al de las puzolanas naturales. Su alta fineza permite llenar espacios entre las partículas de cemento y especialmente los poros generados por los agregados, llamado también zona de interfase, en donde las partículas de cemento no pueden llegar, cuyas propiedades influyen en algunas propiedades del concreto. Como resultado podemos decir que la alta reactividad de la microsilice y su fineza conllevan a

una mayor cohesión de la mezcla, reducción de la segregación, disminución de la porosidad y por tanto el aumento de la resistencia a la compresión y la disminución de la permeabilidad por líquidos.

### **1.2.3.3. Agregados**

Son un conjunto de partículas de origen natural o artificial, que pueden ser tratadas o elaboradas.

Generalmente ocupan el 60% del volumen del concreto, se agrega para otorgarle estabilidad volumétrica al concreto. Su contribución es principalmente física.

#### Clasificación de los agregados para el concreto

- a) Por su Procedencia,
  - Agregados Naturales
  - Agregados Artificiales
- b) Por su Gradación,
  - Agregado Grueso
  - Agregado Fino
- c) Por su Gravedad Especifica (Ge),
  - Normales ( $Ge = 2.5$  a  $2.75$ )
  - Ligeros ( $Ge < 2.5$ )
  - Pesados ( $Ge > 2.75$ )

Los agregados están normalizados bajo la ASTM C33 Standard Specification for Concrete Aggregates. Las características físicas y químicas de los agregados, tienen una influencia determinante sobre las propiedades del concreto tanto en estado fresco (trabajabilidad y consistencia al estado plástico) como endurecido (durabilidad, resistencia y cambio de volumen).

#### Características Físicas y Químicas:

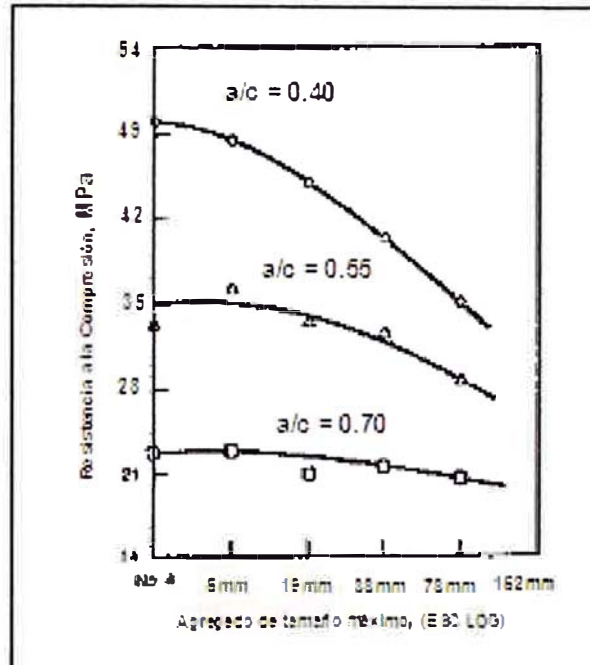
Las fundamentales son las siguientes:

- Granulometría  
*ASTM C136 Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates*
- Peso Específico y Absorción (Porosidad)  
*ASTM C127 Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate*  
*ASTM C128 Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Fine Aggregate*
- Resistencia a la Abrasión  
*ASTM C131 Test Method for Resistance to Degradation of Small-Size Coarse Aggregate by Abrasion and Impact in Los Angeles Machine*
- Humedad  
*ASTM C566 Standard Test Method for Total Evaporable Moisture Content of Aggregate by Drying*
- Material menor a 75  $\mu\text{m}$   
*ASTM C117 Standard Test Method for Materials Finer than 75- $\mu\text{m}$  (No. 200) Sieve in Mineral Aggregates by Washing*
- Peso Unitario  
*ASTM C29 Standard Test Method for Bulk Density ("Unit Weight") and Voids in Aggregate*

Agregados en CAR:

Para CAR se recomienda emplear agregados menores o iguales a 19mm como tamaño del agregado grueso. De acuerdo al ASTM C33 esto correspondería a un agregado Huso 67. Se recomienda este tamaño de agregado debido que agregados gruesos de mayor tamaño forman una zona de interfase cemento agregado más débil conteniendo más microgrietas [19]. Además, los agregados más grandes son más débiles que los agregados de menor tamaño [23]. Como se aprecia en la Gráfica 1.1., la tendencia de la resistencia

del concreto es disminuir conforme aumenta el tamaño del agregado.



**Gráfico 1.1.** Influencia del tamaño del Agregado Grueso y de la relación agua/cemento en la resistencia del concreto (De W. A. Cordon y H. A. Gillespie, I. ACI, Proc., Vol. 60, N° 8, 1963)

#### 1.2.3.4. Aditivos en el Concreto

Son materiales que modifican ciertas características del concreto, gracias a su acción química o fisicoquímica. Estos aditivos mejoran las propiedades del concreto tanto en estado endurecido o en estado fresco, incrementa la eficiencia de las adiciones minerales y mejora la economía de la producción del concreto.

El uso de un aditivo o la combinación de aditivos es solamente una estrategia para alcanzar los resultados deseados.

##### Normalización de los Aditivos:

Hoy en día, los aditivos del concreto son esenciales en la tecnología del concreto. Ellos varían ampliamente en su composición química y muchos realizan más de una función. La ASTM cuenta con especificaciones por separado para los diferentes tipos de aditivos, las cuales se citan a continuación; Cuadro 1.8.

**Cuadro 1.8. Normalización de los Aditivos**

<b>Norma</b>	<b>Clase de Aditivos</b>
ASTM C260	Aditivos Incorporadores de Aire
ASTM C494	Aditivos Químicos Reductores de agua y Controladores de Fragua
ASTM D 98	Cloruro de Calcio
ASTM C 869	Agente Espumoso
ASTM C 1141	Aditivos para Shotcrete
ASTM C 1017	Concreto fluido
ASTM C 937	Grout fluidificante
ASTM C 979	Pigmentos

#### Aditivos Químicos Reductores de Agua y Controladores de Fragua (ASTM C494)

Estos aditivos reductores de agua representan compuestos muy importantes en la tecnología del concreto y para el presente trabajo. También son denominados plastificantes. Su desarrollo ha sido gradual, incrementándose cada vez su capacidad de reducir el volumen de agua por agregar.

De acuerdo al desarrollo de la tecnología de aditivos reductores de agua pueden clasificarse de la siguiente manera:

- Reductores de Agua o de Primera Generación (Lignosulfonatos, Gluconatos o derivados de polisacáridos, Sales de Hidrocarburos y otras resinas). Permiten reducir el agua hasta un 15%.
- Superplastificantes tradicionales. Sales de condensado de naftaleno sulfonado y formaldehído (SNF), Sales de Condensado de melamina sulfonada y formaldehído (SMF) Permiten reducir el agua hasta un 30%.
- Superplastificantes de Tercera Generación. Permiten reducir el agua hasta un 40%. (Como los Copolímeros Vinílicos y los recientes basados en policarboxilatos y poliacrilatos). Utilizados para CAR

### Mecanismo de acción

El cemento Pórtland al entrar en contacto con el agua produce un gran número de iones en disolución que tienden a formar flóculos por afinidad electrostática impidiendo la dispersión uniforme de las partículas del cemento y reteniendo agua en su interior, afectando la trabajabilidad y la hidratación.

Estos efectos de floculación pueden ser contrarrestados con la incorporación de reductores de agua, cuyas moléculas orgánicas neutralizan las cargas eléctricas en la superficie de las partículas de cemento (como el caso de los polímeros de naftaleno, melamina, y lignosulfonatos). Estas moléculas son relativamente cortas porque al no poder recubrir en su totalidad las moléculas de cemento el efecto producido tiene carácter local. El efecto del reductor del agua libera el agua atrapada en los flóculos de los granos de cemento. Como consecuencia, se requiere una menor cantidad de agua de amasado para una determinada trabajabilidad de la mezcla.

En las siguientes figuras se observa el modo de acción de los distintos reductores de agua. Su alta dosificación tiene efectos secundarios como el retraso en la fragua.

**Fig. 1.7.** Efecto de Repulsión de las Partículas y Defloculación con Plastificantes.

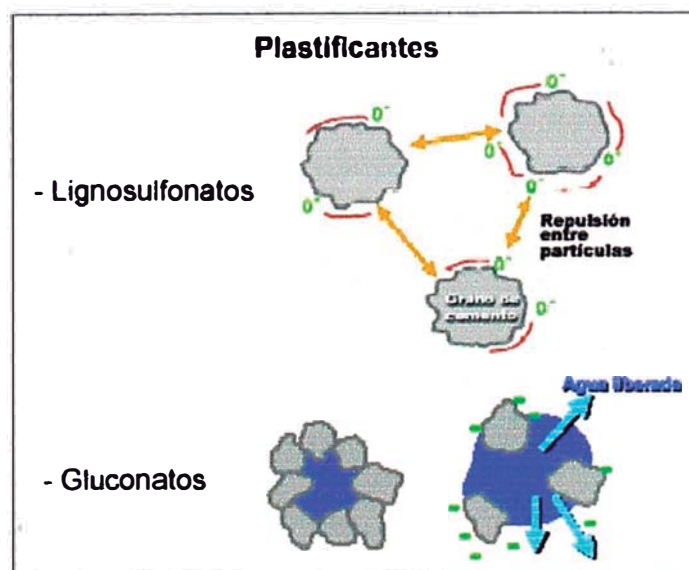


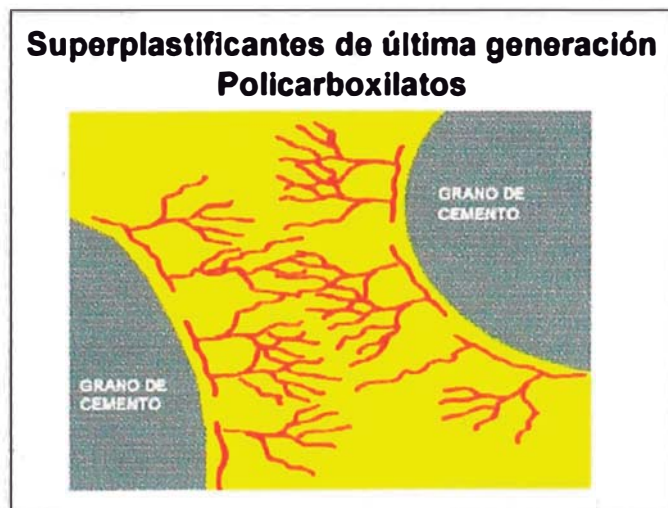


Fig. 1.8. Efecto de Repulsión Electrostática con Superplastificantes Tradicionales.



En los aditivos de última generación, tipo vinílico, pero especialmente con los carboxilatos, no sólo se produce un efecto de dispersión sino también un efecto estérico. Estos aditivos se basan en moléculas complejas con un alto grado de flexibilidad, y comprenden diferentes grupos funcionales, con diferentes longitudes de cadena. Las largas cadenas laterales, Fig. 1.9., crecen más allá de los productos de hidratación del cemento formado, proporcionando un mayor efecto de dispersión. Estas cadenas laterales son responsables del efecto estérico e interactúan con el agua. Al transcurrir el tiempo, una segunda molécula se hace activa mediante el mismo efecto estérico, y así se logra una actividad más duradera en el tiempo.

Fig. 1.9. Efecto reductor de agua de los superplastificantes de última generación (Sika Viscocrete). Dispersión electrostática y acción estérica producto del número y longitud de las cadenas de los compuestos. Reducción del agua hasta 40%



Las interacciones del superfluidificante con las partículas de cemento se clasifican en:

1. Interacciones físicas

La absorción de superfluidificante sobre las partículas de cemento.

2. Interacciones químicas

Bloqueo de sitios reactivos en las partículas de cemento, formación de complejos con  $\text{Ca}^{2+}$ .

### Aditivos Retardantes

Los aditivos retardantes más conocidos son a base de sales de Lignosulfonatos tipo cálcico y sódico su efecto es aun mas pronunciado cuando contiene azucares.

En toda industria de concreto premezclado es primordial la utilización de aditivos retardantes pues alargan el periodo de tiempo en el que el concreto permanece trabajable para el transporte, manejo y colocación del concreto.

### Tipos de Aditivos

La norma ASTM C494, clasifica a los aditivos en las siguientes clases:

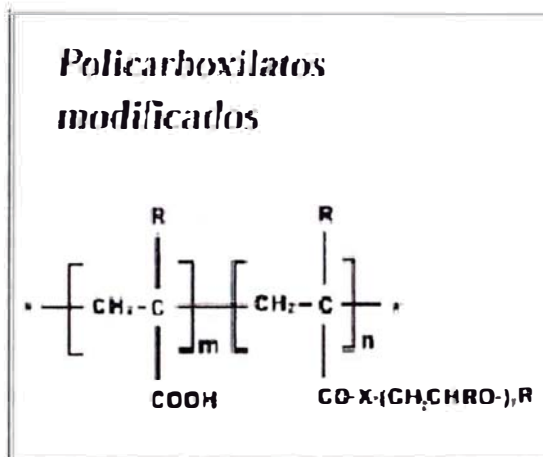
- Tipo A: Aditivos Reductores de agua
- Tipo B: Aditivos Retardantes
- Tipo C: Aditivos Acelerantes
- Tipo D: Aditivos Reductores de Agua y Retardantes
- Tipo E: Aditivos Reductores de agua y Acelerantes
- Tipo F: Aditivos Reductores de agua de Alto rango
- Tipo G: Aditivos Reductores de agua de Alto rango y Retardantes.

Actualmente es común el uso de aditivos Tipo A y B (a base de lignosulfonatos) en concretos convencionales.

### Aditivos en CAR

Los aditivos para producir CAR de acuerdo a la clasificación ASTM C494 son los Tipos F y G. Los últimos aditivos desarrollados para CAR, que reducen hasta 40% la cantidad de agua y se encuentran dentro de esta clasificación, son los aditivos a base de policarboxilatos.

Los Policarboxilatos químicamente se basan en copolímeros de ácido acrílico y grupos éter de ácido acrílico y poseen cadenas laterales a diferencia de los plastificantes tradicionales (macromolécula tipo peine).



Estos aditivos de última generación presentan las siguientes formas:

- Una cadena tipo peine basada en una cadena de policarboxilatos de la que cuelgan cadenas basadas en polímeros de óxido de etileno y óxido de polipropileno unidas por grupos amida o imida.
- Copolímeros tipo peine formados por ácidos acrílicos polimerizables y éteres de estos ácidos.
- Polímeros tipo carboxilato con cadenas laterales basadas en poli éter y poli etilenglicol

El mecanismo de acción de este tipo de aditivos ha sido descrito en el punto anterior.

Su empleo es indispensable cuando se utiliza adiciones minerales para un CAR.

### **1.2.3.5. Agua en el Concreto**

El agua, con relación a su uso en el concreto tiene dos funciones:

- Como componente (agua de mezclado) en donde inicia la reacción química del cemento, produciendo el fraguado y endurecimiento del concreto.
- Como medio de curado del concreto.

El agua de mezclado esta formado por el agua directamente añadida a la mezcla y por la humedad que contengan los agregados. Esta agua primero llena los espacios entre las partículas sólidas (materiales cementicios, y agregados) y crea una película entre ellas que las separa. Este ultimo efecto lubricante es lo que hace una mezcla trabajable (Mindess et al., 2003).

El agua que originalmente era agua de mezclado se transforma en diferentes tipos de agua a medida que la hidratación ocurre. Parte del agua se combina químicamente con los principales compuestos que le otorgan resistencia al concreto, tales como los silicatos de calcio hidratados (C-S-H). Otra parte es adsorbida en la superficie de los productos de hidratación y otra parte queda atrapada en los poros capilares originados durante la hidratación. Dichos capilares se originan debido a que el volumen de los productos de hidratación es menor que el volumen de los materiales previos a la hidratación (Neville, 2003).

El agua empleada en la preparación de concreto cumple con las especificaciones indicadas en la norma ITINTEC 339.088:1982 HORMIGON (CONCRETO). Agua para morteros y hormigones de cementos Pórtland. Actualmente la NTP 339.088 se basa en normas americanas las cuales son menos exigentes.

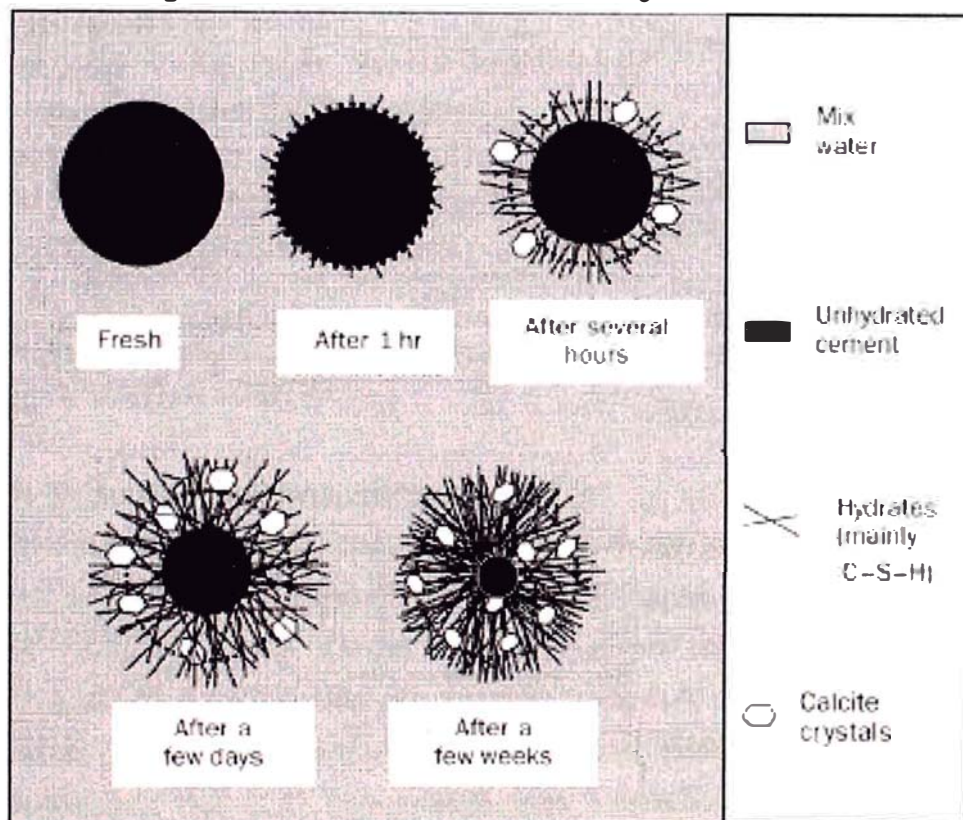
### **1.2.4. Principales Reacciones Químicas**

Las principales reacciones químicas en el concreto son la reacción de hidratación del cemento y la reacción puzolánica, esta última se presenta únicamente en el caso de emplear puzolanas como adición mineral.

### 1.2.4.1. Hidratación del Cemento

La hidratación del Cemento Pórtland es un proceso complejo de disolución-precipitación. La disolución de las fases anhidras del Clínter hace posible la formación de compuestos cuyas solubilidades son menores que ellos mismos, conduciendo a la precipitación de hidratos coloidales y cristalinos que estructuran la pasta de cemento endurecida.

Fig. 1.10. Ilustración de la Hidratación de un grano de Cemento.



### PROCESO DE HIDRATACION

La hidratación del cemento Pórtland a temperatura ambiente es caracterizada por varias etapas o períodos, las reacciones químicas que se llevan a cabo son exotérmicas. Investigaciones [24] han demostrado que se puede deducir el comportamiento de la velocidad a la que están reaccionando los minerales monitoreando la velocidad de calor liberado utilizando un calorímetro. A continuación se muestra un ejemplo de una curva de Velocidad de Liberación de Calor vs. Tiempo, Fig. 1.11.

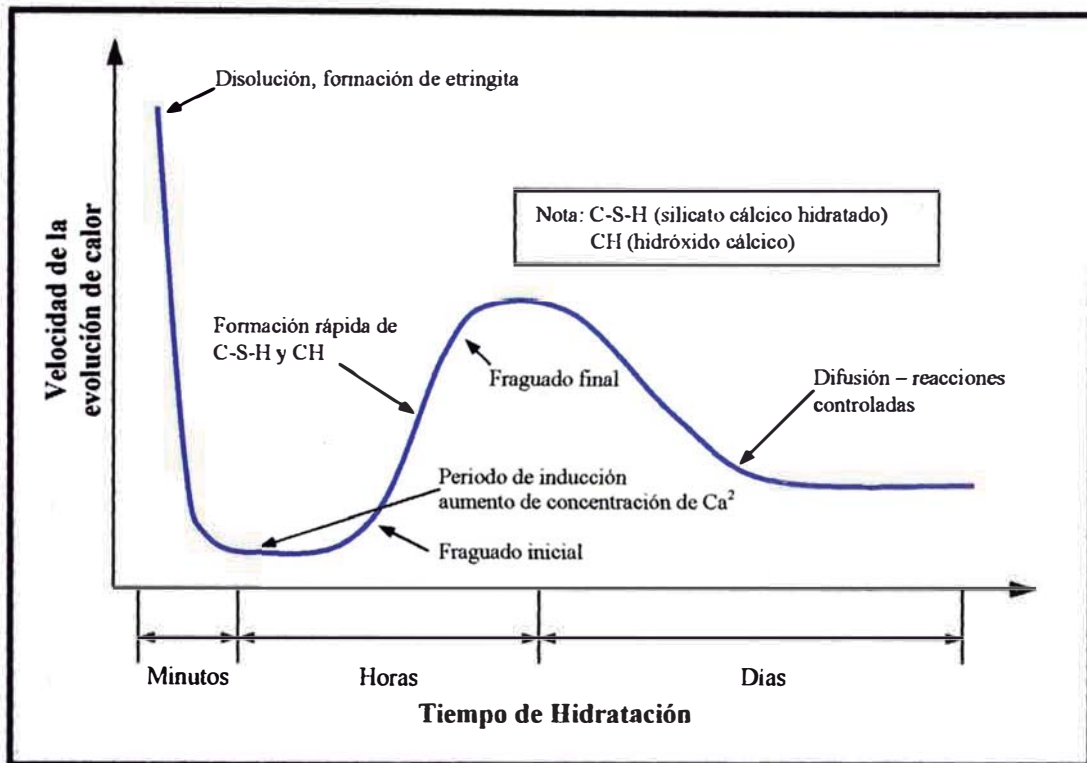


Fig. 1.11. Representación esquemática de la hidratación del cemento en función de la cantidad de liberación de Calor [Jawed et al., 1983]

#### Período de inducción (primeros minutos y horas)

Inmediatamente después del contacto del cemento con el agua, un intercambio de iones se lleva a cabo entre las fases sólida y líquida. La alta solubilidad de algunos compuestos del Clínter, conduce a un rápido aumento en la concentración de la fase líquida con respecto a los  $C_3A$ , sulfatos y álcalis. Gran cantidad de calor es liberada mientras se forman los primeros productos debido a la hidratación del  $C_3A$ , componente más reactivo del cemento, el cual reacciona con el sulfato en solución para formar pequeños cristales de *etringita*. La hidratación del  $C_3A$  es altamente exotérmica pero no dura mucho tiempo, mayormente solo unos pocos minutos y algunos autores la denominan Período de Pre-Inducción).

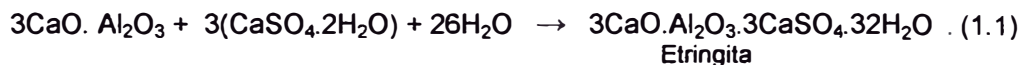
Sin la adición de yeso (retardante), la hidratación del  $C_3A$  puede ocurrir tan rápidamente que el concreto se puede volver espeso al cabo de unos minutos y volverse completamente intrabajable debido al calor emitido.

El C<sub>3</sub>A, yeso y agua forman un recubrimiento protector de sulfoaluminato de calcio hidratado (etringita) sobre las superficies expuestas de aluminato tricálcico que permanece durante varias horas.

Si el cemento Pórtland es Tipo I probablemente contiene más de 8% de C<sub>3</sub>A, que es un porcentaje mucho más alto que el sulfato presente. Una vez que los sulfatos primarios han sido consumidos, el normal fraguado del cemento procede.

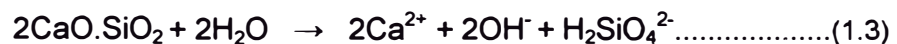
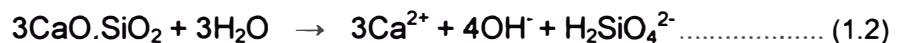
Reacción Principal:

Aluminato Tricálcico (C<sub>3</sub>A) y Yeso:



Posteriormente, la velocidad de evolución de calor disminuye muy rápidamente, la concentración de Ca<sup>2+</sup> de la fase líquida alcanza la sobresaturación.

Reacciones de Disolución:



Al final del periodo de inducción la alita y belita empiezan a hidratar comenzando la nucleación y el crecimiento del C-S-H y el CH.

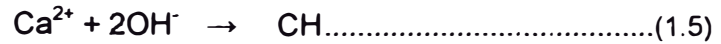
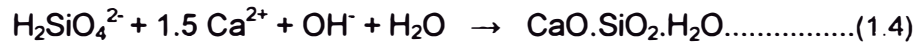
#### Periodo de aceleración (3 a 12 horas después del mezclado)

Esta etapa inicia cuando el fraguado inicial comienza y termina cuando se llega al fraguado final, por ello en esta etapa la resistencia del concreto se incrementa.

La velocidad de hidratación del C<sub>3</sub>S se acelera. Una notable hidratación del C<sub>2</sub>S tiene lugar también. El Ca(OH)<sub>2</sub> (pórtlandita) precipita de la fase líquida y junto con éste, la concentración de Ca<sup>2+</sup> en la fase líquida disminuye gradualmente.

La hidratación del C<sub>3</sub>A continúa. El C<sub>2</sub>S exhibe una reacción de hidratación menos rápida produciendo dos tercios menos de portlandita Ca(OH)<sub>2</sub>.

Principales reacciones:



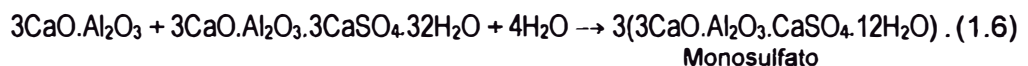
Teniendo en cuenta que la primera reacción esta simplificada, estudios anteriores han demostrado que el factor 1.5 varia desde 1 a 3.

En una mezcla conteniendo solo cemento Pórtland como material cementante, la mayoría de la resistencia se alcanza en un mes. Pero cuando, parte del cemento ha sido reemplazado por una adición mineral, el desarrollo de la resistencia ocurre más lentamente y continua por muchos meses incluso años.

#### Periodo de Post-Aceleración

En este período, la velocidad de hidratación disminuye gradualmente conforme la cantidad de material que todavía no ha reaccionado disminuye y, la velocidad de hidratación llega a ser controlada por la difusión. La fase del C-S-H continúa formándose debido a la hidratación continua del C<sub>3</sub>S y del C<sub>2</sub>S. La contribución del C<sub>2</sub>S en este proceso se incrementa con el tiempo y, consecuentemente, la velocidad con la cual el Ca(OH)<sub>2</sub> adicional es formado se reduce. Después de que el abastecimiento de sulfato cálcico ha sido agotado, la concentración de SO<sub>4</sub><sup>2-</sup> en la fase líquida disminuye. Como consecuencia de esto, la fase Etringita (AFt) que ha sido formada en las etapas iniciales de hidratación, comienza a reaccionar con C<sub>3</sub>A remanente produciendo monosulfato (AFt).

Reacción:



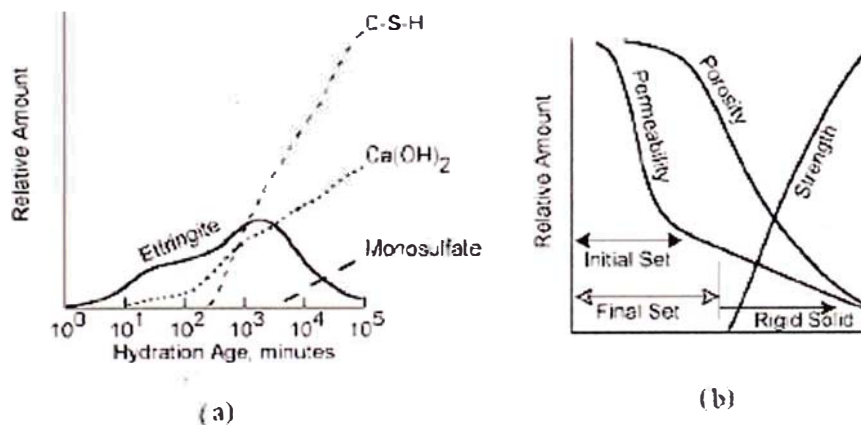
Si hay C<sub>3</sub>A residual presente, en la presencia de hidróxido de calcio en una etapa mas tarde aluminato de calcio hidratado puede ser formado, usualmente de la forma C<sub>4</sub>AH<sub>13</sub>, pero el C<sub>3</sub>AH<sub>6</sub> puede ser formado bajo algunas circunstancias. La hidratación mucho más lenta del C<sub>4</sub>AF es análoga al C<sub>3</sub>A después del consumo de los sulfatos, obteniendo el componente C<sub>4</sub>FH<sub>13</sub>.



A continuación se presenta un resumen de las reacciones de hidratación:

<p><b>(1) Silicato Tricálcico (C<sub>3</sub>S):</b></p> $2(C_3S) + 6H \rightarrow C_3S_2H_3 + 3CH \dots \dots \dots (1.7)$
<p><b>(2) Silicato Dicálcico (C<sub>2</sub>S):</b></p> $2(C_2S) + 4H \rightarrow C_3S_2H_3 + CH \dots \dots \dots (1.8)$
<p><b>(3) Aluminato Tricálcico (C<sub>3</sub>A) y Yeso :</b></p> $C_3A + 3(C\hat{S}H_2) + 26H \rightarrow C_6A\hat{S}_3H_{32} \dots \dots \dots (1.9)$ <p style="text-align: right;">Etringita</p> <p>Luego:</p> $2(C_3A) + C_6A\hat{S}_3H_{32} + 4H \rightarrow 3(C_4A\hat{S}H_{12}) \dots \dots \dots (1.10)$ <p style="text-align: right;">Monosulfato</p> <p>Luego:</p> $C_3A + CH + 12H \rightarrow C_4AH_{13} \dots \dots \dots (1.11)$
<p><b>(4) Aluminoferrito Tetracálcico (C<sub>4</sub>AF):</b></p> $C_4AF + 4CH + 22H \rightarrow C_4AH_{13} + C_4FH_{13} \dots \dots \dots (1.12)$

**Fig. 1.12.** (a) Velocidades típicas de formación de los productos de hidratación en un cemento Pórtland ordinario (b) influencia de los productos de hidratación en el fraguado, porosidad y resistencia de la pasta de cemento. [Adaptado del J. Soroka, Pórtland Cement Paste and Concrete, The Macmillan Press, p 35, 1979]



## **CINETICA DEL PROCESO DE HIDRATACION**

Las reactividades de los compuestos del Clínter en el cemento son aproximadamente en el siguiente orden:  $C_3A > C_3S > C_2S$ .

La cinética del proceso de hidratación depende principalmente de:

- La velocidad de disolución de las fases involucradas;
- La velocidad de nucleación y formación de los cristales de los productos de hidratación; y
- La velocidad de difusión del agua e iones disueltos a través del material hidratado una vez que fue formado.

Además está influenciada por los siguientes factores:

- La composición del Clínter.
- La cantidad de sulfatos cálcicos presentes en el cemento;
- La finura del cemento;
- La relación agua/cemento de la mezcla;
- Las condiciones de curado;
- La temperatura de hidratación; y
- La presencia o no de aditivos químicos.

## **MECANISMOS DE LA REACCION**

Ocurren dos mecanismos durante el proceso de hidratación:

### Hidratación de la solución total

Comprende; la disolución de los compuestos anhidros a sus constituyentes iónicos, la formación de hidratos en la solución y, debido a su baja solubilidad, la precipitación final de los hidratos de la solución.

### Topoquímica o hidratación del estado sólido

Las reacciones se llevan a cabo directamente en la superficie de los compuestos anhidros sin que hayan sido disueltos.

El mecanismo de solución total es dominante en edades tempranas, pero a edades posteriores, cuando la movilidad iónica en solución se ve restringida, la hidratación de las partículas del cemento residual puede ocurrir por reacciones del estado sólido.

### 1.2.4.2. Reacción Puzolánica

En la hidratación del cemento Pórtland las principales fases mineralógicas producen C-S-H (Silicatos de Calcio Hidratado) y además CH (Portlandita) entre un 20 y 30% en masa. La portlandita no aporta un beneficio a la acción mecánica de la matriz cementante, sin embargo, mantienen un ambiente alcalino (alto pH) necesario para evitar la corrosión del acero de refuerzo. Si adicionamos puzolanas al concreto la portlandita reaccionara con estas para producir más compuestos C-S-H similares a los producidos en la hidratación del cemento Pórtland. La reacción puede ser representada por la siguiente ecuación simplificada:



Los C-S-H son los responsables de la resistencia mecánica de las pastas. Este sistema de hidratación es muy complejo.

Adicionalmente se puede decir que la reacción Puzolánica es acompañada por una reducción en los poros grandes, un efecto que es igualmente importante para mejorar la resistencia del sistema y, por tanto, para la aplicación en un CAR.

### 1.2.5. Propiedades del CAR en Estado Fresco

Las propiedades principales que se controlan en estado fresco al CAR son las mismas que se controlan a todos los tipos de concretos y son las siguientes: Trabajabilidad, Peso Unitario, Contenido de Aire, Temperatura y Tiempo de Fragua.

La medición de las propiedades se realiza al término del mezclado del concreto y constituyen una buena información para el control de calidad.

#### 1.2.5.1. Trabajabilidad

Se define como la facilidad que presenta el concreto fresco para ser colocado y vibrado en cualquier molde. Los concretos con baja trabajabilidad presentan problemas de mezclado y compactación dentro de los encofrados lo que puede causar una disminución de la

resistencia. Por ejemplo en el caso de edificaciones con alta densidad de acero.

Para mejorar la trabajabilidad de un concreto se puede añadir agua con la consiguiente disminución de la resistencia o se puede incluir aditivos reductores de agua, que permitirán mejorar sus propiedades manteniendo el nivel de resistencia.

De acuerdo a la ASTM C125, la trabajabilidad es la propiedad que determina el esfuerzo requerido para manipular una mezcla de concreto fresco con una pérdida mínima de homogeneidad. El término manipular incluye las operaciones de colocación, compactación y acabado.

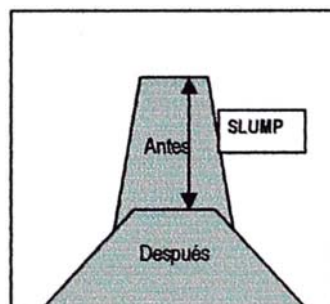
Esta propiedad esta compuesta por la fluidez o consistencia y la cohesividad o resistencia a segregar.

#### Asentamiento (Slump) [9]

*El método de ensayo utilizado es: ASTM C143 Standard Test Method for Slump of Hydraulic-Cement Concrete*

El ensayo de Asentamiento es una medida de la trabajabilidad, además de un indicador de la reología.

El ensayo consiste en llenar de concreto un cono de Metal trunco y soltarlo rápidamente.



En esta prueba, el esfuerzo aplicado lo otorga el mismo peso del concreto (por área). El concreto se asentará solamente cuando el esfuerzo exceda la tensión crítica o tensión de Umbral (Yield Stress) y concluye cuando sea menor a esta. Como resultado, la prueba de Slump puede considerarse estar relacionada a la tensión crítica.

### 1.2.5.2. Contenido de Aire

Generalmente este aire ocupa del 1% al 3% del volumen de la mezcla de concreto normal.

El contenido de aire entrampado en el concreto está en función de las proporciones en que se han combinado los ingredientes en la mezcla, de las características físicas de los agregados, ver Cuadro 1.9, y del método de compactación.

El contenido de aire puede influenciar en la resistencia a la compresión y en la determinación de su porosidad. Es recomendado medir el contenido de aire conjuntamente con el peso unitario. Concretos de alta resistencia presentan contenidos de aire del 1% a 3% (AİTCIN, 2000). El método de ensayo para medir el contenido de aire esta establecido en la norma:

*ASTM C231 Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method*

**Cuadro 1.9.** Contenido de aire atrapado (ACI Committee Report 211.1)

<i>Contenido de Aire Atrapado</i>	
<i>Tamaño Máximo Nominal del Agregado Grueso</i>	<i>Aire Atrapado %</i>
<i>3/8 "</i>	<i>3.0</i>
<i>1/2 "</i>	<i>2.5</i>
<i>3/4 "</i>	<i>2.0</i>
<i>1 "</i>	<i>1.5</i>
<i>1 1/2 "</i>	<i>1.0</i>
<i>2 "</i>	<i>0.5</i>
<i>3 "</i>	<i>0.3</i>
<i>4 "</i>	<i>0.2</i>

### 1.2.5.3. Peso Unitario

Es la masa de concreto fresco por volumen, considerando el volumen de aire aprisionado o incorporado en el concreto. Este ensayo puede ser medido de acuerdo a la norma:

*ASTM C138 Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete*

Para diferentes batch de concreto de un mismo diseño, cuando el valor del Peso Unitario permanece constante, el contenido de aire también.

El uso de la microsílíce para CAR no implica cambios en el peso unitario del concreto (ACI).

### 1.2.5.4. Temperatura

La reacción de hidratación del cemento es exotérmica (Punto 1.2.4.1).

Los cambios considerables de temperatura del concreto pueden causar fisuración debido a que, como cualquier otro material, el concreto se expande a temperaturas elevadas y se contrae a temperaturas bajas.

Cuando las adiciones minerales, como el fly ash, puzolanas, escorias, son utilizadas como reemplazo parcial del cemento Pórtland, el calor de hidratación y la temperatura que alcanza el concreto son menores. Este ensayo se realiza según lo especificado en:

*ASTM C1064/C1064M-01 Standard Test Method for Temperature of Freshly Mixed Portland Cement Concrete*

### 1.2.5.5. Tiempo de Fragua

El ensayo tiene por objeto encontrar el tiempo que demora en ganar el concreto una determinada resistencia desde el contacto cemento – agua. Para tal efecto se consideran dos definiciones: fragua inicial, cuando el concreto alcanza la resistencia de 500 lb/pulg<sup>2</sup>, equivalente a 35 Kg/cm<sup>2</sup>, y fragua final, cuando el concreto alcanza la resistencia de 4.000 lb/pulg<sup>2</sup>, que equivale a 280 kg/cm<sup>2</sup>.

El fraguado está influenciado por; las reacciones de hidratación como se explicó en el punto 1.2.4.1., por el contenido de C<sub>3</sub>A y por la dosificación del aditivo retardante y plastificante

El ensayo para determinar el tiempo de fragua está dada por:

*ASTM C403/C403M-99 Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance*

El campo de aplicación del presente trabajo está orientado al desarrollo de un CAR en la industria de concreto pre-mezclado, por lo tanto, es primordial la incorporación de aditivos que retarden el fraguado.

### **1.2.6. Propiedades del CAR en Estado Endurecido**

Las propiedades más importantes del CAR en estado endurecido son la resistencia a la compresión y la durabilidad.

#### **1.2.6.1. Resistencia a la Compresión**

La resistencia a la compresión es la propiedad principal del concreto, muchas propiedades estructurales del concreto están relacionadas con esta resistencia.

El concreto depende principalmente de su resistencia mecánica una vez fraguado y endurecido, resistencia resultante de la cohesión de las partículas de cemento y su adhesión a los granos de arena u otros áridos con los que se está mezclando.

De acuerdo al ACI un ensayo de resistencia se define como “el resultado del promedio de dos cilindros tomados de una misma mezcla y ensayados a los 28 días, o a la edad especificada en caso que sea diferente de 28 días”.

Los resultados de la prueba de resistencia dependen en alto grado de las condiciones de mezcla, preparaciones de los especímenes y de la forma de hacer los ensayos; es necesario describir la manera exacta de desarrollarlos, con objeto de obtener resultados comparables. La forma de realizar los ensayos está especificada en las siguientes normas ASTM:

*ASTM C39/C39M-01 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*

*ASTM C617-98 Standard Practice for Capping Cylindrical Concrete Specimens*

*ASTM C192: Practice for Making and Curing Concrete Test Specimens in the Laboratory*

Factores que influyen en la resistencia del concreto:

- Relación Agua / Cemento
- Inclusión de Aire
- Tipo de Cemento
- Agregado
- Agua de Mezclado
- Adiciones Minerales
- Condiciones de Curado (Tiempo, Humedad, Temperatura)
- Parámetros de los especímenes

A diferencia de la mayoría de los materiales estructurales, el concreto contiene grietas muy finas aun antes de estar sujeto a esfuerzos externos. Por tanto, la resistencia en el concreto se relaciona con el esfuerzo requerido para causar fractura o cuando el esfuerzo aplicado alcanza su valor máximo.

Distribución de Frecuencias de la Resistencia

Los resultados de resistencia del concreto están afectados por numerosas fuentes de variación como se describió anteriormente, por lo que es absolutamente imposible garantizar que cada espécimen de ensayo (de una misma mezcla) arroje exactamente el mismo valor de resistencia, por lo que generalmente los resultados giran alrededor de un valor central y dentro de un rango de valores. Es todavía más improbable que las pruebas de resistencia obtenidas por varios batch de una misma clase de concreto, producido con igual diseño, materiales y bajo condiciones similares, arrojen el mismo valor de resistencia. Adicionalmente, cada prueba de resistencia indica un valor potencial de la misma y por ello no debe analizarse de manera aislada

Lo anterior implica que para obtener un valor central representativo que caracterice su comportamiento se deben agrupar los resultados



obtenidos de las pruebas de resistencia de varios lotes y buscar cual es la tendencia de esa población de resultados.

De acuerdo a las recomendaciones del ACI 214 y 318, son necesarias 30 pruebas de resistencia como mínimo para realizar un análisis estadístico completo y que sea representativo.

#### **1.2.6.2. Durabilidad**

La durabilidad esta estrechamente relacionada con las condiciones de exposición al medio ambiente, el cual puede generar deterioro por causas físicas o químicas. Por otro lado, la permeabilidad y los cambios de volumen debido a diferencias en propiedades térmicas pueden afectar la durabilidad. Muchas investigaciones han indicado que la permeabilidad del concreto al aire o al agua es una excelente medida de su durabilidad, puesto que mide la resistencia al ingreso de medios agresivos en estado gaseoso o líquido [15]. Los métodos de ensayo para medir la durabilidad del CAR en la presente tesis son Ensayo de Permeabilidad utilizando el Equipo Torrent y Ensayo de Resistencia al Ataque de Sulfatos. Este último se describirá en el ítem 1.4.3.2.

##### Ensayo de Permeabilidad utilizando el Equipo Torrent

Es una característica física que debe de cumplir todo tipo de concreto. Esta se define como los espacios porosos intergranulares o las fisuras de una roca (espacios vacíos) que quedan en la masa del mismo a consecuencia de la evaporación del agua excedente y del aire atrapado en su manipulación. Con un a/c baja se logran resistencias considerablemente altas y menor porosidad capilar.

En el caso de los CAR, los elementos intergranulares que participan son más finos y por tanto los espacios porosos son menores que en los otros tipos de concreto, así como también su permeabilidad

La capacidad de una estructura de concreto depende del comportamiento integral del elemento. Pero su durabilidad con respecto a agentes agresivos del medio ambiente recae en la performance de una delgada capa superficial (20-50 mm de espesor),

el covercrete. Actualmente, criterios de aceptación están basados en los resultados de especímenes de concreto moldeados

El equipo de Torrent es un dispositivo de ensayo no destructivo, diseñado para la medición “in situ” de la permeabilidad al aire del recubrimiento del concreto (covercrete). Se basa en crear vacío dentro de una celda colocada sobre la superficie del concreto y en medir la velocidad con que la presión retorna al valor atmosférico en celda, después que la bomba de vacío ha sido desconectada.

La característica distintiva de este método es que está conformado por una doble celda y un regulador de presión que balancea la presión en ambas cámaras durante la prueba. Este aparato crea un flujo de aire unidireccional y controlado desde los poros hacia la celda interior, mientras que la cámara exterior actúa como un anillo de guarda. Esto permite el cálculo del Coeficiente de Permeabilidad de Torrent (Kt), basado en un modelo teórico [15].

Una determinación simple toma de 1.5 a 12 minutos, dependiendo de la permeabilidad del concreto.

Para concretos secos (la superficie del concreto no ha estado expuesta a humedad por tres semanas), la clase del covercrete se determina directamente a partir de Kt dado por el equipo, usando la siguiente tabla:

**Cuadro 1.10. Valores del Kt**

<b>Calidad</b>	<b>Índice</b>	<b>Kt (*10<sup>-16</sup> m<sup>2</sup>)</b>
Muy Mala	5	> 10
Mala	4	1,0 – 10
Normal	3	0,1 – 1,0
Buena	2	0,01 - 0,1
Muy Buena	1	< 0,01

El procedimiento para la realización del ensayo con este equipo se encuentra en el Anexo E.

### **1.2.7. Proporcionamiento de la Mezcla para CAR (Diseño de Mezcla)**

Para realizar el proporcionamiento de la mezcla para CAR se sigue el método de cálculo establecido en el Informe 211 del ACI, pero este informe sólo tiene recomendaciones (tablas y gráficos de referencia) para concretos convencionales. Para efectos del presente trabajo se ha utilizado:

- Recomendaciones contenidas en el informe 363 del ACI referido a CAR
- Datos históricos pertenecientes a UNICON.
- Métodos de cálculo de acuerdo al informe 211 del Comité ACI. Son los siguientes:
  - Método por peso
  - Método de Volumen absoluto (considerado el más exacto)

UNICON emplea el segundo método, el cual también será empleado en la presente tesis. Los cálculos por realizar requieren de los siguientes datos de entrada:

- Análisis granulométrico de los agregados.
- Peso Específico de los materiales
- Absorción y Humedad de los agregados
- Slump de Diseño de acuerdo a la especificación de la obra de construcción
- Resistencia de Diseño, y
- Relación a/c de acuerdo a la especificación de la obra de construcción

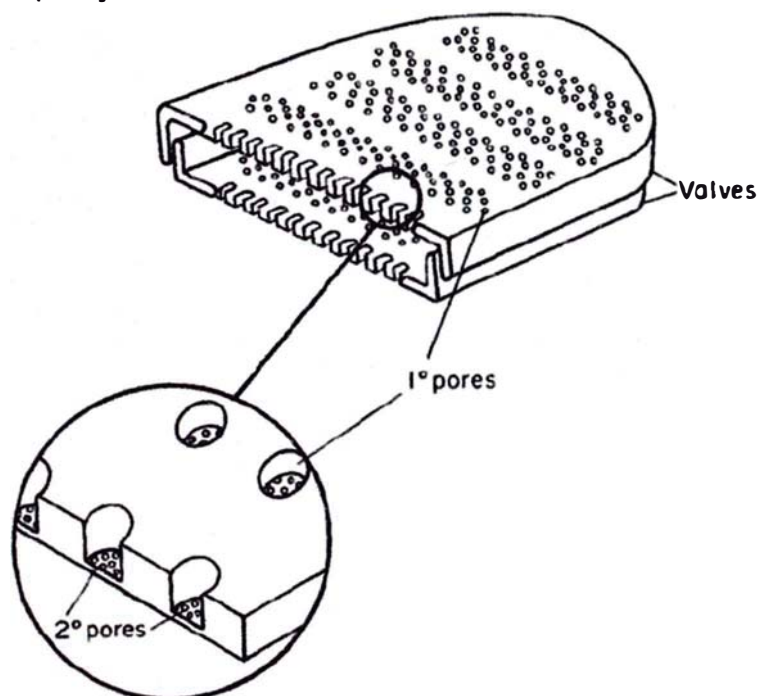
Los tres primeros datos se obtienen de la caracterización de los insumos que se emplearán para preparar el CAR. El Slump y la resistencia son definidos por las especificaciones del cliente y de acuerdo a estas se debe estimar la relación a/c adecuada para el CAR en elaboración. El procedimiento detallado del cálculo se encuentra en el Anexo F.

### 1.3. TIERRAS DIATOMACEAS

#### 1.3.1. Descripción

Las diatomitas se clasifican como una roca sedimentaria silícea de origen orgánico. La fuente de toda diatomita es un organismo vivo denominado diatomea, las cuales son prolíficas y microscópicas algas acuáticas unicelulares, que poseen dos valvas silíceas situadas dentro de la capa externa del plasma. El tamaño de la valva de la diatomea varía según la especie, aproximadamente entre 50 y 100  $\mu\text{m}$  de diámetro, dependiendo de la especie y de las condiciones de crecimiento.

Fig. 1.13. Esquema detallado de un caparazón de Diatomea [Diatomaceous Earth Occurrences in Nova Scotia – Chapter 1]



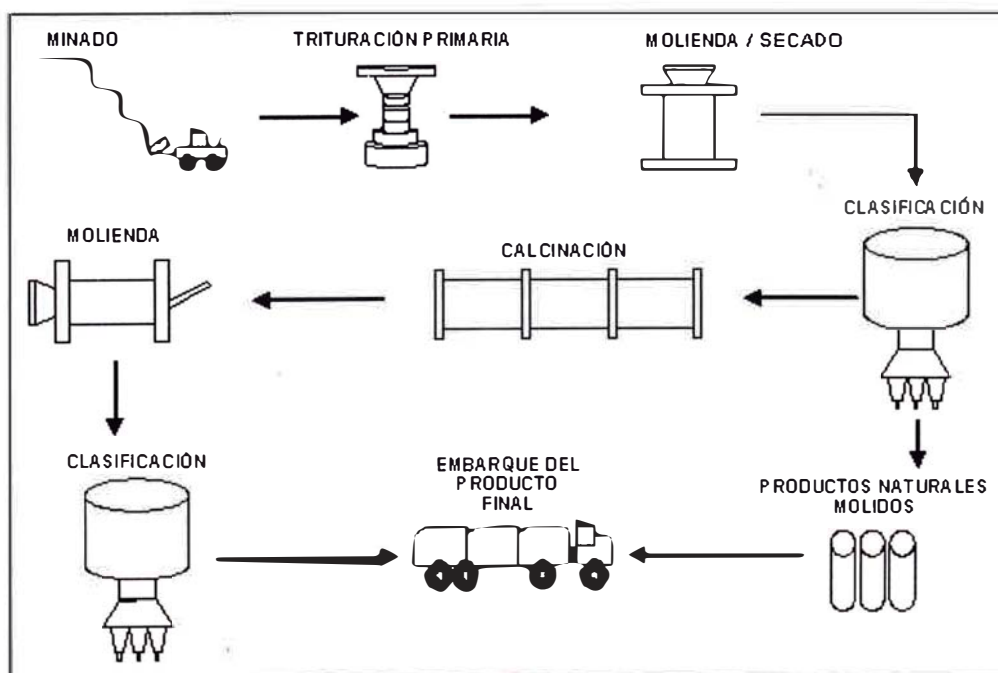
Su función natural es como alimento para otros organismos del mar. Además, su rol en el control del balance geoquímico de la sílica en aguas de mar y lacustre es científicamente reconocida. Cuando el organismo muere se decanta dentro del medio acuoso, formando un sedimento de carácter orgánico. La descomposición del residuo orgánico deja paso a la acumulación de los caparazones silíceos, formados por sílice amorfo (ópalo) casi puro, los cuales se van compactando para convertirse en depósitos potenciales de diatomita.

La ocurrencia de la sílica diatomácea está repartida por todo el mundo. Aunque la alga apareció bastante temprana en la historia geológica, depósitos comerciales son generalmente limitados a formaciones sedimentarias de la edad terciaria y posterior.

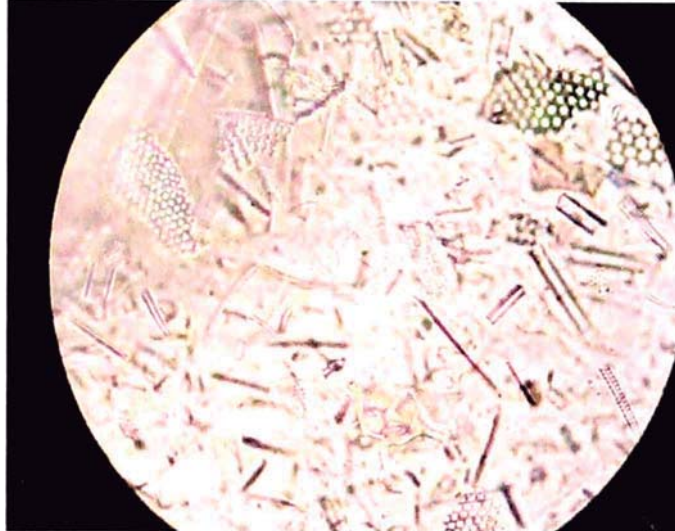
Entre sus características se puede observar que la diatomita pura y seca es una sustancia blanca, de poca densidad aparente (extrema porosidad). Absorbe entre 1.5 y 3 veces su peso de agua, así como también, tiene gran capacidad de absorción de aceite. Es atacada únicamente por álcalis fuertes y por el ácido fluorhídrico.

En nuestro país la explotación de esta sustancia se encuentra en un estado incipiente debido al desconocimiento sobre las técnicas requeridas para su aprovechamiento. Este hecho hace que la demanda de diatomita en la industria nacional sea abastecida totalmente por importaciones. Actualmente, la explotación está limitada solo a procesos físicos de extracción y reducción del material, obteniéndose un mineral que en su mayoría se destina como material de relleno. A continuación se presenta un diagrama de flujo de la explotación y manufactura de la diatomita

Fig. 1.14. Diagrama de Manufactura de la Diatomita



**Fig. 1.15.** Diatomita de origen marino a 40X [11]



### **1.3.2. Variedades**

La frústula o esqueleto silíceo de la diatomea de la clase Bacillariophyceae, y del orden Bacillariaes comprende más de 300 géneros y 12,000 a 16,000 especies. Se podrían clasificar por su forma mediante observaciones microscópicas, y determinar si se formaron en agua dulce o tuvieron su origen en agua de mar. Los depósitos de origen lacustre presentan mayormente diatomeas de forma alargadas, mientras que los depósitos generados en aguas oceánicas presentan diatomeas en forma redondeada o hexagonal.

### **1.3.3. Composición**

La diatomita pura esta formada por sílica opalina o hídrica  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$  presentando, en solución sólida o como parte esencial del complejo de ópalo, pequeñas cantidades de componentes inorgánicos asociados ( $\text{Al}_2\text{O}_3$ , Fe, tierras alcalinas, metales alcalinos) y otros constituyentes menores. También contiene cantidades inusuales de agua químicamente combinada, variando entre 3.5 y 8.0%.

Asociado con la sílice diatomácea, e integrado como parte de la diatomita, puede haber cantidades variables de materia orgánica, sales solubles, y partículas minerales de formación rocosa que fueron depositados o precipitados con las

frústulas de la diatomita. Sus contaminantes típicos son la arena, arcillas, carbonato y ceniza volcánica.

#### **1.3.4. Calidad**

La propiedad más reconocida es el color. Mientras la diatomita presente mayor blancura, más atractiva es su potencial como producto para la industria.

Otra propiedad es la densidad. Una baja densidad indica, entre otras cosas, que esta libre de contaminantes sólidos, tales como la arena y las arcillas.

El análisis químico es útil hasta cierto punto. Es esperado que el porcentaje de componentes no silicios sea bajo y en muchos casos es esencial su ausencia.

Determinaciones mineralógicas e identificación microscópica también es de importancia para la identificación de las frústulas de la diatomita.

#### Propiedades Físico-Químicas

La Diatomita de uso comercial se caracteriza por las siguientes propiedades:

- Color Blanco, variando de acuerdo con las impurezas.
- Baja Densidad
- Punto de Fusión 1000 a 1600°C.
- Brillo Mate y Terroso
- Fractura de pulverulenta a subconcoidal o irregular en estado compacto.
- Transparencia opaco
- Porcentajes de espacios vacíos 60 %
- Área superficial 2,000 a 20,000 m<sup>2</sup>/kg
- Capacidad de absorción de 1.5 a 1.3 veces su peso en agua (roca natural seca).

Dichas propiedades se verán alteradas dependiendo del contenido de impurezas que presenta la diatomita.

#### Composición Química

En cuanto a la composición química característica, el Cuadro 1.11 especifica los contenidos que comúnmente presentan las diatomitas naturales de depósitos que se explotan comercialmente en Estados Unidos y México. En paralelo se muestran los resultados obtenidos para muestras procedentes de Ayacucho - Perú.

**Cuadro 1.11.** Composición Química de Algunas Diatomitas Naturales  
(% en peso)

Componente	Lompoc, USA*	Jalisco, México*	Ayacucho, Perú**	Ayacucho, Perú***
SiO <sub>2</sub> %	89.7	91.2	89.3	87.20
Al <sub>2</sub> O <sub>3</sub> %	3.7	3.2	2.7	3.49
Fe <sub>2</sub> O <sub>3</sub> %	1.1	0.7	0.9	1.22
TiO <sub>2</sub> %	0.1	0.2	1.0	0.16
CaO %	0.3	0.2	0.2	0.47
MgO %	0.6	0.4	0.4	0.79
Na <sub>2</sub> O %	0.3	0.1	0.2	0.07
K <sub>2</sub> O %	0.4	0.2	0.5	0.52
Pérdidas por calcinación %	3.7	3.6	3.8	4.14

\* Lefond 1983 \*\* Naranjo 1994 \*\*\* Agregados Calcareos 2005

### Propiedades Mineralógicas e Identificación Microscópica.

El objetivo de las pruebas mineralógicas (Análisis Térmico, Difracción de Rayos X y Microscopia) es constatar si los contenidos de sílice de la diatomita corresponden en su totalidad a sílice amorfa o a cuarzo. Una diatomita de buena calidad estará constituida básicamente por sílice amorfa.

La diatomita es valiosa por su microporosidad natural, presenta una forma única de sílica por cuanto no se ha encontrado en la naturaleza sus características de estructura física.

#### **1.3.5. Yacimientos de Tierras Diatomáceas en el Perú**

En la Sierra, las diatomeas depositadas son lacustres. El depósito de diatomitas de Tarucani (Arequipa) es un buen ejemplo de depósito lacustre. Las formaciones de Ayacucho cuentan con 85% a 98% de sílice amorfo [8]. Otras rocas con diatomeas lacustre son conocidas en Cajamarca, Ancash, Junín, Cusco, etc.



**Cuadro 1.12. Unidades Mineras con Reservas de Diatomita**

Titular	Unidad	Región	Provincia	Distrito
Cía Mra. Agregados Calcáreos S.A.	San Carlos Nro. 5	Ayacucho	Huamanga	Quinua
Cía Mra. Agregados Calcáreos S.A.	San Carlos 3-85	Ayacucho	Huamanga	Tambillo
Cía Mra. Agregados Calcáreos S.A.	Villa Rica I-88	Ayacucho	Huamanga	Vinchos
Cía Mra. Las Camelias S.A.	Paty 91	Ica	Pisco	San Clemente
Insumex S. A.	Insumex S. A.	Ayacucho	Huamanga	Quinua
Insumex S. A.	Insumex Dos	Ayacucho	Huamanga	Acos Vinchos
Manrique Vda. De Butiler Margarita	Santa Ines 400	Arequipa	Arequipa	Chiguata
Manrique Vda. De Butiler Margarita	Santa Ines Nº 200	Arequipa	Arequipa	Chiguata
Soc. Mra. Celite Perú S. A.	Elisa	Arequipa	Caylloma	Maca
Soc. Mra. Celite Perú S. A.	Zeus - 7	Ayacucho	Huamanga	Carmen Alto

Fuente: MEM DAC 2000. [http://www.minem.gob.pe/minera/mapa\\_formulario.asp](http://www.minem.gob.pe/minera/mapa_formulario.asp)

### 1.3.6. Producción Mundial y Reservas Nacionales

La producción mundial de diatomita se mantuvo estable alrededor de los 1,95 millones de toneladas. En el 2004, el Perú ocupó el segundo lugar en América Latina y el primer lugar en Sudamérica, con una producción de 35,000 toneladas.

**Cuadro 1.13. Producción Mundial de Diatomita y Reservas (Miles de TM)**

Países	Producción Minera 2003	Producción Minera 2004	Reservas
Estados Unidos	620	635	250000
China	380	370	110000
Dinamarca (Procesada)	232	232	n.d.
Japón	180	180	n.d.
Commonwealth	80	80	n.d.
Francia	75	75	n.d.
México	60	65	n.d.
España	35	36	n.d.
Perú	35	35	2000
Total Mundial (redondeado)	1950	1960	920000

Fuente: USGS. Mineral Commodity Summaries January 2005

## **1.4. TIERRAS DIATOMACEAS COMO COMPONENTE EN CAR**

### **1.4.1. Base Teórica**

Estudios realizados en Estados Unidos [28] compararon algunas propiedades de un concreto tradicional (solo con cemento Pórtland) y concretos con cementos adicionados (cenizas volantes, microsílíce o una mezcla de ambas) con un concreto incorporando tierras diatomeas (diatomitas). Se demostró que la incorporación de tierras diatomeas en el concreto puede incrementar sustancialmente la resistencia a la compresión y la resistencia a la reacción álcali-sílíce reduciendo significativamente la porosidad y mejorando la resistencia a los cloruros (ASTM C1202-97). La adición de esta roca en la producción de concreto no favorece el desarrollo adecuado de la porosidad lo que afecta negativamente la resistencia a los ciclos de hielo y deshielo. Todo esto hace que las tierras diatomeas pueden ser usadas como un reemplazo parcial del cemento Pórtland para reducir tanto el costo del concreto como las emisiones de CO<sub>2</sub> que se realizan durante la producción del cemento.

Sobre la base de estos conocimientos y la existencia de grandes reservas de esta roca en el país motivaron a investigar su uso como adición mineral en CAR. Para utilizar la diatomita como componente en un CAR se tiene que evaluar las principales propiedades descritas en los puntos 1.2.5 y 1.2.6, además de ciertas propiedades estructurales tales como: modulo de elasticidad, tracción diametral, contracción y abrasión. Las propiedades estructurales no son objeto de esta tesis y muchas de ellas pueden ser estimadas a partir de la resistencia a la compresión. Complementariamente se realizaran ensayos en Reología (Viscosidad) y durabilidad (Resistencia al Ataque de Sulfatos).

### **1.4.2. Influencia de las Características Químicas y Físicas de la Diatomita en el CAR.**

Actualmente existen normas (ASTM C618) que evalúa algunas características químicas y físicas para verificar si una adición mineral es apta para el uso en el concreto, pero estos ensayos son solo referenciales pues no predicen su comportamiento en el concreto.

Los ensayos que comúnmente se realizan a las adiciones minerales son los análisis químicos y el ensayo de actividad puzolánica (ensayo físico).

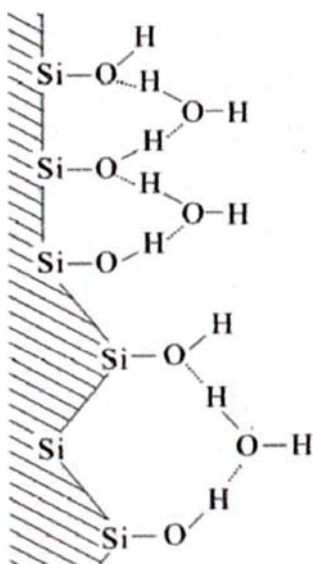
Las características químicas de las puzolanas influyen en el incremento de la resistencia del concreto debido a que estas reaccionan con la portlandita producto de la hidratación del cemento de acuerdo a la reacción 1.13.

La diatomita está clasificada como una adición mineral puzolánica de acuerdo a la norma ASTM C618.

La reacción puzolánica es lenta, el aporte químico de las puzolanas se presenta a edades tardías. A edades tempranas (7 días) el aporte de cualquier adición mineral es netamente física y mejora en estado fresco la trabajabilidad del concreto [7].

De acuerdo a la ASTM C311, el Índice de Actividad de Resistencia sirve para determinar si una adición mineral produce un nivel aceptable de desarrollo de resistencia cuando es usado con cemento hidráulico en el concreto. Este ensayo se realizará para determinar si la diatomita (adición mineral) cumple con las especificaciones de la norma ASTM C618, donde se indica que para todo tipo de puzolana natural, utilizada como adición mineral, el índice de actividad debe ser mayor a 75%. El índice de actividad de resistencia se define como la relación en porcentaje de la resistencia de una muestra con 20% de reemplazo de cemento por adición mineral con una muestra control sin reemplazo de cemento.

Se debe tener en cuenta también que la diatomita es higroscópica, es decir, adsorbe agua del ambiente, debido a la afinidad de la superficie de la diatomita con el agua mediante puentes hidrógeno (Fig. 1.16) y a la gran superficie específica de esta al ser porosa.



**Fig. 1.16.** Estructura de Silice de la Diatomita (The hydroxyl species and acid sites on diatomite surface: a combined IR and Raman study - Yuan, P. and Wu, D. Q. and He, H. P. and Lin, Z. Y. - 2004)

La cantidad de agua necesaria para el concreto con contenido de diatomita será mayor que con microsilice, pero esto será contrarrestado con los aditivos superplastificantes a base de polycarboxilatos, los cuales reducen hasta en 40% la cantidad de agua requerida.

Con respecto a la composición mineralógica, la diatomita tiene una estructura cristalina amorfa, denominada comúnmente ópalo o sílica opalina  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ . Existen numerosas formas de estructuras cristalinas de la sílice. Por ejemplo el cuarzo presenta una estructura ordenada. Otra forma cristalina es la sílice criptocristalina. La microsilice, generalmente utilizada para la producción de CAR, presenta simplemente una estructura de sílice amorfa.

La diatomita, vista al microscopio (40 X), esta conformada por partículas de numerosas formas de acuerdo a su origen desde alargadas hasta hexagonales. En cambio la microsilice son partículas redondeadas.

Estas diferencias en la estructura cristalina de la sílica, y las formas de la partícula tanto de la diatomita como de la microsilice nos dan a entender que, a pesar que contienen un alto contenido de sílice en su composición química, no se espera que se comporten igual físicamente.

Con respecto al aspecto químico, el comportamiento de la diatomita como de la microsilice es similar pues, ambas contienen puntos inestables dentro de sus moléculas, debido a su estructura amorfa, que las hacen más reactivas respecto a una estructura ordenada.

### **1.4.3. Propiedades Exigidas en un CAR**

Anteriormente, en los puntos 1.2.5 y 1.2.6 se han descrito las propiedades en estado fresco y en estado endurecido que se deben medir para evaluar a un CAR. Para estudiar el uso de la diatomita como adición mineral se evaluarán complementariamente dos propiedades: reología (Viscosidad de la pasta) y resistencia al ataque de sulfatos.

#### **1.4.3.1. Reología [5]**

Actualmente existen muchos ensayos a realizar en reología. Las propiedades de un concreto plástico, serán en función de muchos parámetros, tal como tipo y forma de agregados, contenido y

características del cemento, etc. Pero es útil aislar los efectos de la reología de la pasta.

Se escogió el ensayo de viscosidad, para entender el efecto de la pasta de cemento sobre las propiedades plásticas del concreto fresco. Para ello se utilizó el Viscosímetro Brookfield, que mide la viscosidad por torque.

La viscosidad es la medida de la fricción interna de un fluido, causado por la atracción molecular el cual hace resistir a la tendencia a fluir. El viscosímetro Brookfield mide esta fricción. Esta fricción llega a ser aparente cuando una capa de fluido es forzado a moverse en relación a otra capa. Mientras la fricción sea más grande, la cantidad de fuerza requerida será mayor para causar este movimiento, el cual es llamado "Corte". El corte ocurre cuando el fluido es físicamente movido. El principio de funcionamiento del Viscosímetro Brookfield se encuentra explicado en el Anexo D.

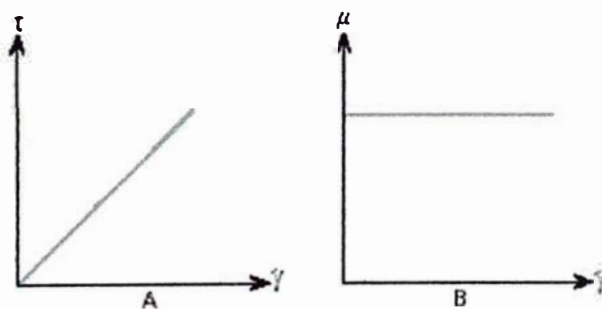
De investigaciones [26], se establece que:

- La consistencia o fluidez del concreto será una función de la viscosidad de la pasta de cemento.
- La cohesión del concreto será una función del umbral de cizallamiento (Yield Stress) de la pasta de cemento.

Otra razón para medir el comportamiento del fluido es por ejemplo determinar la potencia de bombeo requerida.

### Fluidos Newtonianos

Un fluido newtoniano es representado por la siguiente figura:



Donde  $\tau$  : Esfuerzo de Corte (Shear Stress)

$\gamma$  : Velocidad de Corte (Shear Rate),

$\mu$  : Viscosidad

En la práctica para una temperatura dada la viscosidad de un fluido newtoniano permanecerá constante no importando el modelo del viscosímetro, spindle o velocidad que se usa para medir. En términos matemáticos, se expresa:

$$\tau / \gamma = \mu$$

### **Fluidos no newtonianos**

En forma general es aquel donde la relación  $\tau / \gamma$  no es una constante. Las viscosidades de tales fluidos cambiarán cuando la velocidad de corte varía.

Un fluido no newtoniano es como una mezcla de moléculas con diferentes tamaños y formas. Cuando estas moléculas se mueven una con respecto a la otra como sucede durante el flujo, sus tamaños, formas y cohesividad determinaran cuanta fuerza es requerida para moverlos.

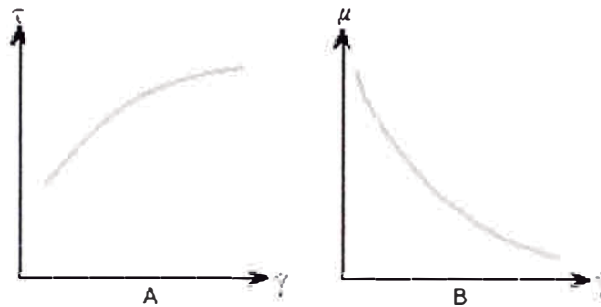
A cada velocidad de corte especifica, la alineación puede ser diferente y la necesidad de mayor o menor fuerza para mantener el movimiento.

Hay muchas formas de comportamiento de fluidos no newtonianos, caracterizados por la forma en el cual una viscosidad de un fluido cambia debido las variaciones de la velocidad de corte. Algunos tipos de fluidos no newtonianos son:

- Pseudoplástico
- Dilatante
- Plástico
- Tixotrópico

- Pseudoplástico

Este tipo de fluido mostrara una disminución en la viscosidad con el incremento de la velocidad de corte.



Donde  $\tau$  : Esfuerzo de Corte (Shear Stress)

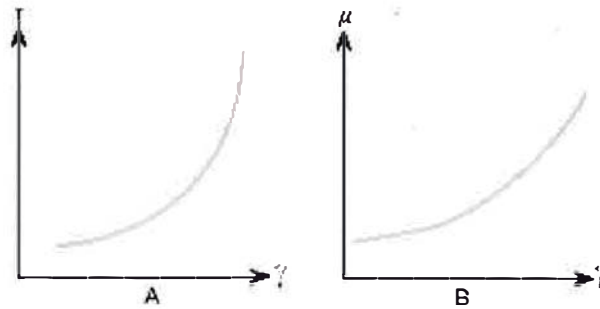
$\gamma$  : Velocidad de Corte (Shear Rate),

$\mu$  : Viscosidad

Un forma fácil de entender es cuando el Spindle del viscosímetro esta girando dentro el fluido, la estructura de las moléculas de la muestra serán temporalmente cambiadas, y la formación de la molécula estará orientada más paralelamente a la superficie de la aguja. Cuando la velocidad de rotación (RPM) se aumenta, entonces la mayor parte de la estructura de las moléculas formadas será destruida y la menor parte de la estructura de las moléculas se moverán juntas y por tanto la viscosidad disminuirá. Las formas mas comunes de un fluido pseudoplástico incluyen, las pinturas, emulsiones y dispersiones de muchos tipos.

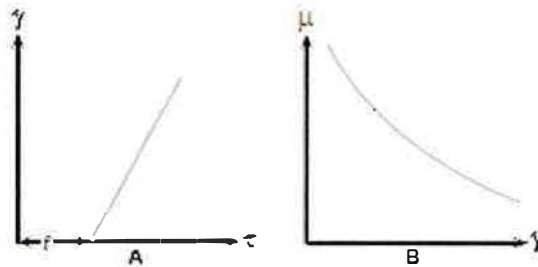
- Dilatante

Un fluido dilatante se caracteriza por incrementar su viscosidad cuando se incrementa la velocidad de corte. Algunas veces se le conoce "Shear Thickening"



- Plástico

Este tipo de fluido se comportara como un sólido bajo condiciones estáticas. Una cierta cantidad de esfuerzo debe ser aplicado al fluido antes de que cualquier flujo sea inducido. Este esfuerzo es llamado "Yield Stress" (esfuerzo critico). Una vez que el valor "Yield Stress" es excedido, empieza el flujo. Los fluidos plásticos pueden mostrar características de un fluido Newtoniano, Pseudoplástico o Dilatante.



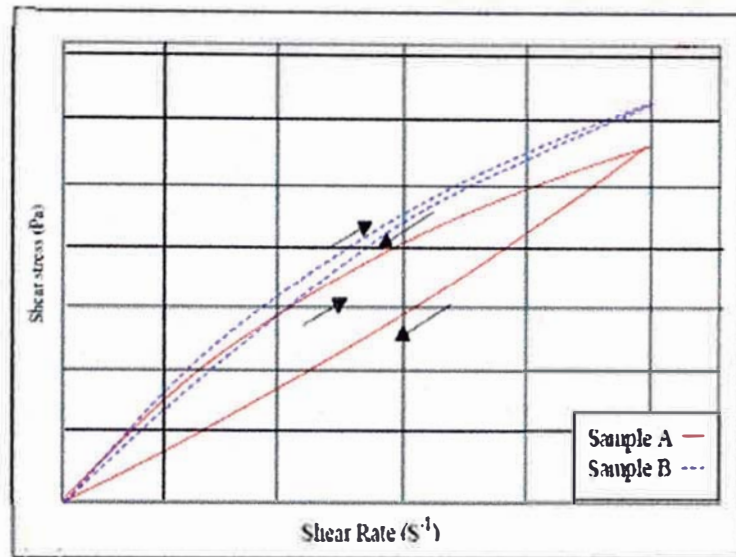
Si se considera el tiempo como otra variable, tendremos dos tipos más de fluidos no newtonianos, los tixotrópicos y reotrópicos.

- Tixotrópico

Se caracteriza por la disminución en la viscosidad aparente bajo movimiento, seguido de un reestablecimiento cuando se detienen el movimiento. Este efecto lo hace un tipo de fluido dependiente del tiempo. Es importante reconocer la tixotropía en los materiales, pues esto puede afectar la precisión y reproducibilidad de las propiedades del concreto.



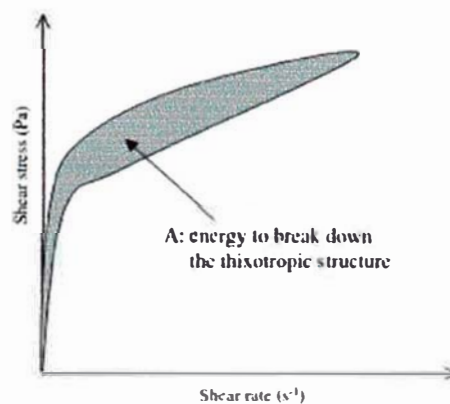
Fig. 1.17. Histéresis o loop de un Fluido tixotrópico.



La histéresis o loop de las muestras A y B en la figura de arriba, han sido formados por un incremento y luego reducción de la velocidad de corte. De acuerdo al área formada se puede decir que la muestra A presenta un alto grado de tixotropía a comparación con la muestra B, la cual no presenta tixotropía, pues tanto la curva hacia arriba y hacia abajo casi se superponen una con otra.

La tixotrópica puede ser evaluada determinando el área, como se muestra en la siguiente figura.

Fig. 1.18. Evaluación de la tixotropía.



De acuerdo a la definición de tixotropía, mientras más grande sea el área tixotrópica o de histéresis, mayor será la cantidad de energía por

volumen necesaria para hacer fluir el concreto luego de un periodo de reposo. [15]

$$\text{Area of hysteresis} = \tau \times \gamma \text{ [Pa. 1/s]} = \text{N/m}^2 \times 1/\text{s} = \text{Nm/s} \times 1/\text{m}^3$$

$$A = \text{work/shear time} \times 1/\text{volume} = \text{energy/volume}$$

Tener en cuenta que las graficas tixotrópicas no dan una medida absoluta de la tixotropía, sino que nos brindan una idea de la tixotropía por medio de la comparación con otras sustancias.

#### 1.4.3.2. Resistencia al Ataque de Sulfatos [17]

Adicionalmente para medir la durabilidad del concreto con incorporación de diatomita se implementó un procedimiento para evaluar el concreto frente al ataque de sulfatos basados en investigaciones realizadas y las normas ASTM.

Uno de los factores que afectan la durabilidad de un concreto es la agresión química. Los compuestos que pueden causar daño importante al concreto son los cloruros y sulfatos. Los primeros tienen acción directa sobre los metales embebidos. Los sulfatos, que normalmente se hallan en el suelo y/o están en disolución en aguas subterráneas o contaminadas por desechos, tienen una acción directa sobre las pastas de cemento produciendo expansión y desintegración.

La resistencia del concreto al ataque de sulfatos depende de:

- Las condiciones de exposición
- La permeabilidad del concreto
- La composición del cemento, tal como la alúmina y la cantidad de Pórtlandita formado durante el proceso de hidratación
- Tipo de sal de sulfato.

El sulfatos alcalinos atacan al concreto de la siguiente manera: Los sulfatos reaccionan con el Hidróxido de Calcio o Portlandita, que es

producido durante la hidratación del cemento, formando yeso, el cual reacciona con el aluminato tricálcico en el concreto para formar compuestos sulfoaluminosos. Estas reacciones resultan en un incremento en el volumen con subsecuente fisuración.

Reacciones frente a la exposición de Sulfato de Sodio ( $\text{Na}_2\text{SO}_4$ ):

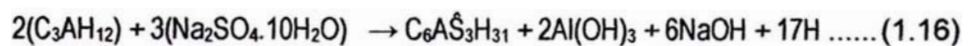
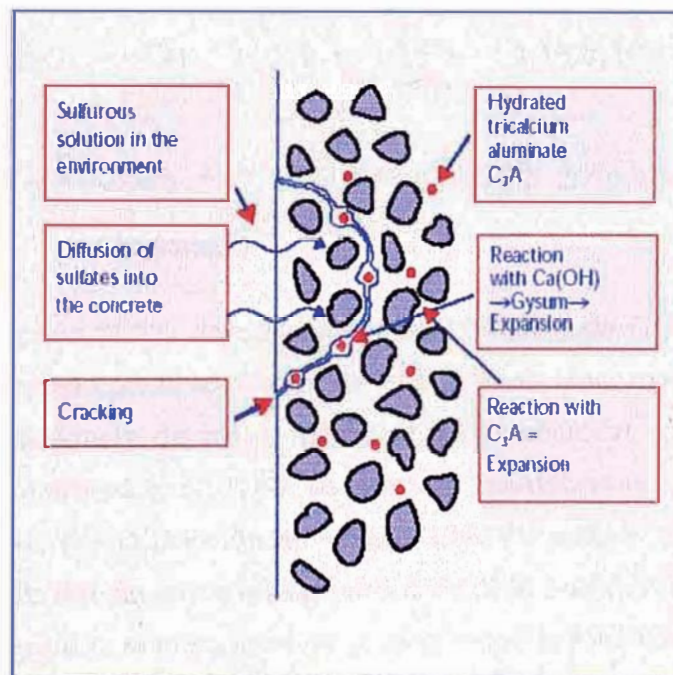


Fig. 1.19. Representación esquemática del Ataque de Sulfato de Sodio en el concreto

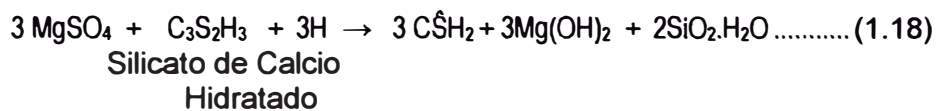


Los C-S-H no se ven afectados cuando las pruebas se realizan con Sulfatos alcalinos, debido a que el pH permanece estable por la generación de NaOH, en cambio cuando las pruebas se realizan con

sulfato de magnesio las condiciones cambian como se vera a continuación.

El ataque por sulfato de magnesio es generalmente un ataque más severo que el causado por los sulfatos alcalinos. En este caso, en presencia de hidróxido de calcio se producen dos nuevos sólidos (yeso e hidróxido de magnesio) que generan esfuerzos, contribuyendo a una expansión del material. Por un lado, la formación de yeso produce compuestos sulfoaluminosos al reaccionar con los aluminatos tricálcicos, y por el otro, precipita el hidróxido de magnesio disminuyendo el pH de la fase líquida, lo cual facilita la descomposición de la fase C-S-H y provoca una pérdida de la capacidad de enlace del material.

Reacciones por ataque con sulfato de magnesio:



La reacción (1.17) de conversión del hidróxido de Calcio a yeso es acompañada por la formación de Hidróxido de Magnesio insoluble.

En la ausencia de iones hidroxilos en la solución, los silicatos de calcio hidratados (C-S-H) se vuelven inestables y por tanto son atacados por la solución de sulfato. Esto no sucede si fuera Sulfato de Sodio debido a que en los productos de la reacción con pórtlandita se asegura la continuación de la alcalinidad al formarse hidróxido de sodio.

No hay una norma con la que se pueda medir la resistencia a los sulfatos en concreto, pero si morteros y cemento. La mayoría de métodos de determinación de la resistencia a los sulfatos, evalúa la expansión de los morteros sometidos a la acción de los sulfatos. La norma ASTM contempla dos ensayos:

- Por sobredosaje de yeso para los Pórtland (ASTM C452), se basa en la mezcla de cemento con yeso).
- Por inmersión en solución de sulfato de sodio para todos los cementos (ASTM C1012), evalúa el cambio de longitud del mortero expuesto, se requiere realizar mediciones por largos periodos de tiempo (desde un año).

De investigaciones realizadas [17], en donde ensayos normalizados se han adaptado de acuerdo al requerimiento, tenemos el método no normalizado de Curado al Vapor, la cual se describe a continuación.

**Método de Curado al vapor:** Similar al Método de la ASTM C1012. Emplea el curado al vapor para el pre-endurecimiento de los especímenes y granulometría incompleta en la arena (retirando la fracción fina) para fomentar su porosidad. El curado a 85°C por dos horas asegura una estructura porosa muy similar a la desarrollada a temperatura normal, facilitando la evaluación. La ejecución del método es sencilla y entrega resultados en 8 semanas.

Para la evaluación del ensayo de Resistencia al Ataque de Sulfatos en la presente tesis se ha tomado como referencia este último método, con la diferencia que los especímenes a utilizar son los provenientes de los diseños en estudio (no se le ha retirado la fracción fina como el método). Y se va a utilizar  $MgSO_4$  para conseguir un medio más severo de ataque.

En resumen, se espera que un CAR sea resistente frente a un ataque de sulfatos, a baja relación agua/cemento la permeabilidad disminuye y no va a permitir el ingreso de sustancias que puedan atacar al concreto.

## **CAPÍTULO II: ENSAYOS EXPERIMENTALES PRELIMINARES**

### **2.1 INTRODUCCIÓN**

Los resultados de estos ensayos preliminares representan una valiosa información, que facilitaran predecir si los diseños elaborados alcanzaran las propiedades mínimas para constituir un Concreto de Alta Resistencia, CAR.

### **2.2 OBJETIVOS**

Esta etapa tiene como finalidad:

- Caracterizar los materiales a utilizar.
- Definir el proporcionamiento de los diferentes diseños por evaluar de acuerdo al Diseño de Referencia (DCR: Microsilíce)
- Evaluar de acuerdo a las normas ASTM y recomendaciones del ACI, la tendencia de las resistencias a la compresión a los 3, 7, 28 días de los diseños obtenidos, dado que es la propiedad principal de un concreto de alta resistencia.

### **2.3 CARACTERIZACIÓN DE LAS MATERIAS PRIMAS, INSUMOS Y ADITIVOS**

Todos los materiales por utilizar fueron designados por la empresa UNICON. Para su caracterización se utilizo los certificados de Calidad otorgados por las empresas proveedoras. Adicionalmente se realizaron algunos ensayos en Laboratorio de Unicon.

### 2.3.1 Cemento Pórtland

- **Procedencia:**

Se utilizó el Cemento Pórtland Tipo IBA  
Este tipo de cemento es un Tipo I de bajo contenido de Álcalis,  
Es manufacturado por Cementos Lima S.A.

- **Ubicación y Embalaje:**

El proveedor transporta este cemento en camiones especiales presurizados (bombonas) a fin de evitar su degradación por efectos de la humedad ambiental.

Este cemento es descargado a los silos de UNICON.

Para los ensayos de la presente tesis:

Se retiró el cemento directamente de las bombonas y se dispuso en cilindros plásticos debidamente tapados.

- **Edad:**

Siendo uno de los principales insumos de las actividades de construcción de UNICON, el cemento disponible siempre es del día.

- **Características Físicas y Químicas:**

De acuerdo al Informe de Control de Calidad, emitido por Cementos Lima S.A., se puede comprobar que este cemento cumple con las especificaciones exigidas por la norma ASTM C150 para un cemento Pórtland Tipo I.

El certificado de calidad del cemento esta adjuntado en la siguiente página



CEMENTOS LIMA S.A.

## INFORME DE CONTROL DE CALIDAD SOBRE CALIDAD DE CEMENTO

TIPO DE CEMENTO: CEMENTO PORTLAND TIPO I BA

REMITIDA A:

COMPOSICION TIPICA DEL MES: DICIEMBRE


FECHA: 23/12/2005

ANALISIS QUIMICO	VALORES	NTP 334.009, ASTM C-150 CEMENTO PORTLAND REQUISITOS	
DIOXIDO DE SILICE (SiO <sub>2</sub> ) %	20.71		
OXIDO DE ALUMINIO (Al <sub>2</sub> O <sub>3</sub> ) %	4.92		
OXIDO DE FIERRO (Fe <sub>2</sub> O <sub>3</sub> ) %	2.23		
OXIDO DE CALCIO (CaO) %	64.26		
OXIDO DE MAGNESIO (MgO) %	3.19	MAX.	6.00 %
TRIOXIDO DE AZUFRE (SO <sub>3</sub> ) %	2.61	MAX.	3.50 %
OXIDO DE POTASIO (K <sub>2</sub> O) %	0.51		
OXIDO DE SODIO (Na <sub>2</sub> O) %	0.19		
PERDIDA POR IGNICION (P.I.) %	0.92	MAX.	3.00 %
Total	99.54		
ALCALIS TOTALES %	0.53		
INSOLUBLES %	0.30	MAX.	0.75 %
CAL LIBRE (CaO (l))	0.80		
C <sub>3</sub> S	60.53		
C <sub>2</sub> S	13.70		
C <sub>3</sub> A	9.26		
<b>ENSAYOS FISICOS</b>			
FINEZA MALLA - 100 %	0.08		
MALLA - 200 %	0.85		
MALLA - 325 %	5.55		
SUPERFICIE ESPECIFICA BLAINE cm <sup>2</sup> /g	3700	MIN.	2800 cm <sup>2</sup> /g
CONTENIDO DE AIRE %		MAX.	12.00 %
EXPANSION AUTOCLAVE %		MAX.	0.80 %
DENSIDAD g/cm <sup>3</sup>			
FRAGUADO VICAT INICIAL (min)	120	MIN.	45 min
FRAGUADO VICAT FINAL (min)	255	MAX.	375min
RESISTENCIA A LA COMPRESION (kg/cm <sup>2</sup> )			
24 HRS	169		
3 DIAS	278	MIN.	122 kg/cm <sup>2</sup>
7 DIAS	365	MIN.	194 kg/cm <sup>2</sup>
28 DIAS	454		
COMENTARIOS: LA RESISTENCIA A LA COMPRESION A LOS 28 DIAS CORRESPONDE AL MES DE NOVIEMBRE 2005.			

Este informe muestra las CARACTERISTICAS TIPICAS DEL PROMEDIO MENSUAL DE LA PRODUCCION confirmando que este cemento cumple las especificaciones de las Normas Técnicas Peruanas NTP 334.009 y ASTM C-150.

División de Control de Calidad

V<sup>o</sup> B<sup>o</sup>

  
Ing. Ruben Gilvonio A.  
Jefe de División de Control de Calidad

  
Ing. E. Veleznoro  
Gerente de Operaciones



## 2.3.2 Adiciones minerales: Diatomita FILLITE y Microsilíce

### 2.3.2.1 Diatomita Fillite

- **Procedencia:**

En el Perú, la diatomita es explotada por tres empresas: Insumex S.A., Agregados Calcáreos S.A. y Celite del Perú S.A. Todos los ensayos experimentales fueron realizados con la diatomita que provee Agregados Calcáreos, quienes producen dos tipos de Diatomita: Fillite y Biocel. Estos materiales solo pasan por proceso de extracción, molienda y embolsado.

- **Ubicación y Embalaje:**

La diatomita ha sido entregada en sacos de papel de 20 Kg., debidamente sellados por el proveedor, que son almacenados a temperatura ambiente, y protegido de la intemperie.

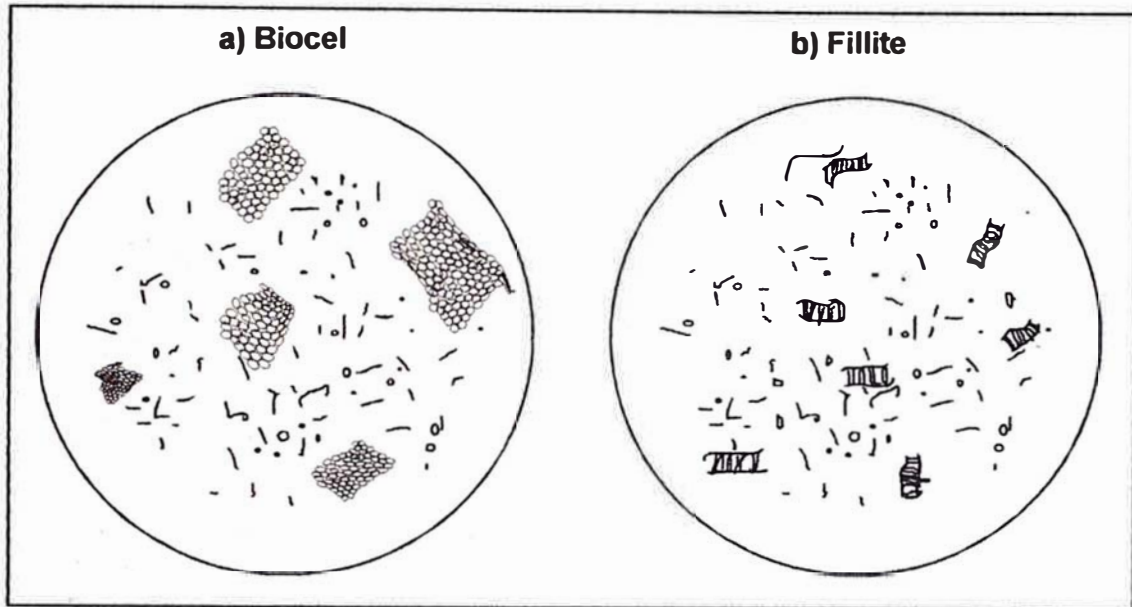
- **Edad:**

Para los Ensayos Preliminares, UNICON S.A. dispuso de dos sacos, recientemente envasados.

- **Caracterización Química y Física:**

De acuerdo a la observación microscópica a 40X, de los dos tipos de diatomita mostradas en la figura 2.1, la diatomita Fillite es de procedencia lacustre y la diatomita Biocel es de procedencia marina. Se seleccionó la diatomita Fillite por ser de mejor calidad, reconociéndola también por el color. La diatomita Fillite es de color blanco y la Biocel de color verdoso. La empresa proveedora indicó que la diatomita Fillite es extraída de las canteras ubicadas en el departamento Ayacucho.

**Fig. 2.1.** Representación Esquemática de las Diatomitas observadas en un Microscopio Óptico a 40X (COMACSA)



Las diatomitas lacustre son de mejor calidad que las diatomitas marinas, el ensayo microscópico nos permitirá hacer un control de calidad a este mineral pues nos indica el tipo de diatomita que se esta entregando. Adicionalmente se puede realizar una inspección visual de la diatomita, cuyo color es blanco, un cambio del color nos dará la indicación de presencia de impurezas en la diatomita.

Para la caracterización química y física, se incluye el certificado de calidad de la diatomita seleccionada entregada por el proveedor. Adicionalmente se incluye los resultados de los análisis químicos realizados por la empresa A.R.P.L Tecnología Industrial S.A.

Acorde con la clasificación de la Norma ASTM C618, la diatomita a utilizar es una puzolana natural perteneciente a la Clase N, por tanto debería cumplir los requisitos químicos y físicos que exige la norma y que se han incluido en el punto 1.2.3.2.

La norma indica que el cumplimiento de estos requisitos no predice la performance de la diatomita en el concreto, pero colectivamente ayuda a describir su composición y su uniformidad en el mineral.



Cía Minera  
AGREGADOS CALCAREOS S.A.

## FILLITE

### ANALISIS QUIMICO TIPICO

SiO <sub>2</sub>	.....	81.33 %
Al <sub>2</sub> O <sub>3</sub>	.....	8.03 %
CaO	.....	1.16 %
MgO	.....	0.00 %
SO <sub>3</sub>	.....	0.14 %
Perdida por Calcificación	.....	8.48 %
No analizado	.....	0.86 %
<b>TOTAL</b>	.....	<b>100.00 %</b>
pH	.....	8.50

### PROPIEDADES FISICAS TIPICAS

Superficie Especifica (Blaine)(ASTM C-204)cm <sup>2</sup> /g	37,200
Densidad Aparente g/l	220
Gravedad especifica	2.2
Humedad %	7.5
Absorción de aceite % (ASTM D-281)	92
Fineza Hegman NS (ASTM D-1210)	4.5 - 5
Granulometría: Menos Malla 325 (% min.)	99.90
Partida (SEDIGRAPH) µm	25 ± 5
Tamaño medio de partículas (SEDIGRAPH) µm	1.50 ± 0.5
Partículas submicrónicas (SEDIGRAPH) %	35 ± 5
Blancura - Photovolt %	82
Abrasividad Valley mg	140

YB:kb

(57)1/02

**INFORME DE ENSAYOS**  
**N° 730-OC/05**

**N° DE SOLICITUD** : 558/05  
**TIPO DE MUESTRAS** : Puzolana  
**PRESENTACIÓN** : Material polvo, en bolsa plástica  
**SOLICITADO POR** : Unión de Concreteras S.A.  
**DIRECCIÓN** : Panamericana Sur Km 11, San Juan de Miraflores  
**CARTA / CORREO E** : Del 15/09/05  
**FECHA DE RECEPCIÓN** : 15-09-05  
**FECHA DE EMISIÓN** : 03-10-05  
**FECHA DE ENSAYO** : Del 19 al 29-09-05

Pág. 1 de 1

**RESULTADOS**

ENSAYOS		Puzolana Diatomita Fillite	Métodos
SiO <sub>2</sub> (dióxido de silicio)	%	90.41	PTQ-RX02-94
Al <sub>2</sub> O <sub>3</sub> (óxido de aluminio)	%	1.47	"
Fe <sub>2</sub> O <sub>3</sub> (óxido férrico)	%	0.61	"
CaO (óxido de calcio)	%	0.31	"
MgO (óxido de magnesio)	%	0.21	"
SO <sub>3</sub> (trioxido de azufre)	%	0.00	"
Na <sub>2</sub> O (óxido de sodio)	%	0.10	"
K <sub>2</sub> O (óxido de potasio)	%	0.26	"
TiO <sub>2</sub> (dióxido de titanio)	%	0.09	"
P <sub>2</sub> O <sub>5</sub> (peróxido de fósforo)	%	0.08	"
Mn <sub>2</sub> O <sub>3</sub> (óxido de manganeso)	%	0.03	"
SrO (óxido de estroncio)	%	0.01	"
Cr <sub>2</sub> O <sub>3</sub> (óxido de cromo)	%	0.02	"
Pérdida al fuego	%	5.48	ASTM C311-00
TOTAL	%	99.08	
Cl <sup>-</sup> (cloruros)	%	0.00	PTQ-RX02-94

ENSAYOS FÍSICOS		Puzolana Diatomita Fillite	Métodos
Retenido Malla 325	%	0.27	ASTM C430-96
Densidad	g/cm <sup>3</sup>	2.01	ASTM C188-95
<b>Índice de actividad puzolanica*</b>			
Requerimiento de agua	%	141.32	ASTM C311-00 Sec.25-29
Índice de Actividad a 7 días	%	64.74	"

**OBSERVACIONES**

El ensayo PTQ-RX02-94, corresponde a una estandarización particular de materiales, de carácter semicuantitativo  
\* Se trabaja con Cemento tipo I,BA de Cementos Lima.

Los resultados obtenidos corresponden a la muestra analizada  
La muestra, la identificación y los datos de referencia fueron proporcionados por el cliente  
El Informe no puede ser utilizado como un certificado del producto

A.R.P.L. TECNOLOGIA INDUSTRIAL S.A.

  
Ing. HUGO VELARDE  
Jefe Departamento de Laboratorio



**INFORME DE ENSAYOS**  
**N° 799-OC/05**

**N° DE SOLICITUD** : 552/05  
**TIPO DE MUESTRAS** : Puzolana  
**PRESENTACIÓN** : Material polvo, en bolsa plástica  
**SOLICITADO POR** : Unión de Concreteras S.A.  
**DIRECCIÓN** : Panamericana Sur Km 11, San Juan de Miraflores  
**CARTA / CORREO E:** : Del 15/09/05  
**FECHA DE RECEPCIÓN** : 15-09-05  
**FECHA DE EMISIÓN** : 31-10-05  
**FECHA DE ENSAYO** : Del 19-09 al 20-10-05

Pág. 1 de 1

**RESULTADOS**

ENSAYOS FÍSICOS		Puzolana Diastomita Fábrica	Métodos
<b>Índice de actividad puzolánica*</b>			
Índice de Actividad a 28 días	%	86.72	ASTM C311-00 Sec 25-29
Índice de Actividad Acelerado (7 días)	%	124.59	ASTM C1240-95 Sec.17

**OBSERVACIONES**

\* Se trabaja con Cemento tipo I BA de Cementos Lima.

Los resultados obtenidos corresponden a la muestra analizada  
La muestra, la identificación y los datos de referencia fueron proporcionados por el cliente  
El Informe no puede ser utilizado como un certificado del producto

ARPL TECNOLOGÍA INDUSTRIAL S.A.  
Ing. HUGO LAZO VELARDE  
Jefe Departamento de Laboratorio

### Requisitos Químicos

El Cuadro 2.1 muestra que la composición química de la diatomita cumple ampliamente con los requisitos químicos para ser usados como adición mineral, a excepción de la humedad. Este requisito es manejable pues se puede solicitar al proveedor Diatomita con un contenido de humedad en un rango determinado, se recomienda utilizar la Diatomita recién procesada. Además, el balance de agua en el diseño de mezcla puede considerar la humedad de la Diatomita. También se aprecia que la variación de la composición química ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  %) entre los dos lotes analizados es de 2.5%.

**Cuadro 2.1.** Comparación de dos Lotes de Diatomita Nacional de acuerdo a los Requisitos Químicos de la ASTM C618

<b>Requisitos ASTM C618</b>	<b>COMACSA (Lote 1)</b>	<b>COMACSA (Lote 2)</b>	<b>ARPL (Lote 1)</b>
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ % - 70 Min.	91.91	89.36	92.49
$\text{SO}_3$ % - 4 Máx.	0.08	0.14	0.00
Humedad % - 3 Máx.	8.09	7.5	-
Pérdida por Ignición % - 10 Máx.	4.14	8.48	5.48

### Requisitos Físicos

Con respecto a los requisitos físicos dados en la Norma ASTM C618, el Índice de Actividad de Resistencia y de ensayo de Fineza Malla 325 se realizaron en el laboratorio de la empresa A.R.P.L. Tecnología Industrial S.A. y los resultados se muestran en el Cuadro 2.2 (El certificado de calidad ha sido mostrado anteriormente). El método de ensayo está incluido en la ASTM C311.

**Cuadro 2.2.** Comparación de los Requisitos Físicos ASTM C618

Requisitos ASTM C618	ARPL (Lote 1) %	COMACSA (Lote 1) %
Requerimiento de Agua – 115% Max.	141.32	-
Índice de Actividad a 7 días – 75% Min.	64.74	-
Índice de Actividad a 28 días – 75% Min.	86.72	-
Retenido Malla 325 – 34% Max.	0.27	0.1

**Fuente:** Informe de Ensayos N° 730-OC/05 y 799-OC/05 – Empresa. A.R.P.L. Tecnología Industrial S.A.

Si comparamos los resultados obtenidos con los valores límites Cuadro 2.2 se concluiría que la Diatomita no cumpliría con las exigencias normativas para ser utilizado como adición mineral en reemplazo de la microsilice.

Para revertir esta situación y de acuerdo a la literatura técnica se procedió a la adición de un aditivo superfluidificante a base de policarboxilato (Viscocrete 1). Como el ensayo de índice de actividad de resistencia con Cemento Pórtland no indica el procedimiento de adición de aditivo, este se adicionó junto con el agua.

Empleando una metodología similar a la indicada en el ASTM C311 se realizaron ensayos de índice de actividad de resistencia con adición de aditivo para reemplazos de 5, 10, 15 y 20% con Diatomita y 10% con Microsilice, para observar el comportamiento de la resistencia del mortero en cada caso. Estas experiencias fueron realizadas en los laboratorios de UNICON.

En el Grafico 2.1 y en el Cuadro 2.3 se muestran los resultados obtenidos de estas pruebas.

De acuerdo a los resultados obtenidos se determina que la diatomita es apta para ser utilizada como adición mineral, siempre y cuando se utilice en conjunto con aditivos reductores de agua, pues cumple con los requisitos máximos y mínimos que señala la norma ASTM C618.

**Cuadro 2.3. Resumen de Índices de Actividad de Resistencia con adición de Aditivo**

<b>Descripción</b>	<b>Limites</b>	<b>Diatomita 5%</b>	<b>Diatomita 10%</b>	<b>Diatomita 15 %</b>	<b>Diatomita 20%</b>	<b>Microsilíce 10%</b>
Requerimiento de Agua	115% Máx.	100.7%	102.1%	104.1%	105.8%	102.1%
Índice de Actividad 7 días	75% Min.	113.3%	129.0%	122.1%	114.6%	114.7%
Índice de Actividad 28 días	75% Min.	103.1%	114.1%	125.9%	111.0%	140.0%

**Nota:**

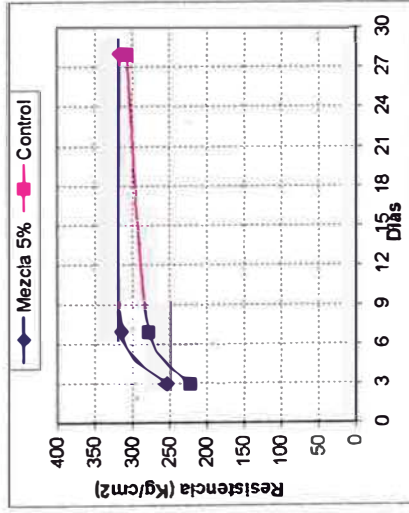
Se define el Índice de Actividad con adición de Aditivo como la relación de las resistencias alcanzadas de cada muestra respecto a resistencia de su respectiva muestra de control.

Los límites mostrados son los indicados en la ASTM C618 para Índices de Actividad de Resistencia.

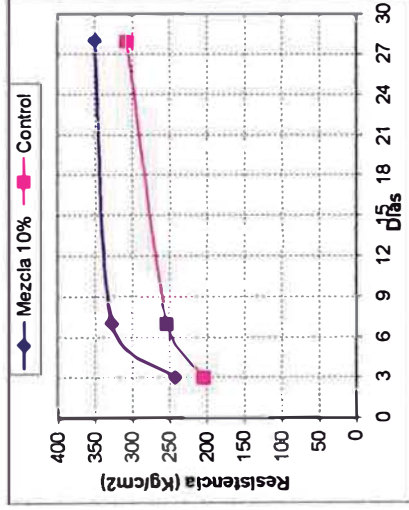


**Grafico 2.1. Resultados Ensayos de Resistencia a la Compresión en Mortero con empleo de Aditivo Superfluidificante**

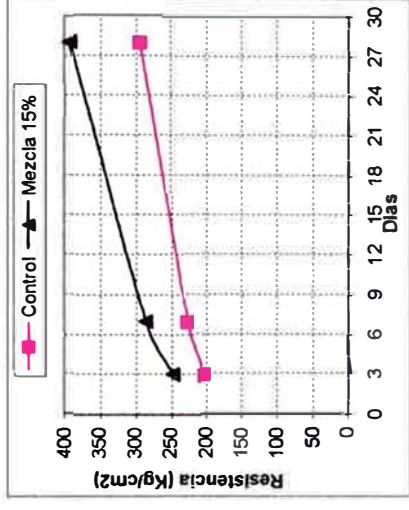
**MEZCLA PRUEBA 5% DIATOMITA**



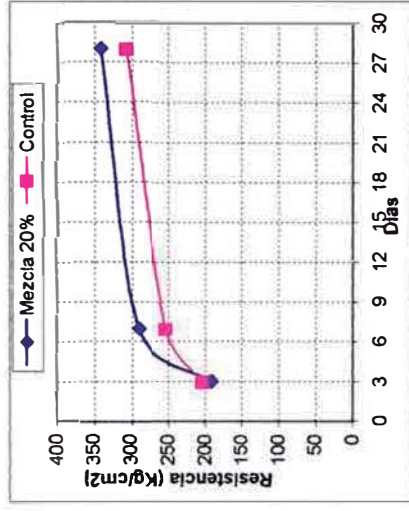
**MEZCLA PRUEBA 10% DIATOMITA**



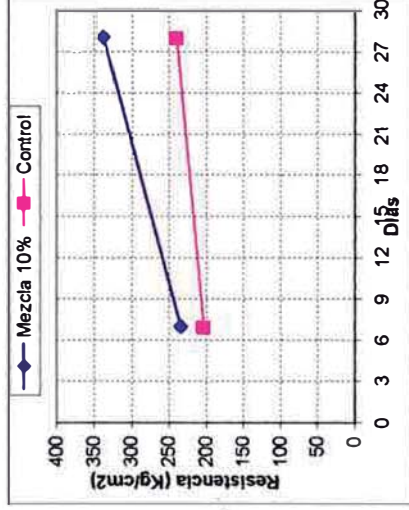
**MEZCLA PRUEBA 15% DIATOMITA**



**MEZCLA PRUEBA 20% DIATOMITA**



**MEZCLA PRUEBA 10% MICROSILICE**



### 2.3.2.2 Microsilíce

- **Procedencia:**

Producto importado cuyo nombre comercial es Sika Fume y es comercializado por Sika Perú S.A.

Este producto es generalmente utilizado para elaborar concretos de Alta Resistencia, éste será utilizado como adición mineral de referencia-patrón para el presente trabajo.

- **Ubicación y Embalaje**

Envasado, debidamente sellados en depósitos de UNICON

- **Caracterización Química y Física**

El uso de esta adición mineral es común en el CAR y esta normalizado por la ASTM C1240, en la cual se incluyen requisitos químicos y físicos exigidos.

Se utilizará el certificado de calidad, otorgada por el proveedor, incluido más adelante.

Los cuadros 2.4 y 2.5 muestran de manera comparativa las características físicas y químicas de la microsilíce y de la diatomita seleccionada versus los requisitos exigidos por la norma ASTM C 1240.

La diatomita no cumple las características de: superficie específica y % humedad que posee la microsilíce.

La forma de la partícula de la microsilíce es esférica, de acuerdo al certificado de calidad.

Estas diferencias van a influenciar en las propiedades del concreto con diatomita y con microsilíce.

Cuadro 2.4. Microsilice: Características Químicas

Requisitos ASTM C1240	Sika Fume SIKA PERU	Diatomita COMACSA (Lote 1)
Silice (SiO <sub>2</sub> ) 85 % min.	96.4	91.91
Contenido de humedad, máx., 3 %	1.5 Máx.	8.09
Pérdida por ignición, máx., 6%	3.5	4.14

Cuadro 2.5. Microsilice: Características Físicas

Requisitos ASTM C1240	Sika Fume SIKA PERU	Diatomita COMACSA (Lote 1)
Tamaño mayor: Porcentaje retenido en tamiz de 45µm (N° 325), máx., 10%	0 - 5	0.1
Índice de actividad puzolánica acelerada*: Con cemento Pórtland a 7 días, 105 mín. % del control	-----	124.59
Superficie Especifica (cm <sup>2</sup> /g), min. 150 000	180 000 – 200 000	37 200

\* Esta prueba solo se la realiza a la diatomita en ARPL, Informe de Ensayos N° 730-OC/05 incluido en anexo A., pues es reconocido que la microsilice SIKA FUME cumple con este requisito.

# Certificado de Calidad

El presente documento presenta el Estado Permisible de las especificaciones técnicas de nuestro producto **SikaFume**

## Resultado de los Análisis:

SiO <sub>2</sub>	96.4
Fe <sub>2</sub> O <sub>3</sub>	0.17
Al <sub>2</sub> O <sub>3</sub>	0.26
CaO	0.41
S	0.10
K <sub>2</sub> O	0.80
C	2.60
MgO	0.27
Na <sub>2</sub> O	0.11
L.O.I.	3.5

## Características Físicas Típicas:

pH al 10%	7.5
Sólidos por Desección (%)	100
Superficie Especifica (m <sup>2</sup> /g)	18 – 20
Gravedad Especifica	2.2
Densidad Bulk (kg/m <sup>3</sup> )	250 – 300
Fineza (ave. dia.) (um)	0.1 – 0.2
Malla 45 um (%)	95 – 100
Forma de la Particula	esférica
% de Humedad	1.5% máximo

Canadian Standard  
Sika Fume es amorfo  
Fecha de Vencimiento

CAN/CSA-A23.5-M86

: 06 meses

## Referencia:

NICC : 1121000  
Edición : 1

Atentamente,

Laboratorio de Control de Calidad e I+D

Lima 17 de Marzo del 2006

Formato CC-F 12  
Autorizado por: MPP  
Fecha: 28.11.01  
Edición: 2

MIRIAM YULI PINTADO PINTADO  
QUIMICO  
C.O.P. 556



Sika Perú S.A. / Av. Los Frutales 253 / Ale / Lima 3 – Perú  
Tel: (51-1) 618-6060 / Fax: (51-1) 618-6070 / [www.sika.com.pe](http://www.sika.com.pe)

### 2.3.3 Agregados

Los agregados grueso y fino que se utilizarán cumplen con las especificaciones de la norma ASTM C33.

Se seleccionó agregado grueso H67, recomendado para CAR según el ítem 1.2.3.3.

#### - **Procedencia:**

Se utilizaron los agregados de la cantera de Jicamarca, ubicada en Huachipa, al este de Lima.

La cantera produce agregado fino, y grueso de los siguientes Husos Granulométricos 5, 57, 67, 89, 3.

Estos agregados son material manufacturado, las cuales pasan por procesos:

- Chancado
- Zarandeo
- Tamizado
- Lavado

Pasan por un estricto control de calidad y monitoreo continuo garantizando la homogeneidad del producto en la granulometría correspondiente.

La planta tiene una producción de 300 TN/hora.

#### - **Ubicación y Embalaje**

En Patios de Acopio en la Planta UNICON

#### - **Caracterización Física y Química:**

La caracterización tanto física como química está incluida en las siguientes páginas.

**INFORME DE ENSAYOS**  
**N° 831-NV/05**

**N° DE SOLICITUD** : 612/05  
**TIPO DE MUESTRAS** : Agregados  
**PRESENTACIÓN** : Bolsas plásticas  
**SOLICITADO POR** : Unión de Concreteras S.A.  
**DIRECCIÓN** : Panamericana Sur Km 11, San Juan de Miraflores  
**CARTA / MEMORANDUM** : USL-077/05  
**FECHA DE RECEPCIÓN** : 12/10/2005  
**FECHA DE EMISIÓN** : 10/11/2005  
**FECHA DE ENSAYO** : Del 05/10 a 04/11/05

Pág. 1 de 2

**RESULTADOS**

ENSAYOS		Agregado Grueso Huso 67 Jicamarca	Métodos
Humedad total	%	1.75	ASTM C566-97
Material menor a 75 µm	%	0.37	ASTM C117-95
Modulo de fineza		6.55	ASTM C136-01
Gravedad Esp. de masa		2.69	ASTM C128/C127-01
GEM superficie sat. Seca		2.72	"
Gravedad Esp. Aparente		2.76	"
Absorción	%	0.85	"
Peso Unitario sin compact.	Kg/m3	1,497	ASTM C29/C29M-97
Peso Unitario compactado	Kg/m3	1,592	"
Vacios	%	40.70	"
Terrones arcilla y friables	%	0.23	ASTM C142-97
Sulfatos solubles	ppm	23	GID-LA-ME-016
Cloruros solubles	ppm	43	GID-LA-ME-015
Sales totales solubles	ppm	237	GID-LA-ME-014
<b>Abrasión Maquina de los Angeles :</b>			
Granulometria empleada		B	ASTM C131-01
Coefficiente de desgaste	%	17.2	"

A.R.P.L. TECNOLOGIA INDUSTRIAL S.A.

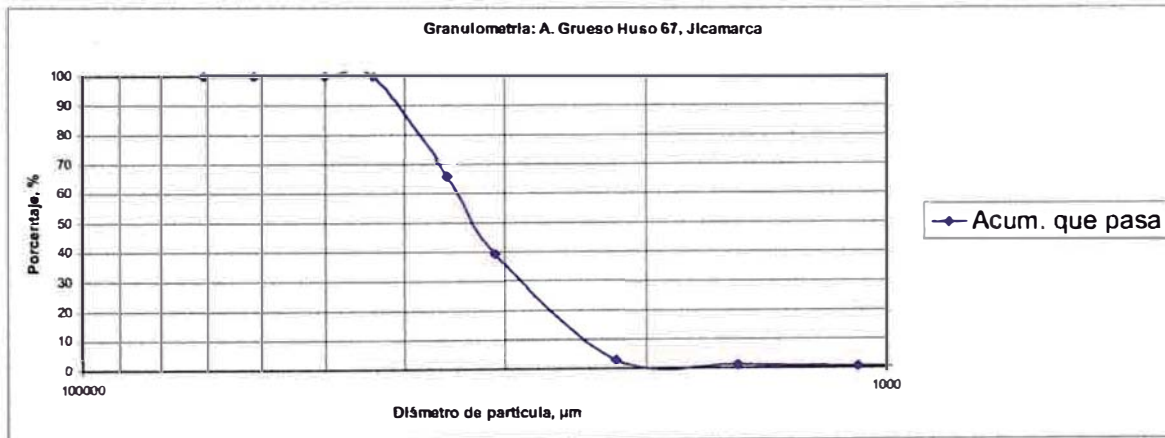
  
Ing. RUSO LUIS VELARDE  
Jefe Departamento de Laboratorio



INFORME DE ENSAYOS  
N° 831-NV/05

Análisis granulométrico de Agregado Grueso Huso 67, Jicamarca

Malla N°	Equivalente $\mu\text{m}$	Fracción Retenida g	Fracción Retenida %	Acumulado Ret %	Acum. que pasa %
2 "	50000	0.0	0.00	0.00	100.00
1 1/2 "	37500	0.0	0.00	0.00	100.00
1 "	25000	0.0	0.00	0.00	100.00
3/4 "	19000	20.5	0.14	0.14	99.86
1/2 "	12500	4838.8	34.17	34.32	65.68
3/8 "	9500	3725.0	26.31	60.63	39.37
No.4	4750	5161.0	36.45	97.08	2.92
No. 8	2360	258.5	1.83	98.90	1.10
No.16	1180	63.8	0.45	99.35	0.65
No.30	600	28.3	0.20	99.55	0.45
No.50	300	16.3	0.11	99.67	0.33
No.100	150	13.8	0.10	99.77	0.23
Base		33.3	0.23	100.00	0.00
Total		14159.0	100.00		



**METODO**

Análisis por tamices de agregado fino y grueso, ASTM C136-01

**OBSERVACIONES**

Métodos UNICON : GID-LA-ME 014, 015 y 016; Abrasión Máquina de los Angeles, Sub-Contrata.

Los resultados obtenidos corresponden a la muestra analizada

La muestra, la identificación y los datos de referencia fueron proporcionados por el cliente

El informe no puede ser utilizado como un certificado del producto

ARPL TECNOLOGIA INDUSTRIAL S.A.  
  
 Ing. RIBO LAST NELARDE  
 Jefe Departamento de Laboratorio



## INFORME DE ENSAYOS N° 827-NV/05

**N° DE SOLICITUD**  
TIPO DE MUESTRAS  
PRESENTACIÓN  
SOLICITADO POR  
DIRECCIÓN  
CARTA / MEMORANDUM  
FECHA DE RECEPCIÓN  
FECHA DE EMISIÓN  
FECHA DE ENSAYO

**612/05**  
Agregados  
Bolsas plásticas  
Unión de Concreteras S.A.  
Panamericana Sur Km 11, San Juan de Miraflores  
USL-077/05  
12/10/2005  
10/11/2005  
Del 05/10 a 04/11/05

Pág. 1 de 2

**RESULTADOS**

ENSAYOS		Agregado Fino Jicamarca	Métodos
Humedad total	%	11.12	ASTM C566-97
Material menor a 75 µm	%	3.74	ASTM C117-95
Modulo de fineza		3.09	ASTM C136-01
Gravedad Esp. de masa		2.69	ASTM C128/C127-01
GEM superficie sat. Seca		2.72	"
Gravedad Esp. Aparente		2.77	"
Absorción	%	0.99	"
Peso Unitario sin compact.	Kg/m3	1,516	ASTM C29/C29M-97
Peso Unitario compactado	Kg/m3	1,733	"
Vacios	%	35.45	"
Impurezas Organica	Ad	1	ASTM C40-99
Terrones arcilla y friables	%	0.25	ASTM C142-97
Sulfatos solubles	ppm	77	GID-LA-ME-016
Cloruros solubles	ppm	48	GID-LA-ME-015
Sales totales solubles	ppm	416	GID-LA-ME-014

A.R.P.L. TECNOLOGÍA INDUSTRIAL S.A.  
  
Jairo Melanda  
Jefe Departamento de Laboratorio

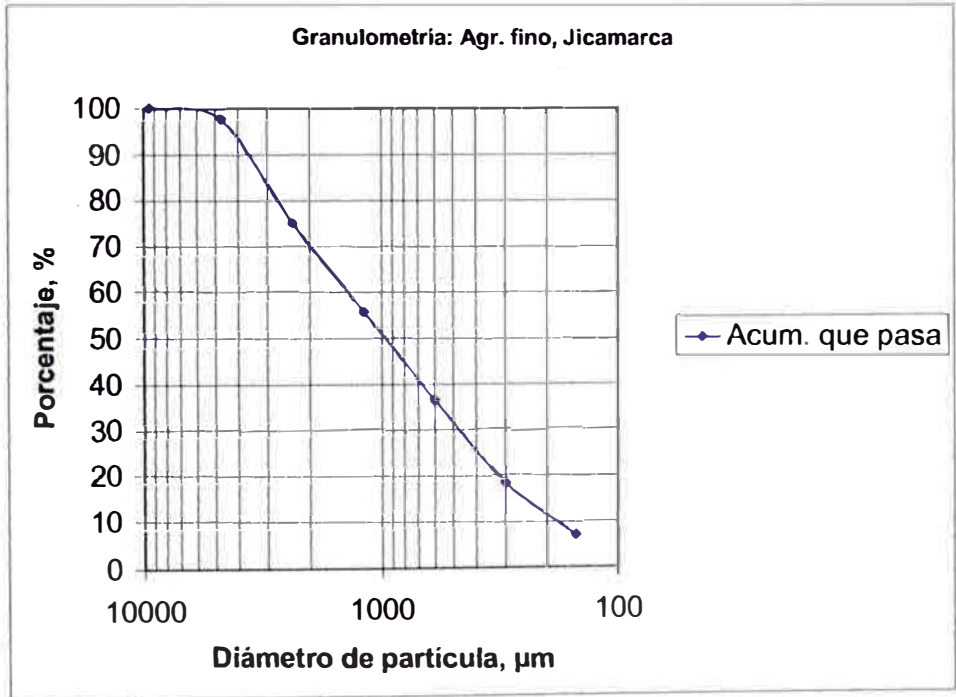




INFORME DE ENSAYOS  
N° 827-NV/05

Análisis granulométrico de Agregado Fino Jicamarca :

Malla N°	Equivalente $\mu\text{m}$	Fracción Retenida g	Fracción Retenida %	Acumulado Ret %	Acum. que pasa %
3/8 "	9500	0.0	0.00	0.00	100.00
No.4	4750	11.1	2.39	2.39	97.61
No. 8	2360	104.2	22.42	24.81	75.19
No.16	1180	90.6	19.49	44.30	55.70
No.30	600	89.0	19.16	63.45	36.55
No.50	300	84.0	18.07	81.52	18.48
No.100	150	51.9	11.17	92.69	7.31
Base		34.0	7.31	100.00	0.00
Total		464.6	100.00		



**METODO**

Análisis por tamices de agregado fino y grueso, ASTM C136-01

**OBSERVACIONES**

Métodos UNICON : GID-LA-ME 014, 015 y 016.

Los resultados obtenidos corresponden a la muestra analizada  
La muestra, la identificación y los datos de referencia fueron proporcionados por el cliente  
El Informe no puede ser utilizado como un certificado del producto

ARPL TECNOLOGIA INDUSTRIAL S.A.

Ing. JUAN CARLOS VELARDE  
Jefe Departamento de Laboratorio

### 2.3.4 Aditivos Químicos: Polyheed RI y Viscocrete I

UNICON seleccionó dos aditivos:

- Un reductor de agua de alto rango (Viscocrete 1) para obtener alta trabajabilidad y;
- Un retardante (Polyheed RI), primordial en toda industria de concreto premezclado.

Los certificados de calidad y las hojas técnicas están incluidos en la siguiente página y en el Anexo A, respectivamente.

#### **VISCOCRETE 1**

Aditivo tipo G, de acuerdo a la norma ASTM C494. Superplastificante a base de una solución acuosa de policarboxilatos modificados.

- ◆ Procedencia: Empresa proveedora: Sika Perú
- ◆ Caracterización: Se utilizó la Hoja Técnica
- ◆ Ubicación y Embalaje: En depósitos de UNICON, envasado

#### **POLYHEED RI**

Es un aditivo retardante, reductor de agua de medio rango a base de lignosulfonatos.

Cumple los requisitos de la norma ASTM C494 para aditivos retardantes Tipo B y reductores de agua y retardantes Tipo D.

- ◆ Procedencia: Empresa proveedora: BASF
- ◆ Caracterización: Se utilizó la Hoja Técnica.
- ◆ Ubicación y Embalaje: En depósitos de UNICON, envasado

# Certificado de Calidad

El presente documento presenta el Estado Permisible de las especificaciones técnicas de nuestro producto **Sika® Viscocrete® 1.**

## Estado Permisible

Aspecto : Líquido, color azul  
Densidad (g/cm<sup>3</sup>) : 1.08 +/- 0.02  
pH al 10% : 6.5 +/- 1.0  
Sólidos por Desecación (%) : 24 +/- 3

Fecha de Vencimiento : 01 año

## Resultados del Lote analizado:

Lote	Aspecto	Densidad	pH al 10%	Sólidos por Desecación (%)	Vencimiento
0507060801	Líquido, color azul	1.08	7.2	21	07 - 08


## Referencia:

NICC : 1081000  
Edición : 2

Atentamente,

Lima 02 de Agosto del 2005

Formato CC-F 12  
Autorizado por: MPP  
Fecha: 28.11.01  
Edición: 2

  
Laboratorio de Control de Calidad e I+D  
MIRTHA YULI PINTADO  
QUIMICA  
C.Q.P. 566



degussa.

*creating essentials*

**MBT UNICON**

Jr. Plácido Jiménez N° 630  
Lima Cercado

Teléfono: 385-0109

Fax: 3852065

### CERTIFICADO CONTROL DE CALIDAD

PRODUCTO: POLYHEED RI  
LOTE: PE-11393-Z5 VENCE: JUNIO 2007  
FECHA: 14 DICIEMBRE 2005

**EL PRODUCTO CUMPLE CON TODOS LOS REQUISITOS DE:  
MASTER BUILDERS TECHNOLOGIES**

PARAMETRO	LOTE	NORMA
ASPECTO FISICO	Líquido	Líquido
COLOR	Marrón oscuro	Marrón oscuro - negro
DENSIDAD	1.23	Min: 1.20 Max: 1.24 @ 20 to 30 ° C
%RESIDUO SOLIDO	45.67	Min: 44.80 Max: 48.40
%RESIDUO INSOLUBLE	0.25	Max: 0.50 (por vol.)
pH	9.33	Min: 7.50 @ 25 ° C

Los datos facilitados solamente reflejan los resultados de los controles realizados sobre una muestra representativa.

La empresa no se hace responsable por el uso que se haga del producto y/o de la información suministrada. La calidad de nuestros productos está garantizada bajo nuestras Condiciones Generales de Venta.

Este certificado es emitido electrónicamente por el laboratorio de Control de Calidad por lo que no requiere firma.



### 2.3.5 Agua para Concreto

◆ **Procedencia:**

El agua que se utilizó es la que brinda Sedapal (Red de Agua Potable).

El agua debe cumplir los requisitos de la siguiente norma:

▬ **ITINTEC 339.088:1982 HORMIGON.**

Agua para morteros y hormigones de cementos Pórtland.

Los requisitos dados en la norma citada están basados en normas europeas.

◆ **Ubicación y Embalaje:**

En tanques de Almacenamiento

◆ **Caracterización:**

Mediante Ensayos Químicos

El certificado de calidad se muestra en la siguiente página.

<b>UNICON</b> PROFESIONALES EN CONCRETO	GID-LA-R-007	<b>INFORME DE ENSAYOS QUÍMICOS</b>	Pág 1 de 1
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**SOLICITUD N°** : INSPECCIÓN : **X**  
**TIPO DE MUESTRA** : AGUA DE PRODUCCION  
**PROCEDENCIA** : PLANTA SAN JUAN  
**METODO DE ENSAYO** : VARIOS  
**SOLICITADO POR** : UNIDAD DE SERVICIOS DE LABORATORIO  
**FECHA RECEPCIÓN** : 12/12/2005  
**FECHA DE ENTREGA** : 14/12/2005

ENSAYO	AGUA	Límite permisible	REFERENCIA MÉTODO
Residuos sólidos totales (ppm)	469.0	5000 Max	ITINTEC 339.071
Contenido de sulfatos (ppm)	139.9	1000 Max	ITINTEC 339.074
Contenido de cloruros (ppm)	68.9	1000 Max	ITINTEC 339.076
pH 21.4 °C	7.77	5,5 Min	ITINTEC 339.073
Alcalinidad 21.0 °C (ppm)	186.8	1000 Max	ASTM D 1067



**Ing. Carlos Guerra Cisneros**  
 Jefe Opto. Laboratorio e Investigación  
 Unión de Concreteras S.A.

GID-LA-R-007  
 Rev.00

## 2.4 CÁLCULO DE LOS PROPORCIONAMIENTOS DE LOS DISEÑOS DE MEZCLA UTILIZANDO DIATOMITA (DCD)

### 2.4.1 Diseño de Referencia – Patrón (DCR)

Como diseño de referencia-patrón para todos los ensayos experimentales a desarrollar en el presente estudio, se utilizará el diseño elaborado por la empresa UNICON (Cuadro 2.6) el cual ha sido desarrollado sobre la base de las recomendaciones dadas por el ACI 363 y 211 así como también por la experiencia práctica que tienen en este campo. El siguiente cuadro presenta el proporcionamiento del Diseño de Referencia, DCR al 10% de Microsilice.

**Cuadro 2.6. DISEÑO DE REFERENCIA-PATRON**

BASE: 1 m<sup>3</sup> de Concreto

<b>CÓDIGO DEL DISEÑO</b>	<b>DCR-10</b>
<b>CEMENTANTE</b>	500 Kg
Cemento	450 Kg (90%)
Microsilice	50 Kg (10%)
<b>a/c</b>	0.34
<b>AGUA</b>	170 mL
<b>% AGREGADOS (Volumen)</b>	64%
% Arena / % Piedra	52.4 / 47.6
<b>ADITIVOS</b>	
Polyheed RI	5 cc / Kg Cementante
Viscocrete 1	20 cc / Kg Cementante
<b>% Aire</b>	1.5 %
<b>CARACTERISTICA MINIMA A CUMPLIR:</b>	
Resistencia a la Compresión a los 28 días	600 Kg-f/cm <sup>2</sup>
Slump	8"

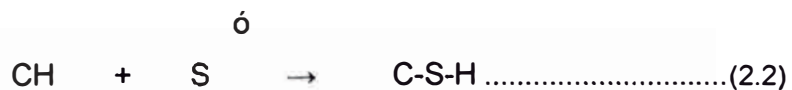
### 2.4.2 Determinación del Porcentaje de Reemplazo de Cemento por Diatomita

La cantidad de Portlandita generada para una hidratación completa del cemento es 0.24 Kg  $\text{Ca(OH)}_2$  / Kg Cemento [13].

La cantidad de puzolana requerida para reaccionar completamente con el  $\text{Ca(OH)}_2$  producido por la hidratación del cemento Portland en mezclas cemento-puzolana puede ser estimada. En hidratación completa, cementos Portland ordinarios producen cerca de:

$$0.20 \frac{\text{kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{hidratos}}} \approx 0.24 \frac{\text{kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{cemento}}}$$

Si asumimos que la composición promedio de la diatomita es: 90%  $\text{SiO}_2$  amorfa, 5% perdidas por calcinación y 5% otros minerales, y que de acuerdo con la reacción puzolánica (reacción 1.13), el ratio promedio molar cal/sílice (C/S) del compuesto C-S-H es 1, tenemos:



La cantidad de  $\text{Ca(OH)}_2$  requerido para reacción completa con la diatomita sería:

$$\frac{\text{Kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{SiO}_2}} = \frac{74}{60}$$

$$\frac{0.9\text{Kg}_{\text{SiO}_2}}{\text{Kg}_{\text{Diatomita}}} \times \frac{1.23\text{Kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{SiO}_2}} = 0.9 \times 1.23 \times \frac{\text{Kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{Diatomita}}}$$

$$= 1.107 \times \frac{\text{Kg}_{\text{Ca(OH)}_2}}{\text{Kg}_{\text{Diatomita}}}$$



La cantidad de diatomita requerida para la reacción completa será:

$$\frac{\frac{0.24 \text{Kg}_{-} \text{Ca(OH)}_2}{\text{Kg}_{-} \text{Cemento}}}{\frac{1.107 \text{Kg}_{-} \text{Ca(OH)}_2}{\text{Kg}_{-} \text{Diatomita}}} = \frac{0.24}{1.107} \times \frac{\text{Kg}_{-} \text{Diatomita}}{\text{Kg}_{-} \text{Cemento}}$$

$$= 0.2168 \frac{\text{Kg}_{-} \text{Diatomita}}{\text{Kg}_{-} \text{Cemento}}$$

Expresado en porcentaje: 17.82 % Diatomita del Cementante.

Por tanto se podría decir que para condiciones ideales de la hidratación del cemento en el concreto, se necesitaría a los más 17.8% de diatomita para que reaccione con la portlandita producida por la hidratación del cemento, cantidades superiores solo actuarían como relleno. Tomando en cuenta también investigaciones utilizando microsilice [23], donde recomiendan utilizar un reemplazo entre 5 y 15%, se realizará los ensayos experimentales para diferentes porcentajes de diatomita hasta 15% de reemplazo por cemento.

### 2.4.3 Determinación de Diseños de Mezcla con Diatomita (DCD)

- Sobre la base del Diseño de Referencia (DCR-10) se elaboraron los diseños de mezclas con diatomita fresca en los siguientes porcentajes 0, 5, 10, 15; Cuadro 2.7.
- Como la diatomita es ávida de agua fue necesario incrementar la dosificación del aditivo superfluidificante (Viscocrete 1). Mediante procedimientos de prueba y error, se determinó el volúmen a considerar en cada diseño y de esta manera asegurar la trabajabilidad de la muestra.
- Los demás insumos conservan las mismas cantidades del diseño de referencia, Cuadro 2.8.

**Cuadro 2.7.** Porcentaje de las Adiciones Minerales y Cemento en el Cementante de los diseños en Estudio

MATERIAL	DISEÑO DE REFERENCIA	DCD			
	DCR10	DCD-00	DCD-05	DCD-10	DCD-15
% Cemento	90	100	95	90	85
% Diatomita	-	0	5	10	15
% Microsilíce	10 %	-	-	-	-

**Cuadro 2.8.** Diseños obtenidos con Diatomita Fresca

Código Mezcla	a/ct	Cementante (Cem) Kg		Agua (L)	Polyheed RI (cc / Kg Cem)	Viscocrete 1 (cc / Kg Cem)	% Agregados	% arena / % piedra
		Cemento	Ad. Mineral					
<b>DISEÑOS DE BASE</b>								
DCD-00	0.34	500	0	170	5	12.0	64	52.4 / 47.6
DCD-05	0.34	475	25	170	5	17.8	64	52.4 / 47.6
DCD-10	0.34	450	50	170	5	28.0	64	52.4 / 47.6
DCD-15	0.34	425	75	170	5	43.0	64	52.4 / 47.6
<b>DISEÑO DE REFERENCIA</b>								
DCR-10	0.34	450	50	170	5	20.0	64	52.4 / 47.6

**Anotaciones:**

- Considerando que la humedad de los agregados no es uniforme en el tiempo y además absorben agua, al momento de preparar las mezclas de concreto (punto 1.2.7), estas variaciones han sido reajustadas mediante un balance de agua.

#### **2.4.4 Preparación de las Mezclas de Concreto**

El procedimiento para preparar la mezcla de concreto, se desarrolló sobre la base de las sugerencias que para estas mezclas propone la ACI, como también por las experiencias propias de UNICON. No hay una norma que indica el orden de adición de los insumos a la mezcla, esto se desarrolla de acuerdo a la práctica.

### Descripción del Procedimiento

#### 1. Equipos e Instrumentos.-

- Mezcladora de 50 litros
- Probeta graduada de 500, 100, 10 mL
- Cucharón, Martillo de goma, Cinta métrica, Carretilla, Pala

#### 2. Mezclado.-

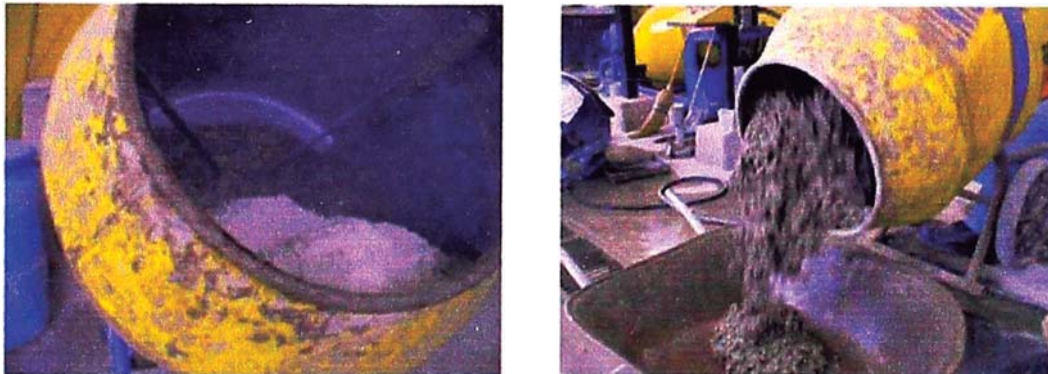
- Pesado de Materiales: Cemento, Diatomita, Arena y Piedra y medir el volumen de agua y aditivos (Polyheed RI y Viscocrete 1), de acuerdo a la formulación de la Mezcla de Concreto.
- Mezclado; El orden de adición de los materiales y el tiempo de Mezclado es según la siguiente tabla:

Tiempo	Orden de Adición
00:00:00	Disolver el Polyheed en el agua. Añadir el agregado grueso a la Mezcladora y la solución de Polyheed. Mezclar por 10 seg.
00:00:10	Adicionar el cemento. Mezclar por 10 seg.
00:00:20	Adicionar la Adición Mineral (Microsilice o Diatomita)
00:03:00	Detener el cronómetro y la mezcladora, mezclar manualmente con una pala, hasta que no haya material pegado en el fondo y en los lados. Tener en cuenta que esto dependerá del porcentaje de diatomita, puesto que a mayor cantidad de la misma, la mezcla tiende a aglomerarse. Cuando ya no haya grumos, poner en marcha el cronómetro y la mezcladora.
00:03:10	Detener la Mezcladora, agregar la arena, mezclar
00:05:00	Adicionar el aditivo Viscocrete 1 a la mezcla, continuar mezclando, observar que la mezcla no contenga grumos.
00:20:00	A este tiempo se observa que el aditivo superfluidificante ha actuado, la mezcla esta fluida y no esta segregada. Fin del Mezclado

**Fig. 2.2.** Insumos. Lado Derecho: Aditivos (Celeste: Viscocrete 1, Marrón: Polyheed RI).  
Lado Izquierdo: Agregados, Cemento y Adiciones minerales



**Fig. 2.3.** Mezclado del Concreto. Lado Izquierdo: Inicio. Lado Derecho: Fin.



## 2.5 ENSAYOS DE RESISTENCIA A LA COMPRESIÓN A LOS 3, 7, 28 DÍAS Y SUS RESULTADOS

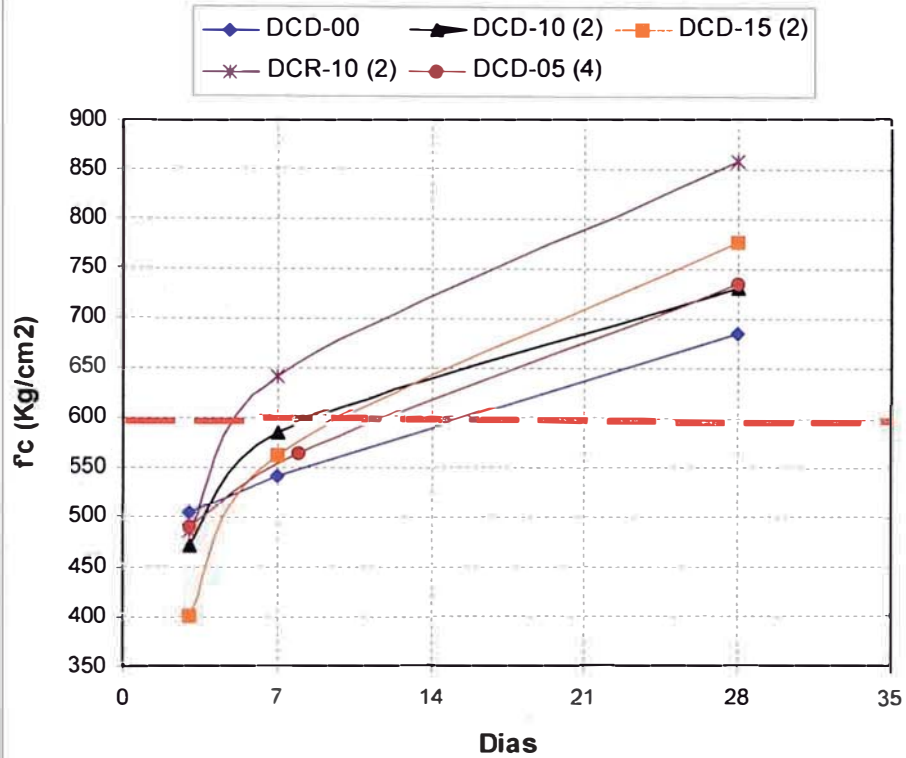
Para llevar a cabo este ensayo se preparó mezclas de concreto de 50 Litros para cada Diseño de Mezcla con Diatomita y de Referencia. Se midieron los parámetros de control (Slump, Contenido de Aire, Peso Unitario) y se elaboraron seis probetas por mezcla, las cuales se midieron su resistencia a la compresión a los 3, 7 y 28 días (2 probetas por fecha).

El Cuadro 2.9 muestra la tabulación de los ensayos de resistencia desarrollados. Para el mejor análisis de estos resultados se han elaborado las curvas: Resistencia vs. Tiempo (Gráfico 2.2) y Resistencia vs. % Diatomita (Gráfico 2.3).

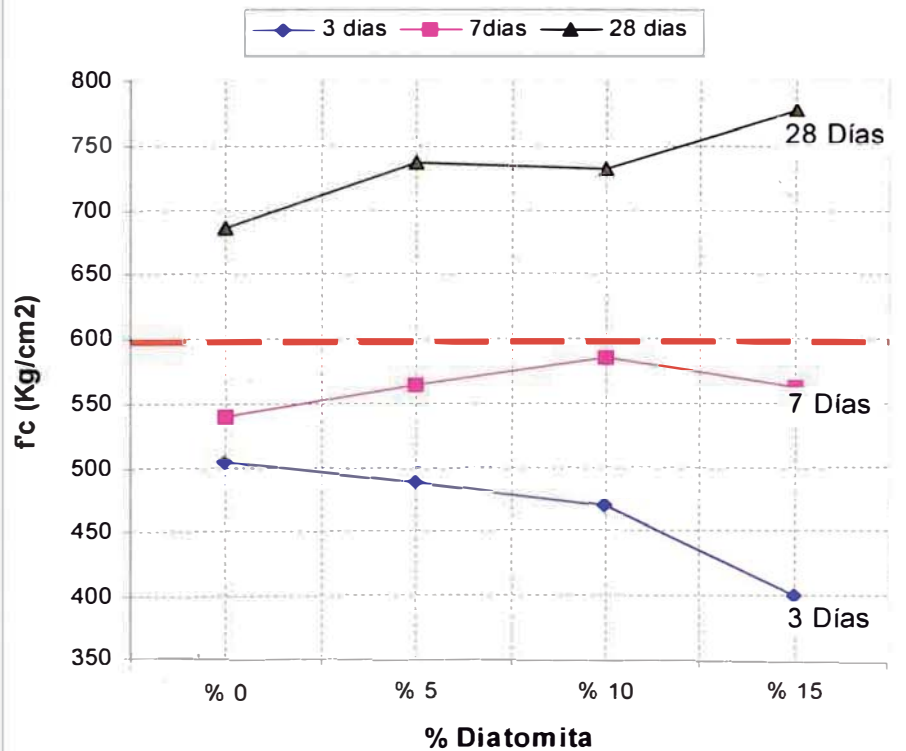
Cuadro 2.9. RESULTADOS DEL ENSAYO DE RESISTENCIA A LA COMPRESION A LOS 3, 7, 28 DIAS

Código Mezcla	a/ct	at/ct	Polyfeed RI (cc)	Viscoconete 1 (cc)	Slump (pulg)	% Aire	P.U Real	Temperatura °C		f'c teórico	f'c					
								Amb.	Cons.		3d <sup>3</sup> %	7d %	28d %			
DISEÑOS DE BASE																
DCD-00	0.34	0.36	5	12.0	9 1/2"	1.5	2454	17.7	24.8	600	504	104%	541	84%	686	80%
DCD-05 (4)	0.34	0.37	5	17.8	9 3/4"	1.2	2439	19.7	20.1	600	489	101%	564	88%	736	86%
DCD-10 (2)	0.34	0.38	5	28.0	10"	1.7	2420	21.7	25.3	600	470	97%	586	91%	731	85%
DCD-15 (2)	0.34	0.40	5	43.0	11"	1.4	2403	20	24	600	400	82%	562	88%	777	91%
DISEÑOS DE REFERENCIA																
DCR-10 (2)	0.34	0.37	5	20.0	9 1/2"	2.0	2428	18	24	600	486	100%	642	100%	858	100%

**Grafico 2.2. Resistencia vs. Tiempo  
(a los 3, 7, 28 dias)**



**Grafico 2.3. Resistencia vs % Diatomita**



### 2.5.1 Resultados Obtenidos

- Conforme se hidrata el cemento, el concreto va aumentando su resistencia, la cual mejorará con la adición de puzolanas, como se puede observar en los Gráficos 2.2 y 2.3.
- A los 3 días, la baja resistencia se debe a la poca hidratación del  $C_3S$ . El diseño DCD-00 a esta edad presenta mayor resistencia a la compresión que los demás diseños incluyendo al de referencia con microsilíce DCR-10, pues es el que contiene mayor cantidad de cemento y por tanto mayor cantidad de  $C_3S$ .
- A los 7 días, el Gráfico 2.3 muestra que hay poca diferencia en la resistencia de los diseños con diferentes porcentajes de diatomita. Se deduce que las partículas de la puzolana por ser más pequeñas llenan los espacios entre los granos de cemento hidratadas reduciendo la porosidad (mejor empaque) y compensando el menor contenido de cemento lo que mejora la resistencia a la compresión (aporte físico), pues si no fuera así las resistencias serían menores al diseño de DCD-00 (diseño de solo cemento).
- En el gráfico 2.2, el diseño DCR-10 a los 7 días presenta una resistencia 18% mayor que el diseño DCD-00, debido principalmente al aporte físico y químico de la puzolana (reacción puzolánica). Varios autores coinciden que la microsilíce es más reactiva que las puzolanas naturales. Esta es la explicación porque la microsilíce desarrolla mayor resistencia que los diseños con diferentes porcentajes de diatomita.
- A los 28 días, la resistencia de los diseños en estudio superan la resistencia esperada de  $600 \text{ Kg/cm}^2$ , debiéndose no solo al aporte físico de la diatomita sino que parte de ella, ha reaccionado formando más silicatos hidratados. Pues como se observa en el Gráfico 2.3, el diseño DCD-15, que contiene menor cantidad de cemento ha desarrollado mayor resistencia a comparación de los otros diseños.

## **CAPÍTULO III: ENSAYOS EXPERIMENTALES FINALES**

### **3.1 OBJETIVO**

Vía ensayos experimentales, analizar el comportamiento de las diferentes características de los diseños anteriormente probados mediante ensayos en pasta, concreto fresco y concreto endurecido, que permitirá seleccionar el Diseño de Mezcla Optimo.

### **3.2 INFORMACIÓN TÉCNICA DE BASE**

Los ensayos finales se realizaron sobre la base de los resultados obtenidos en la Etapa Preliminar. Para obtener un CAR no solo se requiere que tenga una elevada resistencia a la compresión, sino también que en su estado fresco tenga una buena trabajabilidad sin segregación y que sea durable.

La mayoría de los ensayos realizados, se encuentran normalizados por ASTM y otros han sido desarrollados en la tesis tomando como referencia investigaciones anteriores y conocimientos de base; por lo tanto sólo se detallaran los procedimientos de los ensayos que no están normalizados, los otros procedimientos serán incluidos en Anexos respectivos. Los ensayos a realizar son los siguientes:

#### Ensayos en Pasta:

- Viscosidad (Reología) [5]\*

#### Ensayos en Concreto Fresco:

- Tiempo de Fragua
- Asentamiento (Slump)
- Peso Unitario
- Contenido de Aire
- Temperatura

#### Ensayos en concreto Endurecido:

- Resistencia a la Compresión - Medición a los 3, 7, 28, 60 días
- Permeabilidad [15]\*\*
- Resistencia al ataque de Sulfatos [17]\*

\* Ensayos no normalizados. Han sido desarrollados en la presente tesis.

\*\* Ensayo no normalizado desarrollado por UNICON.



Las definiciones de los ensayos anteriormente descritos y las normas correspondientes, están indicadas en los puntos 1.2.5, 1.2.6 y 1.4.3.

### 3.3 ENSAYOS EXPERIMENTALES POR REALIZAR

#### 3.3.1 Ensayos en Pasta

##### 3.3.1.1 Volúmen Necesario y Preparación de la Pasta

Se partió del diseño elaborado según los puntos 2.4.1 y 2.4.3, con la diferencia que se no se agregó los áridos, y se trabajo sobre la base de 600 mL de pasta, pues para hacer las mediciones de viscosidad en el viscosímetro Brookfield se requería de 500 mL de muestra. Este ensayo se realizó por duplicado, y se prepararon dos batch de pasta de 600 mL por diseño.

Preparación de la Pasta (Secuencia):

<b>Tiempo</b>	<b>Orden de Adición</b>
00:00:00	Disolver el Polyheed en el agua, luego adicionar el cementante (cemento y adición mineral). Mezclar por 3 min a Velocidad 1
00:03:00	Detener la mezcladora, mezclar manualmente el material pegado en el fondo y en los lados. Tener en cuenta que esto dependerá del porcentaje de diatomita, a mayor cantidad la mezcla tiende a aglomerarse.
00:03:30	Puesta en marcha de la mezcladora a velocidad 2
00:05:00	Adicionar el aditivo Viscocrete 1, continuar mezclando, observar que la mezcla no tenga grumos.
00:10:00	Detener la mezcladora por 15 s y paletear hasta que no haya material pegado en el fondo. Encender la Mezcladora
00:15:00	Fin del Mezclado

### 3.3.1.2 Desarrollo del Ensayo de Viscosidad

#### A) Objetivos

- Evaluar el comportamiento reológico de las pastas correspondiente a los diseños en estudio.
- Caracterizar las pastas de acuerdo al tipo de fluido que corresponde.

#### B) Equipos y Materiales utilizados

##### MATERIALES:

- Cemento TIPO IBA
- Diatomita Fillite
- Microsílice
- Aditivos: Polyheed RI y Viscocrete 1
- Agua Destilada

##### EQUIPOS:

- Viscosímetro Brookfield  
Modelo: **HBTDV-II**  
Serie: D21823  
Incluye Set de Agujas: HA/HB Spindle Set. (7 agujas)  
Velocidades (RPM): 0.5, 1, 2.5, 5, 10, 20, 50, 100.

El principio de funcionamiento del Equipo está incluido en el anexo D.

Se tiene que tener en cuenta que los resultados obtenidos con el Viscosímetro Brookfield solo se pueden emplear para realizar comparaciones y determinar una tendencia.

- Mezcladora Hobart, de acuerdo a las especificaciones de la Norma ASTM C305.

Fig. 3.1. Mezcladora Hobart



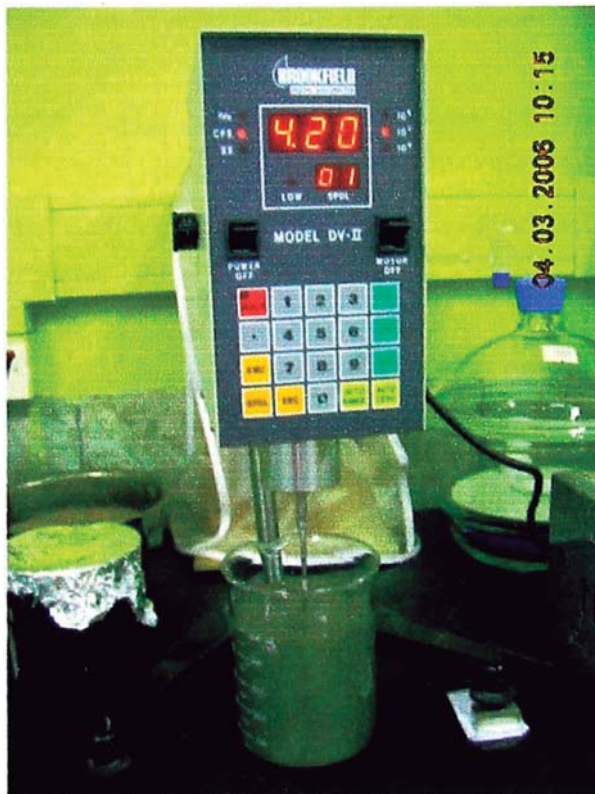
- Termómetro
- Balanza
- Cucharón y Recipientes
- Probetas 10 mL, 20mL, 100mL
- Vaso de Precipitado 600 mL
- Erlenmeyer 300 mL
- Cronómetro

### C) Procedimiento Operativo

#### Medición de las Viscosidades:

- Se traslada la pasta preparada al vaso de precipitado.
- Se mide la temperatura del ambiente y de la pasta (sólo para control).
- Se inicia la medición de la viscosidad a distintas revoluciones aumentándole progresivamente desde 0.5 a 100 RPM y luego disminuyendo de 100 a 0.5 RPM, tomando los datos cuando se estabiliza el dial, después de aproximadamente 2 minutos.
- Después de cada corrida o "loop", se homogeniza la pasta por 5 minutos mezclándolo con una espátula, y se inicia la siguiente corrida. Se realizaron tres corridas, que duraban 35 minutos aproximadamente cada una.

Fig. 3.2. Medición de la Viscosidad usando el Viscosímetro Brookfield



#### D) Trazado de Curvas

Las Gráficas 3.1 al 3.6 muestran las curvas sobre el comportamiento de las viscosidades medidas

Dichas curvas se han trazado considerando las siguientes anotaciones:

- Para caracterizar y evaluar la reología de la pasta, se necesita tener datos de Viscosidad vs. Shear Rate y Shear Stress vs. Shear Rate.
- El equipo permite lecturas desde 0.5 a 100 RPM y sus respectivos % torque.
- Para obtener el Shear Stress se multiplicó el % torque por un factor de corrección.
- Como el RPM es proporcional al Shear Rate, solo se utilizó el RPM.
- Para obtener Shear Stress vs. RPM se graficó la vuelta completa, es decir, desde 0.5 a 100 RPM y de 100 a 0.5 RPM. Las tablas de las mediciones realizadas, se encuentran en el Anexo C

#### Análisis del comportamiento de las Viscosidades Medidas

Comparando las gráficas 3.2 (A), 3.3. (A), 3.4 (A), 3.5 (A):

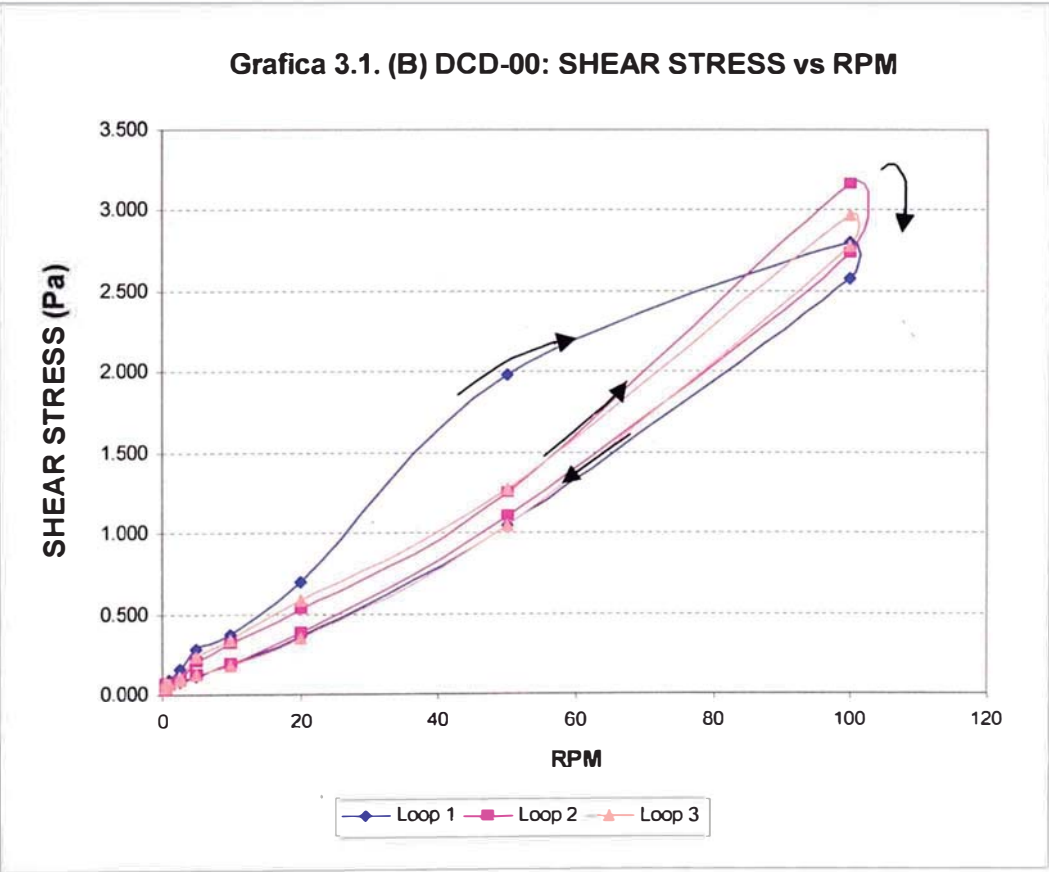
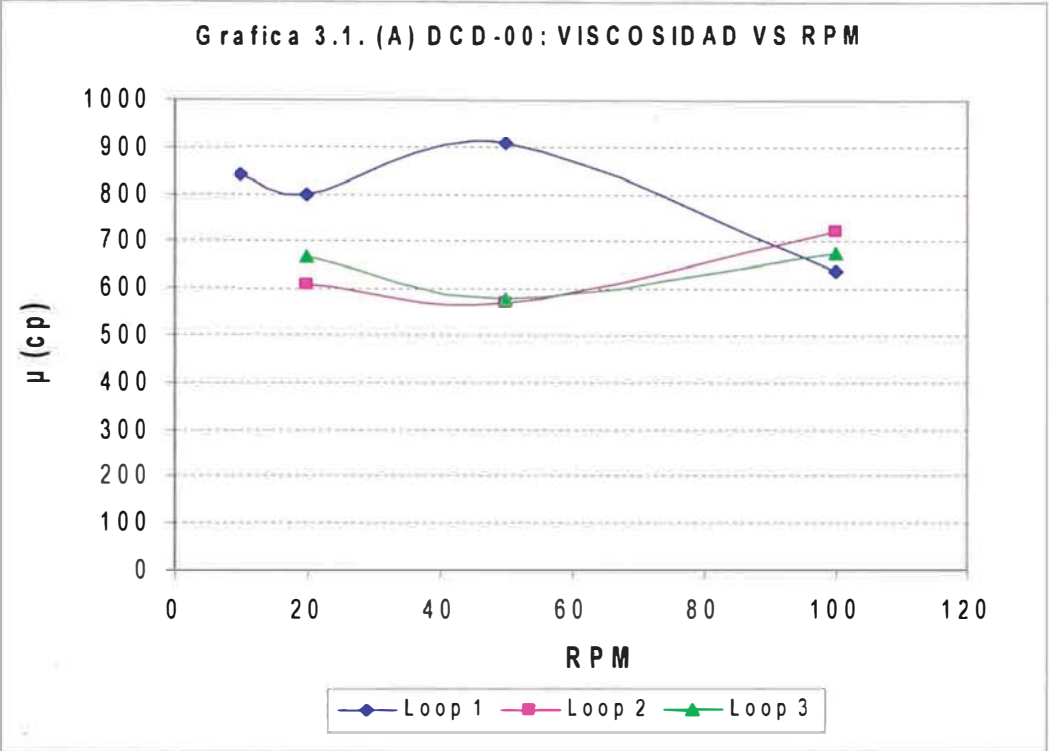
- 1) Comparando las viscosidades aparentes a partir de las 10 RPM, se puede decir que los diseños DCD-10 y DCD-15, presentan mayor viscosidad con respecto al DCD-05 y a la vez las viscosidades de estos diseños incluyendo el DCD-05 son mayores que el diseño DCR-10.  
Esta mayor viscosidad se vio reflejada en los ensayos en concreto pues los diseños con diatomita se mostraron bastantes viscosas a comparación con el diseño con

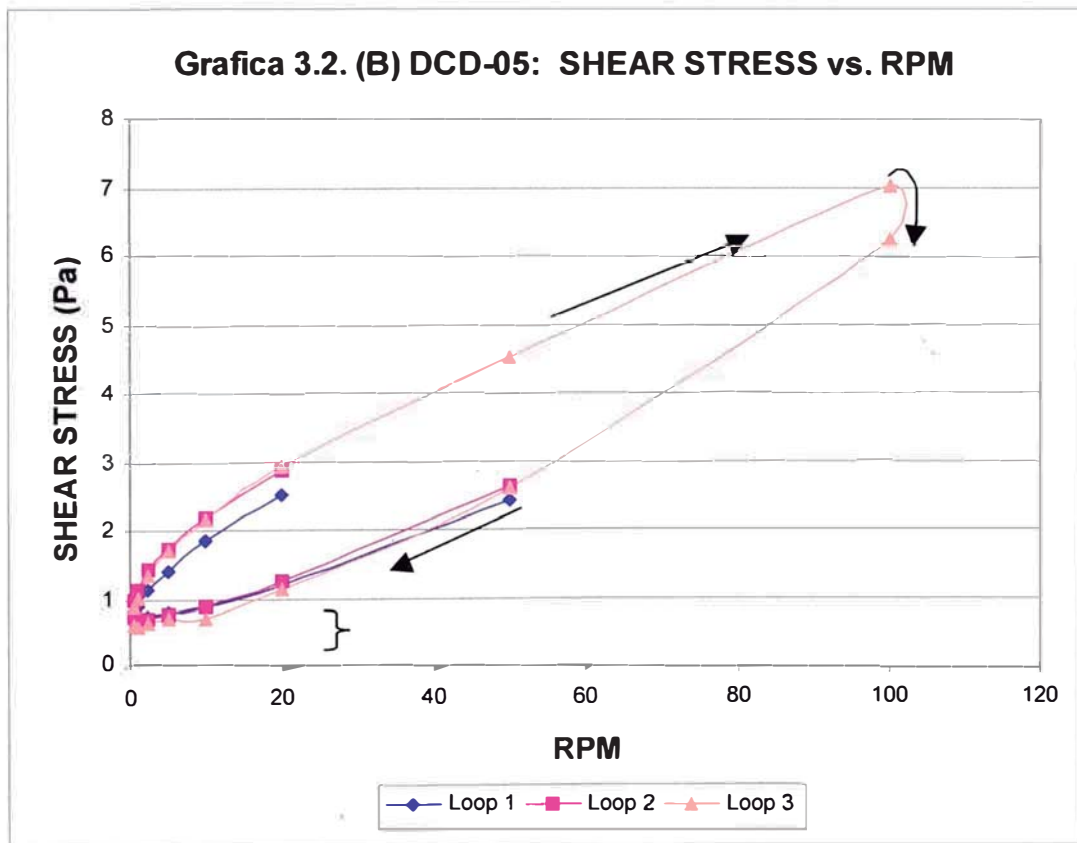
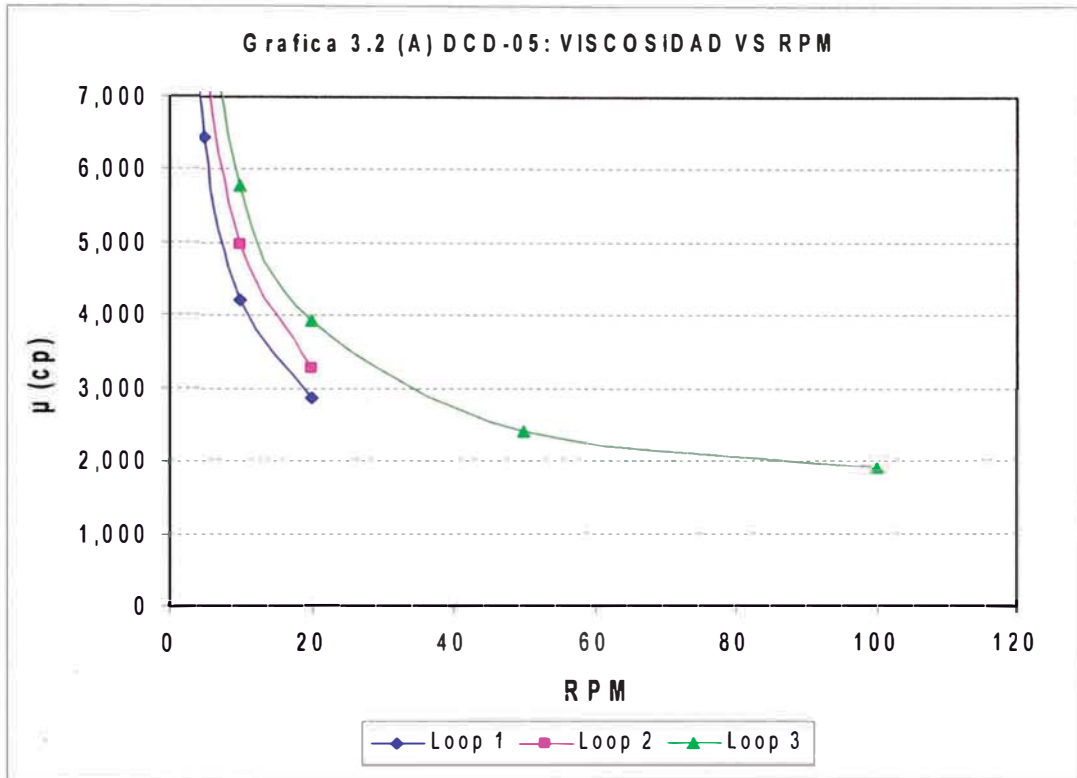
microsílice, además mientras se aumentaba la cantidad de diatomita, la cohesividad de la pasta aumentaba.

La diatomita aumenta la viscosidad de la pasta en mayor grado que la microsíllice, la cual puede deberse a varios factores. Las partículas de diatomita Fillite, al tener diferentes tamaños y formas (Fig. 2.1), harán que los diseños con diatomita requieran un mayor esfuerzo para el mezclado en comparación con el caso de la microsíllice, cuyas partículas redondeadas ofrecerán menor resistencia. Por otro lado, el agua, que actúa como lubricante durante el mezclado, es adsorbida en parte por la diatomita, disminuyendo la lubricación y aumentando la viscosidad.

Este aumento de viscosidad de la pasta influye en la trabajabilidad de la mezcla de concreto y por tanto en el trabajo requerido si se quiere bombear el concreto.

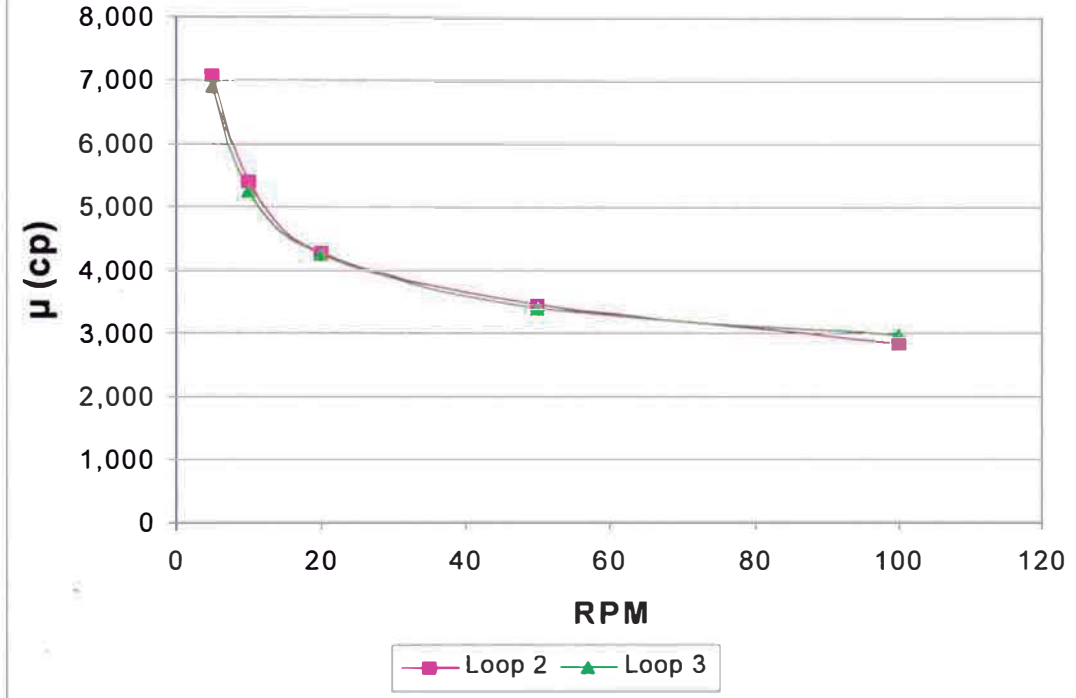
- 2) Se puede comprobar que los diseños con adiciones minerales de tamaño de partícula menor que el cemento mejora la homogeneidad de la pasta y la cohesividad. Al compararlos con el diseño DCD-00 (Gráfico 3.1 (A)), esta curva no mantiene una forma definida de tendencia en el tiempo, en la cual la viscosidad disminuye y aumenta, este comportamiento se debe a la falta de homogeneidad. Durante el ensayo se apreció que la pasta del diseño DCD-00 tiende a sedimentar.
- 3) Conforme aumenta el RPM, la viscosidad disminuye. La pendiente es muy pronunciada al inicio, pero cerca a 100 RPM dicha pendiente disminuye; el mismo comportamiento muestra el diseño con microsíllice. Esta tendencia a disminuir la viscosidad es característica de los fluidos pseudoplásticos.



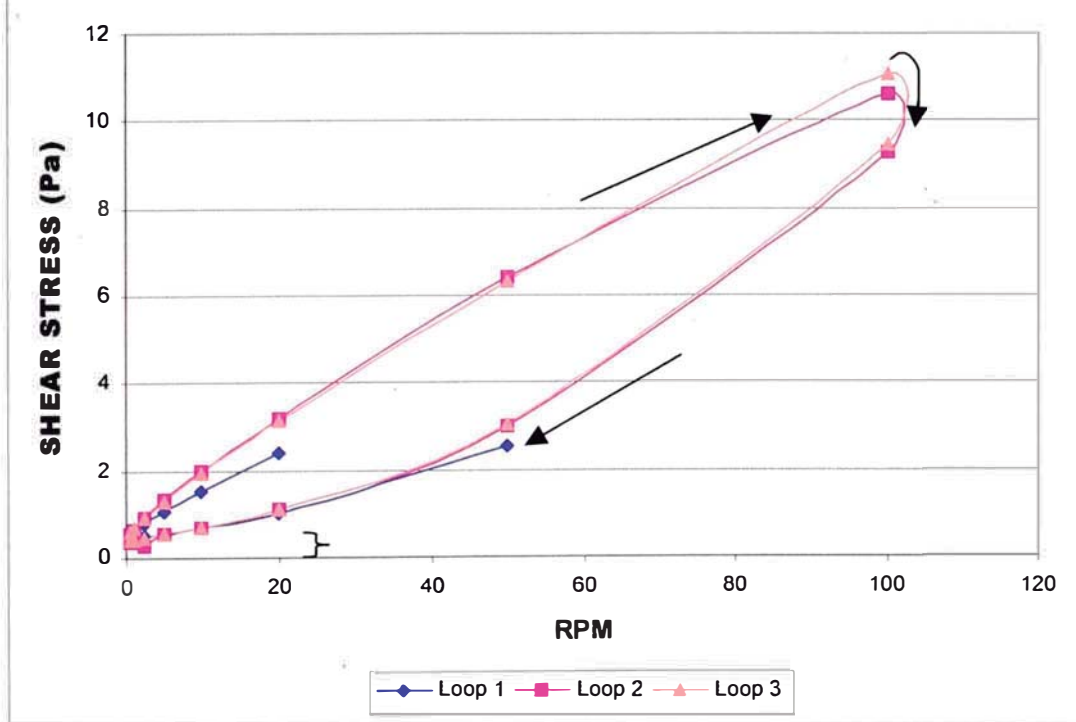




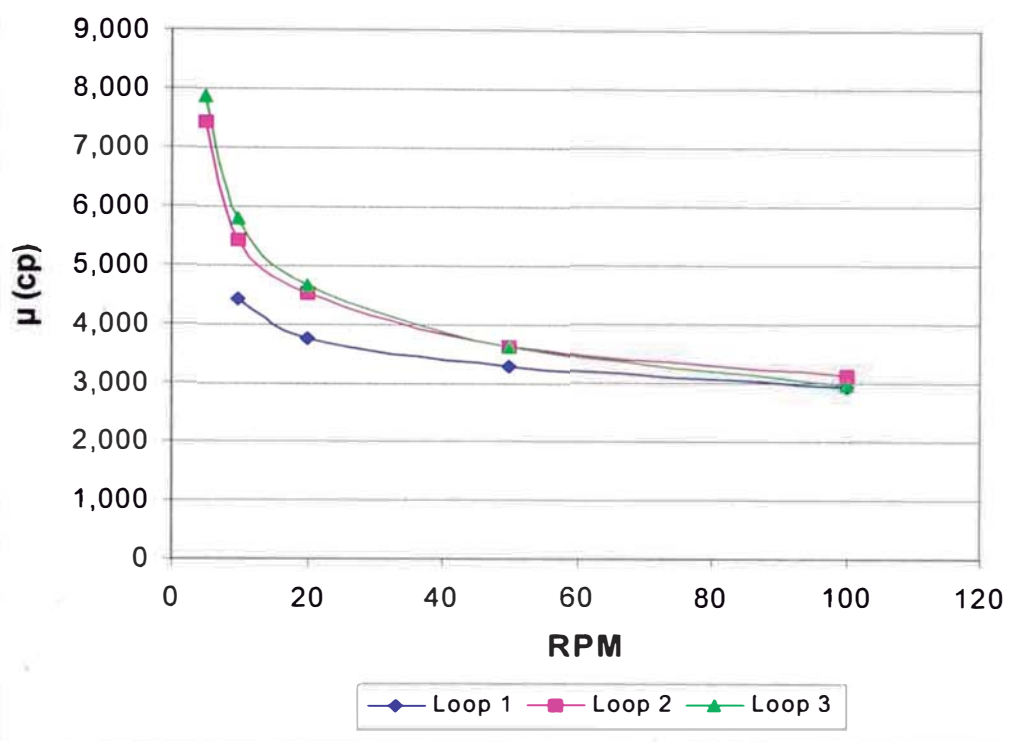
**Grafico 3.3 (A) DCD-10: VISCOSIDAD vs RPM**



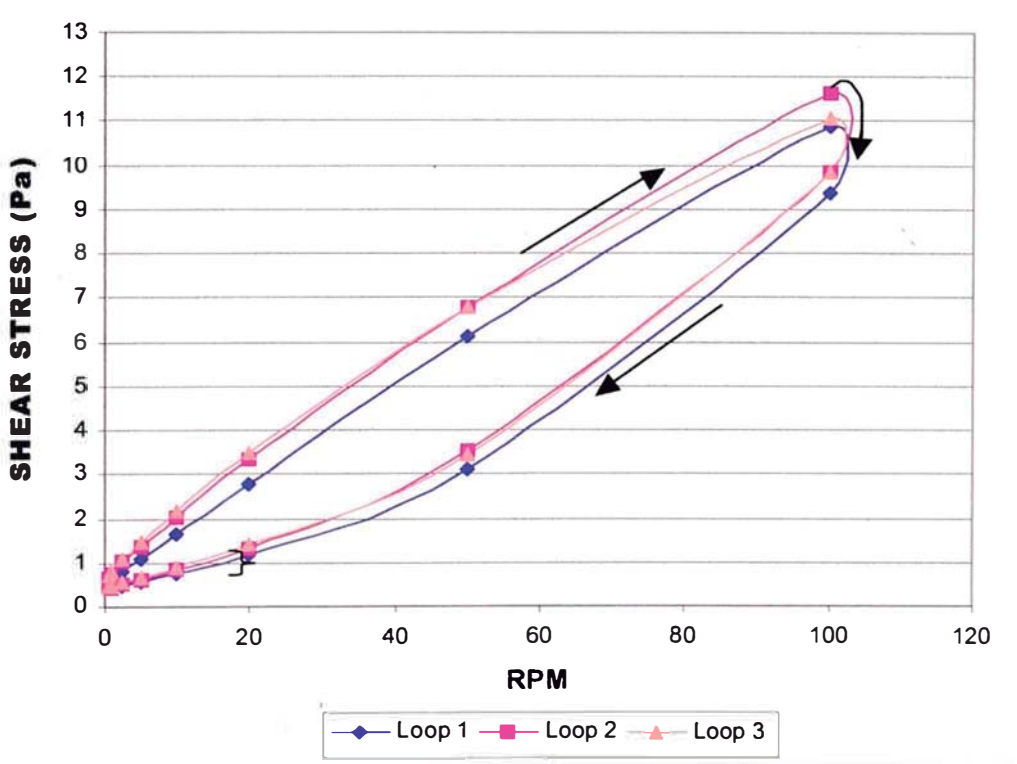
**Gráfico 3.3 (B) DCD-10: SHEAR STRESS vs. RPM**



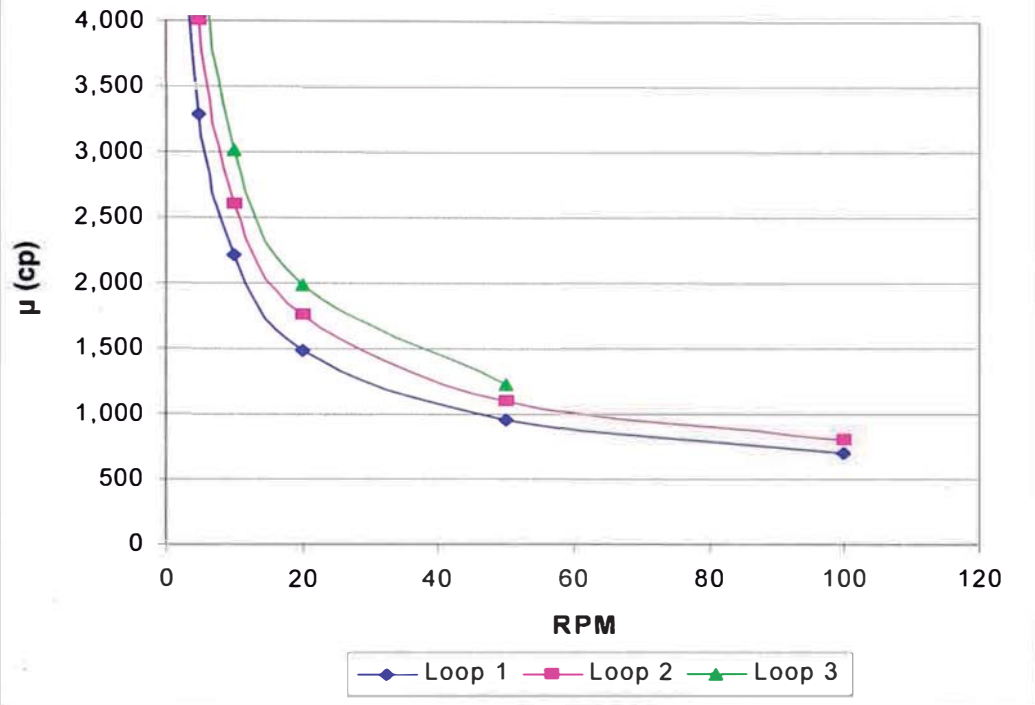
Grafica 3.4 (A) DCD-15: VISCOSIDAD vs RPM



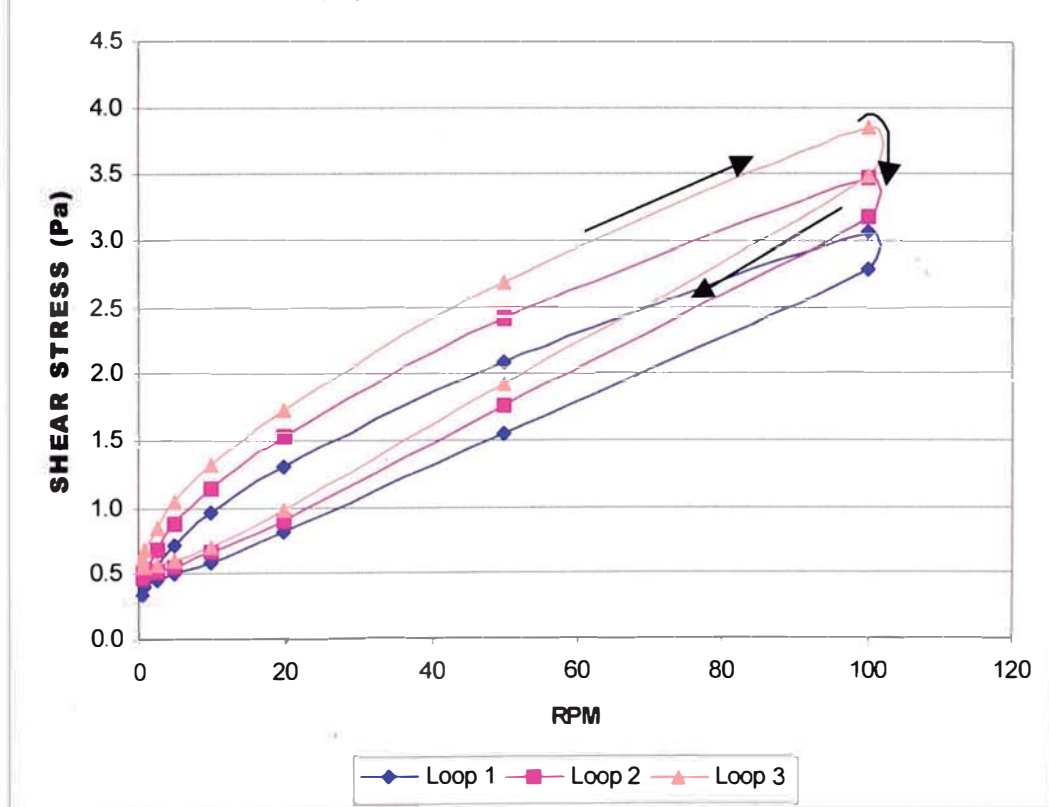
Grafica 3.4 (B) DCD-15: SHEAR STRESS vs RPM



**Grafico 3.5 (A) DCR-10: VISCOSIDAD vs RPM**



**Grafico 3.5 (B) DCR-10: SHEAR STRESS vs. RPM**



- Con respecto a las graficas 3.2 (B), 3.3 (B), 3.4 (B), 3.5 (B):

Se puede apreciar que los diseños con diatomita presentan un comportamiento tixotrópico pues las curvas de subida y de bajada no coinciden. El mismo comportamiento muestra el diseño con microsilíce.

Para cada pasta se realizaron tres corridas o Loops. Se puede apreciar que el Shear Stress aumenta con el RPM. Cuando se mantuvo la velocidad en 100 RPM en todos los casos el Shear Stress disminuyó con el paso del tiempo.

Para la segunda corrida de cada pasta, se dejó reposar por dos minutos antes de iniciar la medición, las curvas de los diseños con diatomita casi se superponen lo que nos lleva a decir que el fluido recupera su comportamiento reológico inicial, esto sucederá hasta que inicie el fraguado.

Para el caso del diseño con microsilíce, aunque el área tixotrópica es menor que los diseños con diatomita, ésta varía con el tiempo como se puede observar en la gráfica 3.5 (B), pues las corridas no se superponen. Estas variaciones no son considerables si se compara con los diseños con diatomita.

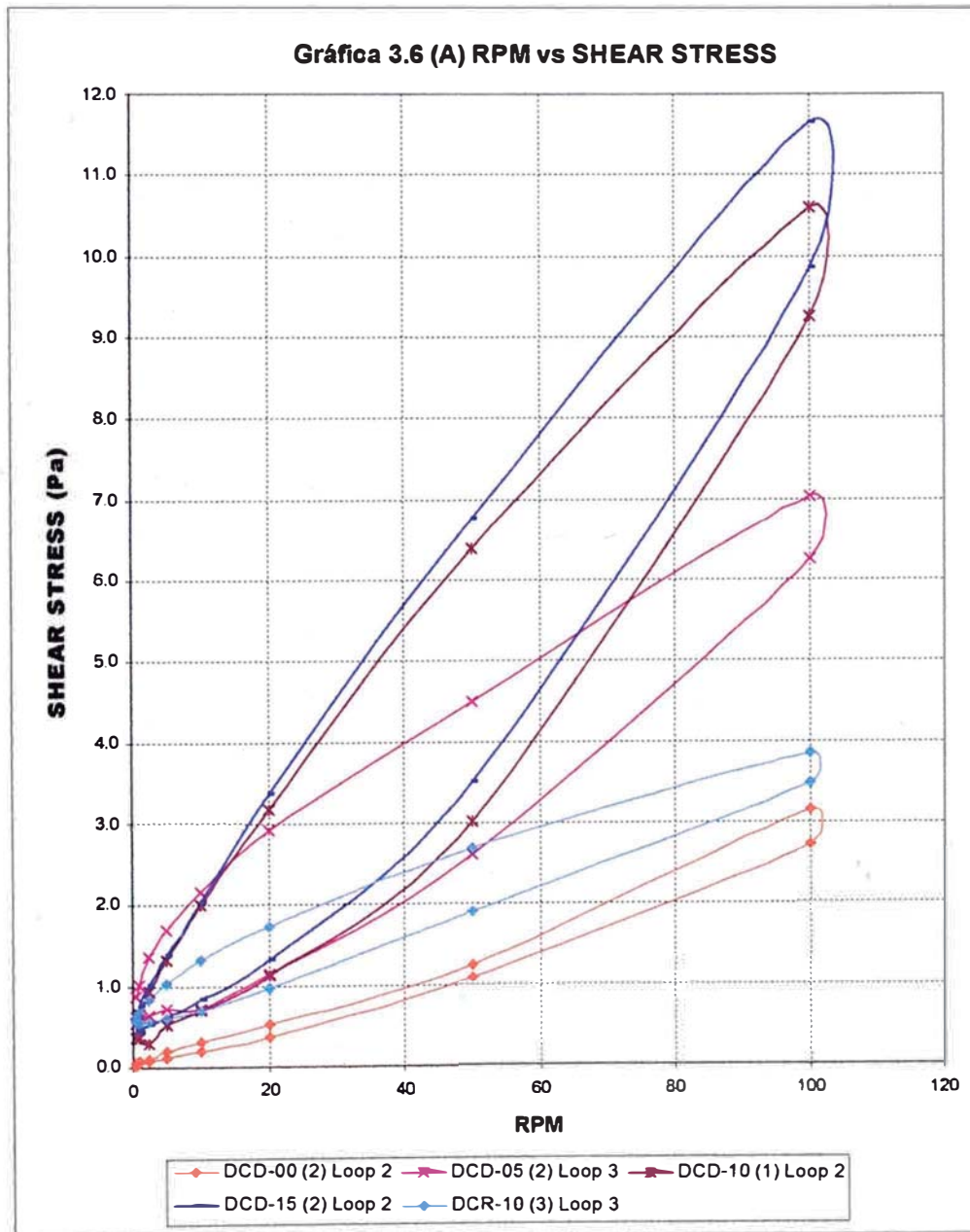
Finalmente se puede observar en las gráficas analizadas que se necesita una fuerza mínima que tiene que ser excedida para que el flujo empiece, este valor llamado tensión umbral (Yield Stress). Este comportamiento nos da a entender que estas pastas presentan un flujo plástico con características de un pseudoplástico.

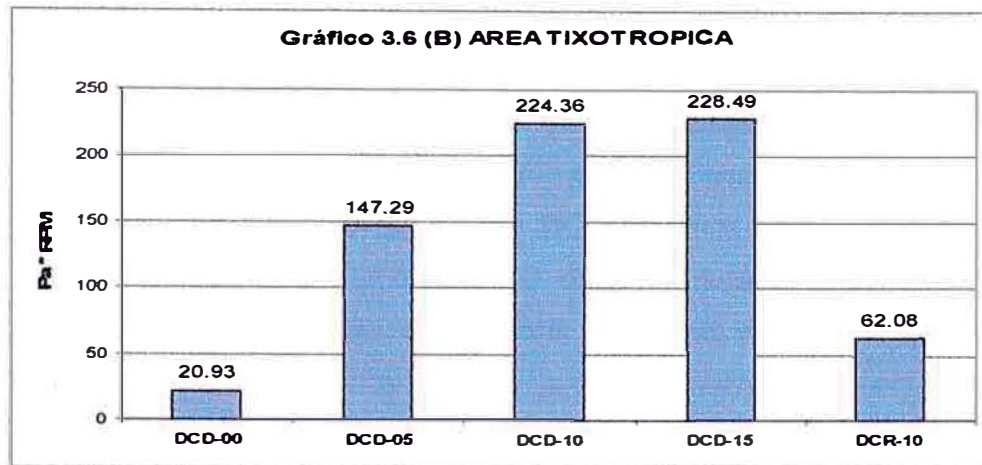
Los valores de Tensión Umbral de las pastas de los diseños son los siguientes:

**Cuadro 3.3.** Cálculo de la Tensión Umbral de las Pastas

DISEÑO	$\zeta_0$ (Tensión Umbral )
DCD-00	0.0 Pa
DCD-05	0.7 Pa
DCD-10	0.4 Pa
DCD-15	0.5 Pa
DCR-10	0.5 Pa

Como la tensión umbral, por definición, es el esfuerzo mínimo que se necesita para que se inicie el movimiento, de la tabla anteriormente descrita, se puede deducir que los agregados en la pasta DCD-00, tienen mayor posibilidad de segregación que los diseño que contienen adición mineral.





- En los grafico 3.6(A) y (B):

Para este gráfico se escogió sólo una corrida (Shear Stress vs. RPM) por cada diseño para hacer una comparación de las tixotropías. En comparación al DCR-10, se puede decir que los DCD presentan un mayor grado de tixotropía, en la grafica 3.6 (B) se muestra las áreas que forma cada curva, estas áreas dan una idea de la cantidad de energía necesaria para hacer fluir la mezcla después de un periodo de reposo para un mismo volumen de pasta en un mismo tiempo.

De lo dicho anteriormente, se puede decir que mientras se mantenga la mezcla en movimiento, la tixotropía del diseño DCD-05 es la que más se acerca al diseño de referencia, pues a más altos contenidos de diatomita, la tixotropía es mayor. Adicionalmente se puede decir que a más altos contenidos de diatomita, si esta se deja en reposo, la energía necesaria para hacer fluir las mezclas serían bastante altas. Por ejemplo, la energía necesaria para hacer fluir la mezcla del DCD-05 sería aproximadamente el doble del DCR-10, a su vez la energía necesaria de los diseños DCD-10 y DCD-15 sería el triple del DCR-10. Por lo tanto al usar, la diatomita en el concreto se deberá evitar en lo posible dejar esta mezcla de concreto en reposo.

Este comportamiento de la diatomita de aumentar la tixotropía en comparación con la microsílíce, puede deberse a la atracción de la diatomita con las moléculas de agua, entonces el esfuerzo para romper este ordenamiento sería mayor a comparación con la microsílíce. Y

mientras más alto sea el contenido de la diatomita, esta atracción aumenta.

Esta propiedad tixotrópica es muy importante porque nos indica que el agregado grueso será soportado por la pasta sin causar segregación. La pasta de solo cemento del diseño DCD-00 en la Gráfica 3.6 (A) no muestra tixotropía y, como se pudo observar durante el ensayo esta pasta sedimentaba. Este efecto será menor en el concreto pues la mezcla contiene agregados finos que le dan una mayor homogeneidad.

También hay que tener en cuenta que no sería conveniente utilizar mezclas que tengan una tixotropía alta, porque esto significaría cambios de viscosidades muy diferenciadas que podría afectar en el bombeo y colocación del concreto.

- En resumen, la diatomita influye en la reología del concreto: por su forma y por la atracción con las moléculas de agua.

Las mediciones de la viscosidad se hicieron en la etapa de pre-inducción, por tanto, los productos de la hidratación del  $C_3A$  también están influenciando en la reología de la pasta.

### **3.3.2 Ensayos en Concreto Fresco**

Estos ensayos son realizados como control del concreto fresco y ayudan a detectar factores que puedan afectar la calidad del producto final, tal como se indicó en el punto 1.2.5. Las normas ASTM (Anexo H) indican el procedimiento para desarrollar estos ensayos.

#### **3.3.2.1 Cálculo del Volúmen Necesario**

Este cálculo es válido para los ensayos del concreto endurecido.

El formato Unicon (Cuadro 3.1) se anota el volumen unitario y total del concreto necesario para los ensayos por realizar tanto en estado fresco como endurecido para un diseño de mezcla.

Para cada diseño se realizaron todos los ensayos por duplicado a excepción de los ensayos de resistencia a la compresión, que se realizaron por triplicado.

De acuerdo al ACI se debería realizar 30 ensayos de resistencia a la compresión (en nuestro caso 60 especímenes por edad) pero la

empresa consideró, debido a su experiencia, que realizar 3 ensayos sería suficiente para determinar una tendencia (en nuestro caso 6 especímenes por edad).

**Cuadro 3.1.** Cálculo del total de especímenes por diseño

Descripción	Realizado en la Tesis	Recomendado por ACI
Edades por Diseño	4	-
Ensayos por Edad	3	30
Especímenes por Ensayo	2	2 (min.)
Total de Especímenes por Diseño	24	-

**Cuadro 3.2.** Formato UNICON para obtener el volumen de concreto necesario para un diseño

ENSAYO		VOLUMEN UNITARIO (L)	Reciclable
1. Reología		7.0	
Fluidez, consistencia (cono de Abrams)		7.1	
2. Peso Unitario y Contenido de Aire		0.0	
3. Temperatura		9.4	
4. Tiempo de Fragua	4.7		
<b>TOTAL</b>		<b>17</b>	

**PRUEBAS PARA CONCRETO FRESCO**

ENSAYO	VOLUMEN UNITARIO (L)	Nº ESPECÍMENES	VOLUMEN TOTAL (L)
1. Resistencia a la Compresión	5.3	8	42.4
2. Permeabilidad	4.0	2	8.0
3. Resistencia al Ataque de Sulfatos	3.0	2	6.0
<b>TOTAL</b>			<b>56</b>

**PRUEBAS PARA CONCRETO ENDURECIDO**

**VOLUMEN TOTAL CONSIDERANDO 10% DE DESPERDICIO: 80 LITROS**

GID-LA-R-XXX Rev.00



**Volumen total de Concreto por Diseño:**

Total por Diseño	80 Litros
Total por Replica	80 Litros
Resistencia a la compresión (Replica adicional) (incluye 10 % de desperdicio)	47 Litros

**Total** **207 Litros**

El volumen total de concreto necesario por diseño fue de 207 Litros. Estos se realizaron en tres batch, dos de 80 Litros y una de 47 Litros.

**3.3.2.2 Desarrollo de los diferentes Ensayos en Concreto Fresco**

A continuación se muestran de manera grafica la ejecución de estos ensayos, realizados en los laboratorios de UNICON, así como también los resultados obtenidos.

**A. CONTENIDO DE AIRE (ASTM C231)**

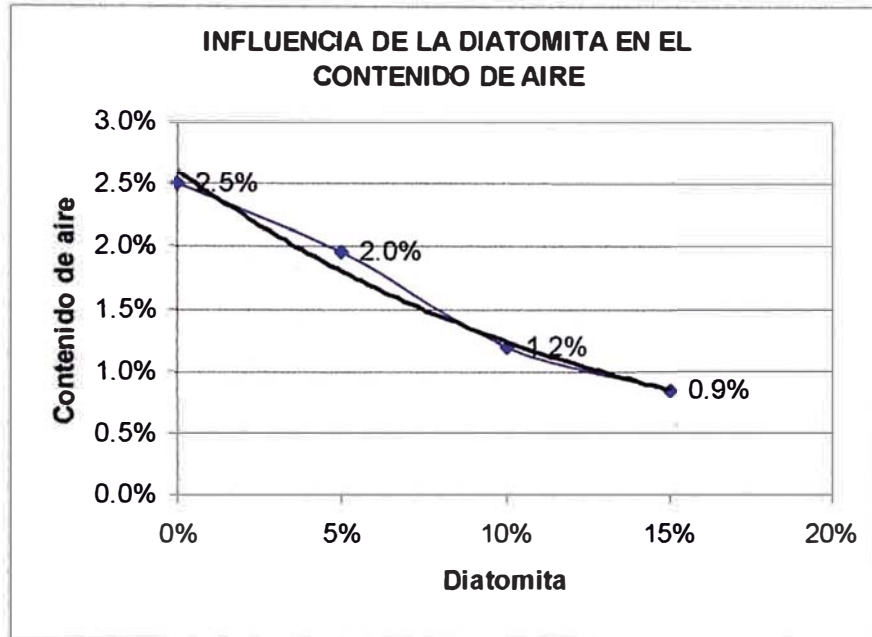
Fig. 3.3. Medición del Contenido de Aire



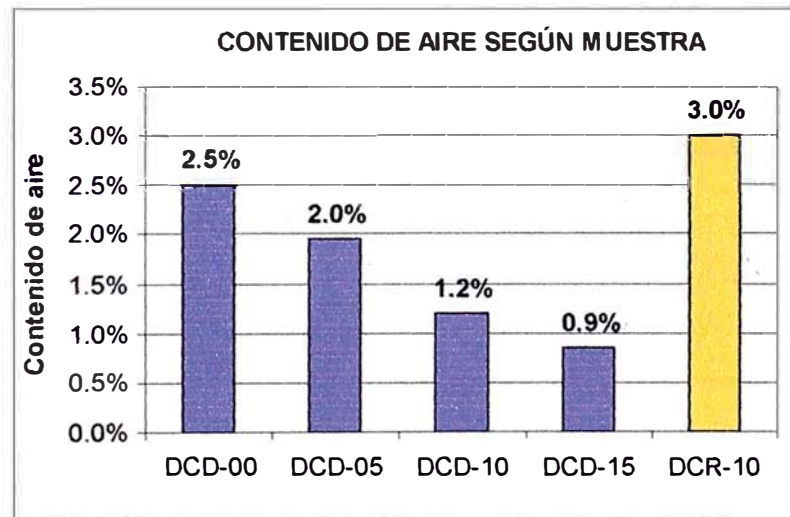
La Gráfica 3.7 muestra que el contenido de aire disminuye cuando se aumenta la cantidad de Diatomita. Al aumentar la cantidad de partículas finas de diatomita, estas no favorecen la formación de porosidad y por tanto la retención de aire disminuye considerablemente, la mezcla se vuelve más compacta, cohesiva y

homogénea. La Gráfica 3.8 ilustra el contenido de aire para cada muestra.

**Grafico 3.7.** Contenido de Aire para diferentes reemplazos de Diatomita



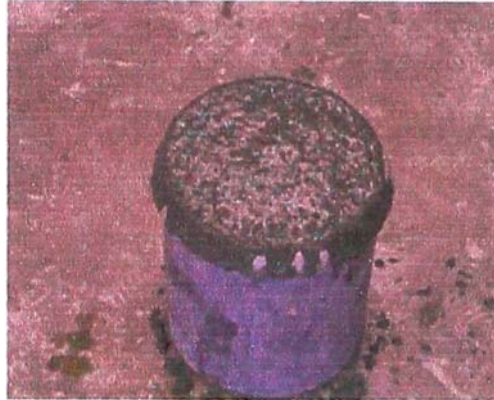
**Grafico 3.8.** Contenido de Aire de los diferentes diseños en estudio



El aire atrapado tanto para los diseños con diatomita y microsilice se encuentran dentro de los límites permisibles (1 a 3%). Este ensayo está relacionado al ensayo de peso unitario del concreto.

## B. PESO UNITARIO (PU) (ASTM C138)

Fig. 3.4. Medición del Peso Unitario

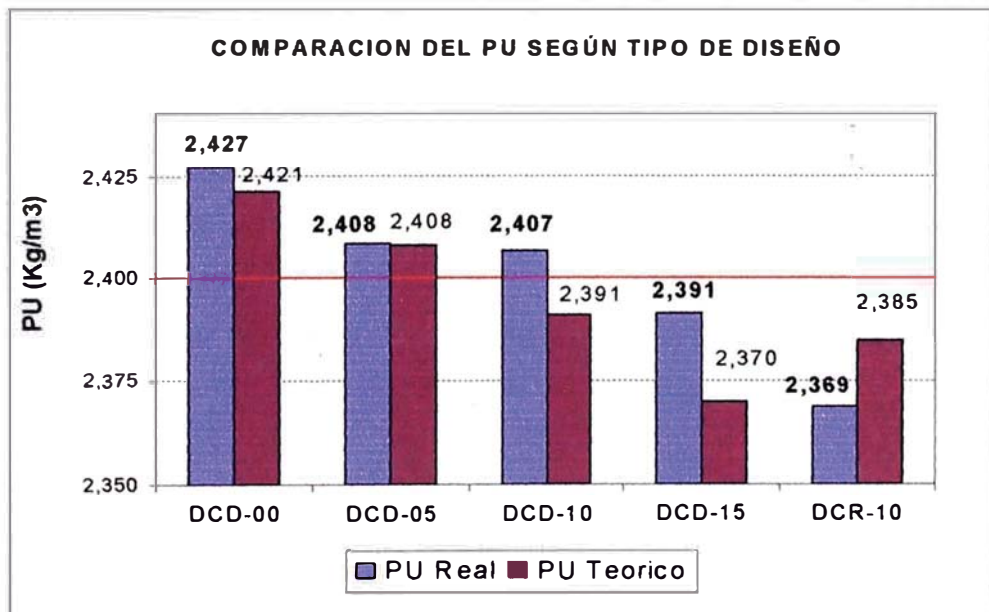


Para el análisis de esta característica técnica primeramente definiremos los siguientes términos::

- **Peso Unitario Real:** Es el peso del concreto fresco menos el peso del recipiente de volumen conocido entre el volumen del recipiente.
- **Peso Unitario Teórico:** Constituye la suma de todos los pesos de los insumos del concreto para un metro cúbico.

Los resultados comparativos de estas medidas para los diseños en estudio están representados en las graficas 3.9.

**Grafico 3.9.** Comparación Peso Unitario Real vs. Peso Unitario Teórico



Para obtener un CAR, si el contenido de cemento aumenta, el PU del concreto aumentará. Sin embargo, si se reemplaza parte del cemento por adición mineral (Diatomita ó Microsílice) de menor densidad que el cemento, los PU de los diseños se mantienen dentro del rango de un concreto normal (2300 – 2400 Kg/m<sup>3</sup>). La Gráfica 3.9. muestra que el PU del diseño DCD-00, es ligeramente superior (1.1% por encima del limite superior), mientras que los diseños con diatomita no implican cambios en el PU del concreto.

#### Comparación del Peso Unitario y Contenido de Aire

En los cálculos del proporcionamiento (punto 2.4.3.) se asumió 1.5% de contenido de aire para todos los diseños. El Cuadro 3.4 muestra de manera comparativa la relación del peso unitario con el contenido de aire de cada muestra. Los comentarios en referencia al porcentaje de aire asumido son los siguientes:

- Para el diseño DCD-00 el porcentaje de aire es 1% mayor al asumido lo que conllevaría a que el peso unitario real sea menor que el teórico, pero los resultados muestran lo contrario. Esto se debe probablemente a la falta de homogeneidad y cohesividad de la mezcla.
- Para el diseño DCD-05 no existe diferencia entre los pesos unitarios real y teórico. El contenido de aire es 0.5% mayor al asumido.
- Para los diseños DCD-10 y DCD-15 el peso unitario real es mayor que el teórico, lo cual coincide pues el contenido de aire real es menor al asumido.
- Por último el DCR-10, el PU real es menor que el teórico, lo cual coincide con el contenido de aire real que es mayor a 1.5%.

**Cuadro 3.4.** Comparación PU y % contenido de Aire

Muestra	PU Real	PU Teórico	% Contenido de Aire
DCD-00	2,427	2,421	2.5
DCD-05	2,408	2,408	2
DCD-10	2,407	2,391	1.2
DCD-15	2,391	2,370	0.9
DCR-10	2,369	2,385	3

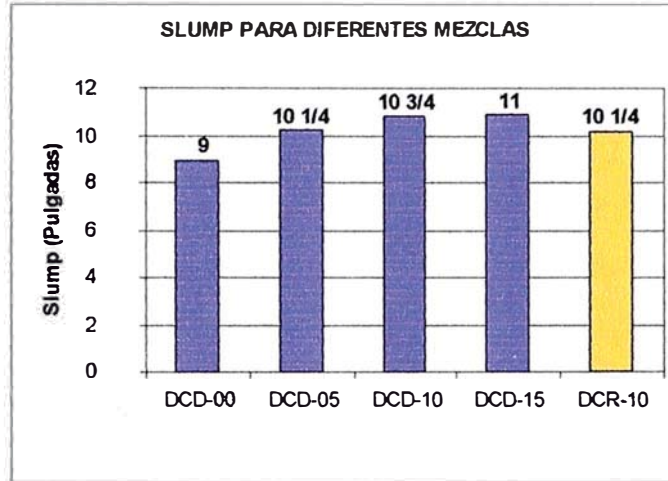
### C. SLUMP (ASTM C143)

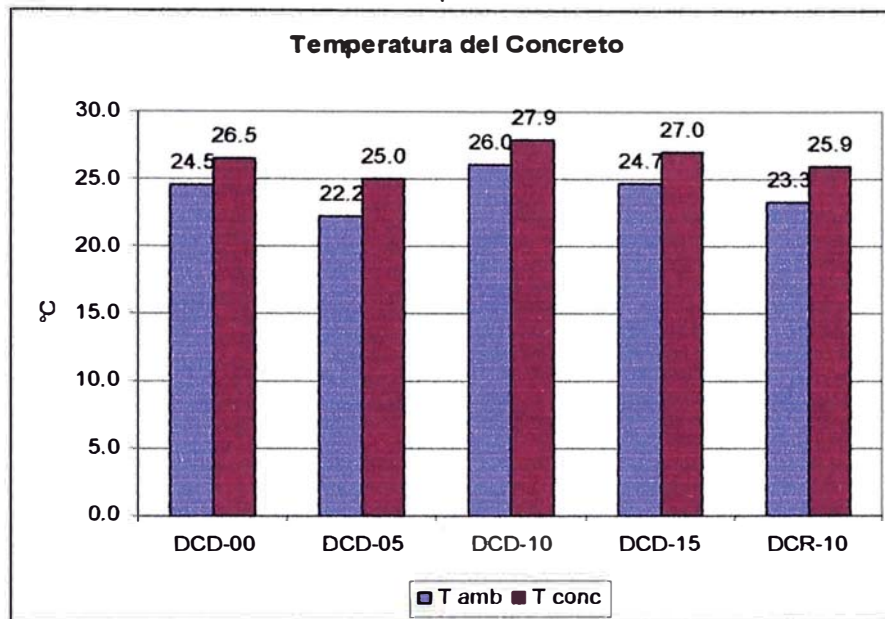
El asentamiento se establece al momento que se define el diseño teórico por alcanzar, que para nuestro caso fué de 600 kg/cm<sup>2</sup> de resistencia a la compresión y Slump de 8" como mínimo (Punto 2.4.3).

**Fig. 3.5.** Medición del Asentamiento usando el cono de Abrams



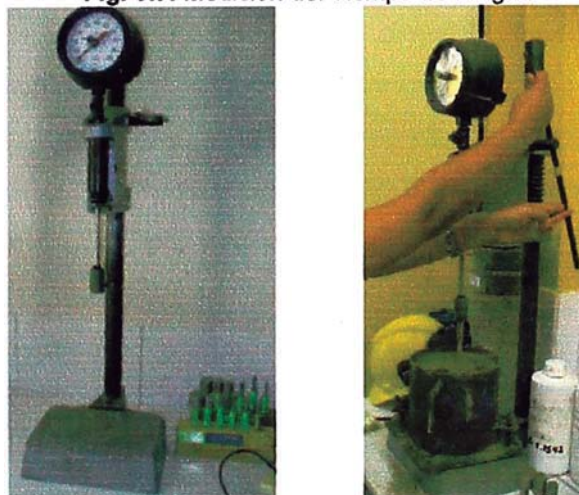
Por los resultados obtenidos, Gráfico 3.10, se puede decir que los diseños con diatomita han llegado a la trabajabilidad requerida.

**Grafico 3.10. Asentamiento****D. TEMPERATURA (ASTM C1064)****Fig. 3.6. Medición de la Temperatura****Termómetro Digital**

**Gráfico 3.11.** Temperatura del concreto

Como se puede observar en la Grafica 3.11, la temperatura del concreto es mayor entre 1°C y 3°C que la temperatura medida del ambiente. Este aumento en temperatura es debido a las primeras reacciones de hidratación del C<sub>3</sub>A (Punto 1.2.5.4.).

### E. TIEMPO DE FRAGUA (ASTM C403)

**Fig. 3.7.** Medición del Tiempo de Fragua

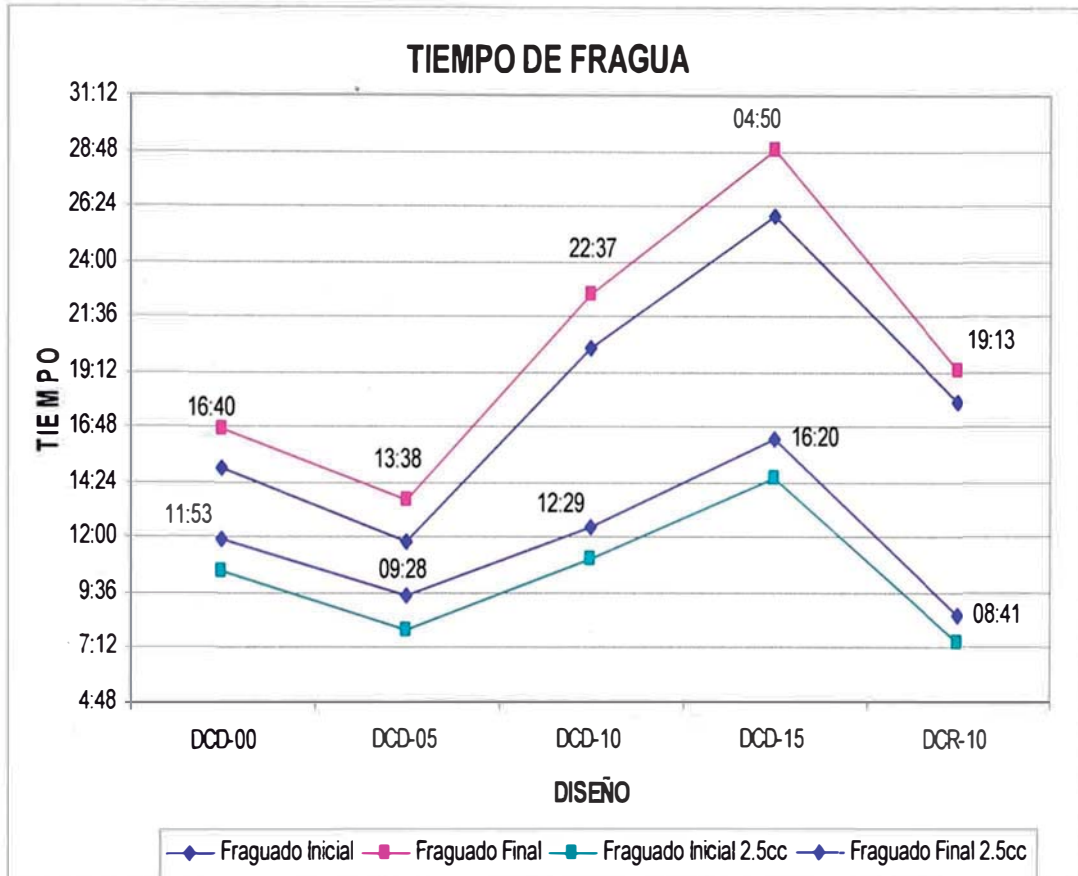
El comportamiento del fraguado para los diferentes diseños se muestra en la Gráfica 3.12. En el proporcionamiento de los diseños, la cantidad

de aditivo retardante era de 5 cc/Kg Cementante, dando como resultado un tiempo de fragua inicial elevado. Por tal motivo se decidió realizar otras pruebas reduciendo la cantidad de retardante a 2.5 cc/Kg Cementante, lo cual dio como resultado un menor tiempo de fragua inicial para cada diseño.

### Comentarios de los Resultados Obtenidos

- Se observa que el tiempo de fraguado es similar para todos los diseños.
- Las tendencias de las curvas del tiempo de fragua inicial y final para 5cc y 2.5 cc de retardante por kilogramo de cementante causan un mismo comportamiento.
- En las curvas de 2.5 cc, al comparar el DCD-00 y el DCD-05, la última presenta un menor tiempo de fraguado inicial, debido al menor contenido cemento (la duración de la formación de etringita es menor).  
De acuerdo a lo dicho anteriormente se esperaría que al aumentar el contenido de diatomita el tiempo de fraguado inicial disminuya por el menor contenido de cemento, pero no sucedió así, al aumentar la cantidad de diatomita la dosificación del aditivo superplastificante también aumenta. Esto se debe probablemente a las altas dosificaciones de aditivo que retrasan la sobresaturación de iones  $Ca^{+}$ , por tanto alargan el periodo de inducción.
- El tiempo de fraguado inicial para el DCD-10 es mayor que para el DCR-10, el contenido de cemento es igual para los dos diseños, pero como la diatomita tiene afinidad por el agua, el requerimiento de aditivo superplastificante es mayor, y por tanto será mayor el tiempo de fragua inicial.



**Gráfico 3.12. Tiempo de Fragua**

### 3.3.3 Ensayos en Concreto Endurecido

#### A. RESISTENCIA A LA COMPRESIÓN

Este ensayo se realizó, de acuerdo a las Normas:

ASTM C39/C39M

ASTM C 617

Estas normas se encuentran en el Anexo G.

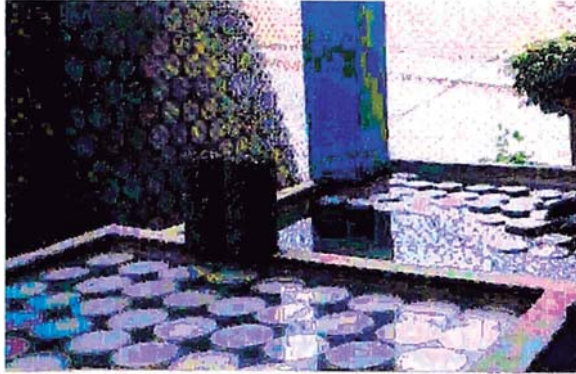
La serie de fotografías que se muestran a continuación ilustran el desarrollo del ensayo de resistencia a la compresión.

Las Gráficas 3.13 y 3.14 muestran el comportamiento de la resistencia a compresión con respecto al tiempo.

**Fig. 3.8. Elaboración de las Probetas de Concreto**



**Fig. 3.9. Curado de las Probetas**

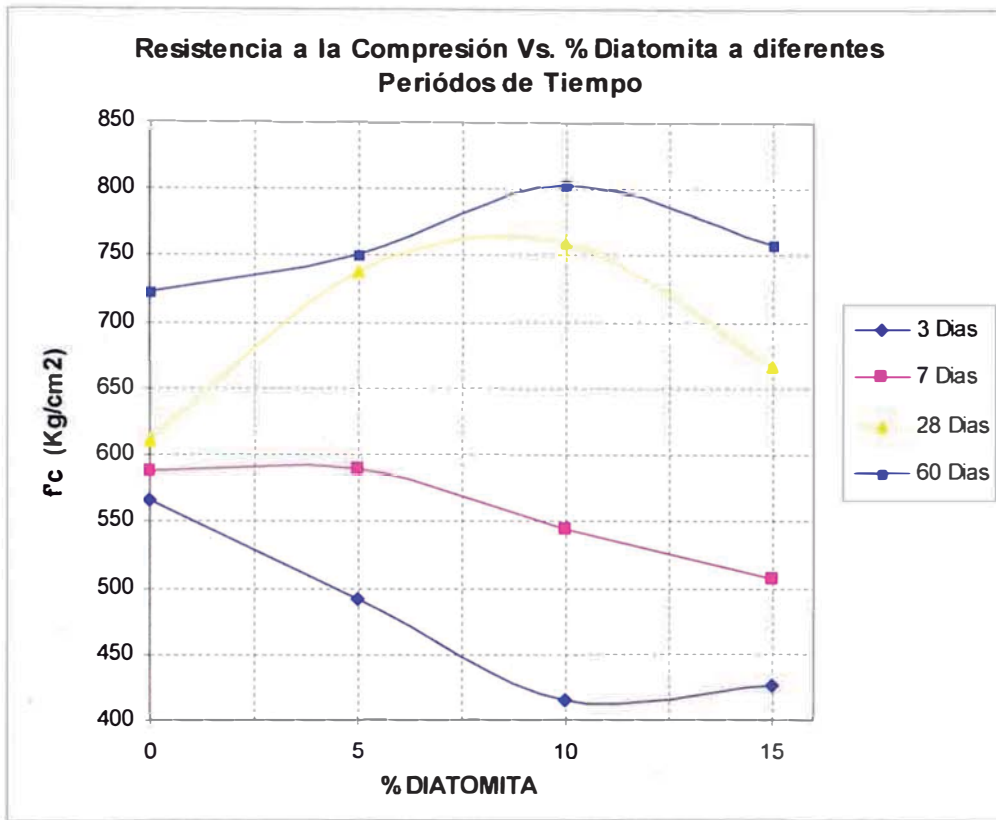


**Fig. 3.10. Medición de la Resistencia a la Compresión**

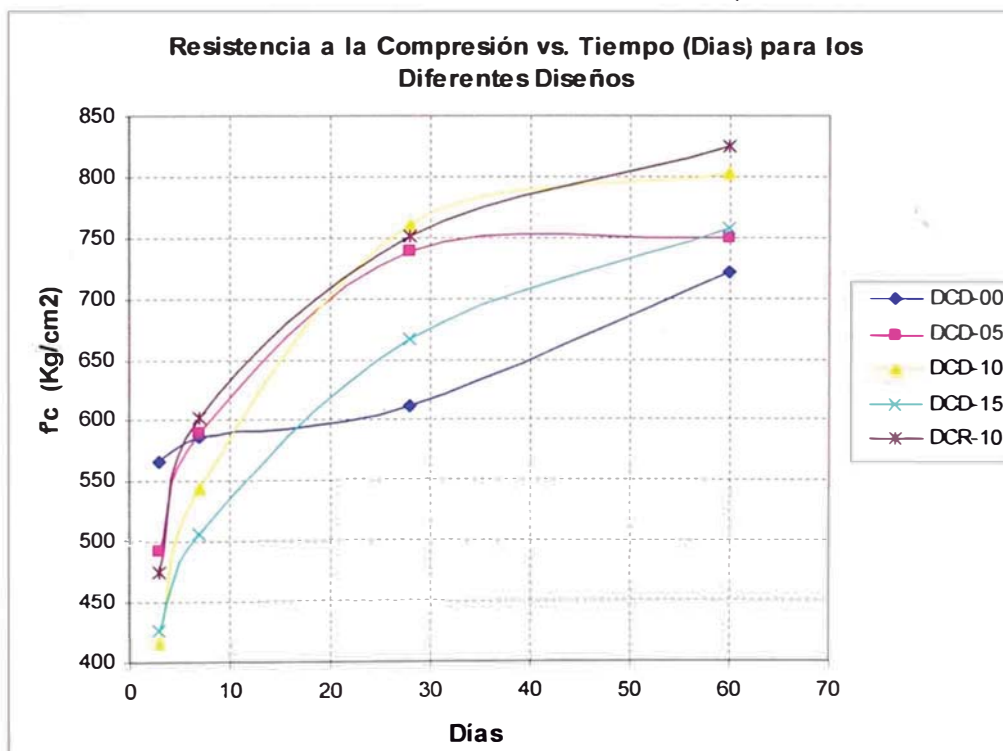
**Capeado de las Probetas**



Gráfica 3.13. Resistencia a la Compresión a diferentes % de Diatomita



Gráfica 3.14. Resultados Resistencia a la Compresión



### Comentarios de los Resultados Obtenidos:

Tomando como referencia las curvas trazadas podemos hacer los siguientes comentarios:

- A los 3 días, la resistencia experimenta la influencia principalmente del proceso de hidratación del  $C_3S$ . En el Gráfico 3.13, el diseño DCD-00 es el que presenta mayor resistencia a la compresión debido a su mayor contenido de cemento. Adicionalmente se puede decir que los otros diseños están influenciados por el contenido del aditivo superplastificante, el que afecta el tiempo de inducción (mayor tiempo de fraguado inicial). Por tanto a esta edad, a mayor contenido de diatomita la resistencia disminuye.
- A los 7 días se observa el mismo fenómeno. Investigadores [7] han demostrado que a esta edad el aporte de los reemplazos del cemento es solamente físico, pues como se puede observar en la Gráfica 3.13 a 5% de reemplazo por Diatomita la resistencia es similar al de 0% de reemplazo. De acuerdo al Gráfico 3.14, el aporte físico de la microsilíce en 10% de reemplazo es muy cercano al aporte físico de la diatomita en 5% (Punto 2.5.1.). Para reemplazos mayores de diatomita el aporte físico es mucho menor.
- A los 28 días se puede observar que el aporte de las puzolanas, ya no es solamente físico, parte de ellas han reaccionado (Punto 1.2.4.2), formando más Silicatos de Calcio Hidratados, disminuyendo la portlandita presente que aporta muy poco a la resistencia. Se debe tener en cuenta que a pesar que la DCD-00 a los 28 días obtuvo una resistencia promedio de  $612 \text{ kg/cm}^2$ , es muy probable obtener un batch con menor resistencia, por lo que se debe considerar que el DCD-00 no alcanza el nivel de Resistencia a la compresión especificado de  $600 \text{ Kg/cm}^2$  (recomendaciones del ACI 214)

Resistencia Promedio Requerida cuando no se tiene Datos Históricos (Referencia: Tabla 4.2-ACI 214R) [25]	
$f'c$ Especificado ( $Kg/cm^2$ )	$f'cr$ ( $Kg/cm^2$ )
<i>Menos de 210</i>	$f'c + 70$
<i>210 a 350</i>	$f'c + 84$
<i>Mayor de 350</i>	$f'c + 98$

Donde:

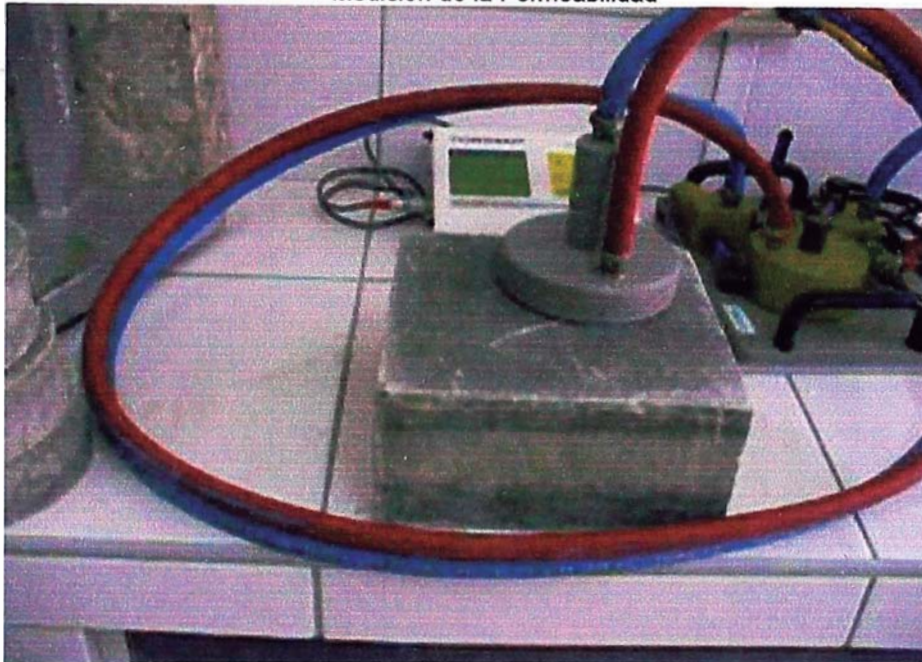
$f'c$  : Resistencia Especificada por el diseñador

$f'cr$ : Resistencia Promedio Requerida en Obra

- A los 60 días el diseño de DCD-00 desarrolla un mayor crecimiento de la resistencia a la compresión respecto a los 28 días, atribuido a la hidratación del  $C_2S$ . La resistencia del diseño DCD-05 es ligeramente mayor con respecto al DCD-00. También se puede observar que el diseño DCD-10 es el que gana mayor resistencia a la compresión en comparación con los demás diseños con diatomita y muestra a los 28 y 60 días un comportamiento similar al diseño DCR-10. Este desempeño del concreto es atribuido tanto al aporte físico como químico de la diatomita.
- Se verificó que la velocidad de reacción a edades tempranas para un reemplazo del 10% de la Microsílice es mayor que para un reemplazo de 10% de diatomita. Es conocida la mayor reactividad de la microsílice a comparación con las puzolanas naturales.

## B. PERMEABILIDAD (Kt)

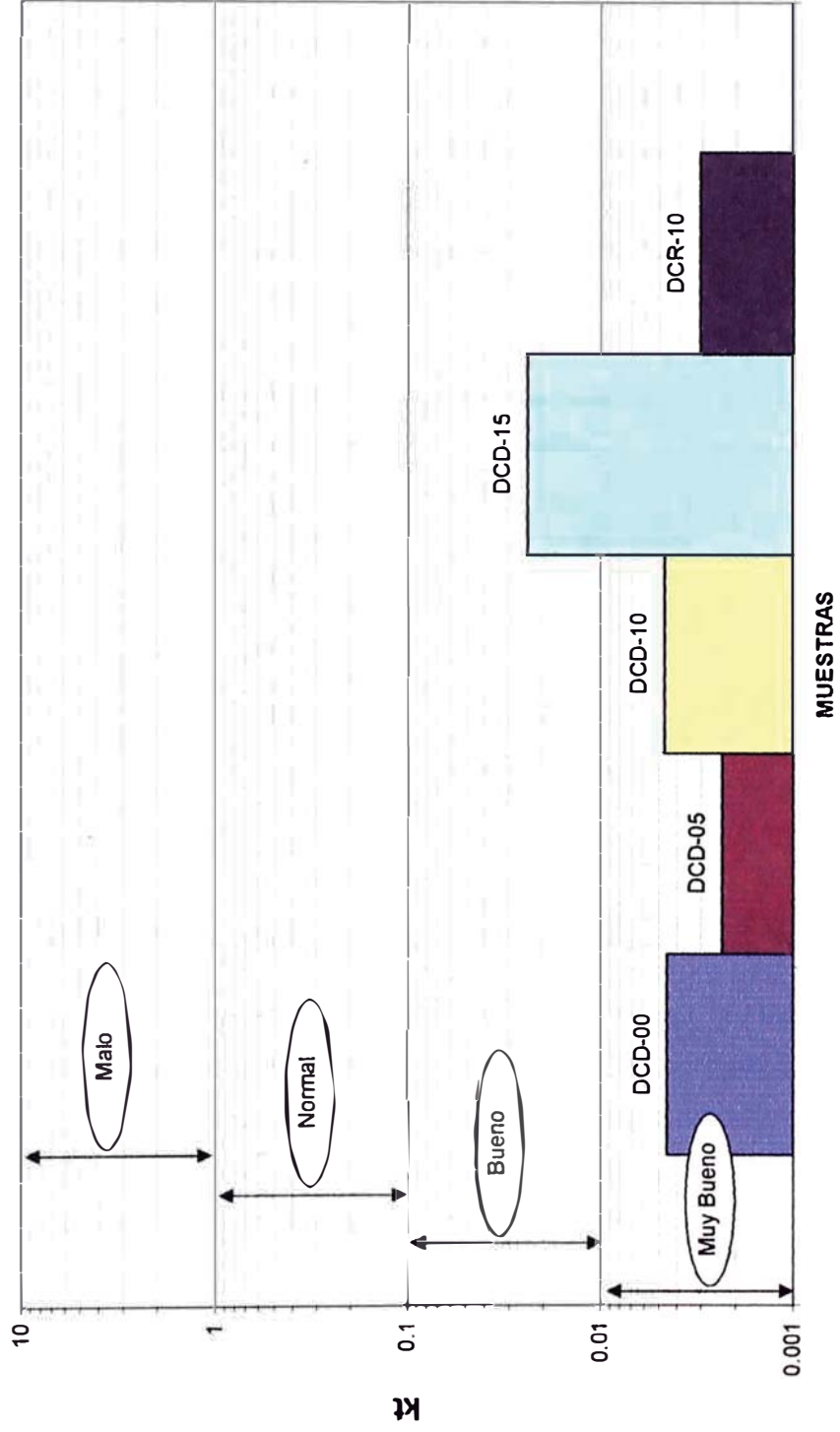
En el punto 1.2.6.2. se indican los conceptos referentes a la permeabilidad. Las siguientes figuras muestran gráficamente el procedimiento para medir la característica de permeabilidad-.

**Fig. 3.11. Resumen del Procedimiento del Ensayo de Permeabilidad.****Preparación de los Especímenes****Medición de la Permeabilidad**

El ensayo de permeabilidad usando el equipo de Torrent permite medir la calidad del covercrete del concreto mediante la medición del Coeficiente de Permeabilidad. La Gráfica 3.15, muestra los resultados obtenidos.

Gráfica 3.15. Resultados del Ensayo de Permeabilidad

### COEFICIENTE DE PERMEABILIDAD PROMEDIO PARA DIFERENTES MUESTRAS DE CONCRETO





Los diseños DCD-00, DCD-05, DCD-10 y DCR-10 se encuentran dentro del rango muy bueno. El diseño DCD-15 presenta mayor permeabilidad a comparación de los demás diseños. Según el modelo de Powers [18], a mayor relación  $a/c$ , mayor será la capacidad capilar (porosidad) y por tanto mostrará mayor permeabilidad. Como se puede observar en la cuadro 3.5, el diseño DCD-15 es el que tiene mayor relación  $a/c$ .

Se trató de mantener a todos los diseños la relación  $a/c$  entre 0.36 y 0.38, pero la demanda de agua de la diatomita no lo permitió, incrementando considerablemente la adición del aditivo superplastificante.

**Cuadro 3.5.** Relación agua total/cementante

<b>Código Diseño</b>	<b><math>a/c</math></b>
DCD-00	0.340
DCD-05	0.358
DCD-10	0.378
DCD-15	0.400
DCR-10	0.378

### **C. RESISTENCIA AL ATAQUE DE SULFATOS EN EL CONCRETO**

El ensayo se realizó tomando como referencia el Método no normalizado de Curado al Vapor (Similar al método normalizado ASTM C1012 [17]), con la diferencia que el ensayo esta orientado a evaluar el concreto frente a un ataque severo de sulfatos. Las reacciones que pudieran ocurrir y definiciones se han descrito en el punto 1.4.3.2.

#### **Equipos:**

- Moldes para Barra sin restricción de acuerdo a la norma ASTM C490 (9 cm. x 9 cm. x 28.5 cm.).
- Comparador de Longitud de acuerdo a la ASTM C490

**Fig. 3.12.** Comparador de Longitud.



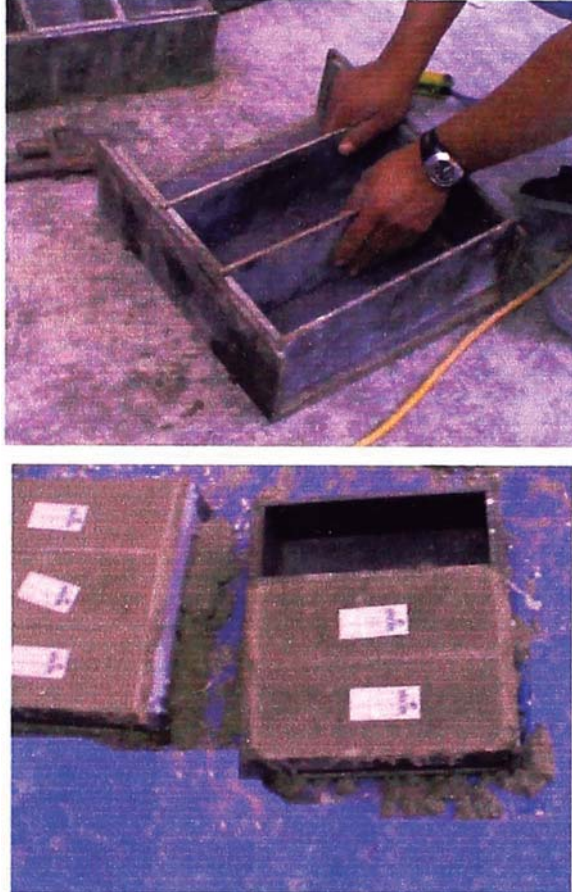
- Contenedor con tapa para evitar contaminación
- Equipo para curado Acelerado (ASTM C684)

**Fig. 3.13.** Equipo para curado Acelerado



**Preparación de lo Especímenes:**

Se prepararon los especímenes de concreto de acuerdo a la norma ASTM C192/C192M *Practice for Making and Curing Concrete Test Specimens in the Laboratory*.

**Fig. 3.14. Preparación de los Especímenes****Ejecución del Ensayo:**

- Se desmolda a las 24 horas. Luego se colocan las barras (dos por diseño) en la cámara de curado acelerado con agua a 85°C y se procede el curado al vapor a esta temperatura por dos horas, conforme al procedimiento del método del curado al vapor.

**Fig. 3.15. Barra de Concreto.**

Retirar las barras, enfriar a temperatura ambiente y tomar la medida inicial de longitud, usando el comparador de longitud de acuerdo a la norma ASTM C490.

Fig. 3.16. Medición de las barras de concreto.



- ☐ Luego, sumergirlas en el contenedor con solución de sulfato.

Fig. 3.17. Barras en solución de sulfato de magnesio.



La solución de sulfato para el curado, tendrá una dilución de sulfato de Magnesio anhidro al 4.4 % en peso y se preparará en un volumen de 4.5 veces el de las barras a sumergir. Esta

permanecerá debidamente tapado en el Laboratorio de Pruebas Físicas a temperatura controlada.

- Se procede a la medición de las barras por semana: 1, 2, 3, 4, 5, 6, 7, 8 y luego a la semana 52, anotándose las medidas de acuerdo a ASTM C490, cuyas mediciones se han graficado (Gráfica 3.16).

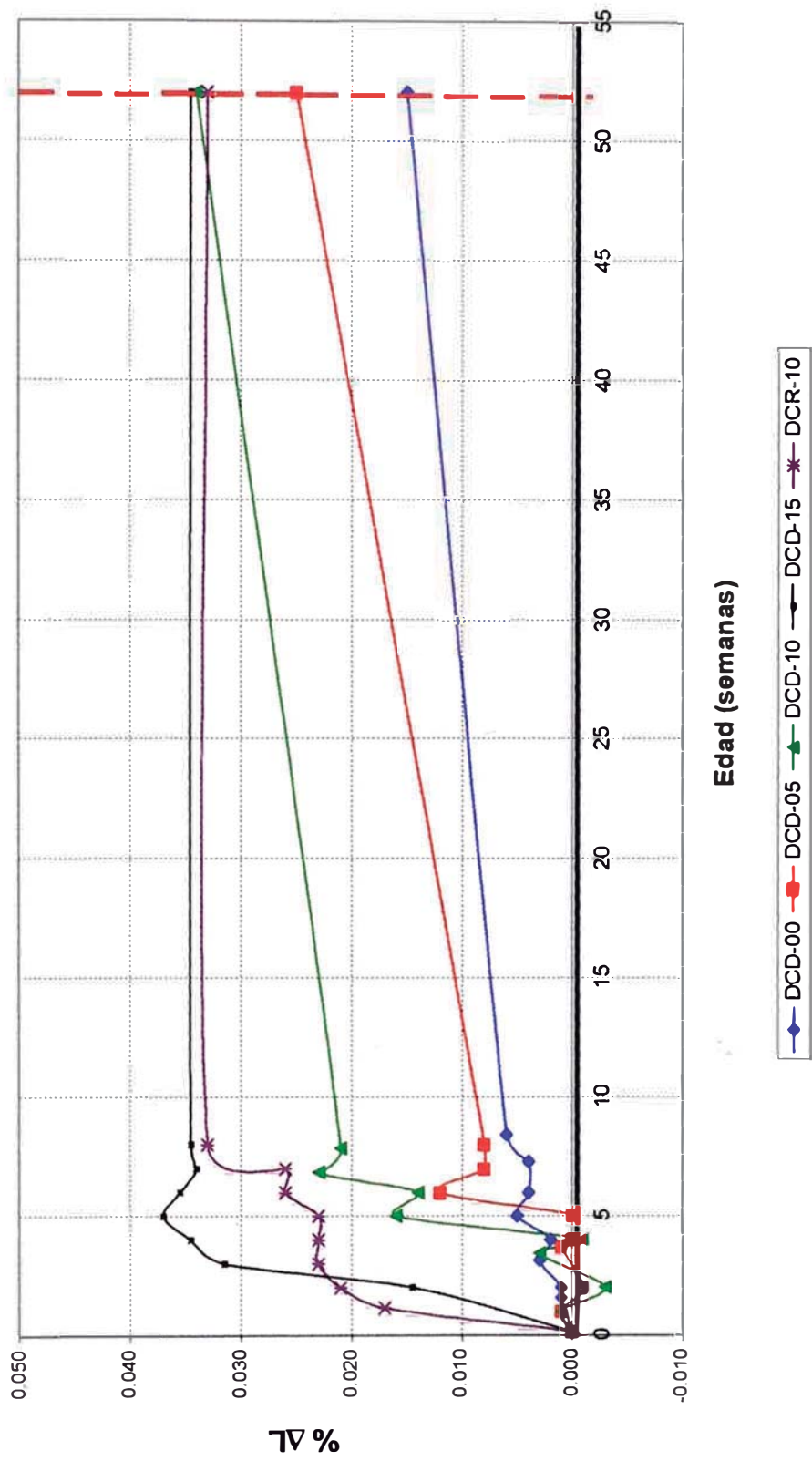
Comentarios a los resultados obtenidos:

- El Gráfico 3.16, presenta la variación de longitud de las barras de concreto de los diferentes diseños como resultado del ataque del  $MgSO_4$ . El medio de exposición con  $MgSO_4$  es severo, la concentración es de aproximadamente 44,000 ppm. Este compuesto puede atacar de dos maneras; formando compuestos sulfoaluminosos (reacción 1.17) y atacando a los silicatos de calcio hidratados (reacción 1.20).
- De acuerdo al gráfico 3.16, todos los diseños, incluido el de referencia, se expanden en el tiempo en pequeños porcentajes.

<b>Diseño</b>	<b>Expansión: 8 Semanas</b>
DCD-00	0.006%
DCD-05	0.008%
DCD-10	0.020%
DCD-15	0.033%
DCR-10	0.034%

- Como se observa a mayor contenido de diatomita la expansión es mayor. El diseño de referencia presenta un grado de expansión similar al diseño DCD-15. La variación de la longitud del DCD-00 es menor que los diseños con adiciones minerales.

Gráfico 3.16. Resultados del Ensayo de Resistencia al Ataque por Sulfatos



- De acuerdo a las reacciones (1.16) y (1.19), los diseños con menor contenido de portlandita y baja permeabilidad son los menos propensos al ataque por sulfatos de sodio, pero esto no sucede cuando se los somete a un ataque severo de sulfatos de magnesio.
- Se puede decir que el compuesto de  $MgSO_4$  esta atacando a los C-S-H en los diseños que contienen puzolanas, debido al bajo pH causado por la reacción Puzolánica (1.13). A simple vista las barras con adiciones minerales muestran deterioro en toda la superficie como se puede apreciar en las fotografías (Fig. x.x a x.y). A mayor cantidad de adición mineral se aprecia mayor deterioro.
- El diseño DCD-00, que contiene mayor cantidad de portlandita, presenta una expansión menor a los demás diseños debido a que mantiene el medio alcalino por mayor tiempo, por tanto los silicatos de calcio hidratados no se han visto afectados a comparación con los diseños con adiciones minerales. En este caso la reacción 1.19 es lenta, pues es un concreto poco permeable.
- Para el caso del DCR-10, el ataque de sulfatos es más rápido que las que contienen diatomita. La microsíllice es bastante reactiva y por lo tanto reacciona rápidamente con la portlandita, causando una disminución en el pH del concreto y por tanto favoreciendo el ataque a los C-S-H por el  $MgSO_4$ . Este fenómeno se observa también en el DCD 15 y DCD10, pero tardío, puesto que la reacción puzolánica en estos diseños es más lenta que en el DCR-10.

- Como este método no está normalizado, no se tiene un límite máximo permisible de expansión.

La norma ASTM C595 para cementos adicionados establece en sus requerimientos que la resistencia a los sulfatos debe tener un límite máximo de expansión de la barra de mortero de 0.1% a los 180 días. (ASTM C1012).

Tomando como referencia este dato, se puede decir que las barras de concreto de todos los diseños están muy por debajo de este valor, alcanzando un máximo de 0.034% de expansión a los 365 días.

La serie de fotografías que se exponen a continuación y que han sido tomadas un año después de comenzado el ensayo muestran las evidencias del ataque del  $MgSO_4$  a las barras diseñadas con puzolanas. Solo las barras DCD-00 están prácticamente intactas.

Cada serie contiene dos fotografías, la superior fue tomada al momento de retirar las barras del contenedor y la de abajo después de haber sido enjuagadas con agua. Las manchas blancas son sal de sulfato de magnesio precipitado.

Se observa después de un año que el  $MgSO_4$  ha reaccionado con la pasta de concreto (cemento y agua) en toda la zona superficial sin deteriorar a los agregados.



Fig. 3.18. Barras de Concreto del Diseño DCD-00 (No contiene adiciones minerales)



Las barras se encuentran en buen estado.

Fig. 3.19. Barras de Concreto del Diseño DCD-05 (Con 5% de Diatomita)



Visualmente se puede decir que el deterioro de este diseño es ligeramente mayor que el diseño DCD-00.

Fig. 3.20. Barras de Concreto del Diseño DCD-10 (Con 10% de Diatomita)



La superficie de las barras se encuentra totalmente deteriorada. Se distingue el agregado.

Fig. 3.21. Barras de Concreto del Diseño DCD-15 (Con 15% de Diatomita)



Se observa deterioro en toda la superficie a tal grado que se puede observar a simple vista los agregados.

Fig. 3.22. Barras de Concreto del Diseño DCR-10 (Con 10% de Microsilíce)



En este diseño con Microsilíce también se aprecia el ataque del  $\text{MgSO}_4$

## CAPÍTULO IV: ANÁLISIS ECONÓMICO COMPARATIVO

A continuación se hace un breve análisis de costos comparativos de los diferentes diseños con diatomita, con respecto al diseño de microsíllice referencial, DCR-10.

### A. Cálculo del Costo Unitario de los Diseños Experimentados

Concepto	Unidad	PU	Código Diseño					
			DCR-10 (Referencia)		DCD-00		DCD-05	
			Cantidad	USD/m <sup>3</sup>	Cantidad	USD/m <sup>3</sup>	Cantidad	USD/m <sup>3</sup>
Cemento	Kg	0.09478	450.00	42.65	500.00	47.39	475.00	45.02
Diatomita - Fillite	Kg	0.25	0.00	0.00	0.00	0.00	25.00	6.25
Silica Fume	Kg	0.91	50.00	45.50	0.00	0.00	0.00	0.00
Agua	L	0.0008398	140.17	0.12	148.33	0.12	138.59	0.12
Arena	Kg	0.00578	920.86	5.32	931.37	5.38	932.24	5.39
Piedra 67	Kg	0.00463	821.09	3.80	831.47	3.85	823.36	3.81
Polyheed RI	L	0.82	2.50	2.05	2.50	2.05	2.50	2.05
Viscocrete	L	2.78	10.00	27.80	6.00	16.68	8.90	24.74
<b>Costo total por m<sup>3</sup></b>			<b>USD</b>	<b>127.24</b>	<b>USD</b>	<b>75.48</b>	<b>USD</b>	<b>87.38</b>

Concepto	Unidad	PU	Código Diseño			
			DCD-10		DCD-15	
			Cantidad	USD/m <sup>3</sup>	Cantidad	USD/m <sup>3</sup>
Cemento	Kg	0.09478	450.00	42.65	425.00	40.28
Diatomita - Fillite	Kg	0.25	50.00	12.50	75.00	18.75
Silica Fume	Kg	0.91	0.00	0.00	0.00	0.00
Agua	L	0.0008398	129.01	0.11	152.79	0.13
Arena	Kg	0.00578	929.81	5.37	894.80	5.17
Piedra 67	Kg	0.00463	812.43	3.76	794.20	3.68
Polyheed RI	L	0.82	2.50	2.05	2.50	2.05
Viscocrete	L	2.78	14.00	38.92	21.50	59.77
<b>Costo total por m<sup>3</sup></b>			<b>USD</b>	<b>105.37</b>	<b>USD</b>	<b>129.83</b>

## B. Ahorro Alcanzado

Diseño	Costo (USD/m3)	Ahorro* (USD/m3)
DCR-10 (Referencia)	127.24	0.00
DCD-00	75.48	51.77
DCD-05	87.38	39.86
DCD-10	105.37	21.88
DCD-15	129.83	-2.59

(\*) Se calcula el ahorro tomando como referencia el diseño (DCR-10)

Se puede concluir que el uso de la Diatomita Fillite de procedencia nacional en reemplazo de la microsilíce aporta ahorros para todos los casos salvo para el DCD-15.

## CONCLUSIONES

1. Se demostró que cuando se reemplaza cemento por diatomita en un porcentaje en peso entre 5% y 10% se obtiene resistencias a la compresión que superan los 600 kg/cm<sup>2</sup>, la permeabilidad disminuye y aumenta la cohesión. Por tanto para este nivel de resistencia a la compresión, se puede reemplazar la Microsilice en la producción de CAR con Diatomita Nacional siempre y cuando su contenido de SiO<sub>2</sub> se encuentre entre 81 y 95%, esté libre de contaminantes y se considere su humedad en el balance de agua al momento de realizar el diseño de mezcla.
2. El empleo de diatomita aumenta la cohesividad de la mezcla, disminuyendo la fluidez. Para lograr una mejor fluidez se requiere el empleo de aditivos superfluidificantes. Porcentajes mayores o iguales a 10% de diatomita llevaría a una mezcla poco trabajable, pues la diatomita tiende a aglomerarse, dificultando la homogeneidad de la mezcla y originando cambios de viscosidad bruscos.
3. De acuerdo a los resultados de los ensayos de viscosidad, el diseño DCD-05 presenta buenas propiedades reológicas de tixotropía y homogeneidad. La primera es muy importante, pues una mezcla tixotrópica mantiene en suspensión a los agregados, y además es la que más se acerca al de referencia (DCR-10). Para los diseños DCD-10 y DCD-15, la tixotropía es de mayor grado en comparación con DCD-05 y DCR-10, lo cual significa cambios de viscosidad considerables, que pueden afectar al producto final. Se verificó que la pasta del DCD-00 presenta problemas de segregación y falta de homogeneidad.
4. En general, las adiciones minerales incrementan el requerimiento de agua, pues éstas presentan mayor superficie específica que el cemento (Microsilice = 180,000 cm<sup>2</sup>/g, Diatomita = 37,200 cm<sup>2</sup>/g y Cemento = 3,700 cm<sup>2</sup>/g), pero durante la ejecución de los ensayos, se observó que los



diseños con diatomita requerían mayor cantidad de agua en comparación del diseño con microsilice. Este incremento se debe a la mayor afinidad de la estructura cristalina de la diatomita (ópalo) con el agua comparándola con la estructura amorfa de la microsilice. Por tanto, para obtener un CAR que sea trabajable, a mayor cantidad de diatomita se necesitaría mayor cantidad de aditivo superplastificante. Pero habrá que tener en cuenta que en la composición del aditivo existe agua, si se agrega una cantidad de aditivo tal que la relación agua/cementante sea mayor al rango recomendado de 0.35 y 0.38 para CAR ( $600 \text{ Kg/cm}^2$ ), entonces tendremos que la resistencia a la compresión del CAR tenderá a disminuir.

5. Por lo anterior a quedado demostrado que para producir CAR con Diatomita superior a  $600 \text{ Kg/cm}^2$  de resistencia a la compresión, será necesario la adición de aditivos en base a policarboxilatos, reductores de agua, pues estos aditivos contrarrestan el efecto de la Diatomita para incrementar el requerimiento de agua.
6. Todos los diseños con Diatomita superaron después de 28 días la resistencia mínima esperada de  $600 \text{ Kg/cm}^2$ . Los diseños DCD-05 y DCD-10 a los 28 días superaron ampliamente (entre 23% y 26%) el valor esperado. A los 60 días los tres diseños con diatomita superaron entre 23% a 33% al valor esperado.
7. El Peso Unitario de las muestras con diatomita son similares a la referencia con Microsilice y se encuentran en el rango de un concreto normal:  $2,300 - 2,400 \text{ Kg / m}^3$ . Esto quiere decir que para pesos unitarios semejantes, el CAR posee mayor resistencia a la compresión.
8. Se puede afirmar que a menor cantidad de diatomita, el tiempo de fraguado disminuye, debido a la menor cantidad de aditivo superfluidificante necesario.
9. Con respecto a su durabilidad, los diseños DCD-00, DCD-05 y DCD-10, se encuentran dentro del rango de permeabilidad muy buenos al igual que el

DCR-10 que es lo que se esperaba. Con respecto a la resistencia al ataque de sulfatos, el uso de esta roca (diatomita) no es favorable frente a ataques severos de sulfato de magnesio ( $\text{MgSO}_4$ ), a pesar que está demostrado [17] que las puzolanas tienden a mejorar la resistencia del mortero frente al ataque de sulfatos de sodio.

10. Del punto de vista económico, salvo el DCD-15, el resto de diseños DCD-05 y DCD-10 experimentados otorgan un ahorro en costos de producción que van del 30 al 17% por metro cúbico producido. Bajo esta perspectiva de costos el DCD-05 sería el que aportaría el mayor ahorro económico.

## RECOMENDACIONES

1. Las variaciones en el contenido de  $\text{SiO}_2$  de la diatomita nacional extraída de canteras, que solo pasa por procesos físicos de reducción (molienda), se han visto reflejadas en los resultados de resistencia a la compresión. Por lo que se recomienda, se efectúe un estudio de sensibilidad del ensayo de resistencia a la compresión tomando en cuenta; el color de la diatomita, el contenido de  $\text{SiO}_2$  y su humedad respectiva.
2. Mientras mas blanca sea la diatomita, mejor será su calidad. Por lo que se debería establecer patrones de referencia de color de la diatomita, pues las variaciones del color significa contenido de impurezas. Esto sería una forma rápida de verificar y monitorear la calidad de la diatomita.
3. Otra manera de realizar un control de calidad de la diatomita sería mediante la observación microscópica. La diatomita utilizada en la presente tesis es de origen lacustre y la forma de las partículas son alargadas (han sido representadas en la Figura 2.1)
4. En referencia a lo anteriormente expresado, en algunos países, la diatomita es purificada mediante lavado - calcinación – precipitación para obtener una mejor concentración de  $\text{SiO}_2$  disminuyendo el rango de variación, obteniéndose una diatomita mas reactiva. En tal sentido se recomienda aplicar este procedimiento de purificación a la diatomita nacional.
5. Para ajustar el valor óptimo de reemplazo de diatomita se recomienda hacer pruebas para reemplazos del 6, 7, 8, 9 % de diatomita y también para periodos de tiempo mas largo (90 días).
6. Los resultados de resistencia a la compresión son datos probabilísticos por lo que para asegurar que la resistencia se mantenga dentro de los límites establecidos en el diseño teórico, se recomienda realizar 30 ensayos para una misma edad.

7. De acuerdo a los resultados del ensayo de Resistencia al Ataque de Sulfatos, se recomienda realizar ensayos comparativos frente a una solución de Sulfato de Sodio y otra de Sulfato de Magnesio, para verificar las reacciones del concreto frente a estas dos sales.
8. Con respecto a la aplicación de este tipo de concreto, se recomendaría que para porcentajes superiores a 5% no se utilice bomba, solo en aplicaciones rápidas de vaciado.
9. De acuerdo a los resultados experimentales, se recomendaría realizar pruebas utilizando un defloculante.
10. La industria del cemento genera grandes cantidades de CO<sub>2</sub> (1 Kg. de CO<sub>2</sub> por cada Kg de Cemento producido) liberándolos al medio ambiente, se debe incentivar el uso de materiales como las adiciones minerales ya que esto favorece el menor el uso del cemento. Al disminuir la demanda de cemento por parte de la industria con concretera se fabricara menos esta materia., reduciendo la contaminación generada. De esta manera, se propicia un desarrollo sostenible que garantice el desarrollo actual sin comprometer las generaciones futuras.
11. Mucho del conocimiento sobre estos procesos (uso de materiales alternativos) se genera en países desarrollados, siendo necesario reducir la dependencia del conocimiento y la tecnología proveniente del extranjero. La formación de recursos humanos calificados ayudará a generar conocimientos y experiencia, por lo que es importante que los sectores industriales privados y públicos inviertan.

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## ANEXO A

**Certificados de Calidad y Hojas Técnicas de la Materia Prima,  
Insumos y Aditivos utilizados**



**Cía Minera  
AGREGADOS CALCAREOS S.A.**

**FILLITE****ANÁLISIS QUÍMICO TÍPICO**

SiO <sub>2</sub>	.....	87.20 %
TiO <sub>2</sub>	.....	0.16 %
Al <sub>2</sub> O <sub>3</sub>	.....	3.49 %
Fe <sub>2</sub> O <sub>3</sub>	.....	1.22 %
Cr <sub>2</sub> O <sub>3</sub>	.....	0.00 %
CaO	.....	0.47 %
MgO	.....	0.79 %
MnO	.....	0.02 %
SO <sub>3</sub>	.....	0.08 %
P <sub>2</sub> O <sub>5</sub>	.....	0.00 %
Na <sub>2</sub> O	.....	0.07 %
K <sub>2</sub> O	.....	0.52 %
Cl-	.....	0.28 %
Pérdida por Calcinación.....		4.14 %
No analizado	.....	1.58 %
<b>TOTAL</b>	.....	<b>100.00 %</b>

**PROPIEDADES FÍSICAS TÍPICAS****RANGO**

Absorción de aceite (ASTM D-281) %	87.02	85.00 - 95.00
Gravedad específica	2.020	2.000 - 2.150
Densidad aparente g/l	152	140 - 170
Fineza Hegman - NS (ASTM D-1210)	3 %	3 - 4
Superficie específica (Blaine)(ASTM C-204) cm <sup>2</sup> /g	30402	28000 - 32000
pH	8.10	7.8 - 8.3
Granulometría : Menos malla 325 (45 μm) % mínimo	99.90	99.90 - 100.00
Tamaño máximo (Sedigraph) μm	31.50	28.0 - 40.0
Tamaño medio de partícula (Sedigraph) μm	1.57	1.40 - 2.00
Partículas submicrónicas (Sedigraph) %	35.0	30.0 - 40.0
Blancura - Photovolt %	84.0	80.0 - 85.8
Abrasividad (Valley) mg	154	140 - 180
Humedad %	8.09	7.50 - 10.00

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<http://www.agregadoscalcareos.com.pe>



# POLYHEED® RI

Aditivo reductor de agua de medio rango y retardante

## USOS RECOMENDADOS

POLYHEED RI se recomienda en todo concreto donde se quiera extender el fraguado, trabajabilidad y acabados superiores, especialmente en climas cálidos. POLYHEED RI es particularmente efectivo en asentamientos de medio rango (150 a 200 mm).

POLYHEED RI puede usarse en concreto arquitectónico y concreto con color.

POLYHEED RI puede usarse con aditivos inclusores de aire, siempre que satisfagan las especificaciones ASTM, AASHTO y CRD.

## DESCRIPCION

POLYHEED RI es un aditivo reductor de agua de medio rango, multicomponente y libre de cloruros formulado para producir:

1. Una verdadera reducción de agua de medio rango (5 a 15%) y un excelente desempeño a través de un amplio rango de asentamiento, especialmente el rango difícil de 150 a 200 mm de asentamiento en el concreto.
1. Aumenta el tiempo de fraguado del concreto a lo largo del rango de dosificación recomendado.
2. Mejor calidad en trabajabilidad, bombeabilidad y acabado aún en mezclas de concreto con bajas cantidades de
3. materiales cementicios y/o mezclas que contengan cenizas volantes.
4. Desarrollo de resistencias comparable con los aditivos reductores y retardantes en todas las edades.
5. Mejor desempeño con un amplio rango de cementos, cenizas volantes, escorias granuladas y agregados (incluyendo la grava y las arenas manufacturadas).

POLYHEED RI cumple con los requisitos de la norma ASTM C494 para aditivos retardantes Tipo B y reductores de agua y retardantes Tipo D, específicamente:

- Reduce el contenido de agua para un asentamiento dado.
- Mejores características del tiempo de fraguado.
- Incremento en el desarrollo de las resistencias a la compresión y a la flexión en todas las edades.

- Mejoramiento en la durabilidad del concreto al daño ocasionado por congelamiento y deshielo.
- Reducción de contracciones.

## VENTAJAS

POLYHEED RI ayuda a la producción de un concreto de calidad proporcionando las siguientes ventajas especiales:

- Trabajabilidad y bombeabilidad superior en aplicaciones en clima caluroso.

Reduce la segregación.

Mejores características de acabado en pisos y en cualquier aplicación donde se utilice encofrado.

- Desempeño consistente en concretos con asentamiento bajo, medio rango de asentamiento de 150 a 200 mm y alto asentamiento del concreto.

POLYHEED RI resulta efectivo ya sea como un aditivo único o como parte de un sistema de aditivos de Degussa Construction Chemicals.

## LIMITACIONES

Si POLYHEED RI se congela, llévese a una temperatura de 2°C o más, y agítese hasta que esté completamente reconstituido. No use aire a presión para agitarlo.

La aplicación adecuada del producto es responsabilidad del usuario. Toda visita de campo realizada por el personal de Degussa tiene como fin único el hacer recomendaciones técnicas y no el supervisar o proporcionar control de calidad en el lugar de la obra.

## DATOS TECNICOS

Tiempo de Fraguado <sup>1</sup>		
Mezcla	Fraguado Inicial Hrs:Mins	Diferencia Hrs:Mins
Normal	3:44	
POLYHEED RI		
195 ml/100 kg	3:55	+0:11
390 ml/100 kg	5:22	+1:38
585 ml/100 kg	8:33	+ 4:49
Resistencia a la Compresión		
Mezcla	7 Días MPa %	28 Días MPa %
Normal	19.8 / 100	28.3 / 100
POLYHEED RI		
195 ml/100 kg	23.9 / 120	29.3 / 111
390 ml/100 kg	25.2 / 127	31.9 / 121
585 ml/100 kg	28.5 / 144	35.9 / 137

<sup>1</sup> Nota: Los datos arriba mostrados están basados en pruebas controladas de laboratorio. Se pueden esperar variaciones con respecto a estos resultados por las condiciones de la obra y de los materiales..

Cuando se vaya a utilizar POLYHEED® RI con otro aditivo, cada uno debe dosificarse por separado en la mezcladora.

## CARACTERISTICAS DE DESEMPEÑO

### Datos de la mezcla

305 kg de cemento Tipo I por m<sup>3</sup>. Asentamiento de 114 mm, sin aire incluido. Temperatura del concreto 32°C, temperatura ambiente 32 °C.

### Dosificación

POLYHEED RI se recomienda en un rango de 200 a 800 ml por cada 100 kg de cemento. en la mayoría de las mezclas de concreto para clima caluroso.

Degussa Construction Chemicals no recomienda usar dosificaciones fuera del rango establecido sin antes realizar pruebas de campo. Consulte a su representante local de Degussa para determinar el rango de dosificación óptimo.

### Sin cloruros, sin corrosión

POLYHEED RI no contiene cloruro de calcio añadido ni algún otro agente químico que contenga cloruros, por lo tanto no provoca la corrosión del acero de refuerzo del concreto.

## ALMACENAMIENTO

La vida útil de POLYHEED RI es de 12 meses como mínimo, si se mantiene en su envase original cerrado y almacenado en un sitio fresco y seco.

## SEGURIDAD

Consulte la Hoja de Datos de Seguridad (MSDS) para este producto

Para información adicional sobre este producto o para su uso en el desarrollo de mezclas de concreto con características especiales de desempeño, consulte a su representante de Degussa.

## EMPAQUE

POLYHEED RI se suministra en tambores de 208 l y a granel.

### DECLARACIÓN DE RESPONSABILIDADES A ISO DE GARANTÍA LIMITADA

Degussa ha realizado todos los esfuerzos posibles para aplicar los estándares exactos tanto en la fabricación de nuestros productos como en la información que emitimos con respecto a los mismos y a su uso. Garantizamos que nuestros productos son de buena calidad y reemplazaremos o, a voluntad, reembolsaremos el precio de compra de cualquier producto que resulte defectuoso. Los resultados satisfactorios dependen no sólo de la calidad de los productos sino de muchos factores que escapan a nuestro control. Por lo tanto, excepto dicho reemplazo o reembolso, Degussa NO DA GARANTÍAS, EXPRESAS O IMPLÍCITAS, INCLUYENDO GARANTÍAS DE ADAPTACIÓN A UN DETERMINADO O DE LA COMERCIABILIDAD DE SUS PRODUCTOS, no teniendo Degussa en el futuro ninguna otra responsabilidad con respecto a ello. Cualquier reclamo con respecto a un defecto del producto debe ser recibido por escrito dentro del año de la fecha de envío. No se considerarán reclamos sin dicho aviso escrito o después de la fecha especificada. El usuario determinará a su exclusivo criterio la conveniencia de los productos para el uso de que se trate y asume todo riesgo y responsabilidad en relación con ello. Todo cambio autorizado en las recomendaciones impresas con respecto al uso de nuestros productos debe llevar la firma del Gerente Técnico de Degussa.

Esta información y todo otro aviso técnico se basan en la real experiencia y conocimiento de Degussa. Sin embargo, Degussa no asume responsabilidad por el suministro de dicha información y avisos incluso en la medida en que dicha información y avisos pueden relacionarse con derechos de propiedad intelectual existentes de terceros. En particular, Degussa se exime de toda GARANTÍA, SEA EXPRESA O IMPLÍCITA, INCLUYENDO LA GARANTÍA IMPLÍCITA DE ADAPTACIÓN A UN DETERMINADO O COMERCIABILIDAD, DE GUSSA NO SER RESPONSABLE POR DAÑOS DIRECTOS O INDIRECTOS O DE CUALQUIER OTRA NATURALEZA (INCLUYENDO PERDIDA DE BENEFICIOS DE CUALQUIER TIPO). Degussa se reserva el derecho de realizar cambios de acuerdo con el progreso tecnológico o ulteriores desarrollos. Es responsabilidad y obligación del cliente inspeccionar cuidadosamente y probar toda mercadería que reciba. El rendimiento de los productos aquí detallados debe ser verificado con pruebas y llevadas a cabo sólo por personal calificado. Es responsabilidad única del cliente llevar a cabo y establecer todo tipo de pruebas. La referencia a marcas utilizadas por otras empresas no es recomendación ni respaldo de producto alguno y no implica que pueda utilizarse ningún producto similar.

### Degussa Construction Chemicals Latin America

Argentina (54 34 8843 3000) Brasil (55 11 108 5555) Chile (56 2 444 97 0) Colombia (57 1 321 7210) Costa Rica (50 440 9110) Ecuador (593 2 25 011) México (52 55 2122 2200) Perú (51 385 0109) Puerto Rico (787 258 2737) República Dominicana (809 957 9303) Venezuela (58 212 7 2 5471)

**CONSUMO**

- Para concretos plásticos suaves: 0.4% – 1.2% del peso del cemento
- Para concretos fluidos y autocompactantes: 1.2% – 2.0% del peso del cemento

**OBSERVACIONES****Para Concretos Fluidos y Concretos Autocompactantes**

Sika® ViscoCrete®-1 también puede usarse para concretos fluidos y autocompactantes mediante la utilización de dosificaciones especiales de mezclado.

**Cuando el Sika® ViscoCrete®-1 está Congelado**

Descongelarlo lentamente a temperatura ambiente y mezclarlo en forma intensiva.

**Combinaciones**

Sika® ViscoCrete®-1 puede combinarse con los siguientes productos Sika®: Sika® Pump, Sika® Ferrogard® 901, Sika® Fume y Sika® Aer.

Se recomienda realizar un ensayo previo si se realizan combinaciones de varios de los productos antes mencionados. Favor consultar a nuestro servicio técnico.

**ALMACENAMIENTO**

12 meses a partir de la fecha de producción, en su envase original y sin abrir, protegido de la luz directa del sol y de las heladas, a temperaturas entre 5°C y 35°C.

**INSTRUCCIONES DE SEGURIDAD****Precauciones**

Durante la manipulación de cualquier producto químico, evite el contacto directo con los ojos, piel y vías respiratorias. Protéjase adecuadamente utilizando guantes de goma natural o sintéticos y anteojos de seguridad.

En caso de contacto con los ojos, lavar inmediatamente con abundante agua durante 15 minutos manteniendo los párpados abiertos.

**Ecología**

No desechar en vías acuáticas ni en el suelo. Cumplir las normas locales al respecto.

**Toxicidad**

No tóxico según los códigos suizos vigentes sobre salud y seguridad.

La Hoja de Seguridad de este producto se encuentra a disposición del interesado. Agradeceremos solicitarla a nuestro Departamento Comercial, teléfonos: 437 7055 ó 437 5888 o descargarla a través de Internet en nuestra página web [www.sika.com.pe](http://www.sika.com.pe)

**PRESENTACIONES**

Cilindro de 180 kg.

Balde x 20 kg.

**“La presente Edición anula y reemplaza la Edición N°2  
la misma que deberá ser destruida”**

**Advertencia:**

La información y, en particular, las recomendaciones sobre la aplicación y el uso final de los productos Sika son proporcionadas de buena fe, en base al conocimiento y experiencia actuales en Sika respecto a sus productos, siempre y cuando éstos sean adecuadamente almacenados, manipulados y transportados; así como aplicados en condiciones normales. En la práctica, las diferencias en los materiales, sustratos y condiciones de la obra en donde se aplicarán los productos Sika son tan particulares que de esta información, de alguna recomendación escrita o de algún asesoramiento técnico, no se puede deducir ninguna garantía respecto a la comercialización o adaptabilidad del producto a una finalidad particular, así como ninguna responsabilidad contractual. Los derechos de propiedad de las terceras partes deben ser respetados.

Todos los pedidos aceptados por Sika Perú S.A. están sujetos a Cláusulas Generales de Contratación para la Venta de Productos de Sika Perú S.A. Los usuarios siempre deben remitirse a la última edición de las Hojas Técnicas de los productos; cuyas copias se entregarán a solicitud del interesado o a las que pueden acceder en Internet a través de nuestra página web [www.sika.com.pe](http://www.sika.com.pe)



# Sika® ViscoCrete®-1

## ADITIVO SUPERPLASTIFICANTE CON RETARDO

Aspecto	· Líquido
Color	· Verde azulado
Densidad	· 1.06 kg/l ± 0.02 kg/l

### DESCRIPCION

Sika® ViscoCrete®-1 es un poderoso superplastificante de tercera generación para concretos y morteros. Cumple con los requerimientos para superplastificantes según las normas SIA 162 (1989) y prEN 934-2.

### CAMPOS DE APLICACIÓN

- Es adecuado para la producción de concreto en obra, así como para el concreto pre-mezclado.
- Se usa en los meses de verano y para concretos sometidos a trayectos largos o espera antes de su utilización.
- Facilita la extrema reducción de agua, tiene excelentes propiedades con los agregados finos, una óptima cohesión y alto comportamiento autocompactante.
- Se usa para los siguientes tipos de concreto:
  - Concreto autocompactante
  - Concreto para climas cálidos y/o sometido a trayectos largos o espera antes de su utilización
  - Concreto de alta reducción de agua (hasta 30%)
  - Concreto de alta resistencia
- La alta reducción de agua y la excelente fluidez tienen una influencia positiva sobre las aplicaciones antes mencionadas.

### VENTAJAS

Sika® ViscoCrete®-1 actúa por diferentes mecanismos. Gracias a la absorción superficial y el efecto de separación espacial sobre las partículas de cemento (paralelos al proceso de hidratación) se obtienen las siguientes propiedades:

- Fuerte comportamiento autocompactante que lo hace adecuado para la producción de concreto autocompactante.
- Extrema reducción de agua (que trae consigo una alta densidad y resistencia).
- Excelente fluidez (reduce en gran medida el esfuerzo de colocación y vibración).
- Mejora la contracción y plasticidad.
- Reduce la carbonatación del concreto.

Sika® ViscoCrete®-1 no contiene cloruros ni otros ingredientes que promuevan la corrosión del acero. Por lo tanto, puede usarse sin restricciones en construcciones de concreto reforzado y pre-tensado.

### DATOS TÉCNICOS

Base	Solución acuosa de policarboxilato modificado
Valor pH	6.5 ± 1.0

### MODO DE EMPLEO

Sika® ViscoCrete®-1 se agrega al agua de amasado o se echa junto con el agua a la mezcladora de concreto. Para un aprovechamiento óptimo de la alta capacidad de reducción de agua, recomendamos un mezclado cuidadoso durante 60 segundos como mínimo.

Para evitar la exudación en el concreto y lograr la consistencia deseada, el agua restante de la mezcla recién se añadirá cuando hayan transcurrido 60 segundos del tiempo de mezclado.

El uso de Sika® ViscoCrete®-1 garantiza un concreto de la más alta calidad. Sin embargo, también en el caso del concreto preparado con Sika® ViscoCrete®-1 debe cumplirse con las normas estándar para la buena producción y colocación de concretos.

El concreto fresco debe ser curado apropiadamente con Antisol®.

## Sika® Fume

### Aditivo para Concreto y Mortero Bajo el Agua

#### Descripción

**General**

Es un aditivo en polvo compuesto por microsilice (Silica Fume) de alta calidad y que acondicionado a la mezcla de concreto o mortero, disminuye el lavado del cemento en el vaciado de la mezcla bajo agua. Sika Fume no contiene cloruros y puede utilizarse en concretos y morteros en conjunto con un superplastificante para obtener la fluidez necesaria para la colocación del concreto.

**Campos de aplicación**

- En el concreto bajo agua en puertos, puentes, presas, reparaciones, rellenos, entre otros.
- En concretos de alta impermeabilidad y durabilidad.
- En concretos de alta resistencia (mayor a 500 kg/cm<sup>2</sup>).
- En concretos bombeados y proyectados.
- En morteros y lechadas de inyección.

**Ventajas**

- Disminuye la pérdida de cemento y elementos finos.
- Aumenta la resistencia mecánica.
- Aumenta la impermeabilidad.
- Aumenta la resistencia química.
- Aumenta la adherencia al acero.
- Permite utilizar mezclas altamente fluidas con alta cohesión.
- Aumenta la cohesión y disminuye la exudación de la mezcla fresca.
- Aumenta la durabilidad frente a agentes agresivos.
- Aumenta la resistencia a abrasión.

#### Datos Básicos

**Aspecto** Polvo

**Color** Gris

**Presentación** Bolsa de 20 kg

**Almacenamiento** Se puede almacenar durante un año en su envase original cerrado en un lugar fresco y bajo techo.

#### Datos Técnicos

**Densidad** 0.3 kg/l

**Blaine (superficie específica)** 180 000 – 200 000 cm<sup>2</sup>/g

**Gravedad específica** 2.2

**Finura (diámetro promedio)** 0.1 – 0.2 μm

**Porcentaje pasando 45 μm** 95 – 100 %

**Partícula** Esférica

**Forma** Amorfa

#### Aplicación

**Consumo**

Puede utilizarse en dosis de aproximadamente 10 % del peso del cemento. Se recomienda realizar ensayos previos para definir el consumo exacto.



<b>Método de aplicación</b>	Se puede mezclar con productos Sikament o ViscoCrete. La dosificación del concreto se realiza de acuerdo a la práctica normal para concreto bajo agua o para la aplicación específica que se requiera. La utilización conjunta de ambos productos asegura las características de cohesión, adherencia y resistencia en el concreto bajo agua. Sika Fume se adiciona a la mezcladora junto con el cemento o la arena. El aditivo Sikament se agrega diluido en el agua de amasado.
<b>Instrucciones de Seguridad</b> <b>Precauciones de manipulación</b>	Durante la manipulación de cualquier producto químico, evite el contacto directo con los ojos, piel y vías respiratorias. Protéjase adecuadamente utilizando guantes de goma natural o sintéticos y anteojos de seguridad. En caso de contacto con los ojos, lavar inmediatamente con abundante agua durante 15 minutos manteniendo los párpados abiertos y consultar a su médico.
<b>Observaciones</b>	La Hoja de Seguridad de este producto se encuentra a disposición del interesado. Agradeceremos solicitarla a nuestro Departamento Comercial, teléfono: 618-6060 o descargarla a través de Internet en nuestra página web: <a href="http://www.sika.com.pe">www.sika.com.pe</a>
<b>Nota Legal</b>	La información y en particular las recomendaciones sobre la aplicación y el uso final de los productos Sika son proporcionadas de buena fe, en base al conocimiento y experiencia actuales en Sika respecto a sus productos, siempre y cuando éstos sean adecuadamente almacenados, manipulados y transportados; así como aplicados en condiciones normales. En la práctica, las diferencias en los materiales, sustratos y condiciones de la obra en donde se aplicarán los productos Sika son tan particulares que de esta información, de alguna recomendación escrita o de algún asesoramiento técnico, no se puede deducir ninguna garantía respecto a la comercialización o adaptabilidad del producto a una finalidad particular, así como ninguna responsabilidad contractual. Los derechos de propiedad de las terceras partes deben ser respetados. Todos los pedidos aceptados por Sika Perú S.A. están sujetos a Cláusulas Generales de Contratación para la Venta de Productos de Sika Perú S.A. Los usuarios siempre deben remitirse a la última edición de las Hojas Técnicas de los productos; cuyas copias se entregarán a solicitud del interesado o a las que pueden acceder en Internet a través de nuestra página web <a href="http://www.sika.com.pe">www.sika.com.pe</a> .

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## ANEXO B

### Cálculo del Costo de la Investigación

#### Costo de Insumos empleados en la investigación

Insumo	Unidad	PU	Total	USD
Cemento	Kg	0.09478	476.10	45.12
Diatomita	Kg	0.25	31.05	7.76
Silica Fume	Kg	0.91	10.35	9.42
Agua	L	0.000839813	146.74	0.12
Arena	Kg	0.00578	954.08	5.51
Piedra 67	Kg	0.00463	845.09	3.91
Polyheed RI	L	0.82	2.59	2.12
Viscocrete	L	2.78	12.50	34.76
<b>Costo total</b>			<b>USD</b>	<b>108.74</b>

#### Costo de los Ensayos en Laboratorio Externo

Tipo	Costo
Químico y Físico	500
<b>Total (USD)</b>	<b>500</b>

#### Costo de los Ensayos Básicos

##### - En Fresco

Ensayo	Por diseño	Diseños	Replicas	Cantidad	Costo Unitario (USD)	Total (USD)
Slump	1	5	2	10	9.00	90.00
Peso Unitario	1	5	2	10	9.00	90.00
Contenido de Aire	1	5	2	10	18.00	180.00
Temperatura	1	5	2	10	2.70	27.00
Fragua	2	5	2	20	31.50	630.00
<b>Total</b>						<b>1,017.00</b>

##### - Endurecido

Ensayo	Por diseño	Diseños	Replicas	Total	Costo Unitario (USD)	Total (USD)
Compresión	8	5	3	120	2.10	252.00
Permeabilidad	2	5	2	20	30.00	600.00
Sulfatos	2	5	2	20	79.20	1,584.00
<b>Total</b>						<b>2,436.00</b>

#### Costos incurridos en el Personal

Personal	Cantidad	Sueldo Mensual (USD)	Meses	Dedicación	Total (USD)
Dirección	1	1,562.50	3	15%	703.13
Asistente	1	625.00	3	45%	843.75
Técnicos (*)	2	312.50	2	50%	625.00
<b>Total</b>					<b>2,171.88</b>

(\*) Costo de los técnicos para realizar las mezclas

#### Resumen

Concepto	USD	%
Insumos	108.74	1.7%
Ensayos Básicos - Fresco	1,017.00	16.3%
Ensayos Básicos - Endurecido	2,436.00	39.1%
Ensayos por Terceros	500.00	8.0%
Personal	2,171.88	34.8%
<b>TOTAL</b>	<b>6,233.61</b>	<b>100.0%</b>

## ANEXO C

### ENSAYOS DE VISCOSIDAD

Los siguientes cuadros muestran los datos obtenidos durante el ensayo de Viscosidad.

#### **DCD-00: Solo Cemento** ( Spindle # 1 para las tres corridas o Loops)

Código Prueba : DCD-00 (1)  
Fecha : 01/03/2006

RPM	Loop 1		Loop 2		Loop 3	
	$\mu$ (cp)	$\zeta$ (Pa)	$\mu$ (cp)	$\zeta$ (Pa)	$\mu$ (cp)	$\zeta$ (Pa)
0.5	1,760	0.039	1,280	0.028	1920	0.042
1	2,000	0.088	1,200	0.053	1440	0.063
2.5	1,470	0.161	864	0.095	1020	0.112
5	1,300	0.284	912	0.200	1100	0.241
10	640	0.368	712	0.312	760	0.333
20	800	0.700	610	0.584	670	0.586
50	908	1.986	570	1.247	581	1.271
100	640	2.800	723	3.163	678	2.966
100	590	2.581	625	2.734	636	2.783
50	476	1.041	502	1.098	480	1.050
20	416	0.364	432	0.378	404	0.354
10	432	0.189	448	0.196	424	0.186
5	528	0.116	544	0.119	544	0.119
2.5	736	0.081	800	0.088	800	0.088
1	1,520	0.067	1,600	0.070	1600	0.070
0.5	3,040	0.067	3,040	0.067	3360	0.074

#### **DCD-05: 5% Diatomita** (Spindle # 1 para las corridas Loop 1 y Loop 2; Spindle # 2 para la corrida Loop 3)

Código Prueba : DCD-05 (2)  
Fecha : 01/03/2006

RPM	Loop 1		Loop 2		Loop 3	
	$\mu$ (cp)	$\zeta$ (Pa)	$\mu$ (cp)	$\zeta$ (Pa)	$\mu$ (cp)	$\zeta$ (Pa)
0.5	35,600	0.779	45,200	0.989	47,400	0.881
1	20,600	0.901	25,800	1.129	27,500	1.023
2.5	10,400	1.138	13,000	1.422	14,600	1.357
5	6,440	1.409	7,880	1.724	9,160	1.703
10	4,220	1.846	4,980	2.179	5,790	2.153
20	2,870	2.511	3,260	2.853	3,930	2.923
50	-	-	-	-	2,420	4.500
100	-	-	-	-	1,890	7.028
100	-	-	-	-	1,680	6.248
50	1,110	2.428	1,200	2.625	1,400	2.603
20	1,370	1.199	1,420	1.243	1,540	1.145
10	2,080	0.910	2,060	0.901	1,950	0.725
5	3,640	0.796	3,500	0.766	3,900	0.725
2.5	6,800	0.744	6,640	0.726	6,920	0.643
1	16,900	0.739	16,400	0.718	16,300	0.606
0.5	35,600	0.779	33,800	0.739	33,200	0.617



**DCD-10: 10% Diatomita** (Spindle # 1 para las corridas Loop 1; Spindle # 2 para la corrida Loop 2 y Loop 3)

Código Prueba : DCD-10 (1)  
Fecha : 01/03/2006

RPM	Loop 1		Loop 2		Loop 3	
	(cp)	$\zeta$ (Pa)	(cp)	$\zeta$ (Pa)	(cp)	$\zeta$ (Pa)
0.5	20,400	0.446	29,400	0.547	31,400	0.584
1	13,000	0.589	17,900	0.668	18,900	0.703
2.5	7,160	0.783	10,000	0.930	10,000	0.930
5	4,800	1.050	7,100	1.320	6,920	1.287
10	3,510	1.536	5,380	2.001	5,250	1.952
20	2,750	2.406	4,280	3.183	4,240	3.154
50	-	-	3,440	6.396	3,400	6.322
100	-	-	2,650	10.596	2,980	11.062
100	-	-	2,480	9.260	2,540	9.446
50	1160	2.538	1,620	3.012	1,630	3.031
20	1160	1.015	1,510	1.123	1,520	1.131
10	1610	0.704	1,690	0.703	1,920	0.714
5	2600	0.589	2,680	0.536	2,880	0.536
2.5	4640	0.506	3,140	0.292	4,860	0.454
1	11000	0.461	10,000	0.372	10,600	0.394
0.5	23400	0.512	20,400	0.379	21,200	0.394

**DCD-15: 15% Diatomita** (Spindle # 2 para las tres corridas)

Código Prueba : DCD-15 (2)  
Fecha : 01/03/2006

RPM	Loop 1		Loop 2		Loop 3	
	(cp)	$\zeta$ (Pa)	(cp)	$\zeta$ (Pa)	(cp)	$\zeta$ (Pa)
0.5	25,600	0.476	35,200	0.655	37,800	0.703
1	15,700	0.584	20,500	0.762	22,100	0.822
2.5	8,440	0.785	10,900	1.013	11,600	1.078
5	5,880	1.093	7,420	1.360	7,860	1.465
10	4,450	1.655	5,440	2.023	5,790	2.153
20	3,760	2.797	4,520	3.362	4,680	3.481
50	3,300	6.136	3,640	6.768	3,640	6.768
100	2,930	10.896	3,130	11.640	2,980	11.062
100	2,520	9.371	2,650	9.655	2,650	9.855
50	1,660	3.087	1,690	3.514	1,860	3.456
20	1,600	1.190	1,790	1.331	1,890	1.406
10	2,050	0.762	2,270	0.844	2,430	0.904
5	3,000	0.558	3,320	0.617	3,640	0.677
2.5	5,120	0.476	5,640	0.524	6,000	0.558
1	11,200	0.417	11,800	0.439	13,100	0.487
0.5	21,800	0.405	24,400	0.454	26,200	0.487

**DCR-10: 10% Microsilice (Spindle # 1 para las tres corridas)**

Código Prueba : DCR-10 (3)

Fecha : 01/03/2006

RPM	Loop 1		Loop 2		Loop 3	
	$\mu$ (cp)		(cp)			$\zeta$ (Pa)
0.5	15,500	0.339	21,600	0.473	28,400	0.621
1	9,280	0.406	12,300	0.538	15,500	0.678
2.5	5,000	0.547	6,320	0.691	7,720	0.844
5	3,280	0.718	4,000	0.875	4,760	1.041
10	2,210	0.967	2,600	1.138	3,010	1.317
20	1,490	1.304	1,760	1.540	1,980	1.733
50	954	2.087	1,100	2.406	1,230	2.691
100	700	3.063	794	3.474	-	3.850
100	638	2.791	728	3.185	798	3.491
50	710	1.553	802	1.754	876	1.916
20	930	0.814	1,030	0.901	1,110	0.971
10	1,350	0.591	1,510	0.661	1,620	0.709
5	2,280	0.499	2,560	0.560	2,760	0.604
2.5	4,240	0.464	4,760	0.521	5,200	0.569
1	10,200	0.446	11,600	0.508	12,600	0.551
0.5	20,800	0.455	23,600	0.516	25,400	0.556

## ANEXO D

### PRINCIPIO DE FUNCIONAMIENTO DEL VISCOSIMETRO BROOKFIELD MODELO HBDV-II

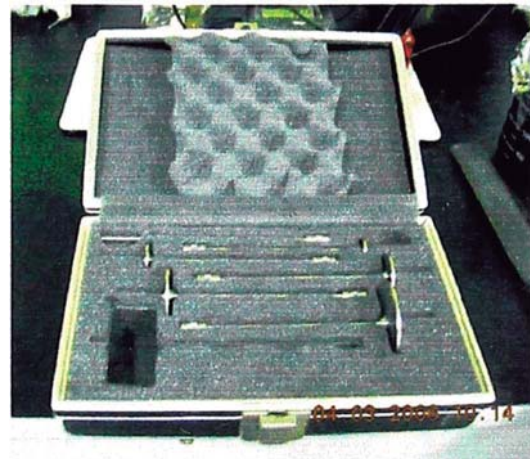
#### Principio de Operación:

- Todos los Viscosímetros Brookfield hacen rotar un elemento de detección en un fluido (Spindle o Aguja) y miden el torque necesario para vencer la resistencia de la viscosidad al movimiento inducido, por la conducción del elemento inmerso, llamado aguja, a través de un resorte de Cobre-Berilio. El grado en el cual el resorte es forzado, el cual es detectado por el transductor rotacional, es proporcional a la viscosidad del fluido. Las mediciones hechas usando la misma aguja a diferentes velocidades son usadas para detectar y evaluar las propiedades reológicas del material de prueba.

Viscosímetro



Spindles (Agujas)



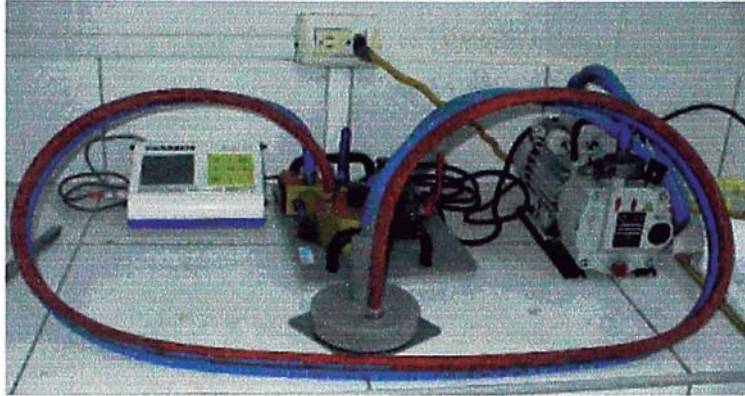
- Los datos de %Torque son convertidos a Shear Stress mediante factores de corrección del equipo.
- El equipo utilizado, no tiene calculado el valor de corrección del RPM a Shear Rate. Por consultas realizadas a los fabricantes, se puede utilizar el RPM en lugar del Shear Rate sólo para ensayos comparativos en ensayos comparativos utilizando el mismo equipo.
- Mide viscosidad y % Torque para un valor de RPM y spindle seleccionado.

El Shear Stress es proporcional al Torque

El Shear Rate es proporcional al RPM

## ANEXO E

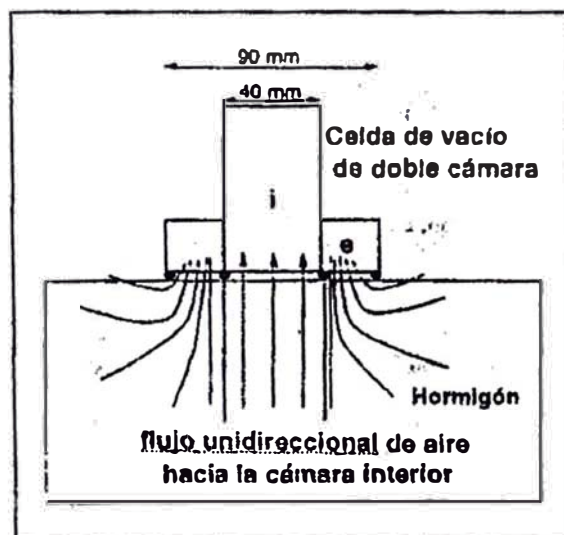
### MEDICIÓN DE LA PERMEABILIDAD UTILIZANDO EL EQUIPO DE TORRENT



Descripción del aparato y de su operación

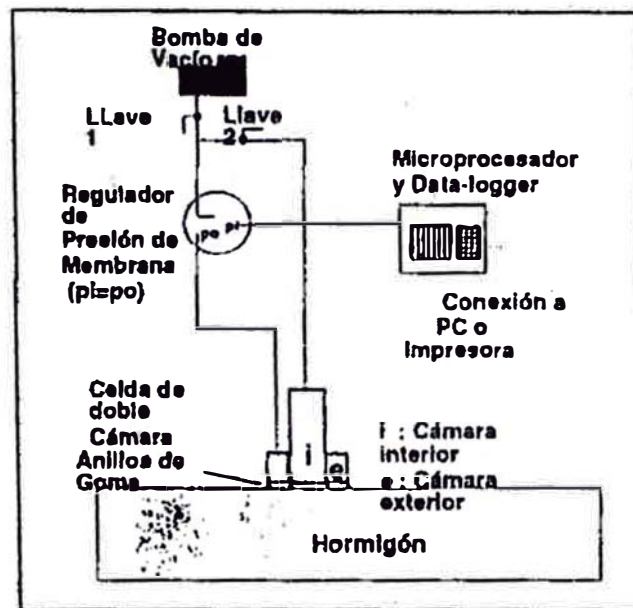
Sus dos características distintivas son:

- a) Una celda con doble cámara; una cámara interna "i" y una cámara externa "e"



- b) Un regulador de presión a membrana, cuya función es mantener a ambas cámaras siempre a la misma presión ( $P_i = P_e$ )

La operación del aparato es como sigue:



Con las llaves 1 y 2 abiertas, se crea vacío en ambas cámaras mediante la bomba. Luego de 1 minuto de crear vacío se cierra la llave 2, momento a partir del cual la bomba solo puede actuar (cuando se lo permita el regulador) sobre la cámara externa, de manera que equilibra, en todo momento, la presión en ambas cámaras. De este modo, todo exceso de aire que ingrese lateralmente en la cámara externa será evacuado. Así se logra que el flujo de aire hacia la cámara central sea básicamente unidireccional y no afectado por el ingreso espurio de aire, sea por un deficiente sellado de la cámara externa o a través de la más permeable piel superficial.

La evolución de la presión  $P_i$  se mide con un sensor de presión comandado por un microprocesador que tiene integrado un cronometro. El microprocesador almacena la información y efectúa los cálculos para mostrar automáticamente, al final del ensayo, el valor del coeficiente de permeabilidad al aire  $kT$  ( $m^2$ ). El fin del ensayo acontece cuando la elevación de la presión en la cámara interna  $P_i$ , alcanza 20 mbar o, en el caso de concreto muy impermeable cuando han transcurrido 12 minutos desde el comienzo del ensayo.

Para concretos secos (superficie que no ha estado expuesta a humedad por tres semanas), la clase del covercrete se determina directamente a partir de  $K_t$  dado por el equipo, ver Cuadro 1.10.

## ANEXO F

### METODO DE CÁLCULO PARA EL PROPORCIONAMIENTO DE LOS MATERIALES (ACI 211)

Asumiendo que se conocen todas las características de los materiales como son el tipo de cemento elegido y sus propiedades, los agregados y sus pesos específicos y pesos unitarios secos, granulometrías, humedades, absorciones y las condiciones particulares de la obra a ejecutar, todos los métodos aplican los siguientes pasos:

1. Establecimiento de la cantidad de agua por m<sup>3</sup> de concreto en función de las condiciones de trabajabilidad, el tamaño máximo de los agregados y ocasionalmente el tipo de cemento.
2. Definición de la relación agua/cemento en peso en base a la resistencia en compresión solicitada o requisitos de durabilidad.
3. Calculo de la cantidad de cemento en peso en función de la relación agua /cemento y la cantidad de agua definidas en (1) y (2):

$$\text{Cemento (kg)} = \frac{\text{Peso del Agua (kg)}}{\text{Relación A/C}}$$

4. Calculo de los volúmenes absolutos del agua y el cemento:

$$\text{Vol. Abs. Cemento (m}^3\text{)} = \frac{\text{Peso del Cemento (kg)}}{\text{Peso Espec. Cemento (Kg/m}^3\text{)}}$$

$$\text{Vol. Abs. Agua (m}^3\text{)} = \frac{\text{Peso del Agua (kg)}}{\text{Peso Espec. Agua (Kg/m}^3\text{)}}$$

5. Estimación del porcentaje de aire por m<sup>3</sup> y el volumen absoluto que atraparé el concreto en función de las características granulométricas de los agregados.
6. Obtención del volumen absoluto que ocuparan los agregados, restando de 1m<sup>3</sup> los volúmenes hallados de cemento, agua y aire.

$$\text{Volumen Absoluto de Agregados} = 1 \text{ m}^3 - \text{Vol. Abs. Cemento (m}^3\text{)} - \text{Vol. Abs. Agua (m}^3\text{)} - \text{Vol. Aire (m}^3\text{)}$$

7. Definición de la proporción en volumen absoluto en que intervendrán el agregado grueso y el fino en la mezcla (Por el método preferido)

$K$  = porcentaje en que interviene la piedra

$K-1$  = porcentaje en que interviene la arena

8. Distribución del volumen obtenido en (6) en la proporción definida en (7)

$\text{Vol. Abs. Piedra (m}^3\text{)} = K \times \text{Vol. Abs. Agregados (m}^3\text{)}$

$\text{Vol. Abs. Arena (m}^3\text{)} = (K-1) \times \text{Vol. Abs. Agregados (m}^3\text{)}$

9. Calculo de los pesos que corresponden a los volúmenes de agregados obtenidos en (8) utilizando los pesos específicos secos:

$\text{Peso Piedra (Kg.)} = \text{Vol. Abs. Piedra (m}^3\text{)} \times \text{Peso Espec. Piedra (Kg/m}^3\text{)}$

$\text{Peso Arena (Kg.)} = \text{Vol. Abs. Arena (m}^3\text{)} \times \text{Peso Espec. Arena (Kg/m}^3\text{)}$

10. Corrección por humedad y absorción del diseño:

- a)  $\text{Peso de la Piedra Húmeda (Kg.)} = \text{Peso seco Piedra (Kg.)} \times (1 + \text{Humedad Piedra en valor absoluto})$
- b)  $\text{Peso de la Arena Húmeda (Kg.)} = \text{Peso seco Arena (Kg.)} \times (1 + \text{Humedad Arena en valor absoluto})$
- c)  $\text{Balance Agua en la Piedra (Puede ser positivo o negativo)} = \text{Humedad Piedra} - \text{Absorción Piedra (Valores absolutos)}$
- d)  $\text{Balance Agua en la Arena (Puede ser positivo o negativo)} = \text{Humedad Arena} - \text{Absorción Arena (Valores absolutos)}$
- e)  $\text{Contribución de Agua por la Piedra (Kg.) (Puede ser positiva o negativa, es decir, añadir o quitar agua)} = \text{Balance Agua en la Piedra} \times \text{Peso Piedra Húmeda (Kg.)}$
- f)  $\text{Contribución de Agua por la Arena (Kg.) (Puede ser positiva o negativa, es decir, añadir o quitar agua)} = \text{Balance Agua en la Arena} \times \text{Peso Arena Húmeda (Kg.)}$
- g)  $\text{Agua Final en el diseño} = \text{Agua (Kg.)} - \text{Contrib. Agua Piedra (Kg.)} - \text{Contrib. Agua Arena (Kg.)}$



11. Diseño final:

Agua Final (Kg.), Peso Húmedo Piedra (Kg.), Peso Húmedo Arena (Kg.)  
Peso Cemento (Kg.)

12. Elaboración del diseño final en laboratorio y a escala de obra para la verificación práctica de sus propiedades con objeto de confirmarlo o corregirlo.

Como se aprecia, los diseños se calculan inicialmente en base a los pesos secos de los agregados para luego corregirlos al establecer su condición de humedad real, sin embargo la experiencia demuestra que esto es sólo una aproximación que hay que corregir en forma práctica al ejecutar las mezclas de prueba.

Hay que tener presente al usar aditivos (que normalmente son líquidos), el incluir su intervención cuantificándolos como parte del agua de mezcla en el cálculo de la relación Agua/Cemento.

## ANEXO G

### GLOSARIO DE TÉRMINOS TÉCNICOS

**Absorción:** proceso por el cual un líquido es atraído hacia un sólido poroso y tiende a llenar los poros permeables del mismo; también el aumento de masa de un sólido poroso que se produce como resultado de la penetración de un líquido en sus poros permeables.

**Agregado:** material granular, tal como la arena, grava, piedra molida, hormigón de cemento hidráulico molido o escoria de alto horno molida, empleado junto con un medio de cemento hidráulico para elaborar hormigón o mortero.

**Agregado grueso:** agregado predominantemente retenido sobre un tamiz de 4,75 mm (No. 4), o aquella porción retenida sobre un tamiz de 4,75 mm (No. 4).

**Agregado fino:** agregado que atraviesa un tamiz de 9,5 mm (3/8 in.) y atraviesa casi totalmente un tamiz de 4,75 mm (No. 4) mientras que es predominantemente retenido sobre el tamiz de 75  $\mu\text{m}$  (No. 200); o aquella porción que atraviesa el tamiz de 4,75 mm (No. 4) y es predominantemente retenida sobre el tamiz de 75  $\mu\text{m}$  (No. 200).

**Agregado de peso normal:** agregado que no es pesado ni liviano

**Aire incorporado:** burbujas de aire microscópicas incorporadas intencionalmente al mortero u hormigón durante el mezclado, generalmente utilizando un agente tensoactivo; típicamente de entre 10 y 1000  $\mu\text{m}$  de diámetro y de forma esférica o casi esférica.

**Alita:** nombre empleado por Tornebohm (1897) para identificar el silicato tricálcico con pequeñas cantidades de  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  y otros óxidos; es un componente principal del Clínter de cemento Pórtland.

Aluminato Tricálcico: compuesto cuya composición química es  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  y se abrevia  $\text{C}_3\text{A}$ .

**Apisonamiento:** operación de compactar hormigón fresco golpeando o penetrando repetidamente con un pisón.

**Arena de Ottawa:** Arena normalizada correctamente graduada entre los tamices U.S. Standard 600  $\mu\text{m}$  (No. 30) y 150  $\mu\text{m}$  (No. 100) que se utiliza para ensayar los cementos. Arena de sílice que se produce procesando el material obtenido

de la explotación los depósitos abiertos de cuarcita próximos a Ottawa, Illinois; está compuesta fundamentalmente por granos de cuarzo casi puro naturalmente redondeados; se emplea en morteros que se usan para ensayar cementos hidráulicos.

**Ataque por sulfatos:** reacción física y/o química entre los sulfatos generalmente presentes en el suelo o el agua del suelo y un mortero u hormigón; la reacción química es fundamentalmente con los aluminatos de calcio hidratados en la matriz de la pasta cementicia, y a menudo provoca deterioros.

**Belita:** nombre empleado por Tornebohm (1897) para identificar una forma del componente del Clínter de cemento Pórtland que en su forma pura ahora se conoce como silicato dicálcio ( $2\text{CaO}\cdot\text{SiO}_2$ ).

**Calor de hidratación:** calor que se desprende por las reacciones químicas con el agua, tal como el calor que se desprende durante el fraguado y endurecimiento del cemento Pórtland, o la diferencia entre el calor de solución del cemento seco y el de un cemento parcialmente hidratado.

**Cámara de curado:** sala o habitación en la cual la atmósfera se mantiene a una temperatura seleccionada (generalmente  $23,0 \pm 1,7$  °C ó  $73,4 \pm 3,0$  °F) y una humedad relativa de al menos 95 por ciento; se usa para curar y almacenar probetas de ensayo; las instalaciones deben ser capaces de mantener continuamente humedad libre sobre el exterior de las probetas de ensayo.

**Camión mezclador (mixer):** mezcladora de hormigón que se puede montar sobre el chasis de un camión y que es capaz de mezclar el hormigón mientras el camión circula.

**Carbonatación:** reacción entre dióxido de carbono y un hidróxido o un óxido para formar un carbonato, especialmente en una pasta de cemento, mortero u hormigón; reacción con compuestos de calcio para producir carbonato de calcio.

**Clínter:** producto de un horno parcialmente fundido que se muele para fabricar cemento; también otros materiales vitrificados o calcinados.

**Compactación:** proceso de inducir una disposición más próxima de las partículas sólidas del hormigón o mortero fresco durante su colocación mediante la reducción de los vacíos; generalmente se lleva a cabo por vibrado, centrifugado, varillado, apisonamiento, o alguna combinación de estos procesos; el término también es aplicable a la manipulación similar de otras mezclas cementicias, suelos, agregados o similares.

**Contenido de aire:** volumen de vacíos de aire en la pasta cementicia, mortero u hormigón, excluyendo el espacio de los poros en las partículas de los agregados; generalmente se expresa como un porcentaje del volumen total de la pasta, mortero u hormigón.

**Concreto:** llamado también hormigón, material compuesto que consiste esencialmente en un medio ligante dentro del cual hay partículas o fragmentos de agregado, generalmente una combinación de agregado fino y agregado grueso; en el hormigón de cemento Pórtland el ligante es una mezcla de cemento Pórtland y agua (pasta).

**Contracción:** disminución de longitud o volumen.

**Curado al vapor:** curado de hormigón, mortero o pasta cementicia en vapor de agua a presión atmosférica o superior y temperaturas entre alrededor de 100 y alrededor de 420 °F (40 y 215 °C).

**Curva de gradación granulométrica:** representación gráfica de las proporciones de los diferentes tamaños de partículas de un material granular; se obtiene graficando los porcentajes acumulados o individuales del material que atraviesa determinados tamices cuyas aberturas forman una serie dada.

**Ensayo de compresión:** ensayo que se realiza sobre una probeta de mortero u hormigón para determinar su resistencia a la compresión; a menos que se especifique lo contrario, en Estados Unidos los ensayos de compresión de los morteros se realizan sobre probetas cúbicas de 2 in. (50 mm) de lado y los ensayos de compresión de los hormigones en probetas cilíndricas de 6 in. (152 mm) de diámetro y 12 in. (305 mm) de altura.

**Escoria de alto horno:** producto no metálico, compuesto fundamentalmente por silicatos y alumino-silicatos de calcio y otras bases, que se produce simultáneamente con la fusión del mineral de hierro en un alto horno.

**Espécimen:** pieza o porción de una muestra que se usa para un ensayo.

**Fineza Blaine:** fineza de los materiales en polvo tales como los cementos y puzolanas; se expresa como superficie específica generalmente en metros cuadrados por kilogramo y se determina por medio del aparato Blaine.

**Fluorescencia de rayos X:** radiación secundaria característica emitida por un elemento como resultado de su excitación por rayos X que se usa para obtener el análisis químico de una muestra.

**Fraguado final:** grado de rigidización de una mezcla de cemento y agua mayor que el correspondiente al fraguado inicial, generalmente expresado como un valor empírico que indica el tiempo en horas y minutos necesario para que una pasta cementicia se rigidice lo suficiente como para resistir la penetración de una aguja de ensayo hasta un punto establecido; también se aplica a las mezclas de hormigón y mortero, usando procedimientos de ensayo adecuados.

- tiempo de fraguado final – tiempo necesario para que una pasta cementicia, mortero u hormigón recién mezclado alcance el fraguado final.

**Fraguado inicial:** grado de rigidización de una mezcla de cemento y agua menor que el correspondiente al fraguado final, generalmente expresado como un valor empírico que indica el tiempo en horas y minutos necesario para que una pasta cementicia se rigidice lo suficiente como para resistir la penetración de una aguja de ensayo hasta un grado establecido; también es aplicable al hormigón o mortero, usando procedimientos de ensayo adecuados.

- tiempo de fraguado inicial – tiempo que se requiere para que una pasta cementicia, mortero u hormigón fresco adquiera el fraguado inicial.

**Fineza:** medida del tamaño de las partículas.

**Granulometría:** distribución de las partículas de un material granular en diferentes tamaños; generalmente se expresa en términos de porcentajes acumulados mayores o menores que cada uno de una serie de tamaños (aberturas de tamiz) o porcentajes entre ciertos rangos de tamaños (aberturas de tamiz).

**Gravedad específica:** relación entre la masa de un volumen de un material a una temperatura dada y la masa del mismo volumen de agua destilada a una temperatura dada.

**Gravedad específica aparente:** relación entre la masa de un volumen de la porción impermeable de un material a una temperatura dada y la masa de un volumen igual de agua destilada a una temperatura dada.

**Gravedad específica de la masa:** relación entre la masa de un volumen de un material (incluyendo los vacíos permeables e impermeables del material, pero excluyendo los vacíos entre partículas del material) a una temperatura dada y la masa de un volumen igual de agua destilada a una temperatura dada;

**Gravedad específica de la masa saturada y superficialmente seca (Bulk):** relación entre la masa de un volumen de un material, incluyendo la masa de

agua dentro de los vacíos (pero excluyendo los vacíos entre partículas) a una temperatura dada y la masa de un volumen igual de agua destilada a una temperatura dada.

**Hidratación:** formación de un compuesto por la combinación de agua con alguna otra sustancia; en el concreto, reacción química entre el cemento hidráulico y el agua.

**Mesa de flujo:** dispositivo plano, circular y con movimiento que se usa para realizar ensayos de fluencia a fin de determinar la consistencia de las pastas cementicias, morteros u hormigones.

**Módulo de fineza (MF):** factor que se obtiene sumando los porcentajes totales de material presente en la muestra mayores que cada uno de los siguientes tamices (porcentajes retenidos acumulados), y dividiendo la sumatoria por 100: 150  $\mu\text{m}$  (No. 100); 300  $\mu\text{m}$  (No. 50); 600  $\mu\text{m}$  (No. 30); 1,18 mm (No. 16); 2,36 mm (No. 8); 4,75 mm (No. 4); 9,5 mm (3/8 in.); 19,0 mm (3/4 in.); 37,5 mm (1½ in.); 75 mm (3 in.); 150 mm (6 in.).

**Mortero:** mezcla de pasta cementicia y agregado fino; en el hormigón fresco, material que ocupa los intersticios entre las partículas de agregado grueso; en las construcciones de mampostería el mortero puede contener cemento de albañilería o cemento hidráulico y cal (y posiblemente otros aditivos) para lograr mayor plasticidad y trabajabilidad que las que se obtienen con el mortero estándar de cemento hidráulico.

**Pasta:** Mezcla de cemento y agua.

**Pérdida por ignición:** pérdida porcentual de una masa inflamada a una temperatura especificada (generalmente 900-1000 °C) hasta alcanzar un peso constante.

**Pisón:** implemento que se usa para compactar el hormigón o mortero en moldes o encofrados;

**Relación agua-cemento (a/c):** relación entre la cantidad de agua, excluyendo solamente aquella absorbida por los agregados, y la cantidad de cemento en un mortero, concreto o pasta cementicia; preferentemente expresada en forma decimal y abreviada.

**Relación agua-material cementicio:** relación entre la cantidad de agua, excluyendo solamente aquella absorbida por los agregados, y la cantidad de material cementicio en un mortero o concreto.

**Resistencia a la abrasión:** capacidad de una superficie de resistir el desgaste por frotación y fricción.

**Silicato Tricálcico:** compuesto cuya composición química es  $3\text{CaO}\cdot\text{SiO}_2$  y se abrevia  $\text{C}_3\text{S}$ ; una de sus formas impuras es un componente principal del cemento Pórtland.

**Superficie específica:** área superficial de las partículas o de los vacíos de aire contenidos en una masa unitaria o volumen unitario de un material; en el caso de los vacíos de aire del hormigón endurecido, área superficial del volumen de vacíos de aire expresada como pulgadas cuadradas por pulgadas cúbicas o como milímetros cuadrados por milímetros cúbicos.

## ANEXO H

### **Métodos de Ensayo Normalizados por la ASTM (American Society for Testing and Materials) utilizados**

ASTM C39/C39M-01: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens

ASTM C192/192M: Practice for Making and Curing Concrete Test Specimens in the Laboratory

ASTM C138: Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete

ASTM C143: Standard Test Method for Slump of Hydraulic-Cement Concrete

ASTM C150: Standard Specification for Portland Cement

ASTM C231: Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method

ASTM C311: Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete

ASTM C403/C403M-99: Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance

ASTM C490: Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete.

ASTM C617-98: Standard Practice for Capping Cylindrical Concrete Specimens

ASTM C618: Standard Specification for fly-ash and raw or calcined Natural Pozzolan for use as a Mineral Admixture in Portland Cement Concrete.

ASTM C1012: Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution

ASTM C1064/C1064M-01: Standard Test Method for Temperature of Freshly Mixed Portland Cement Concrete

ASTM C1240: Standard Specification for Use of Silica Fume as a Mineral Admixture Cement Concrete, Mortar, and Grout





## Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens<sup>1</sup>

This standard is issued under the fixed designation C 39/C 39M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope \*

1.1 This test method covers determination of compressive strength of cylindrical concrete specimens such as molded cylinders and drilled cores. It is limited to concrete having a unit weight in excess of 50 lb/ft<sup>3</sup> [800 kg/m<sup>3</sup>].

1.2 The values stated in either inch-pound or SI units are to be regarded separately as standard. The SI units are shown in brackets. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The text of this standard references notes which provide explanatory material. These notes shall not be considered as requirements of the standard.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 31 Practice for Making and Curing Concrete Test Specimens in the Field<sup>2</sup>

C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete<sup>2</sup>

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory<sup>2</sup>

C 617 Practice for Capping Cylindrical Concrete Specimens<sup>2</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

C 873 Test Method for Compressive Strength of Concrete Cylinders Cast in Place in Cylindrical Molds<sup>2</sup>

C 1077 Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation<sup>2</sup>

C 1231 Practice for Use of Unbonded Caps in Determina-

tion of Compressive Strength of Hardened Concrete Cylinders<sup>2</sup>

E 4 Practices for Force Verification of Testing Machines<sup>3</sup>

E 74 Practice for Calibration of Force-Measuring Instruments for Verifying the Load Indication of Testing Machines<sup>3</sup>

Manual of Aggregate and Concrete Testing<sup>2</sup>

2.2 *American Concrete Institute:*

CP-16 Concrete Laboratory Testing Technician, Grade I<sup>4</sup>

### 3. Summary of Test Method

3.1 This test method consists of applying a compressive axial load to molded cylinders or cores at a rate which is within a prescribed range until failure occurs. The compressive strength of the specimen is calculated by dividing the maximum load attained during the test by the cross-sectional area of the specimen.

### 4. Significance and Use

4.1 Care must be exercised in the interpretation of the significance of compressive strength determinations by this test method since strength is not a fundamental or intrinsic property of concrete made from given materials. Values obtained will depend on the size and shape of the specimen, batching, mixing procedures, the methods of sampling, molding, and fabrication and the age, temperature, and moisture conditions during curing.

4.2 This test method is used to determine compressive strength of cylindrical specimens prepared and cured in accordance with Practices C 31, C 192, C 617 and C 1231 and Test Methods C 42 and C 873.

4.3 The results of this test method are used as a basis for quality control of concrete proportioning, mixing, and placing operations; determination of compliance with specifications; control for evaluating effectiveness of admixtures and similar uses.

4.4 The individual who tests concrete cylinders for acceptance testing shall have demonstrated a knowledge and ability to perform the test procedure equivalent to the minimum guidelines for certification of Concrete Laboratory Technician, Level I, in accordance with ACI CP-16.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.61 on Testing Concrete for Strength.

Current edition approved Feb. 10, 2001. Published March 2001. Originally published as C 39 – 21 T. Last previous edition C 39 – 99.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.01.

<sup>4</sup> Available from American Concrete Institute, P.O. Box 9094, Farmington Hills, MI 48333-9094.

\*A Summary of Changes section appears at the end of this standard.

NOTE 1—The testing laboratory performing this test method should be evaluated in accordance with Practice C 1077.

$$E_p = 100(A - B)/B$$

5. Apparatus

5.1 *Testing Machine*—The testing machine shall be of a type having sufficient capacity and capable of providing the rates of loading prescribed in 7.5.

5.1.1 Verification of calibration of the testing machines in accordance with Practices E 4 is required under the following conditions:

5.1.1.1 After an elapsed interval since the previous verification of 18 months maximum, but preferably after an interval of 12 months,

5.1.1.2 On original installation or relocation of the machine,

5.1.1.3 Immediately after making repairs or adjustments that affect the operation of the force applying system of the machine or the values displayed on the load indicating system, except for zero adjustments that compensate for the mass of bearing blocks, or specimen, or both, or

5.1.1.4 Whenever there is reason to doubt the accuracy of the results, without regard to the time interval since the last verification.

5.1.2 *Design*—The design of the machine must include the following features:

5.1.2.1 The machine must be power operated and must apply the load continuously rather than intermittently, and without shock. If it has only one loading rate (meeting the requirements of 7.5), it must be provided with a supplemental means for loading at a rate suitable for verification. This supplemental means of loading may be power or hand operated.

NOTE 2—High-strength concrete cylinders rupture more intensely than normal strength cylinders. As a safety precaution, it is recommended that the testing machines should be equipped with protective fragment guards.

5.1.2.2 The space provided for test specimens shall be large enough to accommodate, in a readable position, an elastic calibration device which is of sufficient capacity to cover the potential loading range of the testing machine and which complies with the requirements of Practice E 74.

NOTE 3—The types of elastic calibration devices most generally available and most commonly used for this purpose are the circular proving ring or load cell.

5.1.3 *Accuracy*—The accuracy of the testing machine shall be in accordance with the following provisions:

5.1.3.1 The percentage of error for the loads within the proposed range of use of the testing machine shall not exceed  $\pm 1.0\%$  of the indicated load.

5.1.3.2 The accuracy of the testing machine shall be verified by applying five test loads in four approximately equal increments in ascending order. The difference between any two successive test loads shall not exceed one third of the difference between the maximum and minimum test loads.

5.1.3.3 The test load as indicated by the testing machine and the applied load computed from the readings of the verification device shall be recorded at each test point. Calculate the error,  $E$ , and the percentage of error,  $E_p$ , for each point from these data as follows:

$$E = A - B \tag{1}$$

where:

$A$  = load, lbf [kN] indicated by the machine being verified, and

$B$  = applied load, lbf [kN] as determined by the calibrating device.

5.1.3.4 The report on the verification of a testing machine shall state within what loading range it was found to conform to specification requirements rather than reporting a blanket acceptance or rejection. In no case shall the loading range be stated as including loads below the value which is 100 times the smallest change of load estimable on the load-indicating mechanism of the testing machine or loads within that portion of the range below 10 % of the maximum range capacity.

5.1.3.5 In no case shall the loading range be stated as including loads outside the range of loads applied during the verification test.

5.1.3.6 The indicated load of a testing machine shall not be corrected either by calculation or by the use of a calibration diagram to obtain values within the required permissible variation.

5.2 The testing machine shall be equipped with two steel bearing blocks with hardened faces (Note 4), one of which is a spherically seated block that will bear on the upper surface of the specimen, and the other a solid block on which the specimen shall rest. Bearing faces of the blocks shall have a minimum dimension at least 3 % greater than the diameter of the specimen to be tested. Except for the concentric circles described below, the bearing faces shall not depart from a plane by more than 0.001 in. [0.02 mm] in any 6 in. [150 mm] of blocks 6 in. [150 mm] in diameter or larger, or by more than 0.001 in. [0.02 mm] in the diameter of any smaller block; and new blocks shall be manufactured within one half of this tolerance. When the diameter of the bearing face of the spherically seated block exceeds the diameter of the specimen by more than 0.5 in. [13 mm], concentric circles not more than 0.03 in. [0.8 mm] deep and not more than 0.04 in. [1 mm] wide shall be inscribed to facilitate proper centering.

NOTE 4—It is desirable that the bearing faces of blocks used for compression testing of concrete have a Rockwell hardness of not less than 55 HRC.

5.2.1 Bottom bearing blocks shall conform to the following requirements:

5.2.1.1 The bottom bearing block is specified for the purpose of providing a readily machinable surface for maintenance of the specified surface conditions (Note 5). The top and bottom surfaces shall be parallel to each other. If the testing machine is so designed that the platen itself is readily maintained in the specified surface condition, a bottom block is not required. Its least horizontal dimension shall be at least 3 % greater than the diameter of the specimen to be tested. Concentric circles as described in 5.2 are optional on the bottom block.

NOTE 5—The block may be fastened to the platen of the testing machine.

5.2.1.2 Final centering must be made with reference to the upper spherical block. When the lower bearing block is used to

assist in centering the specimen, the center of the concentric rings, when provided, or the center of the block itself must be directly below the center of the spherical head. Provision shall be made on the platen of the machine to assure such a position.

5.2.1.3 The bottom bearing block shall be at least 1 in. [25 mm] thick when new, and at least 0.9 in. [22.5 mm] thick after any resurfacing operations.

5.2.2 The spherically seated bearing block shall conform to the following requirements:

5.2.2.1 The maximum diameter of the bearing face of the suspended spherically seated block shall not exceed the values given below:

Diameter of Test Specimens, in. [mm]	Maximum Diameter of Bearing Face, in. [mm]
2 [50]	4 [105]
3 [75]	5 [130]
4 [100]	6.5 [165]
6 [150]	10 [255]
8 [200]	11 [280]

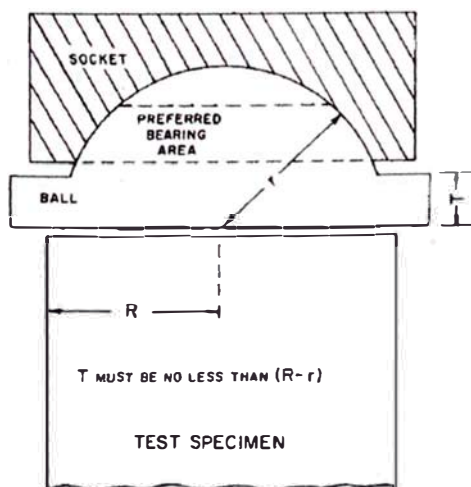
NOTE 6—Square bearing faces are permissible, provided the diameter of the largest possible inscribed circle does not exceed the above diameter.

5.2.2.2 The center of the sphere shall coincide with the surface of the bearing face within a tolerance of  $\pm 5\%$  of the radius of the sphere. The diameter of the sphere shall be at least 75% of the diameter of the specimen to be tested.

5.2.2.3 The ball and the socket must be so designed by the manufacturer that the steel in the contact area does not permanently deform under repeated use, with loads up to 12 000 psi [85 MPa] on the test specimen.

NOTE 7—The preferred contact area is in the form of a ring (described as preferred "bearing" area) as shown on Fig. 1.

5.2.2.4 The curved surfaces of the socket and of the spherical portion shall be kept clean and shall be lubricated with a petroleum-type oil such as conventional motor oil, not with a pressure type grease. After contacting the specimen and application of small initial load, further tilting of the spherically seated block is not intended and is undesirable.



NOTE 1—Provision shall be made for holding the ball in the socket and for holding the entire unit in the testing machine.

FIG. 1 Schematic Sketch of a Typical Spherical Bearing Block

5.2.2.5 If the radius of the sphere is smaller than the radius of the largest specimen to be tested, the portion of the bearing face extending beyond the sphere shall have a thickness not less than the difference between the radius of the sphere and radius of the specimen. The least dimension of the bearing face shall be at least as great as the diameter of the sphere (see Fig. 1).

5.2.2.6 The movable portion of the bearing block shall be held closely in the spherical seat, but the design shall be such that the bearing face can be rotated freely and tilted at least  $4^\circ$  in any direction.

5.3 Load Indication:

5.3.1 If the load of a compression machine used in concrete testing is registered on a dial, the dial shall be provided with a graduated scale that is readable to at least the nearest 0.1% of the full scale load (Note 8). The dial shall be readable within 1% of the indicated load at any given load level within the loading range. In no case shall the loading range of a dial be considered to include loads below the value that is 100 times the smallest change of load that can be read on the scale. The scale shall be provided with a graduation line equal to zero and so numbered. The dial pointer shall be of sufficient length to reach the graduation marks; the width of the end of the pointer shall not exceed the clear distance between the smallest graduations. Each dial shall be equipped with a zero adjustment located outside the dialcase and easily accessible from the front of the machine while observing the zero mark and dial pointer. Each dial shall be equipped with a suitable device that at all times until reset, will indicate to within 1% accuracy the maximum load applied to the specimen.

NOTE 8—Readability is considered to be 0.02 in. [0.5 mm] along the arc described by the end of the pointer. Also, one half of a scale interval is readable with reasonable certainty when the spacing on the load indicating mechanism is between 0.04 in. [1 mm] and 0.06 in. [2 mm]. When the spacing is between 0.06 and 0.12 in. [2 and 3 mm], one third of a scale interval is readable with reasonable certainty. When the spacing is 0.12 in. [3 mm] or more, one fourth of a scale interval is readable with reasonable certainty.

5.3.2 If the testing machine load is indicated in digital form, the numerical display must be large enough to be easily read. The numerical increment must be equal to or less than 0.10% of the full scale load of a given loading range. In no case shall the verified loading range include loads less than the minimum numerical increment multiplied by 100. The accuracy of the indicated load must be within 1.0% for any value displayed within the verified loading range. Provision must be made for adjusting to indicate true zero at zero load. There shall be provided a maximum load indicator that at all times until reset will indicate within 1% system accuracy the maximum load applied to the specimen.

6. Specimens

6.1 Specimens shall not be tested if any individual diameter of a cylinder differs from any other diameter of the same cylinder by more than 2%.

NOTE 9—This may occur when single use molds are damaged or deformed during shipment, when flexible single use molds are deformed during molding or when a core drill deflects or shifts during drilling.

6.2 Neither end of compressive test specimens when tested

## C 39/C 39M

shall depart from perpendicularity to the axis by more than 0.5° (approximately equivalent to 0.12 in. [3 in. 300 mm]). The ends of compression test specimens that are not plane within 0.002 in. [0.050 mm] shall be sawed or ground to meet that tolerance, or capped in accordance with either Practice C 617 or Practice C 1231. The diameter used for calculating the cross-sectional area of the test specimen shall be determined to the nearest 0.01 in. [0.25 mm] by averaging two diameters measured at right angles to each other at about midheight of the specimen.

6.3 The number of individual cylinders measured for determination of average diameter is not prohibited from being reduced to one for each ten specimens or three specimens per day, whichever is greater, if all cylinders are known to have been made from a single lot of reusable or single-use molds which consistently produce specimens with average diameters within a range of 0.02 in. [0.5 mm]. When the average diameters do not fall within the range of 0.02 in. [0.5 mm] or when the cylinders are not made from a single lot of molds, each cylinder tested must be measured and the value used in calculation of the unit compressive strength of that specimen. When the diameters are measured at the reduced frequency, the cross-sectional areas of all cylinders tested on that day shall be computed from the average of the diameters of the three or more cylinders representing the group tested that day.

6.4 The length shall be measured to the nearest 0.05  $D$  when the length to diameter ratio is less than 1.8, or more than 2.2, or when the volume of the cylinder is determined from measured dimensions.

### 7. Procedure

7.1 Compression tests of moist-cured specimens shall be made as soon as practicable after removal from moist storage.

7.2 Test specimens shall be kept moist by any convenient method during the period between removal from moist storage and testing. They shall be tested in the moist condition.

7.3 All test specimens for a given test age shall be broken within the permissible time tolerances prescribed as follows:

Test Age	Permissible Tolerance
24 h	± 0.5 h or 2.1 %
3 days	2 h or 2.8 %
7 days	6 h or 3.6 %
28 days	20 h or 3.0 %
90 days	2 days 2.2 %

7.4 *Placing the Specimen*—Place the plain (lower) bearing block, with its hardened face up, on the table or platen of the testing machine directly under the spherically seated (upper) bearing block. Wipe clean the bearing faces of the upper and lower bearing blocks and of the test specimen and place the test specimen on the lower bearing block. Carefully align the axis of the specimen with the center of thrust of the spherically seated block.

7.4.1 *Zero Verification and Block Seating*—Prior to testing the specimen, verify that the load indicator is set to zero. In cases where the indicator is not properly set to zero, adjust the indicator (Note 10). As the spherically seated block is brought to bear on the specimen, rotate its movable portion gently by hand so that uniform seating is obtained.

NOTE 10—The technique used to verify and adjust load indicator to zero will vary depending on the machine manufacturer. Consult your owner's manual or compression machine calibrator for the proper technique.

7.5 *Rate of Loading*—Apply the load continuously and without shock.

7.5.1 For testing machines of the screw type, the moving head shall travel at a rate of approximately 0.05 in. [1 mm]/min when the machine is running idle. For hydraulically operated machines, the load shall be applied at a rate of movement (platen to crosshead measurement) corresponding to a loading rate on the specimen within the range of 20 to 50 psi/s [0.15 to 0.35 MPa/s]. The designated rate of movement shall be maintained at least during the latter half of the anticipated loading phase of the testing cycle.

7.5.2 During the application of the first half of the anticipated loading phase a higher rate of loading shall be allowed.

7.5.3 Make no adjustment in the rate of movement of the platen at any time while a specimen is yielding rapidly immediately before failure.

7.6 Apply the load until the specimen fails, and record the maximum load carried by the specimen during the test. Note the type of failure and the appearance of the concrete.

### 8. Calculation

8.1 Calculate the compressive strength of the specimen by dividing the maximum load carried by the specimen during the test by the average cross-sectional area determined as described in Section 6 and express the result to the nearest 10 psi [0.1 MPa].

8.2 If the specimen length to diameter ratio is less than 1.8, correct the result obtained in 8.1 by multiplying by the appropriate correction factor shown in the following table:

L/D:	1.75	1.50	1.25	1.00
Factor:	0.98	0.96	0.93	0.87 (Note 11)

NOTE 11—These correction factors apply to lightweight concrete weighing between 100 and 120 lb/ft<sup>3</sup> [1600 and 1920 kg/m<sup>3</sup>] and to normal weight concrete. They are applicable to concrete dry or soaked at the time of loading. Values not given in the table shall be determined by interpolation. The correction factors are applicable for nominal concrete strengths from 2000 to 6000 psi [15 to 45 MPa].

### 9. Report

9.1 Report the following information:

9.1.1 Identification number,

9.1.2 Diameter (and length, if outside the range of 1.8 $D$  to 2.2 $D$ ), in inches [millimetres],

9.1.3 Cross-sectional area, in square inches [square millimetres],

9.1.4 Maximum load, in pounds-force [kilonewtons],

9.1.5 Compressive strength calculated to the nearest 10 psi [0.1 MPa],

9.1.6 Type of fracture, if other than the usual cone (see Fig. 2),

9.1.7 Defects in either specimen or caps, and,

9.1.8 Age of specimen.

### 10. Precision and Bias

10.1 *Precision*—The single operator precision of tests of individual 6 by 12 in. [150 by 300 mm] cylinders made from

ASTM C 39/C 39M

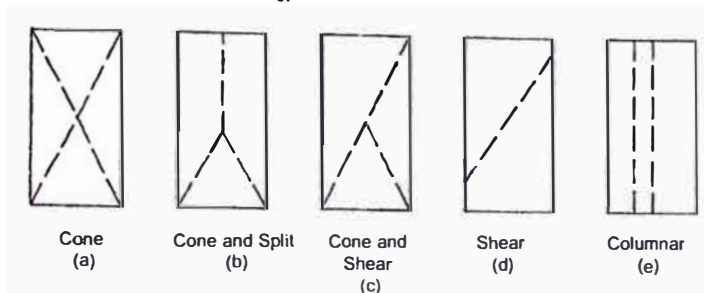


FIG. 2 Sketches of Types of Fracture

-mixed sample of concrete is given for cylinders made in  
 ory environment and under normal field conditions  
 .1.1).

concrete reference sample data for laboratory conditions and a  
 collection of 1265 test reports from 225 commercial testing  
 laboratories in 1978.<sup>5</sup>

rator conditions	Coefficient of Variation <sup>a</sup>	Acceptable Range of <sup>a</sup>	
		2 results	3 results
s	2.37 %	6.6 %	7.8 %
	2.87 %	8.0 %	9.5 %

NOTE 12—Subcommittee C09.03 will re-examine recent CCRL Con-  
 crete Reference Sample Program data and field test data to see if these  
 values are representative of current practice and if they can be extended to  
 cover a wider range of strengths and specimen sizes.

numbers represent respectively the (1s) and (d2s) limits as described  
 C 670.

10.2 *Bias*—Since there is no accepted reference material, no  
 statement on bias is being made.

1 The values given are applicable to 6 by 12 in. [150 by  
 ] cylinders with compressive strength between 2000  
 000 psi [15 to 55 MPa]. They are derived from CCRL

<sup>5</sup> Research report RR:C09-1006 is on file at ASTM Headquarters.

SUMMARY OF CHANGES

This section identifies the location of changes to this test method that have been incorporated since the last  
 issue.

was revised.

(2) 7.5.2 was revised.

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 views known to the ASTM Committee on Standards, at the address shown below.*

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# Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory<sup>1</sup>

This standard is issued under the fixed designation C 192/C 192M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This practice covers procedures for making and curing test specimens of concrete in the laboratory under accurate control of materials and test conditions using concrete that can be consolidated by rodding or vibration as described herein.

1.2 The values stated in either inch-pound units or SI units shall be regarded separately as standard. The SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of each other. Combining values from the two systems may result in nonconformance.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

- C 31/C 31M Practice for Making and Curing Concrete Test Specimens in the Field<sup>2</sup>
- C 70 Test Method for Surface Moisture in Fine Aggregate<sup>2</sup>
- C 125 Terminology Relating to Concrete and Concrete Aggregates<sup>2</sup>
- C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate<sup>2</sup>
- C 128 Test Method for Specific Gravity and Absorption of Fine Aggregate<sup>2</sup>
- C 138 Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete<sup>2</sup>
- C 143/C 143M Test Method for Slump of Hydraulic Cement Concrete<sup>2</sup>
- C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>
- C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method<sup>2</sup>
- C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method<sup>2</sup>

C 330 Specification for Lightweight Aggregates for Structural Concrete<sup>2</sup>

C 403/C 403M Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance<sup>2</sup>

C 470/C 470M Specification for Molds for Forming Concrete Test Cylinders Vertically<sup>2</sup>

C 511 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes<sup>3</sup>

C 566 Test Method for Total Moisture Content of Aggregate by Drying<sup>2</sup>

C 617 Practice for Capping Cylindrical Concrete Specimens<sup>2</sup>

C 1064 Test Method for Temperature of Freshly Mixed Portland-Cement Concrete<sup>2</sup>

C 1077 Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation<sup>2</sup>

### 2.2 American Concrete Institute Publications:<sup>4</sup>

- 211.3 Practice for Selecting Proportions for No-Slump Concrete
- 309 Guide for Concrete Consolidation

## 3. Significance and Use

3.1 This practice provides standardized requirements for preparation of materials, mixing concrete, and making and curing concrete test specimens under laboratory conditions.

3.2 If specimen preparation is controlled as stipulated herein, the specimens may be used to develop information for the following purposes:

- 3.2.1 Mixture proportioning for project concrete,
- 3.2.2 Evaluation of different mixtures and materials,
- 3.2.3 Correlation with nondestructive tests, and
- 3.2.4 Providing specimens for research purposes.

NOTE 1—The concrete test results for concrete specimens made and cured using this practice are widely used. They may be the basis for acceptance testing for project concrete, research evaluations, and other studies. Careful and knowledgeable handling of materials, mixing concrete, molding test specimens, and curing test specimens is necessary.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.61 on Testing Concrete for Strength.

Current edition approved Aug. 10, 2000. Published October 2000. Originally published as C 192 – 44 T. Last previous edition C 192 – 98.

<sup>2</sup> Annual Book of ASTM Standards, Vol 04.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 04.01.

<sup>4</sup> Available from the American Concrete Institute, P.O. Box 9094, Farmington Hills, MI 48333-9094.

Many laboratories performing this important work are independently inspected or accredited. Practice C 1077 identifies and defines duties, responsibilities, including minimum responsibilities of the laboratory personnel and minimum technical requirements for laboratory equipment used. Many laboratories ensure qualified technicians by participating in national certification programs such as the American Concrete Institute Laboratory Technician Program or an equivalent program.

#### 4. Apparatus

4.1 *Molds, General*—Molds for specimens or fastenings hereto in contact with the concrete shall be made of steel, cast iron, or other nonabsorbent material, nonreactive with concrete containing portland or other hydraulic cements. Molds shall conform to the dimensions and tolerances specified in the method for which the specimens are required. Molds shall hold their dimensions and shape under all conditions of use. Watertightness of molds during use shall be judged by their ability to hold water poured into them. Test procedures for watertightness are given in the section on Test Methods for Elongation, Absorption, and Watertightness of Specification C 470. A suitable sealant, such as heavy grease, modeling clay, or microcrystalline wax, shall be used where necessary to prevent leakage through the joints. Positive means shall be provided to hold base plates firmly to the molds. Reusable molds shall be lightly coated with mineral oil or a suitable nonreactive release material before use.

##### 4.2 *Cylinder Molds:*

4.2.1 *Molds for Casting Specimens Vertically* shall conform to the requirements of 4.1 and Specification C 470.

4.2.2 *Horizontal Molds for Creep Test Cylinders* shall conform to the requirements of 4.1 and to the requirements for symmetry and dimensional tolerance in the section on General Requirements except for verticality requirements of Specification C 470. The use of horizontal molds is intended only for creep specimens that contain axially embedded strain gages. Molds for creep cylinders to be filled while supported in a horizontal position shall have a filling slot parallel to the axis of the mold which extends the full length to receive the concrete. The width of the slot shall be one half the diameter of the specimen. If necessary the edges of the slot shall be reinforced to maintain dimensional stability. Unless specimens are to be capped or ground to produce plane ends, the molds shall be provided with two machined metal end plates at least 1 in. [25 mm] thick and the working surfaces shall comply with the requirements for planeness and surface roughness given in the section on Capping Plates of Practice C 617. Provision shall be made for fixing both end plates firmly to the mold. The inside surface of each end plate shall be provided with at least three lugs or studs approximately 1 in. [25 mm] long, firmly fastened to the plate for embedment in the concrete. One base plate shall be drilled from the inside at an angle to permit the lead wire from the strain gage to exit the specimen through the edge of the plate. Provision shall be made for accurately positioning the strain gage. All necessary holes shall be as small as possible to minimize disturbance to subsequent strain measurements and shall be sealed to prevent leakage.

4.3 *Beam and Prism Molds* shall be rectangular in shape (unless otherwise specified) and of the dimensions required to produce the desired specimen size. The inside surfaces of the molds shall be smooth and free from indentations. The sides,

bottom, and ends shall be at right angles to each other and shall be straight and true and free of warpage. Maximum variation from the nominal cross section shall not exceed  $\frac{1}{8}$  in. [3 mm] for molds with depth or breadth of 6 in. [150 mm] or more, or  $\frac{1}{16}$  in. [2 mm] for molds of smaller depth or breadth. Except for flexure specimens, molds shall not vary from the nominal length by more than  $\frac{1}{16}$  in. [2 mm]. Flexure molds shall not be shorter than  $\frac{1}{16}$  in. [2 mm] of the required length, but may exceed it by more than that amount.

4.4 *Tamping Rods*—Two sizes are specified in ASTM methods. Each shall be a round, straight steel rod with at least the tamping end rounded to a hemispherical tip of the same diameter as the rod. Both ends may be rounded, if preferred.

4.4.1 *Larger Rod*,  $\frac{3}{8}$  in. [16 mm] in diameter and approximately 24 in. [600 mm] long.

4.4.2 *Smaller Rod*,  $\frac{3}{8}$  in. [10 mm] in diameter and approximately 12 in. [300 mm] long.

4.5 *Mallets*—A mallet with a rubber or rawhide head weighing  $1.25 \pm 0.50$  lb [ $0.6 \pm 0.20$  kg] shall be used.

##### 4.6 *Vibrators:*

4.6.1 *Internal Vibrators*—The vibrator frequency shall be at least 7000 vibrations per minute [115 Hz] while the vibrator is operating in the concrete. The diameter of a round vibrator shall be no more than one fourth the diameter of the cylinder mold or one fourth the width of the beam or prism mold. Other shaped vibrators shall have a perimeter equivalent to the circumference of an appropriate round vibrator. The combined length of the vibrator shaft and vibrating element shall exceed the depth of the section being vibrated by at least 3 in. [75 mm]. Check the performance of the vibrator in accordance with ACI 309.

4.6.2 *External Vibrators*—The two types of external vibrators permitted are either table or plank. The external vibrator frequency shall be 3600 vibrations per minute [60 Hz] or higher.

4.6.3 Provisions shall be made for clamping the mold securely to the apparatus for both types of vibrators.

NOTE 2—Vibratory impulses are frequently imparted to a table or plank vibrator through electromagnetic means, or by use of an eccentric weight on the shaft of an electric motor or on a separate shaft driven by a motor.

4.7 *Small Tools*—Tools and items such as shovels, pails, trowels, wood float, blunted trowels, straightedge, feeler gage, scoops, rulers, rubber gloves, and metal mixing bowls shall be provided.

4.8 *Slump Apparatus*—The apparatus for measurement of slump shall conform to the requirements of Test Method C 143.

4.9 *Sampling and Mixing Pan*—The pan shall be flat-bottom and of heavy-gage metal, watertight, of convenient depth, and of sufficient capacity to allow easy mixing by shovel or trowel of the entire batch; or, if mixing is by machine, to receive the entire batch on discharge of the mixer and allow remixing in the pan by trowel or shovel.

4.10 *Wet-Sieving Equipment*—If wet-sieving is required, the equipment shall conform to the requirements of Practice C 172.

4.11 *Air Content Apparatus*—The apparatus for measuring air content shall conform to the requirements of either Test Methods C 231 or C 173.

4.12 *Scales*—Scales for determining the mass of batches of

materials and concrete shall be accurate within 0.3 % of the test load at any point within the range of use.

**NOTE 3**—In general the mass of small quantities should not be determined on large capacity scales. In many applications the smallest mass determined on a scale should be greater than about 10 % of the maximum capacity of the scale; however, this will vary with the performance characteristics of the scale and the required accuracy of the determination. Acceptable scales used for determining the mass for concrete materials preferably should determine mass accurately to about 0.1 % of total capacity and the foregoing precaution is applicable. However, certain analytical and precision balances are exceptions to this rule and should weigh accurately to 0.001 %. Particular care must be exercised in measuring small quantities of material by determining the difference between two much larger masses.

**4.13 Temperature Measuring Device**—The temperature measuring device shall conform to the requirements of Test Method C 1064.

**4.14 Concrete Mixer**—A power-driven concrete mixer shall be a revolving drum, tilting mixer, or suitable revolving pan or revolving-paddle mixer capable of thoroughly mixing batches of the prescribed sizes at the required slump.

**NOTE 4**—A pan mixer is usually more suitable for mixing concrete with less than 1-in. [25 mm] slump than a revolving drum mixer. The rate of rotation, degree of tilt, and rated capacity of tilting mixers are not always suitable for laboratory mixed concrete. It may be found desirable to reduce the rate of rotation, decrease the angle of tilt from the horizontal, and use the mixer at somewhat less than the manufacturer's rated capacity.

## 5. Specimens

**5.1 Cylindrical Specimens**—Cylinders for such tests as compressive strength, Young's modulus of elasticity, creep, and splitting tensile strength may be of various sizes with a minimum of 2-in. [50-mm] diameter by 4-in. [100-mm] length. Where correlation or comparison with field-made cylinders (Practice C 31) is desired, the cylinders shall be 6 by 12 in. [or 150 by 300 mm]. Otherwise, dimensions should be governed in accordance with 5.4 and the specific test method concerned.

**NOTE 5**—When molds in SI units are required and not available, equivalent inch-pound unit size mold should be permitted.

**5.1.1 Cylindrical specimens for tests other than creep** shall be molded and allowed to harden with the axis of the cylinder vertical.

**5.1.2 Cylindrical creep specimens** may be cast with the cylindrical axis either vertical or horizontal and allowed to harden in the position in which cast.

**5.2 Prismatic Specimens**—Beams for flexural strength, prisms for freezing and thawing, bond, length change, volume change, etc., shall be formed with their long axes horizontal, unless otherwise required by the method of test in question, and shall conform in dimension to the requirements of the specific test method.

**5.3 Other Specimens**—Other shapes and sizes of specimens for particular tests may be molded as desired following the general procedures set forth in this practice.

**5.4 Specimen Size versus Aggregate Size**—The diameter of a cylindrical specimen or minimum cross-sectional dimension of a rectangular section shall be at least three times the nominal maximum size of the coarse aggregate in the concrete as defined in Terminology C 125. Occasional over-size aggregate

particles (of a size not normally found in the average aggregate grading) shall be removed by hand picking during the molding of the specimens. When the concrete contains aggregate larger than that appropriate for the size of the molds or equipment to be used, wet-sieve the sample as described in Practice C 172.

**5.5 Number of Specimens**—The number of specimens and the number of test batches are dependent on established practice and the nature of the test program. Guidance is usually given in the test method or specification for which the specimens are made. Usually three or more specimens are molded for each test age and test condition unless otherwise specified (Note 6). Specimens involving a given variable should be made from three separate batches mixed on different days. An equal number of specimens for each variable should be made on any given day. When it is impossible to make at least one specimen for each variable on a given day, the mixing of the entire series of specimens should be completed in as few days as possible, and one of the mixtures should be repeated each day as a standard of comparison.

**NOTE 6**—Test ages often used are 7 and 28 days for compressive strength tests, or 14 and 28 days for flexural strength tests. Specimens containing Type III cement are often tested at 1, 3, 7, and 28 days. For later test ages, 3 months, 6 months, and 1 year are often used for both compressive and flexural strength tests. Other test ages may be required for other types of specimens.

## 6. Preparation of Materials

**6.1 Temperature**—Before mixing the concrete, bring the concrete materials to room temperature in the range from 68 to 86°F [20 to 30°C], except when the temperature of the concrete is stipulated. When a concrete temperature is stipulated, the method proposed to obtain the concrete temperature needs approval of the stipulator.

**6.2 Cement**—Store the cement in a dry place, in moisture-proof containers, preferably made of metal. The cement shall be thoroughly mixed to provide a uniform supply throughout the tests. It shall be passed through a 850- $\mu$ m [No. 20] or finer sieve to remove all lumps, remixed on a plastic sheet, and returned to sample containers.

**6.3 Aggregates**—In order to preclude segregation of a coarse aggregate, separate into individual size fractions and for each batch recombine in the proper proportions to produce the desired grading.

**NOTE 7**—Only rarely is a coarse aggregate batched as a single size fraction. The number of size fractions will generally be between 2 and 5 for aggregate smaller than 2½ in. [60 mm]. When a size fraction to be batched is present in amounts in excess of 10 %, the ratio of the opening of the larger to the smaller sieve should not exceed 2.0. More closely sized groups are sometimes advisable.

**6.3.1** Unless fine aggregate is separated into individual size fractions, maintain it in a damp condition or restore to a damp condition until use, to prevent segregation, unless material uniformly graded is subdivided into batch size lots using a sample splitter with proper size openings. If unusual gradings are being studied, the fine aggregate may need to be dried and separated into individual sizes. In this instance, if the total quantity of fine aggregate required is larger than can be efficiently blended in a single unit, then the individual size fractions should be determined in a mass required for each



individual batch. When the total quantity of fine aggregate needed for the complete investigation is such that it can be thoroughly mixed, blended, and maintained in a damp condition, then it should be handled in that manner. Determine the specific gravity and absorption of aggregates in accordance with either Test Methods C 127 or C 128.

6.3.2 Before incorporating in concrete, prepare the aggregate to ensure a definite and uniform condition of moisture. Determine the weight of aggregate to be used in the batch by one of the following procedures:

6.3.2.1 Determine the mass of low-absorption aggregates (absorption less than 1.0 %) in the room-dry condition with allowance made for the amount of water that will be absorbed from the unset concrete (Note 8). This procedure is particularly useful for coarse aggregate which must be batched as individual sizes; because of the danger of segregation it can be used for fine aggregate only when the fine aggregate is separated into individual size fractions.

NOTE 8—When using aggregates with low absorption in room-dry condition the amount of water that will be absorbed by the aggregates before the concrete sets may be assumed to be 80 % of the difference between the 24-h absorption of the aggregates determined by Test Methods C 127 or C 128, and the amount of water in the pores of the aggregates in their room-dry state, as determined by Test Method C 566.

6.3.2.2 Individual size fractions of aggregate may be weighed separately, recombined into a tared container in the amounts required for the batch, and immersed in water for 24 h prior to use. After immersion the excess water is decanted and the combined weight of aggregate and mixing water determined. Allowance shall be made for the amount of water absorbed by the aggregate. The moisture content of the aggregates may be determined in accordance with Test Methods C 70 and C 566.

6.3.2.3 The aggregate may be brought to and maintained in a saturated condition, with surface moisture contained in sufficiently small amounts to preclude loss by draining, at least 24 h prior to use. When this method is used, the moisture content of the aggregate must be determined to permit calculation of proper quantities of the damp aggregate. The quantity of surface moisture present must be counted as a part of the required amount of mixing water. Surface moisture in fine aggregate may be determined in accordance with Test Methods C 70 and C 566, making due allowance for the amount of water absorbed. The method outlined here (moisture content slightly exceeding absorption) is particularly useful for fine aggregate. It is used less frequently for coarse aggregate because of the difficulty of accurately determining the moisture content, but if used, each size fraction must be handled separately to ensure that the proper grading is obtained.

6.3.2.4 Aggregates, fine or coarse, may be brought to and maintained in a saturated surface-dry condition until batched for use. This method is used primarily to prepare material for batches not exceeding  $\frac{1}{4}$  ft<sup>3</sup> [0.007 m<sup>3</sup>] in volume. Care must be taken to prevent drying during weighing and use.

6.4 *Lightweight Aggregates*—The procedures for specific gravity, absorption, and preparation of aggregates mentioned in this practice pertain to materials with normal absorption values. Lightweight aggregates, air-cooled slag, and certain

highly porous or vesicular natural aggregate may be so absorptive as to be difficult to treat as described. The moisture content of lightweight aggregate at the time of mixing may have important effects on properties of freshly mixed and hardened concretes such as slump loss, compressive strength, and resistance to freezing and thawing.

6.5 *Admixtures*—Powdered admixtures that are entirely or largely insoluble, that do not contain hygroscopic salts and are to be added in small quantities, should be mixed with a portion of the cement before introduction into the batch in the mixer so as to ensure thorough distribution throughout the concrete. Essentially insoluble materials which are used in amounts exceeding 10 % by mass of cement, such as pozzolans, should be handled and added to the batch in the same manner as cement. Powdered admixtures which are largely insoluble but contain hygroscopic salts may cause balling of cement and should be mixed with the sand. Water-soluble and liquid admixtures should be added to the mixer in solution in the mixing water. The quantity of such solution used shall be included in the calculation of the water content of the concrete. Admixtures, incompatible in concentrated form, such as solutions of calcium chloride and certain air-entraining and set-retarding admixtures, should not be intermixed prior to their addition to concrete. The time, sequence, and method of adding some admixtures to a batch of concrete can have important effects on concrete properties such as time of set and air content. The method selected must remain unchanged from batch to batch.

NOTE 9—The mixing apparatus and accessories shall be thoroughly cleaned to ensure that chemical additions or admixtures used in dissimilar batches of concrete do not affect subsequent batches.

## 7. Procedure

### 7.1 *Mixing Concrete:*

7.1.1 *General*—Mix concrete in a suitable mixer or by hand in batches of such size as to leave about 10 % excess after molding the test specimens. Hand-mixing procedures are not applicable to air-entrained concrete or concrete with no measurable slump. Hand mixing should be limited to batches of  $\frac{1}{4}$  ft<sup>3</sup> [0.007 m<sup>3</sup>] volume or less. Mixing procedures are given in 7.1.2 and 7.1.3. However, other procedures may be used when it is desired to simulate special conditions or practices, or when the procedures specified are impracticable. A machine-mixing procedure suitable for drum-type mixers is described. It is important not to vary the mixing sequence and procedure from batch to batch unless the effect of such variation is under study.

7.1.2 *Machine Mixing*—Prior to starting rotation of the mixer add the coarse aggregate, some of the mixing water, and the solution of admixture, when required, in accordance with 6.5. When feasible, disperse the admixture in the mixing water before addition. Start the mixer, then add the fine aggregate, cement, and water with the mixer running. If it is impractical for a particular mixer or for a particular test to add the fine aggregate, cement, and water while the mixer is running, these components may be added to the stopped mixer after permitting it to turn a few revolutions following charging with coarse aggregate and some of the water (Note 10). Mix the concrete, after all ingredients are in the mixer, for 3 min followed by a

3-min rest, followed by a 2-min final mixing. Cover the open end or top of the mixer to prevent evaporation during the rest period. Take precautions to compensate for mortar retained by the mixer so that the discharged batch, as used, will be correctly proportioned (Note 11). To eliminate segregation, deposit machine-mixed concrete in the clean, damp mixing pan and remix by shovel or trowel until it appears to be uniform.

NOTE 10—An experienced operator may add water incrementally during mixing to adjust to the desired slump.

NOTE 11—It is difficult to recover all of the mortar from mixers. To compensate for this difficulty one of the following procedures may be used to ensure the correct final proportions in the batch:

(1) "Buttering" the Mixer—Just prior to mixing the test batch, the mixer is "battered" by mixing a batch proportioned to simulate closely the test batch. The mortar adhering to the mixer after discharging is intended to compensate for loss of mortar from the test batch.

(2) "Over-Mortaring" the Mix—The test mix is proportioned by the use of an excess mortar, the amount established in advance, to compensate for that which, on the average, adheres to the mixer. In this case the mixer is cleaned before mixing the test batch.

7.1.3 Hand Mixing—Mix the batch in a watertight, clean (Note 9), damp, metal pan or bowl, with a bricklayer's blunted trowel, using the following procedure when aggregates have been prepared in accordance with 6.3.2.1, 6.3.2.3, and 6.3.2.4.

7.1.3.1 Mix the cement, powdered insoluble admixture, if used, and fine aggregate without addition of water until they are thoroughly blended.

7.1.3.2 Add the coarse aggregate and mix the entire batch without addition of water until the coarse aggregate is uniformly distributed throughout the batch.

7.1.3.3 Add water, and the admixture solution if used, and mix the mass until the concrete is homogeneous in appearance and has the desired consistency. If prolonged mixing is necessary because of the addition of water in increments while adjusting the consistency, discard the batch and make a new batch in which the mixing is not interrupted to make trial consistency tests.

7.1.4 Mixed Concrete—Select the portions of the batch of mixed concrete to be used in tests for molding specimens so as to be representative of the actual proportions and condition of the concrete. When the concrete is not being remixed or sampled cover it to prevent evaporation.

7.2 Slump, Air Content, Yield, and Temperature:

7.2.1 Slump—Measure the slump of each batch of concrete immediately after mixing in accordance with Test Method C 143.

NOTE 12—The slump test is unsuitable for concrete so dry that it slumps less than 1/4 in. [6 mm]. No-slump concrete may be tested by one of several means described in ACI 211.3.

7.2.2 Air Content—Determine the air content, when required, in accordance with either Test Methods C 173 or C 231. Test Method C 231 should not be used with concretes made with lightweight aggregates, air-cooled blast-furnace slag, or aggregates of high porosity. Discard the concrete used for the determination of air content.

7.2.3 Yield—Determine the yield of each batch of concrete, if required, in accordance with Test Method C 138. Concrete used for slump and yield tests may be returned to the mixing pan and remixed into the batch.

7.2.4 Temperature—Determine the temperature of each batch of concrete in accordance with Test Method C 1064.

7.3 Making Specimens:

7.3.1 Place of Molding—Mold specimens as near as practicable to the place where they are to be stored during the first 24 h. If it is not practicable to mold the specimens where they will be stored, move them to the place of storage immediately after being struck off. Place molds on a rigid surface free from vibration and other disturbances. Avoid jarring, striking, tilting, or scarring of the surface of the specimens when moving the specimens to the storage place.

7.3.2 Placing—Place the concrete in the molds using a scoop, blunted trowel, or shovel. Select each scoopful, trowelful, or shovelful of concrete from the mixing pan to ensure that it is representative of the batch. It may be necessary to remix the concrete in the mixing pan with a shovel or trowel to prevent segregation during the molding of specimens. Move the scoop or trowel around the top edge of the mold as the concrete is discharged in order to ensure a symmetrical distribution of the concrete and to minimize segregation of coarse aggregate within the mold. Further distribute the concrete by use of a tamping rod prior to the start of consolidation. In placing the final layer the operator shall attempt to add an amount of concrete that will exactly fill the mold after compaction. Do not add nonrepresentative samples of concrete to an underfilled mold.

7.3.2.1 Number of Layers—Make specimens in layers as indicated in Table 1.

7.4 Consolidation:

7.4.1 Methods of Consolidation—Preparation of satisfactory specimens requires different methods of consolidation. The methods of consolidation are rodding, and internal or external vibration. Base the selection of the method on the slump, unless the method is stated in the specifications under which the work is being performed. Rod or vibrate concrete with slump greater than or equal to 1 in. [25 mm]. Vibrate concrete with slump less than 1 in. (Note 13). Do not use internal vibration for cylinders with a diameter less than 4 in. [100 mm], and for beams or prisms with breadth or depth less than 4 in.

NOTE 13—Concrete of such low water content that it cannot be properly consolidated by the methods described herein is not covered by

TABLE 1 Number of Layers Required for Specimens

Specimen Type and Size	Mode of Consolidation	Numbers of Layers of Approximate Equal Depth
Cylinders:		
Diameter, in. [mm]		
3 or 4 [75 to 100]	rodding	2
6 [150]	rodding	3
9 [225]	rodding	4
up to 9 [225]	vibration	2
Prisms and horizontal creep		
Cylinders:		
Depth, in. [mm]		
up to 8 [200]	rodding	2
over 8 [200]	rodding	3 or more
up to 8 [200]	vibration	1
over 8 [200]	vibration	2 or more

this practice. Provisions for specimens and methods of testing will be found in the standards concerned. There are concretes that can be consolidated by external vibration, but additional forces on the surface are required to embed the coarse aggregate thoroughly and consolidate the mixture. For such mixtures the following procedures may be followed: using external vibration fill 6 by 12-in. [150 by 300-mm] cylinder molds in 3 in. [75 mm] lifts using a 10-lb [4.5-kg] cylindrical surcharge, or 3 by 6-in. [75 by 150-mm] cylinder molds in 2 in. [50 mm] lifts using a 2.5-lb [1-kg] cylindrical surcharge. The surcharge should have a diameter  $\frac{1}{4}$  in. [6 mm] less than the inside of the mold. Simultaneously each lift should be compacted by external vibration with the surcharge on the top surface of the concrete, until the mortar begins to ooze around the bottom of the surcharge.

**7.4.2 Rodding**—Place the concrete in the mold, in the required number of layers of approximately equal volume. Rod each layer with the rounded end of the rod using the number of strokes and size of rod specified in Table 2. Rod the bottom layer throughout its depth. Distribute the strokes uniformly over the cross section of the mold and for each upper layer allow the rod to penetrate through the layer being rodded and into the layer below approximately 1 in. [25 mm]. After each layer is rodded, tap the outsides of the mold lightly 10 to 15 times with the mallet to close any holes left by rodding and to release any large air bubbles that may have been trapped. Use an open hand to tap light-gage single-use molds which are susceptible to damage if tapped with a mallet. After tapping, spade the concrete along the sides and ends of beam and prism molds with a trowel or other suitable tool.

**7.4.3 Vibration**—Maintain a uniform duration of vibration for the particular kind of concrete, vibrator, and specimen mold involved. The duration of vibration required will depend upon the workability of the concrete and the effectiveness of the vibrator. Usually sufficient vibration has been applied as soon as the surface of the concrete becomes relatively smooth and large air bubbles cease to break through the top surface. Continue vibration only long enough to achieve proper consolidation of the concrete (see Note 14). Overvibration may cause segregation. Fill the molds and vibrate in the required number of approximately equal layers (Table 2). Place all the concrete for each layer in the mold before starting vibration of that layer. When placing the final layer, avoid overfilling by more than  $\frac{1}{4}$  in. [6 mm]. When the finish is applied after vibration, add only enough concrete with a trowel to overfill the mold about  $\frac{1}{8}$  in. [3 mm], work it into the surface and then strike it off.

NOTE 14—Generally, no more than 5 s of vibration should be required for each insertion to adequately consolidate the concrete with a slump

greater than 3 in. [75 mm]. Longer times may be required for lower slump concrete, but the vibration time should rarely have to exceed 10 s per insertion.

**7.4.3.1 Internal Vibration**—In compacting the specimen insert the vibrator slowly and do not allow the vibrator to rest on or touch the bottom or sides of the mold or strike embedded items such as strain meters. Slowly withdraw the vibrator so that no large air pockets are left in the specimen.

**7.4.3.2 Cylinders**—The number of insertions of the vibrator is given in Table 3. When more than one insertion per layer is required, distribute the insertions uniformly within each layer. Allow the vibrator to penetrate into the layer below approximately 1 in. [25 mm]. After each layer is vibrated, tap the outside of the mold at least 10 times with the mallet to close the holes that remain and to release entrapped air voids. Use an open hand to tap cardboard or single-use metal molds that are susceptible to damage if tapped with a mallet.

**7.4.3.3 Beams, Prisms, and Horizontal Creep Cylinders**—Insert the vibrator at intervals not exceeding 6 in. [150 mm] along the center line of the long dimension of the specimen, or along both sides but not in contact with the strain gage in the case of creep cylinders. For specimens wider than 6 in. [150 mm], use alternating insertions along two lines. Allow the shaft of the vibrator to penetrate into the bottom layer approximately 1 in. [25 mm]. After each layer is vibrated, tap the outsides of the mold sharply at least 10 times with the mallet to close holes left by vibrating and to release entrapped air voids.

**7.4.4 External Vibration**—When external vibration is used, take care to ensure that the mold is rigidly attached to or securely held against the vibrating element or vibrating surface (Note 13).

**7.5 Finishing**—After consolidation by any of the methods, strike off the surface of the concrete and float or trowel it in accordance with the method concerned. If no finish is specified, finish the surface with a wood or magnesium float. Perform all finishing with the minimum manipulation necessary to produce a flat even surface that is level with the rim or edge of the mold and which has no depressions or projections larger than  $\frac{1}{8}$  in. [3 mm].

**7.5.1 Cylinders**—After consolidation finish the top surfaces by striking them off with the tamping rod where the consistency of the concrete permits, or with a wood float or trowel. If desired, cap the top surface of freshly made cylinders with a thin layer of stiff portland cement paste which is permitted to harden and cure with the specimen. See the section on Capping Materials of Practice C 617.

**TABLE 2 Diameter of Rod and Number of Roddings to be Used in Molding Test Specimens**

Cylinders		
Diameter of Cylinder, in. [mm]	Diameter of Rod in. [mm]	Number of Strokes/Layer
2 [50] to <6 [150]	$\frac{3}{8}$ [10]	25
6 [150]	$\frac{1}{2}$ [16]	25
8 [200]	$\frac{5}{8}$ [16]	50
10 [250]	$\frac{3}{4}$ [16]	75
Beams and Prisms		
Top Surface Area of Specimen, in. <sup>2</sup> [cm <sup>2</sup> ]	Diameter of Rod in. (mm)	Number of Roddings/Layer
25 [160] or less	$\frac{3}{8}$ [10]	25
26 to 49 [165 to 310]	$\frac{1}{2}$ [10]	one for each 1 in. <sup>2</sup> [7 cm <sup>2</sup> ] of surface
50 [320] or more	$\frac{5}{8}$ [16]	one for each 2 in. <sup>2</sup> [14 cm <sup>2</sup> ] of surface
Horizontal Creep Cylinders		
Diameter of Cylinder in. [mm]	Diameter of Rod in. [mm]	Number of Roddings/Layer
6 [150]	$\frac{3}{8}$ [16]	50 total, 25 along both sides of axis

**TABLE 3 Number of Vibrator Insertions per Layer**

Specimen Type and Size	Number of Insertions per Layer
Cylinder Diameter, in. [mm]	
4 in. [200 mm]	1
6 in. [150 mm]	2
9 in. [225 mm]	4

**7.5.2 Horizontally Cast Creep Cylinders**—After consolidation strike off the specimen with a trowel or float, then trowel the minimum amount required to form the concrete in the opening concentrically with the rest of the specimen. Use a screed curved to the radius of the specimen to more precisely shape and finish the concrete in the opening.

**8. Curing**

**8.1 Initial Storage**—To prevent evaporation of water from unhardened concrete, cover the specimens immediately after finishing, preferably with a nonabsorptive, nonreactive plate or a sheet of tough, durable impervious plastic. Specimens shall be stored immediately after finishing until the removal of the molds to prevent loss of moisture from the specimens. Select an appropriate procedure or combination of procedures that will prevent moisture loss and is nonabsorptive and nonreactive with the concrete. When wet burlap is used for covering, the burlap must not be in contact with the fresh concrete surface and care must be exercised to keep the burlap wet until the specimens are removed from the molds. Placing a sheet of plastic over the burlap will facilitate keeping it wet. Protect the outside surfaces of cardboard molds from contact with wet burlap or other sources of water for the first 24 h after the cylinders have been molded in them. Water may cause the molds to expand and damage specimens at this early age.

**8.2 Removal from Molds**—Remove the specimens from the molds  $24 \pm 8$  h after casting. For concrete with prolonged setting time, molds shall not be removed until  $20 \pm 4$  h after final set. If needed, determine the setting times in accordance with Test Method C 403/C 403M.

**8.3 Curing Environment**—Unless otherwise specified all specimens shall be moist cured at  $73 \pm 3^\circ\text{F}$  [ $23 \pm 2^\circ\text{C}$ ] from the time of molding until the moment of test (Note 15). Storage during the first 48 h of curing shall be in a vibration-free environment. As applied to the treatment of demolded specimens, moist curing means that the test specimens shall have free water maintained on the entire surface area at all times. This condition is met by using water storage tanks or a moist room in accordance with the requirements of Specification C 511. When water storage tanks are used, specimens shall be immersed in a water saturated with calcium hydroxide to prevent leaching from the specimens. Specimens shall not be exposed to dripping or running water. Cure structural light-weight concrete cylinders in accordance with Specification C 330.

NOTE 15—The temperature within damp sand and under wet burlap or

similar materials will always be lower than the temperature in the surrounding atmosphere if evaporation takes place.

**8.4 Flexural Strength Test Specimens**—Cure the flexural strength test specimens in accordance with 8.1 and 8.2 except that while in storage for a minimum period of 20 h immediately prior to testing they shall be immersed in water saturated with calcium hydroxide at  $73 \pm 3^\circ\text{F}$  [ $23 \pm 2^\circ\text{C}$ ]. At the end of the curing period, between the time the specimen is removed from curing until testing is completed, drying of the surfaces shall be prevented.

NOTE 16—Relatively small amounts of drying of the surface of flexural strength specimens will induce tensile stresses in the extreme fibers that will markedly reduce the indicated flexural strength.

**9. Precision and Bias**

**9.1** Data to establish precision statements for various testing required by this standard were obtained in the Concrete Proficiency Sample Program of the Cement and Concrete Reference Laboratory.

**9.2** The single-operator standard deviations for slump, unit weight, air content, and 7-day compressive strength of trial batches have been found to be 0.7 in., 0.9 lb/ft<sup>3</sup>, 0.3 %, and 203 psi, respectively; therefore the results of properly conducted tests on two trial batches made in the same laboratory should not differ by more than 2.0 in., 2.5 lb/ft<sup>3</sup>, 0.8 %, and 574 psi, respectively. This precision statement is considered applicable to laboratory trial batches proportioned to contain prescribed quantities of materials and to have a constant water-cement ratio. The values should be used with caution for air-entrained concrete, concrete with slump less than 2 in. [50 mm] or over 6 in. [150 mm], or concrete made with other than normal weight aggregate or aggregate larger than 1 in. [25 mm] nominal maximum size.

**9.3** The multilaboratory standard deviations for slump, unit weight, air content, and 7-day compressive strength of trial batches have been found to be 1.0 in., 1.4 lb/ft<sup>3</sup>, 0.4 %, and 347 psi, respectively; therefore, the results of properly conducted tests on single trial batches made in two different laboratories should not differ by more than 2.8 in., 4.0 lb/ft<sup>3</sup>, 1.1 %, and 981 psi, respectively. This precision statement is considered applicable to laboratory trial batches proportioned to contain prescribed quantities of materials and to have a prescribed water-cement ratio. The values should be used with caution for air-entrained concrete, concrete with slump less than 2 in. [50 mm] or over 6 in. [150 mm], or concrete made with other than normal weight aggregate or aggregate larger than 1 in. [25 mm] nominal maximum size.

**9.4 Bias**—The procedures for the test methods in 9.3 have no bias because the values obtained from each of those test methods are defined only in terms of the test method.

**10. Keywords**

10.1 concrete; cylinders; laboratory; prisms; strength testing



# Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete<sup>1</sup>

This standard is issued under the fixed designation C 138/C 138M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope \*

1.1 This test method covers determination of the density (see Note 1) of freshly mixed concrete and gives formulas for calculating the yield, cement content, and air content of the concrete. Yield is defined as the volume of concrete produced from a mixture of known quantities of the component materials.

1.2 The values stated in either inch-pound or SI units shall be regarded separately as standard. The SI units are shown in brackets. The values stated might not be exact equivalents; therefore each system must be used independently of the other.

NOTE 1—Unit weight was the previous terminology used to describe the property determined by this test method, which is mass per unit volume.

1.3 The text of this test method references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables) shall not be considered as requirements of this test method.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- C 29/C 29M Test Method for Bulk Density (Unit Weight) and Voids in Aggregate<sup>2</sup>
- C 150 Specification for Portland Cement<sup>3</sup>
- C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>
- C 188 Test Method for Density of Hydraulic Cement<sup>3</sup>
- C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method<sup>2</sup>
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

## 3. Terminology

### 3.1 Symbols:

- $A$  = air content (percentage of voids) in the concrete
- $C$  = actual cement content, lb/yd<sup>3</sup> or kg/m<sup>3</sup>
- $C_b$  = mass of cement in the batch, lb or kg
- $D$  = density (unit weight) of concrete, lb/ft<sup>3</sup> or kg/m<sup>3</sup>
- $M$  = total mass of all materials batched, lb or kg (see Note 3)
- $M_c$  = mass of the measure filled with concrete, lb or kg
- $M_m$  = mass of the measure, lb or kg
- $R_y$  = relative yield
- $T'$  = theoretical density of the concrete computed on an airfree basis, lb/ft<sup>3</sup> or kg/m<sup>3</sup> (see Note 2)
- $Y$  = yield, volume of concrete produced per batch, yd<sup>3</sup> or m<sup>3</sup>
- $Y_d$  = volume of concrete which the batch was designed to produce, yd<sup>3</sup> or m<sup>3</sup>
- $Y_f$  = volume of concrete produced per batch, ft<sup>3</sup>
- $V$  = total absolute volume of the component ingredients in the batch, ft<sup>3</sup> or m<sup>3</sup>
- $V_m$  = volume of the measure, ft<sup>3</sup> or m<sup>3</sup>

NOTE 2—The theoretical density is, customarily, a laboratory determination, the value for which is assumed to remain constant for all batches made using identical component ingredients and proportions. It is calculated from the following equation:

$$T = M/V \quad (1)$$

The absolute volume of each ingredient in cubic feet is equal to the quotient of the mass of that ingredient divided by the product of its specific gravity times 62.4. The absolute volume of each ingredient in cubic metres is equal to the mass of the ingredient in kilograms divided by 1000 times its specific gravity. For the aggregate components, the bulk specific gravity and mass should be based on the saturated, surface-dry condition. For cement, the actual specific gravity should be determined by Test Method C 188. A value of 3.15 may be used for cements manufactured to meet the requirements of Specification C 150.

NOTE 3—The total mass of all materials batched is the sum of the masses of the cement, the fine aggregate in the condition used, the coarse aggregate in the condition used, the mixing water added to the batch, and any other solid or liquid materials used.

## 4. Apparatus

4.1 *Balance*—A balance or scale accurate to 0.1 lb [45 g] or to within 0.3 % of the test load, whichever is greater, at any point within the range of use. The range of use shall be

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.60 on Fresh Concrete Testing.

Current edition approved Mar. 10, 2001. Published May 2001. Originally published as C 138 – 38 T. Last previous edition C 138 – 01.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.01.

\*A Summary of Changes section appears at the end of this standard.

considered to extend from the mass of the measure empty to the mass of the measure plus its contents at 160 lb/ft<sup>3</sup> [2600 kg/m<sup>3</sup>].

4.2 *Tamping Rod*—A round, straight steel rod, 5/8 in. [16 mm] in diameter and approximately 24 in. [600 mm] in length, having the tamping end rounded to a hemispherical tip the diameter of which is 5/8 in.

4.3 *Internal Vibrator*—Internal vibrators may have rigid or flexible shafts, preferably powered by electric motors. The frequency of vibration shall be 7000 vibrations per minute or greater while in use. The outside diameter or the side dimension of the vibrating element shall be at least 0.75 in. [19 mm] and not greater than 1.50 in. [38 mm]. The length of the shaft shall be at least 24 in. [600 mm].

4.4 *Measure*—A cylindrical container made of steel or other suitable metal (see Note 4). The minimum capacity of the measure shall conform to the requirements of Table 1 based on the nominal size of aggregate in the concrete to be tested. All measures, except for measuring bowls of air meters which are also used for Test Method C 138 tests, shall conform to the requirements of Test Method C 29/C 29M. When measuring bowls of air meters are used, they shall conform to the requirements of Test Method C 231, and shall be calibrated for volume as described in Test Method C 29/C 29M. The top rim of the air meter bowls shall be smooth and plane within 0.01 in. [0.3 mm] (see Note 5).

NOTE 4—The metal should not be readily subject to attack by cement paste. However, reactive materials such as aluminum alloys may be used in instances where as a consequence of an initial reaction, a surface film is rapidly formed which protects the metal against further corrosion.

NOTE 5—The top rim is satisfactorily plane if a 0.01-in. [0.3-mm] feeler gage cannot be inserted between the rim and a piece of 1/4-in. [6-mm] or thicker plate glass laid over the top of the measure.

4.5 *Strike-Off Plate*—A flat rectangular metal plate at least 1/4 in. [6 mm] thick or a glass or acrylic plate at least 1/2 in. [12 mm] thick with a length and width at least 2 in. [50 mm] greater than the diameter of the measure with which it is to be used. The edges of the plate shall be straight and smooth within a tolerance of 1/16 in. [2 mm].

4.6 *Mallet*—A mallet (with a rubber or rawhide head) having a mass of 1.25 ± 0.50 lb [600 ± 200 g] for use with measures of 0.5 ft<sup>3</sup> [14 L] or smaller, and a mallet having a mass of 2.25 ± 0.50 lb [1000 ± 200 g] for use with measures larger than 0.5 ft<sup>3</sup>.

**5. Sample**

5.1 Obtain the sample of freshly mixed concrete in accordance with Practice C 172.

dance with Practice C 172.

**6. Procedure**

6.1 Base the selection of the method of consolidation on the slump, unless the method is stated in the specifications under which the work is being performed. The methods of consolidation are rodding and internal vibration. Rod concretes with a slump greater than 3 in. [75 mm]. Rod or vibrate concrete with a slump of 1 to 3 in. [25 to 75 mm]. Consolidate concretes with a slump less than 1 in. by vibration.

NOTE 6—Nonplastic concrete, such as is commonly used in the manufacture of pipe and unit masonry, is not covered by this test method.

6.2 *Rodding*—Place the concrete in the measure in three layers of approximately equal volume. Rod each layer with 25 strokes of the tamping rod when nominal 0.5-ft<sup>3</sup> [14-L] or smaller measures are used, 50 strokes when nominal 1-ft<sup>3</sup> [28-L] measures are used, and one stroke per 3 in.<sup>2</sup> [20 cm<sup>2</sup>] of surface for larger measures. Rod the bottom layer throughout its depth but the rod shall not forcibly strike the bottom of the measure. Distribute the strokes uniformly over the cross section of the measure and for the top two layers, penetrate about 1 in. [25 mm] into the underlying layer. After each layer is rodded, tap the sides of the measure 10 to 15 times with the appropriate mallet (see 4.6) using such force so as to close any voids left by the tamping rod and to release any large bubbles of air that may have been trapped. Add the final layer so as to avoid overfilling.

6.3 *Internal Vibration*—Fill and vibrate the measure in two approximately equal layers. Place all of the concrete for each layer in the measure before starting vibration of that layer. Insert the vibrator at three different points for each layer. In compacting the bottom layer, do not allow the vibrator to rest on or touch the bottom or sides of the measure. In compacting the final layer, the vibrator shall penetrate into the underlying layer approximately 1 in. [25 mm]. Take care that the vibrator is withdrawn in such a manner that no air pockets are left in the specimen. The duration of vibration required will depend upon the workability of the concrete and the effectiveness of the vibrator (see Note 7). Continue vibration only long enough to achieve proper consolidation of the concrete (see Note 8). Observe a constant duration of vibration for the particular kind of concrete, vibrator, and measure involved.

NOTE 7—Usually, sufficient vibration has been applied as soon as the surface of the concrete becomes relatively smooth.

NOTE 8—Overvibration may cause segregation and loss of appreciable quantities of intentionally entrained air.

6.4 On completion of consolidation the measure must not contain a substantial excess or deficiency of concrete. An excess of concrete protruding approximately 1/8 in. [3 mm] above the top of the mold is optimum. A small quantity of concrete may be added to correct a deficiency. If the measure contains a great excess of concrete at completion of consolidation, remove a representative portion of the excess concrete with a trowel or scoop immediately following completion of consolidation and before the measure is struck-off.

6.5 *Strike-Off*—After consolidation, strike-off the top surface of the concrete and finish it smoothly with the flat strike-off plate using great care to leave the measure just level

**TABLE 1 Capacity of Measures**

Nominal Maximum Size of Coarse Aggregate		Capacity of Measure <sup>A</sup>	
in.	mm	ft <sup>3</sup>	L
1	25.0	0.2	6
1½	37.5	0.4	11
2	50	0.5	14
3	75	1.0	28
4½	112	2.5	70
6	150	3.5	100

<sup>A</sup>The indicated size of measure shall be used to test concrete containing aggregates of a nominal maximum size equal to or smaller than that listed. The actual volume of the measure shall be at least 95 % of the nominal volume listed.

full. The strike-off is best accomplished by pressing the strike-off plate on the top surface of the measure to cover about two thirds of the surface and withdrawing the plate with a sawing motion to finish only the area originally covered. Then place the plate on the top of the measure to cover the original two thirds of the surface and advance it with a vertical pressure and a sawing motion to cover the whole surface of the measure and continue to advance it until it slides completely off the measure. Several final strokes with the inclined edge of the plate will produce a smooth finished surface.

6.6 *Cleaning and Weighing*—After strike-off, clean all excess concrete from the exterior of the measure and determine the mass of the concrete and measure to an accuracy consistent with the requirements of 4.1.

### 7. Calculation

7.1 *Density (Unit Weight)*—Calculate the net mass of the concrete in pounds or kilograms by subtracting the mass of the measure,  $M_m$ , from the mass of the measure filled with concrete,  $M_c$ . Calculate the density,  $D$ ,  $\text{ft}^3$  or  $\text{yd}^3$ , by dividing the net mass of concrete by the volume of the measure,  $V_m$  as follows:

$$D = (M_c - M_m) / V_m \quad (2)$$

7.2 *Yield*—Calculate the yield as follows:

$$Y(\text{yd}^3) = M / (D \times 27) \quad (3)$$

or

$$Y(\text{m}^3) = M / D \quad (4)$$

7.3 *Relative Yield*—Relative yield is the ratio of the actual volume of concrete obtained to the volume as designed for the batch (see Note 9) calculated as follows:

$$R_y = Y / Y_d \quad (5)$$

NOTE 9—A value for  $R_y$  greater than 1.00 indicates an excess of concrete being produced whereas a value less than this indicates the batch to be “short” of its designed volume. In practice, a ratio of yield in cubic feet per cubic yard of design concrete mixture is frequently used, for example, 27.3  $\text{ft}^3/\text{yd}^3$ .

7.4 *Cement Content*—Calculate the actual cement content as follows:

$$C = C_b / Y \quad (6)$$

7.5 *Air Content*—Calculate the air content as follows:

$$A = [(T - D) / T] \times 100 \quad (7)$$

or

$$A = [(Y_f - V) / Y_f] \times 100 \text{ (inch-pound units)} \quad (8)$$

or

$$A = [(Y - V) / Y] \times 100 \text{ (SI units)} \quad (9)$$

### 8. Precision and Bias

8.1 The following estimates of precision for this test method are based on a collection of data from various locations by the National Ready Mixed Concrete Association.<sup>4</sup> The data represent concrete mixtures with slump ranging from 3 to 6 in. [75 to 150 mm] and density ranging from 115 to 155  $\text{lb}/\text{ft}^3$  [1842 to 2483  $\text{kg}/\text{m}^3$ ] and included air-entrained and non air-entrained concrete. The study was conducted using 0.25  $\text{ft}^3$  [7-L] and 0.5  $\text{ft}^3$  [14-L] measures.

8.1.1 *Single-Operator Precision*—The single operator standard deviation of density of freshly mixed concrete has been found to be 0.65  $\text{lb}/\text{ft}^3$  [10.4  $\text{kg}/\text{m}^3$ ] (1s). Therefore, results of two properly conducted by the same operator on the same sample of concrete should not differ by more than 1.85  $\text{lb}/\text{ft}^3$  [29.6  $\text{kg}/\text{m}^3$ ] (d2s).

8.1.2 *Multi-Operator Precision*—The multi-operator standard deviation of density of freshly mixed concrete has been found to be 0.82  $\text{lb}/\text{ft}^3$  [13.1  $\text{kg}/\text{m}^3$ ] (1s). Therefore, results of two properly conducted tests by the two operators on the same sample of concrete should not differ by more than 2.31  $\text{lb}/\text{ft}^3$  [37.0  $\text{kg}/\text{m}^3$ ] (d2s).

8.2 *Bias*—This test method has no bias since the density is defined only in terms of this test method.

### 9. Keywords

9.1 air content; cement content; concrete; relative yield; unit weight; yield

<sup>4</sup> Mullings, G. M., NRMCA/NAA Joint Research Lab Study “Series D 324 Accuracy of Concrete Density Test,” Feb. 17, 2000.

## SUMMARY OF CHANGES

This section identifies the location of changes to this test method that have been incorporated since the last issue.

- |  |   |
|--|---|
| <ul style="list-style-type: none"> <li>(1) Designation was revised.</li> <li>(2) Title was revised.</li> <li>(3) Paragraphs 1.1 and 1.2 were revised.</li> <li>(3) Note 1 and paragraph 1.3 were added. Subsequent notes were renumbered.</li> <li>(4) Section 2 was updated.</li> </ul> | <ul style="list-style-type: none"> <li>(5) Section 3 was renamed “Terminology” and revised.</li> <li>(6) Paragraphs 4.1-4.6 were revised.</li> <li>(7) Paragraphs 6.1-6.6 were revised.</li> <li>(8) Equations in Section 7 were revised.</li> <li>(9) Paragraphs 7.1-7.5 were revised.</li> <li>(10) Paragraph 8.1 was revised.</li> </ul> |
|--|---|



# Standard Test Method for Slump of Hydraulic-Cement Concrete<sup>1</sup>

This standard is issued under the fixed designation C 143/C 143M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This test method covers determination of slump of hydraulic-cement concrete, both in the laboratory and in the field.

1.2 The values stated in either inch-pound units or SI units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

## 3. Summary of Test Method

3.1 A sample of freshly mixed concrete is placed and compacted by rodding in a mold shaped as the frustum of a cone. The mold is raised, and the concrete allowed to subside. The vertical distance between the original and displaced position of the center of the top surface of the concrete is measured and reported as the slump of the concrete.

## 4. Significance and Use

4.1 This test method is intended to provide the user with a procedure to determine slump of plastic hydraulic-cement concretes.

NOTE 1—This test method was originally developed to provide a technique to monitor the consistency of unhardened concrete. Under laboratory conditions, with strict control of all concrete materials, the slump is generally found to increase proportionally with the water content of a given concrete mixture, and thus to be inversely related to concrete strength. Under field conditions, however, such a strength relationship is not clearly and consistently shown. Care should therefore be taken in relating slump results obtained under field conditions to strength.

4.2 This test method is considered applicable to plastic concrete having coarse aggregate up to 1 1/2 in. [37.5 mm] in size. If the coarse aggregate is larger than 1 1/2 in. [37.5 mm] in size, the test method is applicable when it is performed on the fraction of concrete passing a 1 1/2-in. [37.5-mm] sieve, with the larger aggregate being removed in accordance with the section titled "Additional Procedure for Large Maximum Size Aggregate Concrete" in Practice C 172.

4.3 This test method is not considered applicable to non-plastic and non-cohesive concrete.

NOTE 2—Concretes having slumps less than 1/2 in. [15 mm] may not be adequately plastic and concretes having slumps greater than about 9 in. [230 mm] may not be adequately cohesive for this test to have significance. Caution should be exercised in interpreting such results.

## 5. Apparatus

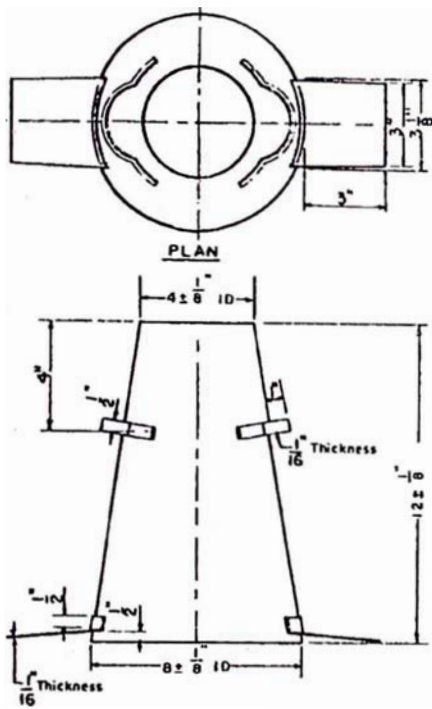
5.1 *Mold*—The test specimen shall be formed in a mold made of metal not readily attacked by the cement paste. The metal shall not be thinner than 0.060 in. [1.5 mm] and if formed by the spinning process, there shall be no point on the mold at which the thickness is less than 0.045 in. [1.15 mm]. The mold shall be in the form of the lateral surface of the frustum of a cone with the base 8 in. [200 mm] in diameter, the top 4 in. [100 mm] in diameter, and the height 12 in. [300 mm]. Individual diameters and heights shall be within  $\pm 1/8$  in. [3 mm] of the prescribed dimensions. The base and the top shall be open and parallel to each other and at right angles to the axis of the cone. The mold shall be provided with foot pieces and handles similar to those shown in Fig. 1. The mold shall be constructed without a seam. The interior of the mold shall be relatively smooth and free from projections. The mold shall be free from dents, deformation or adhered mortar. A mold which clamps to a nonabsorbent base plate is acceptable instead of the one illustrated provided the clamping arrangement is such that it can be fully released without movement of the mold and the base is large enough to contain all of the slumped concrete in an acceptable test.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.60 on Fresh Concrete Testing.

Current edition approved Aug. 10, 2000. Published October 2000. Originally published as D 138 – 22 T. Last previous edition C 143 – 98.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol. 04.02.





		Dimensional Units									
in.	1/16	1/8	1/4	1/2	1	3	3 1/2	4	8	12	
mm	[2]	[3]	[6]	[12]	[25]	[75]	[90]	[100]	[200]	[300]	

FIG. 1 Mold for Slump Test

5.1.1 Mold with alternative materials.

5.1.1.1 Molds other than metal are allowed if the following requirements are met: The mold shall meet the shape, height, and internal dimensional requirements of 5.1. The mold shall be sufficiently rigid to maintain the specified dimensions and tolerances during use, resistant to impact forces, and shall be nonabsorbent. The mold shall be demonstrated to provide test results comparable to those obtained when using a metal mold meeting the requirements of 5.1. Comparability shall be demonstrated on behalf of the manufacturer by an independent testing laboratory. Test for comparability shall consist of not less than 10 pairs of comparisons performed at each of 3 different slumps ranging from 2 in. [50 mm] to 6 in. [150 mm]. No individual test results shall vary by more than 0.50 in. [15 mm] from that obtained using the metal mold. The average test results of each slump range obtained using the mold constructed of alternative material shall not vary by more than 0.30 in. [10 mm] from the average of test results obtained using the metal mold. Manufacturer comparability test data shall be available to users and laboratory inspection authorities (see Note 3). If any changes in material or method of manufacture are made, tests for comparability shall be repeated.

NOTE 3—Because the slump of concrete decreases with time and higher temperatures, it will be advantageous for the comparability tests to be performed by alternating the use of metal cones and alternative material cones, to utilize several technicians, and to minimize the time between test procedures.

5.1.1.2 If the condition of any individual mold is suspected of being out of tolerance from the as manufactured condition,

a single comparative test shall be performed. If the test results differ by more than 0.50 in. [15 mm] from that obtained using the metal mold, the mold shall be removed from service.

5.2 Tamping Rod—A round, straight steel rod 3/8 in. [16 mm] in diameter and approximately 24 in. [600 mm] in length, having the tamping end or both ends rounded to a hemispherical tip, the diameter of which is 5/8 in. [16 mm].

6. Sample

6.1 The sample of concrete from which test specimens are made shall be representative of the entire batch. It shall be obtained in accordance with Practice C 172.

7. Procedure

7.1 Dampen the mold and place it on a flat, moist, nonabsorbent (rigid) surface. It shall be held firmly in place during filling by the operator standing on the two foot pieces. From the sample of concrete obtained in accordance with Section 6, immediately fill the mold in three layers, each approximately one third the volume of the mold.

NOTE 4—One third of the volume of the slump mold fills it to a depth of 2 3/4 in. [70 mm]; two thirds of the volume fills it to a depth of 6 1/4 in. [160 mm].

7.2 Rod each layer with 25 strokes of the tamping rod. Uniformly distribute the strokes over the cross section of each layer. For the bottom layer this will necessitate inclining the rod slightly and making approximately half of the strokes near the perimeter, and then progressing with vertical strokes spirally toward the center. Rod the second layer and the top layer each throughout its depth, so that the strokes just penetrate into the underlying layer.

7.3 In filling and rodding the top layer, heap the concrete above the mold before rodding is started. If the rodding operation results in subsidence of the concrete below the top edge of the mold, add additional concrete to keep an excess of concrete above the top of the mold at all times. After the top layer has been rodded, strike off the surface of the concrete by means of a screeding and rolling motion of the tamping rod. Remove concrete from the area surrounding the base of the slump cone to preclude interference with the movement of slumping concrete. Remove the mold immediately from the concrete by raising it carefully in a vertical direction. Raise the mold a distance of 12 in. [300 mm] in 5 ± 2 s by a steady upward lift with no lateral or torsional motion. Complete the entire test from the start of the filling through removal of the mold without interruption and complete it within an elapsed time of 2 1/2 min.

7.4 Immediately measure the slump by determining the vertical difference between the top of the mold and the displaced original center of the top surface of the specimen. If a decided falling away or shearing off of concrete from one side or portion of the mass occurs (Note 5), disregard the test and make a new test on another portion of the sample.

NOTE 5—If two consecutive tests on a sample of concrete show a falling away or shearing off of a portion of the concrete from the mass of the specimen, the concrete probably lacks necessary plasticity and cohesiveness for the slump test to be applicable.

8. Report

8.1 Report the slump in terms of inches [millimetres] to the nearest 1/4 in. [5 mm] of subsidence of the specimen during the test.

9. Precision and Bias<sup>3</sup>

9.1 Precision—The estimates of precision for this test method are based upon results from tests conducted in Fayetteville, Arkansas by 15 technicians from 14 laboratories representing 3 states. All tests at 3 different slump ranges, from 1.0 in. [25 mm] to 6.5 in. [160 mm], were performed using one load of truck-mixed concrete. The concrete was delivered and tested at a low slump, with water then being added and mixed into the remaining concrete to independently produce moderate and finally high-slump concrete. The concrete mixture that used a No. 67 crushed limestone aggregate and a washed river sand, contained 500 lb of cementitious materials per cubic yard [297 kg of cementitious material per cubic metre]. The 500 lb [227 kg] were equally divided between a C150, Type I/II cement and a Class C fly ash. A double dosage of a chemical retarder was used in an attempt to minimize slump losses and maintain workability of the concrete. Concrete temperatures ranged from 86°F [30°C] to 93°F [34°C]. Slump losses averaged 0.68 in. [17 mm] during the 20 min required to perform a series of 6 tests at 1 slump range. Testing was performed alternately using metal and plastic slump cones, which were determined to produce comparable results. Precision data thus applies to both metal and plastic molds. A total of 270 slump tests were performed.

9.1.1 Inch-Pound [SI]—The data used to develop the precision statement were obtained using metric units (millimetres). The precision values shown in inch-pound units are conversions from the millimetre measurements, which were recorded to the nearest 1 mm.

9.1.2 Measure of Variability—The standard deviation was determined to be the most consistent measure of variability and was found to vary with the slump value.

<sup>3</sup>The test data used to develop this precision statement were based on tests performed in September 1997. A report of test results is on file at ASTM Headquarters. Request RR: C09-1022.

9.1.3 Single-Operator Precision—The single-operator standard deviation represented by (1s) is shown in Table 1 by average slump values. The reported results for the replicate readings apply to tests conducted by the same operator performing successive tests, one immediately following the other. Acceptable results of two properly conducted tests by the same operator on the same material (Note 6) will not differ from each other by more than the (d2s) value of the last column of Table 1 for the appropriate slump value and single-operator precision.

9.1.4 Multilaboratory Precision—The multilaboratory standard deviation represented by (1s) is shown in Table 1 by average slump values. The reported results for the replicate readings apply to tests conducted by different operators from different laboratories performing tests less than 4 min apart. Therefore, acceptable results of two properly conducted slump tests on the same material (Note 6) by two different laboratories will not differ from each other by more than the (d2s) value of the last column of Table 1 for the appropriate slump value and multilaboratory precision.

NOTE 6—“Same materials,” is used to mean freshly mixed concrete from one batch.

9.2 Bias—This test method has no bias since slump is defined only in terms of this test method.

10. Keywords

10.1 concrete; cone; consistency; plasticity; slump; workability

TABLE 1 Precision

Slump and Type Index	Standard Deviation (1s) <sup>A</sup>		Acceptable Range of Two Results (d2s) <sup>A</sup>	
	in.	[mm]	in.	[mm]
<i>Single-Operator Precision:</i>				
Slump 1.2 in. [30 mm]	0.23	[6]	0.65	[17]
Slump 3.4 in. [85 mm]	0.38	[9]	1.07	[25]
Slump 6.5 in. [160 mm]	0.40	[10]	1.13	[28]
<i>Multilaboratory Precision:</i>				
Slump 1.2 in. [30 mm]	0.29	[7]	0.82	[20]
Slump 3.4 in. [85 mm]	0.39	[10]	1.10	[28]
Slump 6.5 in. [160 mm]	0.53	[13]	1.50	[37]

<sup>A</sup>These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.

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# Standard Specification for Portland Cement<sup>1</sup>

This standard is issued under the fixed designation C 150; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This specification covers eight types of portland cement, as follows (see **Note 2**):

1.1.1 *Type I*—For use when the special properties specified for any other type are not required.

1.1.2 *Type IA*—Air-entraining cement for the same uses as Type I, where air-entrainment is desired.

1.1.3 *Type II*—For general use, more especially when moderate sulfate resistance or moderate heat of hydration is desired.

1.1.4 *Type IIA*—Air-entraining cement for the same uses as Type II, where air-entrainment is desired.

1.1.5 *Type III*—For use when high early strength is desired.

1.1.6 *Type IIIA*—Air-entraining cement for the same use as Type III, where air-entrainment is desired.

1.1.7 *Type IV*—For use when a low heat of hydration is desired.

1.1.8 *Type V*—For use when high sulfate resistance is desired.

**NOTE 1**—Some cements are designated with a combined type classification, such as *Type IIII*, indicating that the cement meets the requirements of the indicated types and is being offered as suitable for use when either type is desired.

1.2 When both SI and inch-pound units are present, the SI units are the standard. The inch-pound units are approximations listed for information only.

1.3 The text of this standard references notes and footnotes<sup>2</sup> which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

C 33 Specification for Concrete Aggregates

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C 114 Test Methods for Chemical Analysis of Hydraulic Cement

C 115 Test Method for Fineness of Portland Cement by the Turbidimeter

C 151 Test Method for Autoclave Expansion of Hydraulic Cement

C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement

C 185 Test Method for Air Content of Hydraulic Cement Mortar

C 186 Test Method for Heat of Hydration of Hydraulic Cement

C 191 Test Method for Time of Setting of Hydraulic Cement by Vicat Needle

C 204 Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus

C 219 Terminology Relating to Hydraulic Cement

C 226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Hydraulic Cement

C 266 Test Method for Time of Setting of Hydraulic Cement Paste by Gillmore Needles

C 451 Test Method for Early Stiffening of Hydraulic Cement (Paste Method)

C 452 Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate

C 465 Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements

C 563 Test Method for Optimum SO<sub>3</sub> in Hydraulic Cement Using 24-h Compressive Strength

C 1038 Test Method for Expansion of Hydraulic Cement Mortar Bars Stored in Water

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

## 3. Terminology

3.1 *Definitions*—See Terminology C 219.

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.10 on Hydraulic Cements for General Concrete Construction.

Current edition approved July 15, 2005. Published August 2005. Originally approved in 1940. Last previous edition approved in 2004 as C 150 – 04a<sup>1</sup>.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**4. Ordering Information**

4.1 Orders for material under this specification shall include the following:

- 4.1.1 This specification number and date,
- 4.1.2 Type or types allowable. If no type is specified, Type I shall be supplied,
- 4.1.3 Any optional chemical requirements from Table 2, if desired, and
- 4.1.4 Any optional physical requirements from Table 4, if desired.

NOTE 2—Cement conforming to the requirements for all types are not carried in stock in some areas. In advance of specifying the use of cement other than Type I, determine whether the proposed type of cement is, or can be made, available.

**5. Additions**

5.1 The cement covered by this specification shall contain no addition except as follows:

5.1.1 Water or calcium sulfate, or both, if added, shall be in amounts such that the limits shown in Table 1 for sulfur trioxide and loss-on-ignition are not exceeded.

5.1.2 Processing additions used in the manufacture of the cement shall have been shown to meet the requirements of Specification C 465 in the amounts used or greater.

5.1.3 Up to 5.0 % limestone by mass is permitted in amounts such that the chemical and physical requirements of this standard are met (See Note 3). The limestone shall be naturally occurring, consisting of at least 70 % by mass of one or more of the mineral forms of calcium carbonate.

NOTE 3—The standard permits up to 5 % by mass of the final cement product to be naturally occurring, finely ground limestone, but does not require that limestone be added to the cement. Cement without ground limestone can be specified in the contract or order.

5.1.4 Air-entraining portland cement shall contain an inter-ground addition conforming to the requirements of Specification C 226.

**6. Chemical Composition**

6.1 Portland cement of each of the eight types shown in Section 1 shall conform to the respective standard chemical requirements prescribed in Table 1. In addition, optional chemical requirements are shown in Table 2.

**7. Physical Properties**

7.1 Portland cement of each of the eight types shown in Section 1 shall conform to the respective standard physical requirements prescribed in Table 3. In addition, optional physical requirements are shown in Table 4.

**8. Sampling**

8.1 When the purchaser desires that the cement be sampled and tested to verify compliance with this specification, perform sampling and testing in accordance with Practice C 183.

8.2 Practice C 183 is not designed for manufacturing quality control and is not required for manufacturer’s certification.

**9. Test Methods**

9.1 Determine the applicable properties enumerated in this specification in accordance with the following test methods:

- 9.1.1 Air Content of Mortar—Test Method C 185.
- 9.1.2 Chemical Analysis—Test Methods C 114.
- 9.1.3 Strength—Test Method C 109/C 109M.
- 9.1.4 False Set—Test Method C 451.
- 9.1.5 Fineness by Air Permeability—Test Method C 204.
- 9.1.6 Fineness by Turbidimeter—Test Method C 115.
- 9.1.7 Heat of Hydration—Test Method C 186.
- 9.1.8 Autoclave Expansion—Test Method C 151.
- 9.1.9 Time of Setting by Gillmore Needles—Test Method C 266.
- 9.1.10 Time of Setting by Vicat Needles—Test Method C 191.
- 9.1.11 Sulfate Resistance—Test Method C 452 (sulfate expansion).

**TABLE 1 Standard Composition Requirements**

Cement Type <sup>A</sup>	Applicable Test Method	I and IA	II and IIA	III and IIIA	IV	V
Aluminum oxide (Al <sub>2</sub> O <sub>3</sub> ), max, %	C 114	...	6.0	...	...	...
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ), max, %	C 114	...	6.0 <sup>B,C</sup>	...	6.5	...
Magnesium oxide (MgO), max, %	C 114	6.0	6.0	6.0	6.0	6.0
Sulfur trioxide (SO <sub>3</sub> ), <sup>D</sup> max, %	C 114	...	...	...	...	...
When (C <sub>3</sub> A) <sup>E</sup> is 8 % or less		3.0	3.0	3.5	2.3	2.3
When (C <sub>3</sub> A) <sup>E</sup> is more than 8 %		3.5	<sup>F</sup>	4.5	<sup>F</sup>	<sup>F</sup>
Loss on ignition, max, %	C 114	3.0	3.0	3.0	2.5	3.0
Insoluble residue, max, %	C 114	0.75	0.75	0.75	0.75	0.75
Tricalcium silicate (C <sub>3</sub> S) <sup>E</sup> , max, %	See Annex A1	...	...	...	35 <sup>B</sup>	...
Dicalcium silicate (C <sub>2</sub> S) <sup>E</sup> , min, %	See Annex A1	...	...	...	40 <sup>B</sup>	...
Tricalcium aluminate (C <sub>3</sub> A) <sup>E</sup> , max, %	See Annex A1	...	8	15	7 <sup>B</sup>	5 <sup>C</sup>
Tetracalcium aluminoferrite plus twice the tricalcium aluminate (C <sub>4</sub> AF + 2(C <sub>3</sub> A)), or solid solution (C <sub>4</sub> AF + C <sub>2</sub> F), as applicable, max, %	See Annex A1	...	...	...	...	25 <sup>C</sup>

<sup>A</sup> See Note 2.

<sup>B</sup> Does not apply when the heat of hydration limit in Table 4 is specified.

<sup>C</sup> Does not apply when the sulfate resistance limit in Table 4 is specified.

<sup>D</sup> There are cases where optimum SO<sub>3</sub> (using Test Method C 563) for a particular cement is close to or in excess of the limit in this specification. In such cases where properties of a cement can be improved by exceeding the SO<sub>3</sub> limits stated in this table, it is permissible to exceed the values in the table, provided it has been demonstrated by Test Method C 1038 that the cement with the increased SO<sub>3</sub> will not develop expansion in water exceeding 0.020 % at 14 days. When the manufacturer supplies cement under this provision, he shall, upon request, supply supporting data to the purchaser.

<sup>E</sup> See Annex A1 for calculation.

<sup>F</sup> Not applicable.

**TABLE 2 Optional Composition Requirements<sup>A</sup>**

Cement Type	Applicable Test Method	I and IA	II and IIA	III and IIIA	IV	V	Remarks
Tricalcium aluminate (C <sub>3</sub> A) <sup>B</sup> , max, %	See Annex A1	...	...	8	...	...	for moderate sulfate resistance
Tricalcium aluminate (C <sub>3</sub> A) <sup>B</sup> , max, %	See Annex A1	...	...	5	...	...	for high sulfate resistance
Sum of tricalcium silicate and tricalcium aluminate (C <sub>3</sub> S + C <sub>3</sub> A), max, %	See Annex A1	...	58 <sup>C</sup>	...	...	...	for moderate heat of hydration
Equivalent alkalies (Na <sub>2</sub> O + 0.658K <sub>2</sub> O), max, %	C 114	0.60 <sup>D</sup>	0.60 <sup>D</sup>	0.60 <sup>D</sup>	0.60 <sup>D</sup>	0.60 <sup>D</sup>	low-alkali cement

<sup>A</sup> These optional requirements apply only when specifically requested. Verify availability before ordering. See Note 2 in Section 4.

<sup>B</sup> See Annex A1 for calculation.

<sup>C</sup> The optional limit for heat of hydration in Table 4 shall not be requested when this optional limit is requested.

<sup>D</sup> Specify this limit when the cement is to be used in concrete with aggregates that are potentially reactive and no other provisions have been made to protect the concrete from deleteriously reactive aggregates. Refer to Specification C 33 for information on potential reactivity of aggregates.

**TABLE 3 Standard Physical Requirements**

Cement Type <sup>A</sup>	Applicable Test Method	I	IA	II	IIA	III	IIIA	IV	V
Air content of mortar, <sup>B</sup> volume %:	C 185								
max		12	22	12	22	12	22	12	12
min		...	16	...	16	...	16	...	...
Fineness, <sup>C</sup> specific surface, m <sup>2</sup> /kg (alternative methods):									
Turbidimeter test, min	C 115	160	160	160	160	...	...	160	160
Air permeability test, min	C 204	280	280	280	280	...	...	280	280
Autoclave expansion, max, %	C 151	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80
Strength, not less than the values shown for the ages indicated as follows: <sup>D</sup>									
Compressive strength, MPa (psi):	C 109/ C 109M								
1 day		...	...	...	...	12.0 (1740)	10.0 (1450)	...	...
3 days		12.0 (1740)	10.0 (1450)	10.0 (1450) 7.0 <sup>E</sup> (1020) <sup>E</sup>	8.0 (1160) 6.0 <sup>E</sup> (870) <sup>E</sup>	24.0 (3480)	19.0 (2760)	...	8.0 (1160)
7 days		19.0 (2760)	16.0 (2320)	17.0 (2470) 12.0 <sup>E</sup> (1740) <sup>E</sup>	14.0 (2030) 9.0 <sup>E</sup> (1310) <sup>E</sup>	...	...	7.0 (1020)	15.0 (2180)
28 days		...	...	...	...	...	...	17.0 (2470)	21.0 (3050)
Time of setting; Vicat test: <sup>F</sup>	C 191								
Time of setting, min, not less than		45	45	45	45	45	45	45	45
Time of setting, min, not more than		375	375	375	375	375	375	375	375

<sup>A</sup> See Note 2.

<sup>B</sup> Compliance with the requirements of this specification does not necessarily ensure that the desired air content will be obtained in concrete.

<sup>C</sup> The testing laboratory shall select the fineness method to be used. However, when the sample fails to meet the requirements of the air-permeability test, the turbidimeter test shall be used, and the requirements in this table for the turbidimetric method shall govern.

<sup>D</sup> The strength at any specified test age shall be not less than that attained at any previous specified test age.

<sup>E</sup> When the optional heat of hydration or the chemical limit on the sum of the tricalcium silicate and tricalcium aluminate is specified.

<sup>F</sup> The time of setting is that described as initial setting time in Test Method C 191.

9.1.12 *Calcium Sulfate (expansion of) Mortar*—Test Method C 1038.

9.1.13 *Optimum SO<sub>3</sub>*—Test Method C 563.

## 10. Inspection

10.1 Inspection of the material shall be made as agreed upon between the purchaser and the seller as part of the purchase contract.

## 11. Rejection

11.1 The cement shall be rejected if it fails to meet any of the requirements of this specification.

11.2 At the option of the purchaser, retest, before using, cement remaining in bulk storage for more than 6 months or cement in bags in local storage in the custody of a vendor for

more than 3 months after completion of tests and reject the cement if it fails to conform to any of the requirements of this specification. Cement so rejected shall be the responsibility of the owner of record at the time of resampling for retest.

11.3 Packages shall identify the mass contained as net weight. At the option of the purchaser, packages more than 2 % below the mass marked thereon shall be rejected and if the average mass of packages in any shipment, as shown by determining the mass of 50 packages selected at random, is less than that marked on the packages, the entire shipment shall be rejected.

## 12. Manufacturer's Statement

12.1 At the request of the purchaser, the manufacturer shall state in writing the nature, amount, and identity of any

**TABLE 4 Optional Physical Requirements<sup>A</sup>**

Cement Type	Applicable Test Method	I	IA	II	IIA	III	IIIA	IV	V
False set, final penetration, min, %	C 451	50	50	50	50	50	50	50	50
Heat of hydration:	C 186								
7 days, max, kJ/kg (cal/g)		...	...	290 (70) <sup>B</sup>	290 (70) <sup>B</sup>	...	...	250 (60) <sup>C</sup>	...
28 days, max, kJ/kg (cal/g)		...	...	...	...	...	...	290 (70) <sup>C</sup>	...
Strength, not less than the values shown:									
Compressive strength, MPa (psi)	C 109/ C 109M								
28 days		28.0 (4060)	22.0 (3190)	28.0 (4060)	22.0 (3190)	...	...	...	...
Sulfate resistance, <sup>D</sup> 14 days, max, % expansion	C 452	...	...	22.0 <sup>B</sup> (3190) <sup>B</sup>	18.0 <sup>B</sup> (2610) <sup>B</sup>	...	...	...	0.040
Gillmore test:	C 266			...	...	...	...	...	...
Initial set, min, not less than		60	60	60	60	60	60	60	60
Final set, min, not more than		600	600	600	600	600	600	600	600

<sup>A</sup> These optional requirements apply only when specifically requested. Verify availability before ordering. See Note 2 in Section 4.

<sup>B</sup> The optional limit for the sum of the tricalcium silicate and tricalcium aluminate in Table 2 shall not be requested when this optional limit is requested. These strength requirements apply when either heat of hydration or the sum of tricalcium silicate and tricalcium aluminate requirements are requested.

<sup>C</sup> When the heat of hydration limit is specified, it shall be instead of the limits of C<sub>3</sub>S, C<sub>2</sub>S, C<sub>3</sub>A, SiO<sub>2</sub>, and Fe<sub>2</sub>O<sub>3</sub> listed in Table 1.

<sup>D</sup> When the sulfate resistance is specified, it shall be instead of the limits of C<sub>3</sub>A, C<sub>4</sub>AF + 2 C<sub>3</sub>A, SiO<sub>2</sub>, and Fe<sub>2</sub>O<sub>3</sub> listed in Table 1.

<sup>E</sup> Cement meeting the high sulfate resistance limit for Type V is deemed to meet the moderate sulfate resistance requirement of Type II.

air-entraining addition and of any processing addition used, and also, if requested, shall supply test data showing compliance of such air-entraining addition with Specification C 226 and of such processing addition with Specification C 465.

12.2 When limestone is used, the manufacturer shall state in writing the amount thereof and, if requested by the purchaser, shall supply comparative test data on chemical and physical properties of the cement with and without the limestone (See Note 4). The comparative tests do not supersede the normal testing to confirm that the cement meets chemical and physical requirements of this standard. The amount of limestone in cement shall be determined in accordance with Annex A2.

Note 4—Comparative test data may be from qualification tests performed by the manufacturer during formulation of the cement with limestone.

### 13. Packaging and Package Marking

13.1 When the cement is delivered in packages, the words “Portland Cement,” the type of cement, the name and brand of the manufacturer, and the mass of the cement contained therein shall be plainly marked on each package. When the cement is an air-entraining type, the words “air-entraining” shall be plainly marked on each package. Similar information shall be provided in the shipping documents accompanying the shipment of packaged or bulk cement. All packages shall be in good condition at the time of inspection.

Note 5—With the change to SI units, it is desirable to establish a standard SI package for portland cements. To that end 42 kg (92.6 lb) provides a convenient, even-numbered mass reasonably similar to the traditional 94-lb (42.6-kg) package.

### 14. Storage

14.1 The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building that will protect the cement from dampness and minimize warehouse set.

### 15. Manufacturer’s Certification

15.1 Upon request of the purchaser in the contract or order, a manufacturer’s report shall be furnished at the time of shipment stating the results of tests made on samples of the material taken during production or transfer and certifying that the cement conforms to applicable requirements of this specification.

Note 6—Guidance on preparing the manufacturer’s report is provided in Appendix XI.

### 16. Keywords

16.1 hydraulic cement; portland cement; specification

(Mandatory Information)

**A1. CALCULATION OF POTENTIAL CEMENT PHASE COMPOSITION**

A1.1 All values calculated as described in this annex shall be rounded according to Practice E 29. When evaluating conformance to a specification, round values to the same number of places as the corresponding table entry before making comparisons. The expressing of chemical limitations by means of calculated assumed phases does not necessarily mean that the oxides are actually or entirely present as such phases.

A1.2 When expressing phases, C = CaO, S = SiO<sub>2</sub>, A = Al<sub>2</sub>O<sub>3</sub>, F = Fe<sub>2</sub>O<sub>3</sub>. For example, C<sub>3</sub>A = 3CaO·Al<sub>2</sub>O<sub>3</sub>. Titanium dioxide and phosphorus pentoxide (TiO<sub>2</sub> and P<sub>2</sub>O<sub>5</sub>) shall not be included with the Al<sub>2</sub>O<sub>3</sub> content. See Note A1.1.

NOTE A1.1—When comparing oxide analyses and calculated phases from different sources or from different historic times, be aware that they may not have been reported on exactly the same basis. Chemical data obtained by Reference and Alternate Test Methods of Test Methods C 114 (wet chemistry) may include titania and phosphorus as alumina unless proper correction has been made (see Test Methods C 114), while data obtained by rapid instrumental methods usually do not. This can result in small differences in the calculated phases. Such differences are usually within the precision of the analytical methods, even when the methods are properly qualified under the requirements of Test Methods C 114.

A1.3 When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite shall be calculated from the chemical analysis as follows:

$$\begin{aligned} \text{Tricalcium silicate (C}_3\text{S)} = & (4.071 \times \% \text{ CaO}) - (7.600 \times \% \text{ SiO}_2) - \\ & (6.718 \times \% \text{ Al}_2\text{O}_3) - (1.430 \times \% \text{ Fe}_2\text{O}_3) - \\ & (2.852 \times \% \text{ SO}_3) - (5.188 \times \% \text{ CO}_2) \end{aligned} \quad (\text{A1.1})$$

$$\text{Dicalcium silicate (C}_2\text{S)} = (2.867 \times \% \text{ SiO}_2) - (0.7544 \times \% \text{ C}_3\text{S}) \quad (\text{A1.2})$$

$$\text{Tricalcium aluminate (C}_3\text{A)} = (2.650 \times \% \text{ Al}_2\text{O}_3) - (1.692 \times \% \text{ Fe}_2\text{O}_3) \quad (\text{A1.3})$$

$$\text{Tetracalcium aluminoferrite (C}_4\text{AF)} = 3.043 \times \% \text{ Fe}_2\text{O}_3 \quad (\text{A1.4})$$

Unless limestone is used in the cement, the carbon dioxide content shall be considered to be equal to zero when calculating potential tricalcium silicate. In the absence of information on the limestone content of the cement sample, results shall note that no correction has been made for possible use of limestone.

A1.3.1 When the alumina-ferric oxide ratio is less than 0.64, a calcium aluminoferrite solid solution (expressed as ss(C<sub>4</sub>AF + C<sub>2</sub>F)) is formed. No tricalcium aluminate will be present in cements of this composition. Dicalcium silicate shall be calculated as in Eq A1.2. Contents of this solid solution and of tricalcium silicate shall be calculated by the following formulas:

$$\text{ss (C}_4\text{AF} + \text{C}_2\text{F)} = (2.100 \times \% \text{ Al}_2\text{O}_3) + (1.702 \times \% \text{ Fe}_2\text{O}_3) \quad (\text{A1.5})$$

$$\begin{aligned} \text{Tricalcium silicate (C}_3\text{S)} = & (4.071 \times \% \text{ CaO}) - (7.600 \times \% \text{ SiO}_2) - \\ & (4.479 \times \% \text{ Al}_2\text{O}_3) - (2.859 \times \% \text{ Fe}_2\text{O}_3) - \\ & (2.852 \times \% \text{ SO}_3) - (5.188 \times \% \text{ CO}_2) \end{aligned} \quad (\text{A1.6})$$

Unless limestone is used in the cement, the carbon dioxide content shall be considered to be equal to zero when calculating potential tricalcium silicate. In the absence of information on the limestone content of the cement sample, results shall note that no correction has been made for possible use of limestone.

**A2. LIMESTONE CONTENT OF PORTLAND CEMENT**

A2.1 When limestone is used, the limestone content in portland cement shall be derived from the determination of CO<sub>2</sub> in the finished cement. Analysis of CO<sub>2</sub> shall be based on methods described in Test Methods C 114. The percent limestone in the cement is calculated from the CO<sub>2</sub> analysis based on the CO<sub>2</sub> content of the limestone used.

The manufacturer shall include the CO<sub>2</sub> content and calculated limestone content of the cement on the Mill Test Report. The limestone content of the cement is calculated as follows:

$$\frac{\% \text{ CO}_2 \text{ in the cement}}{\% \text{ CO}_2 \text{ in the limestone}} \times 100 = \% \text{ limestone in cement}$$

NOTE A2.1—For example:

Where the determined CO<sub>2</sub> content in the finished cement = 1.5 % and the CO<sub>2</sub> content of the limestone = 43 % (CaCO<sub>3</sub> in limestone = 98 %) Then:

$$\frac{1.5}{43} \times 100 = 3.5 \% \text{ limestone content in cement}$$

A2.2 This specification requires that the limestone to be used must contain a minimum of 70 % CaCO<sub>3</sub>. The manufacturer shall include the CaCO<sub>3</sub> content of the limestone on the manufacturer's report. Calculate the CaCO<sub>3</sub> content of the limestone as follows: % CaCO<sub>3</sub> = 2.274 × % CO<sub>2</sub>.

NOTE A2.2—For verification of limestone content of cement, the purchaser must analyze for CO<sub>2</sub> content and make a correction for the amount of CaCO<sub>3</sub> in the limestone in order for the data to be comparable to the manufacturer's report.

A2.3 Portland cements that do not contain limestone can maintain baseline levels of CO<sub>2</sub> inherent in manufacture, for

example, due to carbonation. This baseline CO<sub>2</sub> content is included as part of any calculated limestone content.

## APPENDIX

### (Nonmandatory Information)

#### X1. MANUFACTURER'S CERTIFICATION (MILL TEST REPORT)

X1.1 To provide uniformity for reporting the results of tests performed on cements under this specification, as required by Section 15 of Specification C 150 entitled "Manufacturer's Certification," an example Mill Test Report is shown in Fig. 1.1.

X1.2 The identity information given should unambiguously identify the cement production represented by the Mill Test Report and may vary depending upon the manufacturer's designation and purchaser's requirements.

X1.3 The Manufacturer's Certification statement may vary depending upon the manufacturer's procurement order, or legal requirements, but should certify that the cement shipped is presented by the certificate and that the cement conforms to applicable requirements of the specification at the time it was tested (or retested) or shipped.

X1.4 The sample Mill Test Report has been developed to reflect the chemical and physical requirements of this specification and recommends reporting all analyses and tests normally performed on cements meeting Specification C 150. Purchaser reporting requirements should govern if different from normal reporting by the manufacturer or from those recommended here.

X1.5 Cements may be shipped prior to later-age test data being available. In such cases, the test value may be left blank. Alternatively, the manufacturer can generally provide estimates based on historical production data. The report should indicate if such estimates are provided.

X1.6 In reporting limits from the tables in Specification C 150 on the Mill Test Report, only those limits specifically applicable should be listed. In some cases, Specification C 150 table limits are superseded by other provisions.



**ASTM C 150 – 05**

ABC Portland Cement Company  
Qualitytown, N.J.

Plant Example

Cement Type II

Date March 9, 1998

Production Period March 2, 1998 – March 8, 1998

**STANDARD REQUIREMENTS**  
ASTM C 150 Tables 1 and 3

CHEMICAL			PHYSICAL		
Item	Spec. Limit	Test Result	Item	Spec. Limit	Test Result
SiO <sub>2</sub> (%)	<sup>A</sup>	20.6	Air content of mortar (volume %)	12 max	8
Al <sub>2</sub> O <sub>3</sub> (%)	6.0 max	4.4	Blaine fineness (m <sup>2</sup> /kg)	280 min	377
Fe <sub>2</sub> O <sub>3</sub> (%)	6.0 max	3.3	Autoclave expansion (%)	0.80 max	0.04
CaO (%)	<sup>A</sup>	62.9	Compressive strength (MPa)	min:	
MgO (%)	6.0 max	2.2	1 day	<sup>A</sup>	
SO <sub>3</sub> (%)	3.0 max	2.7	3 days	7.0	23.4
Ignition loss (%)	3.0 max	2.7	7 days	12.0	29.8
Na <sub>2</sub> O (%)	<sup>A</sup>	0.19	28 days	<sup>A</sup>	
K <sub>2</sub> O (%)	<sup>A</sup>	0.50	Time of setting (minutes)		
Insoluble residue (%)	0.75 max	0.27	(Vicat)		
CO <sub>2</sub> (%)	<sup>A</sup>	1.5	Initial Not less than	45	124
Limestone (%)	5.0 max	3.5	Not more than	375	
CaCO <sub>3</sub> in limestone (%)	70 min	98			
Potential (%)					
C <sub>3</sub> S	<sup>A</sup>	50			
C <sub>2</sub> S	<sup>A</sup>	21			
C <sub>3</sub> A	8 max	6			
C <sub>4</sub> AF	<sup>A</sup>	10			
C <sub>4</sub> AF + 2(C <sub>3</sub> A)	<sup>A</sup>	22			

<sup>A</sup>Not applicable.

**OPTIONAL REQUIREMENTS**  
ASTM C 150 Tables 2 and 4

CHEMICAL			PHYSICAL		
Item	Spec. Limit	Test Result	Item	Spec. Limit	Test Result
C <sub>3</sub> S + C <sub>3</sub> A (%)	58 max	56	False set (%)	50 min	82
Equivalent alkalis (%)	<sup>B</sup>	0.52	Heat of hydration (kJ/kg)		
			7 days	<sup>B</sup>	300
			Compressive strength (MPa)		
			28 days	28.0 min	39.7

mit not specified by purchaser. Test result provided for information only.

We certify that the above described cement, at the time of shipment, meets the chemical and physical requirements of the ASTM C 150 – XX or (other) \_\_\_\_\_ specification.

Signature: \_\_\_\_\_

Title: \_\_\_\_\_

**FIG. X1.1 Example Mill Test Report**

 **C 150 – 05**

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## Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method<sup>1</sup>

This standard is issued under the fixed designation C 231; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

<sup>ε1</sup> NOTE—Section 1.4 was updated editorially in April 1999.

### 1. Scope

1.1 This test method covers determination of the air content of freshly mixed concrete from observation of the change in volume of concrete with a change in pressure.

1.2 This test method is intended for use with concretes and mortars made with relatively dense aggregates for which the aggregate correction factor can be satisfactorily determined by the technique described in Section 6. It is not applicable to concretes made with lightweight aggregates, air-cooled blast-furnace slag, or aggregates of high porosity. In these cases, Test Method C 173 should be used. This test method is also not applicable to nonplastic concrete such as is commonly used in the manufacture of pipe and concrete masonry units.

1.3 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of this standard.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Note A1.7 for a specific caution statement.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 138 Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete<sup>2</sup>

C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>

C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method<sup>2</sup>

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory<sup>2</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods of Construction Materials<sup>2</sup>

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>3</sup>

### 3. Significance and Use

3.1 This test method covers the determination of the air content of freshly mixed concrete. The test determines the air content of freshly mixed concrete exclusive of any air that may be inside voids within aggregate particles. For this reason, it is applicable to concrete made with relatively dense aggregate particles and requires determination of the aggregate correction factor (see 6.1 and 9.1).

3.2 This test method and Test Method C 138 and C 173 provide pressure, gravimetric, and volumetric procedures, respectively, for determining the air content of freshly mixed concrete. The pressure procedure of this test method gives substantially the same air contents as the other two test methods for concretes made with dense aggregates.

3.3 The air content of hardened concrete may be either higher or lower than that determined by this test method. This depends upon the methods and amount of consolidation effort applied to the concrete from which the hardened concrete specimen is taken; uniformity and stability of the air bubbles in the fresh and hardened concrete; accuracy of the microscopic examination, if used; time of comparison; environmental exposure; stage in the delivery, placement and consolidation processes at which the air content of the unhardened concrete is determined, that is, before or after the concrete goes through a pump; and other factors.

### 4. Apparatus

4.1 *Air Meters*—There are available satisfactory apparatus of two basic operational designs employing the principle of Boyle's law. For purposes of reference herein these are designated Meter Type A and Meter Type B.

4.1.1 *Meter Type A*—An air meter consisting of a measuring bowl and cover assembly (see Fig. 1) conforming to the

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates, and is the direct responsibility of Subcommittee C09.60 on Fresh Concrete Testing.

Current edition approved Jan. 10, 1997. Published March 1997. Originally published as C 231 – 49 T. Last previous edition C 231 – 97.

<sup>2</sup> Annual Book of ASTM Standards, Vol 04.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

4.4 *Calibration Vessel*—A measure having an internal volume equal to a percent of the volume of the measuring bowl corresponding to the approximate percent of air in the concrete to be tested; or, if smaller, it shall be possible to check calibration of the meter indicator at the approximate percent of air in the concrete to be tested by repeated filling of the measure. When the design of the meter requires placing the calibration vessel within the measuring bowl to check calibration, the measure shall be cylindrical in shape and of an inside depth ½ in. (13 mm) less than that of the bowl.

NOTE 1—A satisfactory calibration vessel to place within the measure bowl may be machined from No. 16 gage brass tubing, of a diameter to provide the volume desired, to which a brass disk ½ in. in thickness is soldered to form an end. When design of the meter requires withdrawing of water from the water-filled bowl and cover assembly to check calibration, the measure may be an integral part of the cover assembly or may be a separate cylindrical measure similar to the above described cylinder.

4.5 The designs of various available types of airmeters are such that they differ in operating techniques and therefore, all of the items described in 4.6-4.16 may not be required. The items required shall be those necessary for use with the particular design of apparatus used to satisfactorily determine air content in accordance with the procedures prescribed herein.

4.6 *Coil Spring or Other Device for Holding Calibration Cylinder in Place.*

4.7 *Spray Tube*—A brass tube of appropriate diameter, which may be an integral part of the cover assembly or which may be provided separately. It shall be so constructed that when water is added to the container, it is sprayed to the walls of the cover in such a manner as to flow down the sides causing a minimum of disturbance to the concrete.

4.8 *Trowel*—A standard brick mason's trowel.

4.9 *Tamping Rod*—The tamping rod shall be a round straight steel rod ⅝ in. (16 mm) in diameter and not less than 16 in. (400 mm) in length, having the tamping end rounded to a hemispherical tip the diameter of which is ⅝ in. (16 mm).

4.10 *Mallet*—A mallet (with a rubber or rawhide head) weighing approximately 1.25 ± 0.50 lb (0.57 ± 0.23 kg) for use with measures of 0.5 ft<sup>3</sup> (14 dm<sup>3</sup>) or smaller, and a mallet weighing approximately 2.25 ± 0.50 lb (1.02 ± 0.23 kg) for use with measures larger than 0.5 ft<sup>3</sup>.

4.11 *Strike-Off Bar*—A flat straight bar of steel or other suitable metal at least ⅛ in. (3 mm) thick and ¾ in. (20 mm) wide by 12 in. (300 mm) long.

4.12 *Strike-Off Plate*—A flat rectangular metal plate at least ¼ in. (6 mm) thick or a glass or acrylic plate at least ½ in. (12 mm) thick with a length and width at least 2 in. (50 mm) greater than the diameter of the measure with which it is to be used. The edges of the plate shall be straight and smooth within a tolerance of ⅛ in. (1.5 mm).

4.13 *Funnel*, with the spout fitting into spray tube.

4.14 *Measure for Water*, having the necessary capacity to fill the indicator with water from the top of the concrete to the zero mark.

4.15 *Vibrator*, as described in Practice C 192.

4.16 *Sieves*, 1½-in. (37.5-mm) with not less than 2 ft<sup>2</sup> (0.19 m<sup>2</sup>) of sieving area.

## 5. Calibration of Apparatus

5.1 Make calibration tests in accordance with procedures prescribed in the annex. Rough handling will affect the calibration of both Types A and B meters. Changes in barometric pressure will affect the calibration of Type A meter but not Type B meter. The steps described A1.2 to A1.6, as applicable to the meter type under consideration, are prerequisites for the final calibration test to determine the operating pressure, *P*, on the pressure gage of the Type A meter as described in A1.7, or to determine the accuracy of the graduations indicating air content on the dial face of the pressure gage of the Type B meter. The steps in A1.2 to A1.6 need be made only once (at the time of initial calibration), or only occasionally to check volume constancy of the calibration cylinder and measuring bowl. The calibration test described in A1.7 and A1.9, as applicable to the meter type being checked, must be made as frequently as necessary to ensure that the proper gage pressure, *P*, is being used for the Type A meter or that the correct air contents are being indicated on the pressure gage air content scale for the Type B meter. A change in elevation of more than 600 ft (183 m) from the location at which a Type-A meter was last calibrated will require recalibration in accordance with A1.7.

## 6. Determination of Aggregate Correction Factor

6.1 *Procedure*—Determine the aggregate correction factor on a combined sample of fine and coarse aggregate as directed in 6.2 to 6.4. It is determined independently by applying the calibrated pressure to a sample of inundated fine and coarse aggregate in approximately the same moisture condition, amount, and proportions occurring in the concrete sample under test.

6.2 *Aggregate Sample Size*—Calculate the weights of fine and coarse aggregate present in the sample of fresh concrete whose air content is to be determined, as follows:

$$F_s = (S/B) \times F_b \quad (1)$$

$$C_s = (S/B) \times C_b \quad (2)$$

where:

$F_s$  = weight of fine aggregate in concrete sample under test, lb (kg),

$S$  = volume of concrete sample (same as volume of measuring bowl), ft<sup>3</sup> (m<sup>3</sup>),

$B$  = volume of concrete produced per batch (Note 1), ft<sup>3</sup> (m<sup>3</sup>),

$F_b$  = total weight of fine aggregate in the moisture condition used in batch, lb (kg),

$C_s$  = weight of coarse aggregate in concrete sample under test, lb (kg), and

$C_b$  = total weight of coarse aggregate in the moisture condition used in batch, lb (kg).

NOTE 2—The volume of concrete produced per batch can be determined in accordance with applicable provisions of Test Method C 138.

NOTE 3—The term "weight" is temporarily used in this standard because of established trade usage. The word is used to mean both "force" and "mass," and care must be taken to determine which is meant in each case (SI unit for force = newton and for mass = kilogram).

6.3 *Placement of Aggregate in Measuring Bowl*—Mix representative samples of fine aggregate  $F_s$  and coarse aggregate

$C_s$ , and place in the measuring bowl filled one-third full with water. Place the mixed aggregate, a small amount at a time, into the measuring bowl; if necessary, add additional water so as to inundate all of the aggregate. Add each scoopful in a manner that will entrap as little air as possible and remove accumulations of foam promptly. Tap the sides of the bowl and lightly rod the upper 1 in. (25 mm) of the aggregate eight to twelve times. Stir after each addition of aggregate to eliminate entrapped air.

#### 6.4 Aggregate Correction Factor Determination:

6.4.1 *Initial Procedure for Types A and B Meters*—When all of the aggregate has been placed in the measuring bowl, remove excess foam and keep the aggregate inundated for a period of time approximately equal to the time between introduction of the water into the mixer and the time of performing the test for air content before proceeding with the determination as directed in 6.4.2 or 6.4.3.

6.4.2 *Type A Meter*—Complete the test as described in 8.2.1 and 8.2.2. The aggregate correction factor,  $G$ , is equal to  $h_1 - h_2$  (see Fig. 1) (Note 4).

6.4.3 *Type B Meter*—Perform the procedures as described in 8.3.1. Remove a volume of water from the assembled and filled apparatus approximately equivalent to the volume of air that would be contained in a typical concrete sample of a size equal to the volume of the bowl. Remove the water in the manner described in A1.9 for the calibration tests. Complete the test as described in 8.3.2. The aggregate correction factor,  $G$ , is equal to the reading on the air-content scale minus the volume of water removed from the bowl expressed as a percent of the volume of the bowl (see Fig. 1).

NOTE 4—The aggregate correction factor will vary with different aggregates. It can be determined only by test, since apparently it is not directly related to absorption of the particles. The test can be easily made and must not be ignored. Ordinarily the factor will remain reasonably constant for given aggregates, but an occasional check test is recommended.

## 7. Preparation of Concrete Test Sample

7.1 Obtain the sample of freshly mixed concrete in accordance with applicable procedures of Practice C 172. If the concrete contains coarse aggregate particles that would be retained on a 2-in. (50-mm) sieve, wet-sieve a sufficient amount of the representative sample over a 1½-in. (37.5-mm) sieve, as described in Practice C 172, to yield sufficient material to completely fill the measuring bowl of the size selected for use. Carry out the wet-sieving operation with the minimum practicable disturbance of the mortar. Make no attempt to wipe adhering mortar from coarse aggregate particles retained on the sieve.

## 8. Procedure for Determining Air Content of Concrete

### 8.1 Placement and Consolidation of Sample:

8.1.1 Dampen the interior of the measuring bowl and place it on a flat, level, firm surface. Place a representative sample of the concrete, prepared as described in Section 7, in the measuring bowl in equal layers. Consolidate each layer by the rodding procedure (8.1.2) or by vibration (8.1.3). Strike-off the finally consolidated layer (8.1.4). Rod concretes with a slump greater than 3 in. (75 mm). Rod or vibrate concrete with a

slump of 1 to 3 in. (25 to 75 mm). Consolidate concretes with a slump less than 1 in. (25 mm) by vibration.

8.1.2 *Rodding*—Place the concrete in the measuring bowl in three layers of approximately equal volume. Consolidate each layer of concrete by 25 strokes of the tamping rod evenly distributed over the cross section. After each layer is rodded, tap the sides of the measure smartly 10 to 15 times with the mallet to close any voids left by the tamping rod and to release any large bubbles of air that may have been trapped. Rod the bottom layer throughout its depth, but the rod shall not forcibly strike the bottom of the measure. In rodding the second and final layers, use only enough force to cause the rod to penetrate the surface of the previous layer about 1 in. (25 mm). Add the final layer of concrete in a manner to avoid excessive overfilling (8.1.4).

8.1.3 *Vibration*—Place the concrete in the measuring bowl in two layers of approximately equal volume. Place all of the concrete for each layer before starting vibration of that layer. Consolidate each layer by three insertions of the vibrator evenly distributed over the cross section. Add the final layer in a manner to avoid excessive overfilling (8.1.4). In consolidating the bottom layer, do not allow the vibrator to rest on or touch the bottom or sides of the measuring bowl. Take care in withdrawing the vibrator to ensure that no air pockets are left in the specimen. Observe a standard duration of vibration for the particular kind of concrete, vibrator, and measuring bowl involved. The duration of vibration required will depend upon the workability of the concrete and the effectiveness of the vibrator. Continue vibration until the concrete is properly consolidated. Never continue vibration long enough to cause escape of froth from the sample.

NOTE 5—Overvibration may cause segregation and loss of intentionally entrained air. Usually, sufficient vibration has been applied as soon as the surface of the concrete becomes relatively smooth and has a glazed appearance.

8.1.4 *Strike Off*—After consolidation of the concrete, strike off the top surface by sliding the strike-off bar across the top flange or rim of the measuring bowl with a sawing motion until the bowl is just level full. On completion of consolidation, the bowl must not contain an excess or deficiency of concrete. Removal of ¼ in. (3 mm) during strike off is optimum. When a strike-off plate is used, strike off concrete as prescribed in Test Method C 138.

NOTE 6—A small quantity of representative concrete may be added to correct a deficiency. If the measure contains a great excess, remove a representative portion of concrete with a trowel or scoop before the measure is struck off.

NOTE 7—The use of the strike-off plate on cast aluminum or other relatively soft metal air meter bases may cause rapid wear of the rim and require frequent maintenance, calibration, and ultimately, replacement.

8.1.5 *Application of Test Method*—Any portion of the test method not specifically designated as pertaining to Type A or Type B meter shall apply to both types.

### 8.2 Procedure—Type A Meter:

8.2.1 *Preparation for Test*—Thoroughly clean the flanges or rims of the bowl and of the cover assembly so that when the cover is clamped in place a pressure-tight seal will be obtained. Assemble the apparatus and add water over the concrete by

means of the tube until it rises to about the halfway mark in the standpipe. Incline the apparatus assembly about 30° from vertical and, using the bottom of the bowl as a pivot, describe several complete circles with the upper end of the column, simultaneously tapping the cover lightly to remove any entrapped air bubbles above the concrete sample. Return the apparatus assembly to a vertical position and fill the water column slightly above the zero mark, while lightly tapping the sides of the bowl. Bring the water level to the zero mark of the graduated tube before closing the vent at the top of the water column (see Fig. 1 A).

NOTE 8—Some Type A meters have a calibrated starting fill mark above the zero mark. Generally, this starting mark should not be used since, as noted in 8.2.3, the apparent air content is the difference between the water level reading  $h_1$  at pressure  $P$  and the water level  $h_2$  at zero pressure after release of pressure  $P$ .

8.2.2 The internal surface of the cover assembly shall be kept clean and free from oil or grease; the surface shall be wet to prevent adherence of air bubbles that might be difficult to dislodge after assembly of the apparatus.

8.2.3 *Test Procedure*—Apply more than the desired test pressure,  $P$ , (about 0.2 psi (1380 Pa) more) to the concrete by means of the small hand pump. To relieve local restraints, tap the sides of the measure sharply and, when the pressure gage indicates the exact test pressure,  $P$ , as determined in accordance with 7.2.2, read the water level,  $h_1$ , and record to the nearest division or half-division on the graduated precision-bore tube or gage glass of the standpipe (see Fig. 1 B). For extremely harsh mixes tap the bowl vigorously until further tapping produces no change in the indicated air content. Gradually release the air pressure through the vent at the top of the water column and tap the sides of the bowl lightly for about 1 min. Record the water level,  $h_2$ , to the nearest division or half-division (see Fig. 1 C). Calculate the apparent air content as follows:

$$A_1 = h_1 - h_2 \quad (3)$$

where:

$A_1$  = apparent air content,  
 $h_1$  = water level reading at pressure,  $P$  (see Note 6), and  
 $h_2$  = water level reading at zero pressure after release of pressure,  $P$ .

8.2.4 *Check Test*—Repeat the steps described in 8.2.3 without adding water to reestablish the water level at the zero mark. The two consecutive determinations of apparent air content should check within 0.2 % of air and shall be averaged to give the value  $A_1$  to be used in calculating the air content,  $A_s$ , in accordance with Section 9.

8.2.5 In the event the air content exceeds the range of the meter when it is operated at the normal test pressure  $P$ , reduce the test pressure to the alternative test pressure  $P_1$  and repeat the steps outlined in 8.2.2 and 8.2.3.

NOTE 9—See A1.7 for exact calibration procedures. An approximate value of the alternative pressure,  $P_1$ , such that the apparent air content will equal twice the meter reading can be computed from the following relationship:

$$P_1 = P_a P / (2P_a + P) \quad (4)$$

where:

$P_1$  = alternative test pressure, psi (or kPa),  
 $P_a$  = atmospheric pressure, psi (approximately 14.7 psi (101 kPa) but will vary with altitude and weather conditions) (or kPa), and  
 $P$  = normal test or operating gage pressure, psi (or kPa).

### 8.3 Procedure—Type B Meter

8.3.1 *Preparation for Test*—Thoroughly clean the flanges or rims of the bowl and the cover assembly so that when the cover is clamped in place a pressure-tight seal will be obtained. Assemble the apparatus. Close the air valve between the air chamber and the measuring bowl and open both petcocks on the holes through the cover. Using a rubber syringe, inject water through one petcock until water emerges from the opposite petcock. Jar the meter gently until all air is expelled from this same petcock.

8.3.2 *Test Procedure*—Close the airbleeder valve on the air chamber and pump air into the air chamber until the gage hand is on the initial pressure line. Allow a few seconds for the compressed air to cool to normal temperature. Stabilize the gage hand at the initial pressure line by pumping or bleeding-off air as necessary, tapping the gage lightly by hand. Close both petcocks on the holes through the cover. Open the air valve between the air chamber and the measuring bowl. Tap the sides of the measuring bowl smartly with the mallet to relieve local restraints. Lightly tap the pressure gage by hand to stabilize the gage hand. Read the percentage of air on the dial of the pressure gage. Failure to close the main air valve before releasing the pressure from either the container or the air chamber will result in water being drawn into the air chamber, thus introducing error in subsequent measurements. In the event water enters the air chamber it must be bled from the air chamber through the bleeder valve followed by several strokes of the pump to blow out the last traces of water. Release the pressure by opening both petcocks (Fig. 1, A and B) before removing the cover.

## 9. Calculation

9.1 *Air Content of Sample Tested*—Calculate the air content of the concrete in the measuring bowl as follows:

$$A_s = A_1 - G \quad (5)$$

where:

$A_s$  = air content of the sample tested, %,  
 $A_1$  = apparent air content of the sample tested, % (see section 7.2.2 and 8.3.2), and  
 $G$  = aggregate correction factor, % (Section 6).

9.2 *Air Content of Full Mixture*—When the sample tested represents that portion of the mixture that is obtained by wet sieving to remove aggregate particles larger than a 1½-in. (37.5-mm) sieve, the air content of the full mixture is calculated as follows:

$$A_f = 100 A_s V_c / (100 V_f - A_s V_a) \quad (6)$$

where: (Note 10):

$A_f$  = air content of the full mixture, %,  
 $V_c$  = absolute volume of the ingredients of the mixture passing a 1½-in. sieve, airfree, as determined from the original batch weights, ft<sup>3</sup> (m<sup>3</sup>),

$V_i$  = absolute volume of all ingredients of the mixture, airfree, ft<sup>3</sup> (m<sup>3</sup>), and

$V_a$  = absolute volume of the aggregate in the mixture coarser than a 1½-in. sieve, as determined from original batch weights, ft<sup>3</sup> (m<sup>3</sup>).

9.3 *Air Content of the Mortar Fraction*—When it is desired to know the air content of the mortar fraction of the mixture, calculate it as follows:

$$A_m = 100 A_s V_c / [100 V_m + A_s (V_c - V_m)] \quad (7)$$

where: (Note 10):

$A_m$  = air content of the mortar fraction, %, and

$V_m$  = absolute volume of the ingredients of the mortar fraction of the mixture, airfree, ft<sup>3</sup> (m<sup>3</sup>).

NOTE 10—The values for use in Eq 6 and Eq 7 are most conveniently obtained from data on the concrete mixture tabulated as follows for a batch of any size:

	Absolute Volume, ft <sup>3</sup> (m <sup>3</sup> )	
Cement	=====	} $V_m$ } $V_c$
Water	=====	
Fine aggregate	=====	} $V_a$
Coarse aggregate (No. 4 (4.75-mm) to 1½-in. (37.5-mm))	=====	
Coarse aggregate (1½-in.)	=====	} $V_i$
Total	=====	

## 10. Precision and Bias

### 10.1 Precision:

10.1.1 *Single-Operator Precision*—The single-operator standard deviation cannot be established because the sampling requirements for this test, as established in Practice C 172, do

not allow a single operator time to conduct more than one test on a sample.

10.1.2 *Multilaboratory Precision*—The multilaboratory standard deviation has not been established.

10.1.3 *Multioperator Precision*—The multioperator standard deviation of a single test result has been found to be 0.28 % air by volume of concrete for Type A air meters as long as the air content does not exceed 7 %. Therefore results of two tests properly conducted by different operators but on the same material should not differ by more than 0.8 % air by volume of concrete (see Practice E 177, Note 8 and Note 9).

NOTE 11—These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C 670. The precision statements are based on the variations in tests on three different concretes, each tested by eleven different operators.<sup>4</sup>

NOTE 12—The precision of this test method using Type B air meters has not been determined.

10.2 *Bias*—This test method has no bias because the air content of freshly mixed concrete can only be defined in terms of the test methods.

## 11. Keywords

11.1 air content; calibration; concrete; correction factor; measuring bowl; meter; pressure; pump; unit weight

<sup>4</sup> Reidenour, D. R., and Howe, R. H., "Air Content of Plastic and Hardened Concrete," presented at the 2nd International Conference on "Durability of Building Materials and Components" Sept. 14-16, 1981. Reprints compiled by: G. Frohnsdorff and B. Homer, National Institute for Standards and Technology, Gaithersburg, MD 20899, formerly National Bureau of Standards, Washington, DC 20234.

## ANNEX

### (Mandatory Information)

#### A1. CALIBRATION OF APPARATUS

A1.1 Calibration tests shall be performed in accordance with the following procedures as applicable to the meter type being employed.

A1.2 *Calibration of the Calibration Vessel*—Determine accurately the weight of water,  $w$ , required to fill the calibration vessel, using a scale accurate to 0.1 % of the weight of the vessel filled with water. This step shall be performed for Type A and B meters.

A1.3 *Calibration of the Measuring Bowl*—Determine the weight of water,  $W$ , required to fill the measuring bowl, using a scale accurate to 0.1 % of the weight of the bowl filled with water. Slide a glass plate carefully over the flange of the bowl in a manner to ensure that the bowl is completely filled with water. A thin film of cup grease smeared on the flange of the bowl will make a watertight joint between the glass plate and the top of the bowl. This step shall be performed for Type A and B meters.

A1.4 *Effective Volume of the Calibration Vessel, R*—The constant  $R$  represents the effective volume of the calibration vessel expressed as a percentage of the volume of the measuring bowl.

A1.4.1 For meter Types A, calculate  $R$  as follows (Note A1):

$$R = 0.98 w/W \quad (A1.1)$$

where:

$w$  = weight of water required to fill the calibration vessel, and

$W$  = weight of water required to fill the measuring bowl.

NOTE A1.1—The factor 0.98 is used to correct for the reduction in the volume of air in the calibration vessel when it is compressed by a depth of water equal to the depth of the measuring bowl. This factor is approximately 0.98 for an 8-in. (203-mm) deep measuring bowl at sea level. Its value decreases to approximately 0.975 at 5000 ft (1524 m) above sea level and 0.970 at 13 000 ft (3962 m) above sea level. The value of this constant will decrease by about 0.01 for each 4-in. (102-mm)

increase in bowl depth. The depth of the measuring bowl and atmospheric pressure do not affect the effective volume of the calibration vessel for meter Types B.

A1.4.2 For meter Types B calculate  $R$  as follows (Note A1.1):

$$R = w/W \quad (A1.2)$$

A1.5 *Determination of, or Check of, Allowance for Expansion Factor, D:*

A1.5.1 For meter assemblies of Type A determine the expansion factor,  $D$  (Note A1.2) by filling the apparatus with water only (making certain that all entrapped air has been removed and the water level is exactly on the zero mark (Note A1.3) and applying an air pressure approximately equal to the operating pressure,  $P$ , determined by the calibration test described in A1.7. The amount the water column lowers will be the equivalent expansion factor,  $D$ , for that particular apparatus and pressure (Note A1.5).

NOTE A1.2—Although the bowl, cover, and clamping mechanism of the apparatus must of necessity be sturdily constructed so that it will be pressure-tight, the application of internal pressure will result in a small increase in volume. This expansion will not affect the test results because, with the procedure described in Sections 6 and 8, the amount of expansion is the same for the test for air in concrete as for the test for aggregate correction factor on combined fine and coarse aggregates, and is thereby automatically cancelled. However, it does enter into the calibration test to determine the air pressure to be used in testing fresh concrete.

NOTE A1.3—The water columns on some meters of Type-A design are marked with an initial water level and a zero mark, the difference between the two marks being the allowance for the expansion factor. This allowance should be checked in the same manner as for meters not so marked and in such a case, the expansion factor should be omitted in computing the calibration readings in A1.7.

NOTE A1.4—It will be sufficiently accurate for this purpose to use an approximate value for  $P$  determined by making a preliminary calibration test as described in A1.7 except that an approximate value for the calibration factor,  $K$ , should be used. For this test  $K = 0.98R$  which is the same as Eq A1.2 except that the expansion reading,  $D$ , as yet unknown, is assumed to be zero.

A1.5.2 For meters of Type B design, the allowance for the expansion factor,  $D$ , is included in the difference between the initial pressure indicated on the pressure gage and the zero percent mark on the air-content scale on the pressure gage. This allowance shall be checked by filling the apparatus with water (making certain that all entrapped air has been removed), pumping air into the air chamber until the gage hand is stabilized at the indicated initial pressure line, and then releasing the air to the measuring bowl (Note A1.5). If the initial pressure line is correctly positioned, the gage should read zero percent. The initial pressure line shall be adjusted if two or more determinations show the same variation from zero percent and the test repeated to check the adjusted initial pressure line.

NOTE A1.5—This procedure may be accomplished in conjunction with the calibration test described in A1.9.

A1.6 *Calibration Reading, K*—The calibration reading,  $K$ , is the final meter reading to be obtained when the meter is operated at the correct calibration pressure.

A1.6.1 For meter Types A, the calibration reading,  $K$ , is as follows:

$$K = R + D \quad (A1.3)$$

where:

$R$  = effective volume of the calibration vessel (A1.4.1), and

$D$  = expansion factor (A1.5.1, Note A1.6).

A1.6.2 For meter Types B the calibration reading,  $K$ , equals the effective volume of the calibration vessel (A4.2) as follows:

$$K = R \quad (A1.4)$$

NOTE A1.6—If the water column indicator is graduated to include an initial water level and a zero mark, the difference between the two marks being equivalent to the expansion factor, the term  $D$  shall be omitted from Eq A1.3.

A1.7 *Calibration Test to Determine Operating Pressure, P, on Pressure Gage, Type A Meter*—If the rim of the calibration cylinder contains no recesses or projections, fit it with three or more spacers equally spaced around the circumference. Invert the cylinder and place it at the center of the dry bottom of the measuring bowl. The spacers will provide an opening for flow of water into the calibration cylinder when pressure is applied. Secure the inverted cylinder against displacement and carefully lower the cover assembly. After the cover is clamped in place, carefully adjust the apparatus assembly to a vertical position and add water at air temperature, by means of the tube and funnel, until it rises above the zero mark on the standpipe. Close the vent and pump air into the apparatus to the approximate operating pressure. Incline the assembly about 30° from vertical and, using the bottom of the bowl as a pivot, describe several complete circles with the upper end of the standpipe, simultaneously tapping the cover and sides of the bowl lightly to remove any entrapped air adhering to the inner surfaces of the apparatus. Return the apparatus to a vertical position, gradually release the pressure (to avoid loss of air from the calibration vessel), and open the vent. Bring the water level exactly to the zero mark by bleeding water through the petcock in the top of the conical cover. After closing the vent, apply pressure until the water level has dropped an amount equivalent to about 0.1 to 0.2 % of air more than the value of the calibration reading,  $K$ , determined as described in A1.6. To relieve local restraints, lightly tap the sides of the bowl, and when the water level is exactly at the value of the calibration reading,  $K$ , read the pressure,  $P$ , indicated by the gage and record to the nearest 0.1 psi (690 Pa). Gradually release the pressure and open the vent to determine whether the water level returns to the zero mark when the sides of the bowl are tapped lightly (failure to do so indicates loss of air from the calibration vessel or loss of water due to a leak in the assembly). If the water levels fails to return to within 0.05 % air of the zero mark and no leakage beyond a few drops of water is found, some air probably was lost from the calibration cylinder. In this case, repeat the calibration procedure step by step from the beginning of this paragraph. If the leakage is more than a few drops of water, tighten the leaking joint before repeating the calibration procedure. Check the indicated pressure reading promptly by bringing the water level exactly to the zero mark, closing the vent, and applying the pressure,  $P$ , just determined. Tap the gage lightly with a finger. When the gage indicates the exact pressure,  $P$ , the water column should read



the value of the calibration factor,  $K$ , used in the first pressure application within about 0.05 % of air.

**NOTE A1.7—Caution:** The apparatus assembly must not be moved from the vertical position until pressure has been applied which will force water about one third of the way up into the calibration cylinder. Any loss of air from this cylinder will nullify the calibration.

**A1.8 Calibration Test to Determine Alternative Operating Pressure  $P_1$ —Meter Type A**—The range of air contents which can be measured with a given meter can be doubled by determining an alternative operating pressure  $P_1$  such that the meter reads half of the calibration reading,  $K$ , (Eq A1.3). Exact calibration will require determination of the expansion factor at the reduced pressure in A1.5. For most purposes the change in expansion factor can be disregarded and the alternative operating pressure determined during the determination of the regular operating pressure in A1.7.

**A1.9 Calibration Test to Check the Air Content Graduations on the Pressure Gage, Type B Meter**—Fill the measuring bowl with water as described in A1.3. Screw the short piece of tubing or pipe furnished with the apparatus into the threaded petcock hole on the underside of the cover assembly. Assemble the apparatus. Close the air valve between the air chamber and the measuring bowl and open the two petcocks on holes through the cover assembly. Add water through the petcock on the cover assembly having the extension below until all air is expelled from the second petcock. Pump air into the air chamber until the pressure reaches the indicated initial pressure line. Allow a few seconds for the compressed air to cool to normal temperature. Stabilize the gage hand at the initial pressure line by pumping or bleeding off air as necessary, tapping the gage lightly. Close the petcock not provided with the tube or pipe extension on the under side of the cover. Remove water from the assembly to the calibrating vessel controlling the flow, depending on the particular meter design, by opening the petcock provided with the tube or pipe extension and cracking the air valve between the air chamber and the measuring bowl, or by opening the air valve and using the petcock to control flow. Perform the calibration at an air content which is within the normal range of use. If the

calibration vessel (A1.2) has a capacity within the normal range of use, remove exactly that amount of water. With some meters the calibrating vessel is quite small and it will be necessary to remove several times that volume to obtain an air content within the normal range of use. In this instance, carefully collect the water in an auxiliary container and determine the amount removed by weighing to the nearest 0.1 %. Calculate the correct air content,  $R$ , by using Eq A1.2. Release the air from the apparatus at the petcock not used for filling the calibration vessel and if the apparatus employs an auxiliary tube for filling the calibration container, open the petcock to which the tube is connected to drain the tube back into the measuring bowl (Note A1.7). At this point of procedure the measuring bowl contains the percentage of air determined by the calibration test of the calibrating vessel. Pump air into the air chamber until the pressure reaches the initial pressure line marked on the pressure gage, close both petcocks in the cover assembly, and then open the valve between the air chamber and the measuring bowl. The indicated air content on the pressure gage dial should correspond to the percentage of air determined to be in the measuring bowl. If two or more determinations show the same variation from the correct air content, the dial hand shall be reset to the correct air content and the test repeated until the gage reading corresponds to the calibrated air content within 0.1 %. If the dial hand was reset to obtain the correct air content, recheck the initial pressure mark as in A1.5.2. If a new initial pressure reading is required, repeat the calibration to check the accuracy of the graduation on the pressure gage described earlier in this section. If difficulty is encountered in obtaining consistent readings, check for leaks, for the presence of water inside the air chamber (see Fig. 2), or the presence of air bubbles clinging to the inside surfaces of the meter from the use of cool aerated water. In this latter instance use deaerated water which can be obtained by cooling hot water to room temperature.

**NOTE A1.8**—If the calibrating vessel is an integral part of the cover assembly, the petcock used in filling the vessel should be closed immediately after filling the calibration vessel and not opened until the test is complete.

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*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.*



# Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete<sup>1</sup>

This standard is issued under the fixed designation C 311; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

<sup>ε1</sup> NOTE—Eq 10 was editorially corrected November 2001.

## 1. Scope

1.1 These test methods cover procedures for sampling and testing fly ash and raw or calcined pozzolans for use as a mineral admixture in portland-cement concrete.

1.2 The procedures appear in the following order:

	Sections
Sampling	7
CHEMICAL ANALYSIS	
Reagents and apparatus	10
Moisture content	11 and 12
Loss on ignition	13 and 14
Silicon dioxide, aluminum oxide, iron oxide, calcium oxide, magnesium oxide, sulfur trioxide, sodium oxide and potassium oxide	15
Available alkali	16 and 17
PHYSICAL TESTS	
Density	18
Fineness	19
Increase of drying shrinkage of mortar bars	20 to 22
Soundness	23
Air-entrainment of mortar	24 and 25
Strength activity index with portland cement	26 to 29
Water requirement	30
Effectiveness of Mineral Admixture in Controlling Alkali-Silica Reactions	31

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables) shall not be considered as requirements of this standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- C 33 Specification for Concrete Aggregates<sup>2</sup>
- C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)<sup>3</sup>
- C 114 Test Methods for Chemical Analysis of Hydraulic Cement<sup>3</sup>
- C 150 Specification for Portland Cement<sup>3</sup>
- C 151 Test Method for Autoclave Expansion of Portland Cement<sup>3</sup>
- C 157 Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete<sup>2</sup>
- C 185 Test Method for Air Content of Hydraulic Cement Mortar<sup>3</sup>
- C 188 Test Method for Density of Hydraulic Cement<sup>3</sup>
- C 204 Test Method for Fineness of Portland Cement by Air Permeability Apparatus<sup>3</sup>
- C 226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement<sup>3</sup>
- C 227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)<sup>2</sup>
- C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency<sup>3</sup>
- C 430 Test Method for Fineness of Hydraulic Cement by the 45-μm (No. 325) Sieve<sup>3</sup>
- C 441 Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction<sup>2</sup>
- C 618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete<sup>2</sup>
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>
- C 778 Specification for Standard Sand<sup>3</sup>
- C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution<sup>3</sup>

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and are the direct responsibility of Subcommittee C09.24 on Ground Slag and Pozzolonic Admixtures.

Current edition approved July 10, 2000. Published September 2000. Originally published as C 311 – 53. Last previous edition C 311 – 98b.

<sup>2</sup> Annual Book of ASTM Standards, Vol 04.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 04.01.

C1157 Performance Specification for Hydraulic Cement<sup>3</sup>  
 D4326 Test Method for Major and Minor Elements in Coal  
 and Coke Ash By X-Ray Fluorescence<sup>4</sup>

1. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *composite sample*—a sample that is constructed by combining equal portions of grab or regular samples.

3.1.2 *established source*—a source for which at least six months of continuous production quality assurance records from a test frequency required for a new source are available, sampled at the source.

3.1.3 *grab sample*—a sample that is taken in a single operation from a conveyor delivering to bulk storage, from bags, or from a bulk shipment. Such a sample may, or may not, reflect the composition or physical properties of a single lot of mineral admixture. This type of sample can be used to characterize small amounts of mineral admixture.

3.1.4 *jobsite or new source*—a source for which less than six months of production records are available, sampled at the source.

3.1.5 *lot*—specific quantity of fly ash or natural pozzolan offered for inspection at any one time. A lot may be one storage bin or the contents of one or more transport units representing mineral admixture drawn from the same storage bin.

3.1.6 *regular sample*—a sample that is constructed by combining equal portions of grab samples that were taken at predetermined times or locations from any single lot of mineral admixture.

4. Significance and Use

4.1 These test methods are used to develop data for comparison with the requirements of Specification C 618. These test methods are based on standardized testing in the laboratory and are not intended to simulate job conditions.

4.1.1 *Strength Activity Index*—The test for strength activity index is used to determine whether a mineral admixture results in an acceptable level of strength development when used with hydraulic cement in concrete. Since the test is performed with mortar, the results may not provide a direct correlation of how the mineral admixture will contribute to strength in concrete.

4.1.2 *Chemical Tests*— The chemical component determinations and the limits placed on each do not predict the performance of a mineral admixture with hydraulic cement in concrete, but collectively help describe composition and uniformity of the mineral admixture.

5. Materials

5.1 *Graded Standard Sand*—The sand used for making test specimens for the activity index with lime or portland cement shall be natural silica sand conforming to the requirements for graded standard sand in Specification C 778.

NOTE 1—*Segregation of Graded Sand*—The graded standard sand should be handled in such a manner as to prevent segregation, since variations in the grading of the sand cause variations in the consistency of the mortar. In emptying bins or sacks, care should be exercised to prevent

the formation of mounds of sand or craters in the sand, down the slopes of which the coarser particles will roll. Bins should be of sufficient size to permit these precautions. Devices for drawing the sand from bins by gravity should not be used.

5.2 *Hydrated Lime*— The hydrated lime used in the tests shall be reagent-grade calcium hydroxide, 95 % minimum calculated as Ca(OH)<sub>2</sub>(Note 2), and have a minimum fineness of 2500 m<sup>2</sup>/kg as determined in accordance with Test Method C 204.

NOTE 2—The calcium hydroxide should be protected from exposure to carbon dioxide. Material remaining in an opened container after a test should not be used for subsequent tests.

5.3 *Portland Cement*— The portland cement used in the Strength Activity Index with Portland Cement test shall comply with the requirements of Specification C 150 and have a minimum compressive strength of 35 MPa (5000 psi) at 28 days and total alkalis (Na<sub>2</sub>O + 0.658 K<sub>2</sub>O) not less than 0.50 % nor more than 0.80 %.

5.3.1 The use of a locally available portland cement in the Strength Activity Index or a project cement that does not meet the requirements of the section on Materials is permitted when the variations from the requirements of the section on Materials are reported and when the use of such portland cement is requested.

6. Sample Type and Size

6.1 Grab samples and regular samples shall have a mass of at least 2 kg (4 lb).

6.2 Grab samples or regular samples taken at prescribed intervals over a period of time (see Table 1), may be combined to form a composite sample representative of the mineral admixture produced during that period of time.

6.3 Composite samples shall have a mass of at least 4 kg (8 lb).

6.4 The sampling shall be done by, or under the direction of, a responsible representative of the purchaser.

7. Sampling Procedure

7.1 The mineral admixture may be sampled by any one of the following methods:

7.1.1 *From Bulk Storage at Point of Discharge or from Rail Cars and Road Tankers*. A sample may be taken by siphon tube during loading or by sampling tube from each loaded car or tanker. If the load is sampled at the point of discharge into the rail car or tanker, the top surface shall be removed to a depth

TABLE 1 Minimum Sampling and Testing Frequency<sup>A</sup>

Test	Sample Type	Jobsite or New Source <sup>B</sup>	Established Source <sup>B</sup>
Moisture content	Regular	Daily or each (90 Mg <sup>C</sup> (100 Tons)	Daily or each 360 Mg <sup>C</sup> (400 Tons)
Loss on ignition			
Fineness			
Density and the other tests in Specification C 618, Tables 1 and 2	Composite	Monthly or each 1 800 Mg <sup>C</sup> (2 000 Tons)	Monthly or each 2 900 Mg <sup>C</sup> (3 200 Tons)

<sup>A</sup> It should be noted that the minimum test frequency given in Table 1 is not necessarily the frequency needed for quality control programs on some mineral admixtures.

<sup>B</sup> For definitions, refer to the Terminology section.

<sup>C</sup> Whichever comes first.

<sup>4</sup> Annual Book of ASTM Standards, Vol 05.05.

of at least 200 mm (8 in.) before sampling. The sample shall be identified with at least the date and shipment number.

7.1.2 *From Bags in Storage.* The regular sample shall comprise increments of equal size taken by sampling tube from three bags selected at random from one lot of bagged material.

The sample shall be identified with date and lot number.

7.1.3 *From Conveyor Delivering to Bulk Storage.* Take one sample of 2 kg (4 lb) or more of the material passing over the conveyor. This may be secured by taking the entire test sample in a single operation, known as the grab sample method, or by combining several equal portions taken at regular intervals, known as the regular sample method. Automatic samplers may be used to obtain samples.

7.2 Samples shall be treated as described in Section 8.

NOTE 3—Some methods of loading or delivery of mineral admixtures, particularly from an airstream or conveyor belt, may create stratification or segregation in the material stream. Sampling techniques must be designed to ensure that the sample is representative of the mineral admixture shipped.

## 8. Preparation and Storage of Samples

8.1 Prepare composite samples for the tests required in Section 9, by arranging all grab or regular samples into groups covering the period or quantity to be represented by the sample. Take equal portions from each, sufficient to produce a composite sample large enough for the tests required. Mix the composite sample thoroughly.

8.2 Samples shall be stored in clean, airtight containers identified with the source and lot or period of time represented. Untested portions of the sample shall be retained for at least one month after all test results have been reported.

## 9. Testing Frequency

9.1 *General*—When required, the purchaser shall specify the amount of testing for available alkalis, reactivity with cement alkalis, drying shrinkage, and air-entrainment. Make all other tests on regular or composite samples chosen as specified in Table 1.

## CHEMICAL ANALYSIS

### 10. General

10.1 All apparatus, reagents and techniques shall comply with the requirements of Test Methods C 114.

10.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

### MOISTURE CONTENT

### 11. Procedure

11.1 Dry a weighed sample, as received, to constant weight in an oven at 105 to 110°C (221 to 230°F).

### 12. Calculation

12.1 Calculate the percentage of moisture to the nearest 0.1 %, as follows:

$$\text{Moisture content, \%} = (A/B) \times 100 \quad (1)$$

where:

$A$  = mass loss during drying, and

$B$  = mass as received.

### LOSS ON IGNITION

### 13. Procedure

13.1 Determine loss on ignition in accordance with the procedures outlined in Test Methods C 114, except that the material remaining from the determination of moisture content shall be ignited to constant mass in an uncovered porcelain, not platinum, crucible at 750 ± 50°C (1382 ± 190°F).

### 14. Calculation

14.1 Calculate the percentage of loss on ignition to the nearest 0.1, as follows:

$$\text{Loss on ignition, \%} = (A/B) \times 100 \quad (2)$$

where:

$A$  = loss in mass between 105 and 750°C (221 and 1382°F),

$B$  = mass of moisture-free sample used.

*SILICON DIOXIDE, ALUMINUM OXIDE, IRON OXIDE, CALCIUM OXIDE, MAGNESIUM OXIDE, SULFUR TRIOXIDE, SODIUM OXIDE AND POTASSIUM OXIDE*

### 15. Procedure

15.1 Determine the percentages of these oxides as required in accordance with the applicable sections of Test Methods C 114 for materials having an insoluble residue greater than 1 % (Note 4). Analysts performing sodium oxide and potassium oxide determinations shall observe the precautions outlined in the applicable section of C 1157 (refer to the section on Test Methods). Most pozzolans dissolve completely in lithium borate fluxes.

NOTE 4—Rapid and instrumental methods may be employed similar to those in Test Methods C 114 and D 4326.

### AVAILABLE ALKALI

### 16. Procedure

16.1 Weigh 5.0 g of the sample and 2.0 g of hydrated lime on a piece of weighing paper, carefully mix using a metal spatula, and transfer to a small plastic vial of approximately 25-mL capacity. Add 10.0 mL of water to this mixture, seal the vial by securing the cap or lid to the vial with tape (Note 5), blend by shaking until the mixture is uniform, and store at 38 ± 2°C.

NOTE 5—To ensure that moisture loss from the paste does not occur, place the sealed vial in a sealable container (such as a small sample or mason jar), add sufficient water to cover the bottom of the container, and seal.

16.2 Open the vial at the age of 28 days and transfer the contents to a 250-mL casserole. Break up and grind the cake with a pestle, adding a small amount of water, if necessary, so that a uniform slurry containing no lumps is obtained (Note 6). Add sufficient water to make the total volume 200 mL. Let stand 1 h at room temperature with frequent stirring. Filter through a medium-textured filter paper onto a 500-mL volumetric flask. Wash thoroughly with hot water (eight to ten times).

NOTE 6—At times it may be necessary to break the vial and peel off the plastic from the solid cake. In such cases, care should be exercised to avoid the loss of material and to remove all solid material from the fragments of the vial. If the cake is too hard to break up and grind in the asserole, a mortar should be used.

16.3 Neutralize the filtrate with dilute HCl (1 + 3), using 1 to 2 drops of phenolphthalein solution as the indicator. Add exactly 5 mL of dilute HCl (1 + 3) in excess. Cool the solution to room temperature and fill the flask to the mark with distilled water. Determine the amount of sodium and potassium oxides in the solution using the flame photometric procedure, described in Test Methods C 114, except that the standard solutions shall be made up to contain 8 mL of calcium chloride (CaCl<sub>2</sub>) stock solution per litre of standard solution, and the solution as prepared shall be used in place of the solution of cement.

NOTE 7—The standard solutions made up with 8 mL of calcium chloride (CaCl<sub>2</sub>) stock solution contain the equivalent of 504 ppm of CaO. Tests have shown that this amount closely approximates the amount of calcium dissolved in the test solution.

### 17. Calculation and Report

17.1 Calculate the results as weight percent of the original sample material. Report as equivalent percentage of sodium oxide (Na<sub>2</sub>O), calculated as follows:

$$\text{Equivalent Na}_2\text{O, \%} = \text{Na}_2\text{O, \%} + 0.658 \times \text{K}_2\text{O, \%} \quad (3)$$

## PHYSICAL TESTS

### DENSITY

#### 18. Procedure

18.1 Determine the density of the sample in accordance with the procedure described in Test Method C 188, except use about 50 g of mineral admixture instead of approximately 64 g of cement as recommended in Test Method C 188.

### FINESS, AMOUNT RETAINED WHEN WET-SIEVED ON A 45- $\mu\text{m}$ (NO. 325) SIEVE

#### 19. Procedure

19.1 Determine the amount of the sample retained when wet-sieved on a 45- $\mu\text{m}$  (No. 325) sieve, in accordance with Test Method C 430, with the following exceptions.

19.1.1 Calibrate the 45- $\mu\text{m}$  (No. 325) sieve using a cement standard (SRM 114).<sup>5</sup> Calculate the sieve correction factors as follows:

$$CF = \text{std} - \text{obs} \quad (4)$$

where:

$CF$  = the sieve correction factor, %, (include a negative sign when appropriate),

$\text{std}$  = the certified residue value for the SRM, %, and

$\text{obs}$  = the observed residue value for the SRM, %.

19.1.2 Calculate the fineness of the mineral admixture to the nearest 0.1 % as follows:

$$R_c = R_s + CF \quad (5)$$

where:

$R_c$  = corrected sieve residue, %, and

$R_s$  = observed residue for the test sample, %, and

$CF$  = the sieve correction factor, %.

NOTE 8—Test Method C 430 has been adopted for testing fly ash fineness. However, certain requirements, such as cleaning of sieves and interpretation of the test results, are sometimes not appropriate for fly ashes. The C 311 task group is currently evaluating the use of SRM fly ashes for performing sieve calibrations.

### INCREASE OF DRYING SHRINKAGE OF MORTAR BARS

#### 20. Test Specimen

20.1 Prepare test specimens in accordance with the procedures described in Test Method C 157, except mold three mortar bars from both the control mix and the test mix using the following proportions:

	Control Mix	Test Mix
Portland cement, g	500	500
Mineral admixture, g	None	125
Graded standard sand, g	1375	1250
Water	sufficient to produce a flow of 100 to 115 %	

#### 21. Procedure

21.1 Cure and measure the test specimens in accordance with Test Method C 157, except that the moist-curing period (including the period in the molds) shall be 7 days, and the comparator reading at the age of  $24 \pm \frac{1}{2}$  h shall be omitted. Immediately after taking the comparator reading at the end of the 7-day moist-curing period, store the specimens in accordance with Test Method C 157, and after 28 days of air storage, take a comparator reading for the specimens in accordance with Test Method C 157.

#### 22. Calculation and Report

22.1 Calculate the increase in drying shrinkage of the mortar bars,  $S_p$ , as follows:

$$S_p = S_t - S_c \quad (6)$$

where:

$S_t$  = average drying shrinkage of the test specimens calculated as follows, and

$S_c$  = average drying shrinkage of the control specimens calculated as follows:

$$S = \frac{[\text{initial CRD} - \text{CRD}] \times 100}{G} \quad (7)$$

where:

$S$  = drying shrinkage of test or control specimens, %, and

$\text{initial CRD}$  = difference between the comparator reading of the specimen and the reference bar at 7 days of moist curing,

$\text{CRD}$  = difference between the comparator reading of the specimen and the reference bar at 28 days of drying, and

<sup>5</sup> Available from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

$G$  = the gage length of the specimens 250 mm (10 in.).

22.2 Report the results to the nearest 0.01. If the average drying shrinkage of the control specimens is larger than the average drying shrinkage of the test specimens, prefix a minus sign to the increase of drying shrinkage of mortar bars reported.

### SOUNDNESS

#### 23. Procedure

23.1 Conduct the soundness test in accordance with Test Method C 151, except that the specimens shall be molded from a paste composed of 25 parts by weight of mineral admixture and 100 parts by weight of a portland cement conforming to Specification C 150.

### AIR-ENTRAINMENT OF MORTAR

#### 24. Procedure

24.1 Using portland cement conforming to the requirements for Type I or Type II of Specification C 150, prepare a test mixture in accordance with Test Method C 185, using the following proportions:

	Test Mix
Portland cement, g	300
Mineral admixture	75
20–30 standard sand, g	1125
Water, mL, sufficient to give a flow of 80 to 95	Y
Neutralized Vinsol <sup>6</sup> resin solution, mL, <sup>A</sup> sufficient to produce an air content of 18 ± 3 %	Z

<sup>A</sup>The amount of Vinsol resin solution used shall be considered as part of the mixing water.

24.2 The neutralized Vinsol resin solution used in this section on Air-Entrainment of Mortar shall be either a commercial neutralized Vinsol resin solution or a neutralized Vinsol resin solution prepared in accordance with Specification C 226. If it is necessary to dilute either of these solutions, use distilled or demineralized water. (Note 9.)

NOTE 9—Dissolved minerals in drinking water may precipitate Vinsol resin solutions and greatly diminish its air-entraining characteristics.

24.3 Prepare two test mixtures with sufficient neutralized Vinsol<sup>6</sup> resin to produce an air content of 15 to 18 % in the first mix and 18 to 21 % in the second mix. Then, determine by interpolation the amount of Vinsol resin, expressed as weight percent of the cement, required to produce an air content of 18 %.

#### 25. Calculation

25.1 Calculate the air content of the test mixtures as follows:

$$\text{Air content, volume \%} = 100[1 - (W_a/W_c)] \quad (8)$$

$$W_a = W/400 \quad (9)$$

<sup>6</sup> Available from Hercules, Inc., 917 King St., Wilmington, DE 19899.

$$W_c = \frac{300 + 1125 + 75 + (300 \times P \times 0.01)}{\left[ \left( \frac{300}{3.15} \right) + \left( \frac{1125}{2.65} \right) + \left( \frac{75}{D} \right) + \left( \frac{300 \times P \times 0.01}{1} \right) \right]} \quad (10)$$

where:

$W_a$  = actual weight per unit of volume of mortar as determined by Test Method C 185, g/mL,

$W$  = weight of the specified 400 mL of mortar (see Test Method C 185), g,

$W_c$  = theoretical weight per unit of volume, calculated on an air-free basis and using the values for density and quantities of the materials in the mix, g/mL,

$P$  = percentage of mixing water plus Vinsol resin solution based on weight of cement, and

$D$  = density of mineral admixture in the mixture, Mg/m<sup>3</sup>.

### STRENGTH ACTIVITY INDEX WITH PORTLAND CEMENT

#### 26. Specimens

26.1 Mold the specimens from a control mixture and from a test mixture in accordance with Test Method C 109/C 109M. The portland cement used in the Strength Activity Index test shall comply with the requirements of Specification C 150 and with the alkali and strength limits given in the section on Materials. In the test mixture, replace 20 % of the mass of the amount of cement used in the control mixture by the same mass of the test sample. Make six-cube batches as follows:

##### 26.1.1 Control Mixture:

500 g of portland cement  
1375 g of graded standard sand  
242 mL of water

##### 26.1.2 Test Mixture:

400 g of portland cement  
100 g of test sample  
1375 g of graded standard sand  
mL of water required for flow ±5 of control mixture

#### 26.2 Number of Specimens:

26.2.1 Since Specification C 618 specifies that “meeting the 7 day or 28 day Strength Activity Index will indicate specification compliance” only one age might be required. At the option of the producer or the user after preparing six-cube batches, only three cubes of control and test mixtures need to be molded for either 7 or 28 day testing.

#### 27. Storage of Specimens

27.1 After molding, place the specimens and molds (on the base plates) in the moist room or closet at 23.0 ± 2.0°C (73.4 ± 3°F) for 20 to 24 h. While in the moist room or closet, protect the surface from dripping water. Remove the molds from the moist room or closet and remove the cubes from the molds. Place and store the cubes in saturated lime water as specified in Test Method C 109/C 109M.

NOTE 10—Take care to ensure against zones of stratification or pockets of variation in temperature in the curing chamber.

#### 28. Compressive Strength Test

28.1 Determine the compressive strength, as specified in

Test Method C 109/C 109M, of three specimens of the control mixture and three specimens of the test mixture at ages of 7 days, or 28 days, or both, depending upon how many specimens were molded as prescribed in the section on Number of Specimens.

### 9. Calculation

29.1 Calculate the strength activity index with portland cement as follows:

$$\text{Strength activity index with portland cement} = (A/B) \times 100 \quad (11)$$

where:

*A* = average compressive strength of test mixture cubes, MPa (psi), and

*B* = average compressive strength of control mix cubes, MPa (psi).

### WATER REQUIREMENT

### 30. Calculation

30.1 Calculate the water requirement for the Strength Activity Index with Portland Cement as follows:

$$\text{Water requirement, percentage of control} = (Y/242 \times 100) \quad (12)$$

where:

*Y* = water required for the test mixture to be  $\pm 5$  of control flow.

### EFFECTIVENESS OF MINERAL ADMIXTURE IN CONTROLLING ALKALI SILICA-REACTIONS (SEE Appendix X1)

### 31. Procedure

31.1 Determine expansion of mortar made with a mineral admixture and a test cement as a percent of expansion of mortar made with low alkali cement in accordance with Test Method C 441 as modified in the following paragraphs:

31.1.1 *Control Mixture*—The control mixture will be made as required in Test Method C 441 except that the control cement shall have an alkali content (as equivalent  $\text{Na}_2\text{O}$ ) less than 0.60 % (Note 11).

NOTE 11—Generally, the control cement should have an alkali content as equivalent  $\text{Na}_2\text{O}$  between 0.50 and 0.60 %. However, lower alkali control cement may be used, if desired, to demonstrate equivalence.

31.1.2 *Test Mixture Using Mineral Admixture*—The combined quantity of cement plus mineral admixture shall total 400 g (see Appendix X1). Use 900 g of borosilicate glass<sup>7</sup> aggregate and sufficient mixing water to produce a flow between 100 and 115 % as determined in accordance with Test Method C 109/C 109M. The cement used in the test mixture shall have an alkali content greater than that of the cement in the control mixture (Note 12).

<sup>7</sup> Pyrex brand glass No. 7740 is available from Corning Glass Works, Corning, NY.

NOTE 12—Generally, this test cement will have an alkali content equal to or higher than that used in the job.

31.1.3 Store and measure specimens as required in Test Method C 227. Measure length of specimens at ages of 1 and 14 days.

### 32. Report

32.1 Include in the report for each test mixture comparison made:

32.1.1 The 14 day expansion of the test mixture as a percent of the control mixture at that age,

32.1.2 The identification and chemical analysis of the mineral admixture,

32.1.3 The mass percentage of mineral admixture based on the total mass of cement plus mineral admixture in the test mixture, and

32.1.4 The alkali content of control and test mixture cements as equivalent alkali ( $\text{Na}_2\text{O} + 0.658 \text{K}_2\text{O}$ ).

### EFFECTIVENESS OF MINERAL ADMIXTURES IN CONTRIBUTING TO SULFATE RESISTANCE

### 33. Procedure

33.1 Compare the length change of mortar bars with absolute expansion limits or compare the length change of mortar bars made with a control cement with the length change of mortar bars made with fly ash or natural pozzolan and a test cement, in accordance with Test Method C 1012, as modified in the following paragraphs. Results shall be evaluated using absolute limits (Procedure A) or the relative expansion limits (Procedure B) in Specification C 618, Table 2A.

33.2 *Control Mixture for Procedure A*—A control mixture is not mandatory since performance measured using Procedure A is based on maximum expansion of the test mixture. If a control mixture is made for Procedure A then proportion it as required in Test Method C 1012 using a cement meeting the requirements of Specification C 150, Type II or Type V.

33.3 *Test Mixture Using Fly Ash or Natural Pozzolan for Procedure A*—Make the combined quantity of cement plus fly ash or natural pozzolan, by mass, the same as the total cement content of the control mixture described in the Making Mortars section of Test Method C 1012. The proportion of fly ash or natural pozzolan may be varied using from 15 % to 50 %, by mass, of the total cement plus fly ash or natural pozzolan. Any type of portland cement may be used to prepare test mixtures.

33.4 *Control Mixture for Procedure B*—Make the control mixture for Procedure B as required in Test Method C 1012 with the cement that is proposed for use in the project or a cement that through performance or definition (Specification C 150, Section 1) is expected to give satisfactory results (or a cement for which the contribution to sulfate resistance is known and is satisfactory).

NOTE 13—The control cement should be chosen to give sulfate resistance for the expected level of sulfate exposure. Experience has shown that Type II cements are often used for moderate levels of exposure. Type V cement is commonly used for severe exposures (see X2.2 of Appendix X2).

33.5 *Test Mixture for Using Fly Ash or Natural Pozzolan for Procedure B*—Make the combined quantity of cement plus fly

ash or natural pozzolan, by mass, the same as the total cement used in the control mixture. The proportion of fly ash or natural pozzolan may be varied from 15 % to 50 % of the total cement plus fly ash or natural pozzolan by mass. Any type of portland cement may be used to prepare test mixtures.

33.6 For either Procedure A or B, store and measure specimens as required in Test Method C 1012 for at least 6 months.

NOTE 14—Evaluation of the sulfate resistance of cementitious materials for use in certain situations or critical structures may require longer periods of storage and additional length measurements. Refer to Test Method C 1012 for guidance on this matter.

### 34. Report

34.1 In addition to the information required in Test Method C 1012, report the following information for each mixture comparison made:

34.2 For Procedure A, the age and expansion as a specific amount,

34.3 For Procedure B, the age and expansion as a percent of the control mixture at that age,

34.4 The identification and chemical analysis including  $C_3A$  content of the cements used in both the control and test mixtures,

34.5 The identification and chemical analysis of the fly ash or natural pozzolan used in the test mixtures,

34.6 The mass percentages of the fly ash or natural pozzolan based on the total mass of cement plus fly ash or natural pozzolan in the test mixture.

### PRECISION AND BIAS

### 35. Precision and Bias

#### 35.1 Strength Activity Index Test:

**TABLE 2 Within and Between Lab Precision Estimates for C 114 Methods Applied to the Analysis of Fly Ash<sup>A</sup>**

Oxide	1s		d2s	
	W/L	B/L	W/L	B/L
SiO <sub>2</sub>	0.62	0.86	1.75	2.45
Al <sub>2</sub> O <sub>3</sub>	1.46	2.29	4.12	6.48
Fe <sub>2</sub> O <sub>3</sub>				
<2 %	0.12	0.11	0.34	0.31
≥2 %	0.12	0.25	0.34	0.71
CaO	0.63	0.74	1.79	2.09
MgO	0.20	0.21	0.55	0.61
SO <sub>3</sub>	0.10	0.16	0.28	0.45
Moisture	0.05	0.06	0.15	0.17
LOI	0.09	0.12	0.25	0.35

<sup>A</sup>The four values for SO<sub>3</sub> were editorially corrected to reflect updated research reports associated with this standard.

35.1.1 Precision was determined from two interlaboratory studies involving two Class C fly ashes, two Class F fly ashes, and one Class N pozzolan. Participating laboratories numbered 12 and 7 in the two studies.<sup>8</sup>

35.1.2 The single-operator standard deviation for the Strength Activity Index test has been found to be 3.7 % (1s). This does not appear to vary either with material or with test age, over the range of 7 to 28 days. Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than 10.5 % (d2s) of the average of the two results.

35.1.3 The multilaboratory standard deviation for the Strength Activity Index test has been found to be 4.5 % (1s). This does not appear to vary either with material or with test age, over the range of 7 to 28 days. Therefore, results of two properly conducted tests in different laboratories on the same material should not differ by more than 12.7 % (d2s) of the average of the two results.

35.1.4 Since there is no accepted reference material suitable for determining the bias for this procedure, no statement on bias is being made.

#### 35.2 Chemical Analysis:

35.2.1 Precision and bias estimates for the test methods in C 114 when applied to the analysis of pozzolans were calculated from an interlaboratory study involving 7 laboratories each analysing 4 NIST SRM fly ashes. A research report describing the results of this study is available from ASTM.<sup>9</sup>

35.2.2 *Precision*—Within-laboratory (W/L) and between-laboratory (B/L) estimates of standard deviation and estimates of maximum differences expected between duplicate determinations in 95 % of comparisons<sup>10</sup> are summarized in Table 2.

35.2.3 *Bias*—Statistically significant bias was found in the determination of CaO and MgO. CaO determinations averaged 0.46 % higher than the value certified for the SRM. MgO determinations averaged 0.19 % higher than the value certified for the SRM.

### 36. Keywords

36.1 fly ash; natural pozzolan

<sup>8</sup> A research report summarizing the statistical analysis is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Request RR: C09-1001.

<sup>9</sup> A research report is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Request RR: C09-1008.

<sup>10</sup> These values represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.



APPENDIXES

(Nonmandatory Information)

X1. COMMENTARY ON SECTION 31

X1.1 Test procedures in Section 31 are designed to determine the effectiveness of fly ash, or natural pozzolan, in preventing excessive expansion resulting from a reaction between certain aggregates and alkalis in portland-cement mixtures. Tests are made in accordance with Test Method C 441 using:

- X1.1.1 400 g of portland cement or a combined total of cement plus fly ash or natural pozzolan of 400 g,
- X1.1.2 900 g of borosilicate glass,<sup>7</sup> and
- X1.1.3 Sufficient water to obtain a flow of 100 to 115 %.

X1.2 The control mixture is made with a low-alkali portland cement. The test mixture, made of a test percentage of the fly ash or natural pozzolan, can be made with the same cement or any other cement having an equivalent alkali content greater than 0.60 %.

X1.3 *Interpretation of Results*—The fly ash or natural pozzolan should be considered “effective” when used at percentages equal to or greater than the percentages used in the test mixture with cements having alkali contents that do not exceed by more than 0.05 percentage points the alkali content of the cement used in the test mixture.

X1.4 *Selection of the Percentage of Fly Ash or Natural Pozzolan*—It may be necessary to make test mixtures with several percentages of fly ash or natural pozzolan to determine the minimum amount necessary to be considered “effective” in reducing expansion to the level produced by the “control” low-alkali cement. The minimum amount of fly ash or natural pozzolan should be 15 % by weight of cementitious material.

X1.5 *Selection of the Alkali Content of the Cement Used in the Test Mixture*—In some instances it may not be necessary to demonstrate that the fly ash or natural pozzolan will reduce expansion, but rather that it will not increase expansion. In this instance the test and control mixtures should be made with the same low alkali cement used in the control mixture to better

delineate any increase in expansion. The fly ash or natural pozzolan percentage used may need to be sufficiently high to demonstrate that the percentage exceeds the pessimum if such pessimum exists for the combination.

X1.6 *Selection of the Low Alkali Control Cement*—The test procedure in this test method and requirements in Specification C 618 are designed to measure the relative effectiveness of the fly ash or natural pozzolan in reducing expansion due to alkali-silica reactions. The effectiveness is a function of both the alkali content of the cement used in the test mixture and the percentage of fly ash or natural pozzolan. The higher the alkali content of the test mixture cement and the lower the percentage of fly ash or natural pozzolan used, the more effective the fly ash or natural pozzolan. This test procedure and the 100 % criterion of Specification C 618 can be used as a guide to enhance field performance of job mixtures if it is assumed that use of the low alkali control cement will prevent excessive expansion in service with materials proposed for use in the job. This is the situation when the aggregates proposed for use contain rapidly reactive constituents but do not exceed 0.05 % expansion in three months or 0.10 % expansion in six months when tested with the control cement in Test Method C 227. In such instances the control cement used in this test method should have an alkali content as required to control the expansive reactions of the aggregates being used, for example in the 0.50 to 0.60 % range. See the appendix to Specification C 33.

X1.6.1 Certain reactive aggregates have been identified which produce deleterious expansion after many years of service when used with cements with equivalent alkali contents well below 0.60 %. Often these reactive aggregates do not produce expansions in Test Method C 227 exceeding those discussed in the appendix of Specification C 33 until a much later age, perhaps one or two years. In such instances the use of a control cement with an alkali content significantly less than 0.60 % is appropriate.

X2. COMMENTARY ON SECTION 33

X2.1 The test procedures in Section 33 are designed to evaluate the performance of particular fly ashes or natural pozzolans in contributing to the resistance or durability of concrete in a sulfate environment. Tests are made on mortars in accordance with Test Method C 1012.

X2.2 The control mixture should be made with a cement that by performance or definition (Specification C 150, Section 1.1) is expected to give satisfactory results for the anticipated level of sulfate exposure. The American Concrete Institute Guide to Durable Concrete (ACI 201.2R) recommends Type II cement when the sulfate content, expressed as SO<sub>4</sub>, is 0.10 to

0.20 percent in soils or 150 to 1500 ppm in waters. The American Concrete Institute Guide to Durable Concrete (ACI 201.2R) recommends Type V cements when the sulfate content, expressed as SO<sub>4</sub>, is 0.20 to 2.0 % in soils, or 1500 to 10 000 ppm in waters. The test mixtures for both procedure may be made with varying percentages of fly ash or natural pozzolan. Test mixtures can use cements with equal or higher or lower C<sub>3</sub>A contents that might not have given satisfactory results when used alone.

X2.3 *Interpretation of Results*—The absolute expansion limits used in Procedure A make it difficult to ascertain how the

addition of fly ash or natural pozzolan influences sulfate resistance. However, as referenced in Test Method C 1012, research has indicated that mortars meeting the expansion criteria will perform adequately. Under Procedure B, the fly ash or natural pozzolan will be considered to be able to contribute to sulfate resistance if the expansion of the test mixture does not exceed that of the control mixture at an exposure time of at least 6 months. It is recommended that the proportion of fly ash or natural pozzolan used in the test mixture be the same one proposed for use in the project  $\pm 2\%$ , and that the  $C_3A$  content of the project cement be equal to, or less than, that which was

used in the test mixtures. See Appendix X1, Technical Background, Test Method C 1012.

*X2.4 Selection of the Percentage of Fly Ash or Natural Pozzolan*—It may be necessary to make test mixtures with several percentages of fly ash or natural pozzolan to determine the amount necessary to obtain adequate resistance to different concentrations of sulfates. Percentages used in a project should be within 2 % of those that are successful in the test mixtures or should be between two percentages that are successful.

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# Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance<sup>1</sup>

This standard is issued under the fixed designation C 403/C 403M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This test method covers the determination of the time of setting of concrete, with slump greater than zero, by means of penetration resistance measurements on mortar sieved from the concrete mixture.

1.2 This test method is suitable for use only when tests of the mortar fraction will provide the information required.

1.3 This test method may also be applied to prepared mortars and grouts.

1.4 This test method is applicable under controlled laboratory conditions, as well as under field conditions.

1.5 The values stated in either inch-pound units or SI units shall be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining values in any way.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 125 Terminology Relating to Concrete and Concrete Aggregates<sup>2</sup>

C 143/C 143M Test Method for Slump of Hydraulic Cement Concrete<sup>2</sup>

C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>

C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method<sup>2</sup>

C 192/C 192M Practice for Making and Curing Concrete Test Specimens in the Laboratory<sup>2</sup>

C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method<sup>2</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

D 1558 Test Method for Moisture Content Penetration Re-

sistance Relationships of Fine Grained Soils<sup>3</sup>

E 1 Specification for ASTM Thermometers<sup>4</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>5</sup>

## 3. Terminology

3.1 *Definitions*—Definitions are given in Terminology C 125.

## 4. Summary of Test Method

4.1 A mortar sample is obtained by sieving a representative sample of fresh concrete. The mortar is placed in a container and stored at a specified ambient temperature. At regular time intervals, the resistance of the mortar to penetration by standard needles is measured. From a plot of penetration resistance versus elapsed time, the times of initial and final setting are determined.

## 5. Significance and Use

5.1 Since the setting of concrete is a gradual process, any definition of time of setting must necessarily be arbitrary. In this test method, the times required for the mortar to reach specified values of resistance to penetration are used to define times of setting.

5.2 This test method can be used to determine the effects of variables, such as water content; brand, type and amount of cementitious material; or admixtures, upon the time of setting of concrete. This test method may also be used to determine compliance with specified time-of-setting requirements.

5.3 This test method may also be applied to prepared mortars and grouts. However, when the setting time of concrete is desired, the test shall be performed on mortar sieved from the concrete mixture and not on a prepared mortar intended to simulate the mortar fraction of the concrete; it has been shown that the initial and final setting times may be increased when using the prepared mortar.

## 6. Apparatus

6.1 *Containers for Mortar Specimens*—The containers shall be rigid, watertight, nonabsorptive, free of oil or grease, and

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.23 on Chemical Admixtures.

Current edition approved Sept. 10, 1999. Published September 1999. Originally published as C 403-57 T. Last previous edition C 403/C 403M-97.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.08.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 14.02.

either cylindrical or rectangular in cross section. Mortar surface area shall be provided for ten undisturbed readings of penetration resistance in accordance with clear distance requirements specified in Procedure. The lateral dimension shall be at least 6 in. [150 mm] and the height at least 6 in. [150 mm].

6.2 *Penetration Needles*—Needles shall be provided which can be attached to the loading apparatus and which have the following bearing areas: 1, 1/2, 1/4, 1/10, 1/20, and 1/40 in.<sup>2</sup> [645, 323, 161, 65, 32, and 16 mm<sup>2</sup>]. Each needle shank shall be scribed circumferentially at a distance 1 in. [25 mm] from the bearing area. The length of the 1/40 in.<sup>2</sup> [16-mm<sup>2</sup>] needle shall be not more than 3 1/2 in. [90 mm].

6.3 *Loading Apparatus*—A device shall be provided to measure the force required to cause penetration of the needles. The device shall be capable of measuring the penetration force with an accuracy of  $\pm 2$  lbf [10 N] and shall have a capacity of at least 130 lbf [600 N].

NOTE 1—Suitable loading apparatus can be of the spring-reaction type as described in Test Method D 1558, or of other types with a calibrated force measuring device, such as an electronic load cell or a hydraulic pressure gage.

6.4 *Tamping Rod*—The tamping rod shall be a round, straight, steel rod 5/8 in. [16 mm] in diameter and approximately 24 in. [600 mm] in length, having the tamping end or both ends rounded to a hemispherical tip, the diameter of which is 5/8 in. [16 mm].

6.5 *Pipet*—A pipet or other suitable instrument shall be used for drawing off bleed water from the surface of the test specimen.

6.6 *Thermometer*—The thermometer shall be capable of measuring the temperature of the fresh mortar to  $\pm 1^\circ\text{F}$  [ $\pm 0.5^\circ\text{C}$ ]. ASTM liquid-in-glass thermometers having a temperature range from 0 to 120°F [−20 to 50°C], and conforming to the requirements of Thermometer 97F (or 97C) as prescribed in Specification E 1 are satisfactory. Other thermometers of the required accuracy, including the metal immersion type, are acceptable.

## 7. Sampling, Test Specimens, and Test Units

7.1 For tests under field conditions, prepare three specimens from each sample of concrete.

7.2 For tests under laboratory conditions, the requirements depend upon the purpose of the tests.

7.2.1 For testing to prove compliance of a material with performance requirements, make at least three separate concrete batches for each variable under investigation. Perform one time of setting test on each batch. Make an equal number of batches for each variable on any one day. When it is impossible to perform at least one test for each variable on any one day, mix the entire series of batches in as few days as possible, and repeat one of the mixtures each day as a standard for comparison.

7.2.2 For other tests, prepare three test specimens from one batch of concrete for each test variable.

7.3 Record the time at which initial contact was made between cement and mixing water.

7.4 For tests under field conditions, obtain a representative sample of the fresh concrete in accordance with Practice C 172.

For tests under laboratory conditions, make the concrete in accordance with Practice C 192. Determine and record the slump (Test Method C 143) and air content (Test Method C 173 or C 231) of the fresh concrete.

7.5 From the concrete not used in the slump and air content tests, select a representative portion of sufficient volume to provide enough mortar to fill the test container, or containers, to a depth of at least 5 1/2 in. [140 mm].

7.6 Using the procedure in Practice C 172, obtain a mortar sample by wet-sieving the selected portion of concrete through a 4.75-mm sieve<sup>6</sup> and onto a nonabsorptive surface.

7.7 Thoroughly remix the mortar by hand methods on the nonabsorptive surface. Measure and record the temperature of the mortar. Place the mortar in the container, or containers, using a single layer. Consolidate the mortar to eliminate air pockets in the specimen and level the top surface. This may be accomplished by rocking the container back and forth on a solid surface, by tapping the sides of the container with the tamping rod, by rodding the mortar, or by placing the container on a vibrating table (see Note 2). If rodding is used, rod the mortar with the hemispherical end of the tamping rod. Rod the mortar once for each 1 in.<sup>2</sup> [645 mm<sup>2</sup>] of top surface area of the specimen and distribute the strokes uniformly over the cross section of the specimen. After completion of the rodding, tap the sides of the containers lightly with the tamping rod to close voids left by the tamping rod and to further level the surface of the specimen. Upon completion of specimen preparation, the mortar surface shall be at least 1/2 in. [10 mm] below the top edge of the container to provide space for the collection and removal of bleed water and to avoid contact between the mortar surface and the protective covering specified in Section 8.

NOTE 2—Sieved mortar is generally of fluid consistency and air pockets are readily removed by the listed consolidation methods. The user should exercise judgment in the selection of the consolidation method. Rocking the container or tapping of the sides should be sufficient for fluid mortars. Rodding or using a vibrating table may be desirable for stiffer mortars. When using a vibrating table, use low-amplitude vibration so that portions of the sample are not ejected from the container.

## 8. Conditioning

8.1 For tests under laboratory conditions, the storage temperature for specimens shall be within the range 68 to 77°F [20 to 25°C], or as specified by the user.

8.2 For tests under field conditions, store the specimens under ambient conditions, or as specified by the user. Shield the specimens from direct sunlight.

8.3 Measure and record the ambient air temperature at the start and finish of the test. To prevent excessive evaporation of moisture, keep the specimens covered with a suitable material such as damp burlap or a tight-fitting, water-impermeable cover for the duration of the test, except when bleed water is being removed or penetration tests are being made.

## 9. Procedure

9.1 Just prior to making a penetration test, remove bleed water from the surface of the mortar specimens by means of a

<sup>6</sup> Detailed requirements for this sieve are given in Specification E 11.

pipet or suitable instrument. To facilitate collection of bleed water, tilt the specimen carefully to an angle of about 10° from the horizontal by placing a block under one side 2 min prior to removal of the water.

9.2 Insert a needle of appropriate size, depending upon the degree of setting of the mortar, in the penetration resistance apparatus and bring the bearing surface of the needle into contact with the mortar surface. Gradually and uniformly apply a vertical force downward on the apparatus until the needle penetrates the mortar to a depth of  $1 \pm \frac{1}{16}$  in. [ $25 \pm 2$  mm], as indicated by the scribe mark (Note 3). The time required to penetrate to the 1-in. [25-mm] depth shall be  $10 \pm 2$  s. Record the force required to produce the 1-in. [25-mm] penetration and the time of application, measured as elapsed time after initial contact of cement and water. Calculate the penetration resistance by dividing the recorded force by the bearing area of the needle, and record the penetration resistance. In subsequent penetration tests take care to avoid areas where the mortar has been disturbed by previous tests. The clear distance between needle impressions shall be at least two diameters of the needle being used, but not less than  $\frac{1}{2}$  in. [15 mm]. The clear distance between any needle impression and the side of the container shall be at least 1 in. [25 mm].

NOTE 3—To facilitate determination of when the required penetration has been attained, a sliding marker may be attached to the needle shaft. For example, a paper clip or masking tape may be placed on the shaft so that it coincides with the scribe mark. The marker should not interfere with the penetration of the needle into the mortar. The position of the marker should be checked prior to making a penetration.

9.3 For conventional concrete mixtures at laboratory temperatures of 68 to 77°F [20 to 25°C], make the initial test after an elapsed time of 3 to 4 h after initial contact between cement and water. Subsequent tests should be made at  $\frac{1}{2}$ - to 1-h intervals. For concrete mixtures containing accelerators, or at temperatures higher than laboratory, it is advisable to make the initial test after an elapsed time of 1 to 2 h and subsequent tests at  $\frac{1}{2}$ -h intervals. For concrete mixtures containing retarders, or at temperatures lower than laboratory, the initial test may be deferred until an elapsed time of 4 to 6 h. In all cases, time intervals between subsequent tests may be adjusted as necessary, depending upon the rate of setting, to obtain the required number of penetrations.

9.4 Make at least six penetrations for each time-of-setting test, with time intervals of such duration as to provide a satisfactory curve of penetration resistance versus elapsed time (Note 4). Continue testing until one at least penetration resistance reading equals or exceeds 4000 psi [27.6 MPa].

NOTE 4—A satisfactory curve is one which represents the overall development of penetration resistance and includes points before and after the times of initial and final setting to improve the accuracy of the required interpolation. For normal setting mixtures, test points are usually at equally spaced time intervals. Premature penetration testing will result in too many data points earlier than the initial setting time. This may decrease the accuracy of the estimated setting time by biasing the best fit line when regression analysis is used to analyze the penetration resistance data.

9.5 *Plotting Test Results*—One of the following alternative procedures may be used to plot the test results and obtain times

of setting (Note 5). Appendix X1 illustrates the application of these procedures.

NOTE 5—The plot of penetration resistance versus elapsed time provides information on the rate of setting. The plot may be used to select the time for subsequent penetration tests and it can assist in identifying spurious test results. Therefore, it is recommended that the data be plotted as they are being accumulated.

9.5.1 Use the following plotting procedure to determine the times of setting by hand-fitting a smooth curve through the data. Prepare a graph of penetration resistance, as the ordinate, versus elapsed time, as the abscissa, using a scale such that 500 psi [3.5 MPa] and 1 h are each represented by a distance of at least  $\frac{1}{2}$  in. [15 mm]. Plot the values of penetration resistance as a function of elapsed time.

9.5.2 Use the following plotting procedure to determine the times of setting by linear regression analysis of the logarithms of the data by using a suitable calculator. Using log-log graph paper, prepare a graph of penetration resistance, as the ordinate, versus elapsed time in minutes, as the abscissa. The limits of penetration resistance on the ordinate should extend from 10 psi [0.1 MPa] to 10 000 psi [100 MPa], and the limits of elapsed time on the abscissa should extend from 10 to 1000 min. If slow setting mixtures are used, the time limits may have to be 100 to 10 000 min. Plot the values of penetration resistance as a function of elapsed time (Note 4).

9.5.3 Use the following procedure if a computer is used to plot test results and obtain the times of setting by regression analysis of the data. As the test results are obtained, enter the time and penetration resistance into the computer and plot the penetration resistance as the ordinate and the elapsed time as the abscissa. For software that permits only linear regression analysis, convert the data by taking their logarithms. The converted data will be fitted by a straight line<sup>7</sup> (see Eq 1):

$$\text{Log}(PR) = a + b \text{Log}(t) \quad (1)$$

where:

$PR$  = penetration resistance  
 $t$  = elapsed time  
 $a$  and  $b$  = regression constants

The data do not have to be converted if the software permits direct fitting of a power function:

$$PR = ct^d \quad (2)$$

where:

$c$  and  $d$  = regression constants

9.5.4 The procedures in 9.5.2 and 9.5.3 assume that the data obey (Eq 1) or (Eq 2). Verify that the data obey one of these relationships. If the correlation coefficient for the regression analysis, after removal of outliers (see Note 6), is less than 0.98, use the procedure in 9.5.1.

## 10. Calculation

10.1 For each variable under investigation, separately plot the results of three or more time-of-setting tests. For each plot prepared according to 9.5.1, hand fit a smooth curve to the data

<sup>7</sup> Popovics, S., 1971, "Physical Aspects of the Setting of Portland Cement Concrete," *Journal of Materials*, JMLSA, Vol. 6, No. 1, March, pp. 150-162.

points. For each plot prepared according to 9.5.2 or 9.5.3, use the method of least squares to obtain the constants of the best-fit relationship given by (Eq 1) or (Eq 2), whichever is applicable. Disregard data points that are obvious outliers from the trend defined by the rest of the points (Note 6).

**NOTE 6**—Outliers may occur because of factors such as: interferences due to the larger particles in the mortar; presence of large voids within the penetration zone; interferences from the impressions created by adjacent penetrations; failure to maintain the instrument perpendicular to the test surface during penetration; errors in reading the load; variations in the penetration depths; or variations in rate of loading. Judgement of the operator is required to identify those points that should not be included in the data analysis.

10.2 For each plot, determine the times of initial and final setting as the times when the penetration resistance equals 500 psi [3.5 MPa] and 4000 psi [27.6 MPa], respectively. For plots made according to 9.5.1, determine times of setting by visual inspection of the drawn curves. For plots made according to 9.5.2 or 9.5.3, determine the times of setting by interpolation using the best-fit regression equation. Record the times of setting in hours and minutes to the nearest 5 min.

10.3 For each variable under investigation, calculate the times of initial and final setting as the average values of the individual test results. Record the average times in hours and minutes to the nearest 5 min.

## 11. Report

11.1 *Data on Concrete Mixture*—Report the following information on the concrete mixture:

11.1.1 Brand and type of cementitious materials, amounts (mass) of cementitious materials, fine aggregate and coarse aggregate per cubic yard [per cubic metre] of concrete, nominal maximum aggregate size, and water-cement or water-cementitious material ratio,

11.1.2 The name, type, and amount of admixture(s) used,

11.1.3 Air content of fresh concrete and method of determination,

11.1.4 Slump of concrete,

11.1.5 Temperature of mortar after sieving,

11.1.6 Record of ambient temperature during the test period, and

11.1.7 Date of test.

11.2 *Time of Setting Results*—Report the following information on the time of setting tests:

11.2.1 A plot of penetration resistance versus elapsed time for each time of setting test,

11.2.2 The times of initial and final setting for each test, reported in hours and minutes to the nearest minute, and

11.2.3 The average times of initial and final setting for each

test condition, reported in hours and minutes to the nearest 5 min.

## 12. Precision and Bias

12.1 The following precision statements are based on testing of prepared mortar using the inch-pound system and for data analysis using the hand-plotting procedure (see 9.5.1, 10.1, and 10.2).

12.2 The single-operator coefficient of variation for time of initial setting for single tests on each of three batches made on separate days has been found to be 7.1 % (Note 7). Therefore, the range of results obtained on three separate batches by the same operator with the same apparatus, using the same materials and temperature conditions, on three different days should not exceed 23 % of their average.

12.3 The multilaboratory coefficient of variation for the average time of initial setting, based on three tests from separate batches made on separate days, has been found to be 5.2 %. Therefore, the averages of three tests from two different laboratories using the same materials and temperature conditions should not differ by more than 15 % of their average (Note 8).

12.4 The single-operator coefficient of variation for time of final setting for single tests on each of three batches made on separate days has been found to be 4.7 % (Note 7). Therefore, the range of results obtained on three separate batches by the same operator with the same apparatus, using the same materials and temperature conditions, on three different days should not exceed 16 % of their average.

12.5 The multilaboratory coefficient of variation for the average time of final setting, based on three tests from separate batches made on separate days, has been found to be 4.5 %. Therefore, the averages of three tests from two different laboratories using the same materials and temperature conditions should not differ by more than 13 % of their average (Note 8).

**NOTE 7**—These numbers are in a strict sense not the single-operator (1s%) limits described in Practice C 670 because the results were obtained from separate batches made on different days.

**NOTE 8**—These numbers represent, respectively, the (1s%) and (d2s%) limits as described in Practice C 670.

12.6 The bias of this test method cannot be determined because times of setting can be defined only in terms of the test method.

## 13. Keywords

13.1 concrete; mortar; penetration resistance; time of final setting; time of initial setting

APPENDIX

(Nonmandatory Information)

XI. ILLUSTRATIVE EXAMPLES

X1.1 The penetration resistance (*PR*) and the elapsed time (*t*) data in Table X1.1 will be used to illustrate the procedures for determining times of setting.

X1.2 *Hand Fit*—Fig. X1.1 is a plot of the penetration resistance versus elapsed time values in Table X1.1. The smooth curve was drawn by-hand using a flexible drawing curve. The curve was drawn so as to achieve the visual best-fit to the data. Note that the penetration resistance at an elapsed time of 335 min is an obvious outlier, and this point was disregarded in drawing the best-fit curve. Horizontal lines are drawn at penetration resistance values of 500 and 4000 psi. The intersections of the horizontal lines with the curve define the times of initial and final setting, which in this case are 289 min and 389 min, respectively.

X1.3 *Regression Analysis:*

X1.3.1 Fig. X1.2 is a log-log plot of the penetration resistance versus elapsed time values. The plot shows that, with the exception of the outlier, there is approximately a straight line relationship between the logarithms of penetration resistance and elapsed time. The straight line is obtained by linear regression analysis using the logarithms shown in the third and fourth columns of Table X1.1. The equation for this line is:

$$\text{Log}(PR) = -14.196 + 6.871 \text{Log}(t) \quad (X1.1)$$

where:

*PR* = penetration resistance, and  
*t* = elapsed time.

The correlation coefficient is 0.999, and it is, therefore, acceptable to use linear regression analysis.

X1.3.2 To obtain the times of setting, the equation is rewritten as:

$$\text{Log}(t) = \frac{\text{Log}(PR) + 14.196}{6.871} \quad (X1.2)$$

X1.3.3 For time of initial setting, substitute the value 500 for *PR*:

$$\text{Log}(t) = \frac{\text{Log}(500) + 14.196}{6.871} = \frac{2.699 + 14.196}{6.871} = 2.458 \quad (X1.3)$$

TABLE X1.1 Penetration Resistance

Penetration Resistance ( <i>PR</i> ) (psi) <sup>A</sup>	Elapsed Time ( <i>t</i> ) (min)	Log( <i>PR</i> )	Log( <i>t</i> )
44	200	1.643	2.301
110	230	2.041	2.362
216	260	2.334	2.415
540	290	2.732	2.462
1000	320	3.000	2.505
1000	335	3.000	2.525
2000	350	3.301	2.544
2560	365	3.408	2.562
3520	380	3.547	2.580
4440	395	3.647	2.597

<sup>A</sup>MPa = psi × 0.00689.

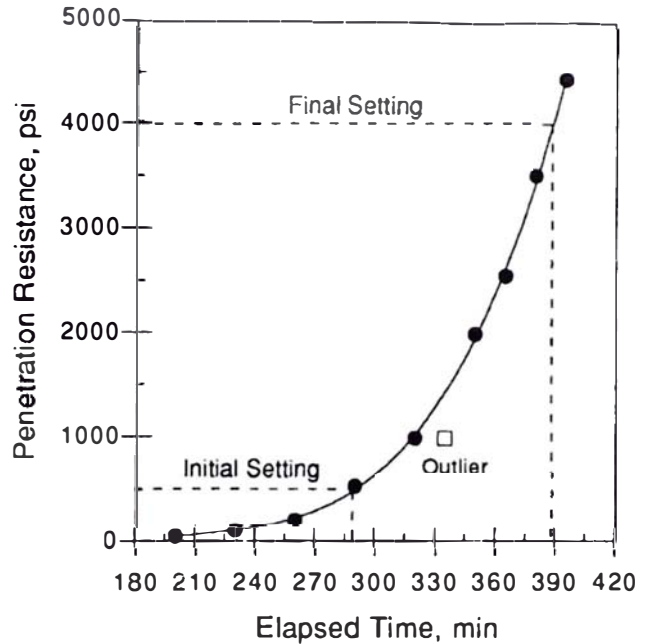


FIG. X1.1 Plot of Penetration Resistance Values Versus Elapsed Time and Hand Fit Curve Used to Determine Time of Setting (Note: Not drawn to actual scale)

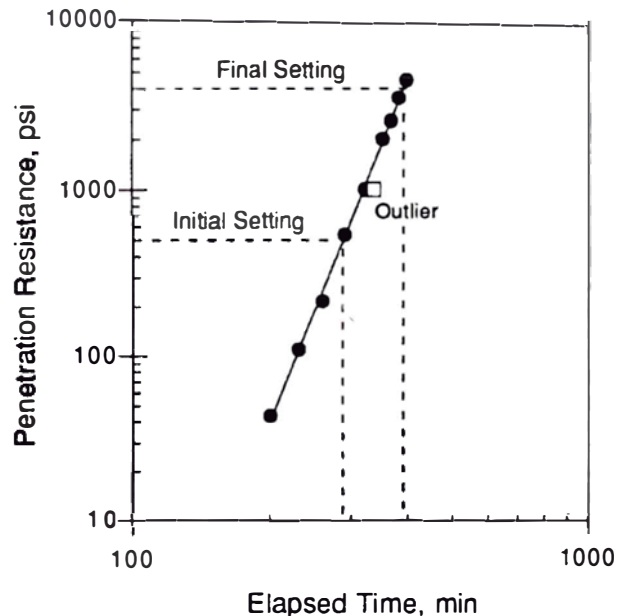


FIG. X1.2 Log-Log Plot Showing Straight Line to Determine Times of Setting by Using Regression Analysis

therefore:  
 $t = (10)^{2.458} = 287 \text{ min.}$

X1.3.4 For time of final setting, substitute the value 4000  
r PR:

therefore:  
 $t = (10)^{2.590} = 389 \text{ min.}$

$$\text{Log}(t) = \frac{\text{Log}(4000) + 14.196}{6.871} = \frac{3.602 + 14.196}{6.871} = 2.590$$

(X1.4)

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# Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete<sup>1</sup>

This standard is issued under the fixed designation C 490; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the requirements for the apparatus and equipment used to prepare specimens for the determination of length change in hardened cement paste, mortar, and concrete, the apparatus and equipment used for the determination of these length changes, and the procedures for its use.

1.2 Methods for the preparation and curing of test specimens, conditions of testing and curing, and detailed procedures for calculating and reporting test results are contained in applicable test methods.

1.3 The values stated in SI units or inch-pound units are to be regarded as standard. The values in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 511 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes<sup>2</sup>

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements<sup>2</sup>

## 3. Terminology

3.1 *length change*—an increase or decrease in the linear dimension of a test specimen, measured along the longitudinal axis, due to causes other than applied load.

## 4. Significance and Use

4.1 This practice is intended to provide standard requirements for apparatus common to many test methods used in connection with cement and concrete and standardized procedures for its use. The detailed requirements as to materials, mixtures, specimens, conditioning of specimens, number of specimens, ages at which measurements are to be made, interpretation of results, and precision and bias are left to be dealt with in specific test methods.

## 5. Apparatus

5.1 *Reference Masses and Devices for Determining Mass and Volume*, shall conform to the requirements of Specification C 1005.

5.2 *Molds*, shall have either one or two compartments and shall be constructed as shown in Fig. 1 or Fig. 2. Molds for test specimens used in determining the length change of cement pastes and mortars shall provide for 25 by 25 by 285-mm prisms having a 250-mm gage length, or for 1 by 1 by 11¼-in. prisms having a 10-in. gage length. Molds for test specimens used in the length change of concretes shall provide for prisms of the desired cross section having a 10-in. or 250-mm gage length. In some routine tests, 25 by 25 by 160-mm specimens with a gage length of 125 mm, or 1 by 1 by 6¼-in. specimens with a gage length of 5-in. are permitted, but in case of dispute, results obtained with specimens of 250-mm [10-in.] gage length shall govern.

5.2.1 The gage length shall be considered as the nominal length between the innermost ends of the gage studs. The parts of the molds shall be tight fitting and firmly held together when assembled, and their surfaces shall be smooth and free of pits. The molds shall be made of steel or other hard metal not readily attacked by the cement paste, mortar, or concrete. The sides of the molds shall be sufficiently rigid to prevent spreading or warping. For the molds shown in Fig. 1, the tolerance on dimension A is  $\pm 0.7$  mm. For the molds shown in Fig. 2, the tolerance on dimension A is  $\pm 0.03$  in.

5.2.2 Each end plate of the mold shall be equipped to hold properly in place, during the setting period, one of the gage studs shown in Fig. 1 or Fig. 2. The gage studs shall be of American Iron and Steel Institute (AISI)<sup>3</sup> Type 316 stainless steel or other corrosion-resistant metal of similar hardness. Gage studs of Invar or similar metal shall be used when specimens are tested at widely different temperatures. To prevent restraint of the gage studs before demolding of the specimen, the device for holding the gage studs in position shall be so arranged that, if necessary, it can be partially or completely released after the compaction of the paste or mortar into place in the mold. The gage studs shall be set so that their principal axes coincide with the principal axis of the test specimen. For the molds shown in Fig. 1, gage studs shall

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.95 on Coordination of Standards.

Current edition approved Nov. 10, 2000. Published January 2001. Originally published as C 490 – 62. Last previous edition C 490 – 00.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.01.

<sup>3</sup> Details on this material are available from the American Iron and Steel Institute, 1133 15th St. N.W., Washington, DC 20005.

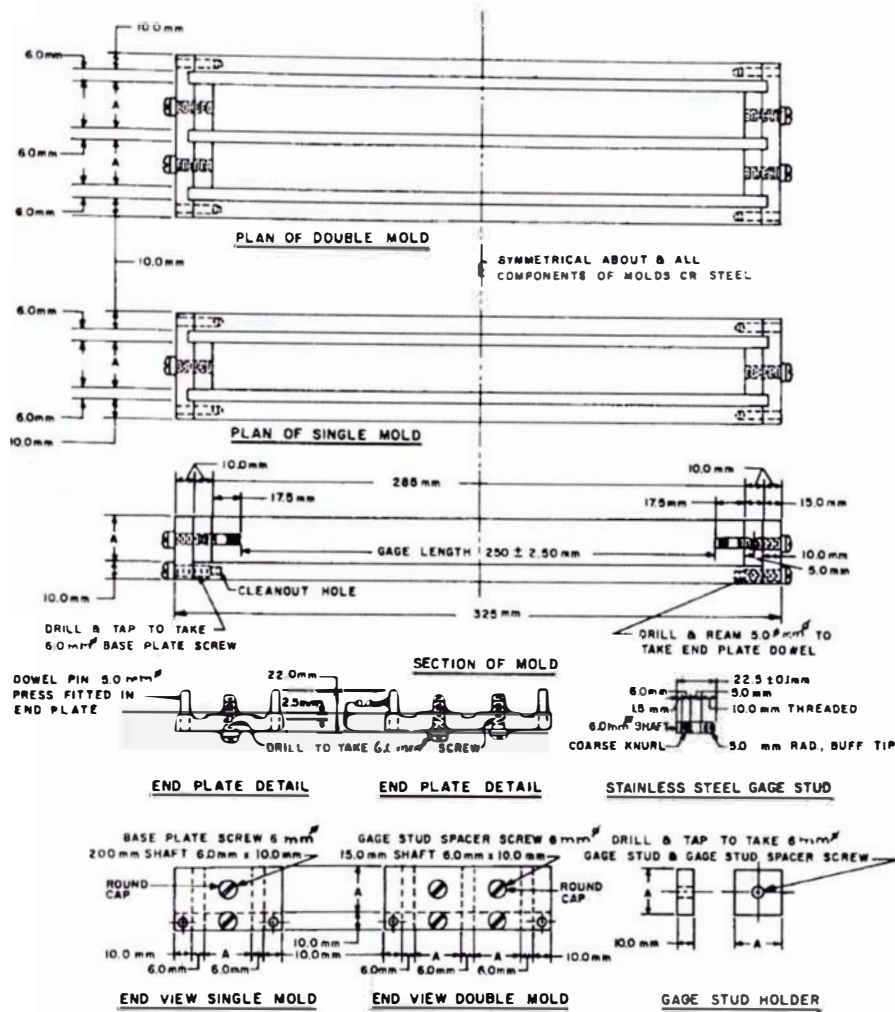


FIG. 1 Molds (SI Units)

extend into the specimen  $17.5 \pm 0.5$  mm and the distance between the inner ends of the gage studs shall be  $250.0 \pm 2.5$  mm and 250 mm shall be considered the gage length for calculating length change. For the molds shown in Fig. 2, gage studs shall extend into the specimen  $0.625 \pm 0.025$  in. and the distance between the inner ends of the gage studs shall be  $10.00 \pm 0.10$  in. and 10 in. shall be considered the gage length for calculating length change.

5.3 *Length Comparator*, for determining length change of specimens, shall be designed to accommodate the size of specimen employed and to provide or permit a positive means of contact with the gage studs and the convenient and rapid obtaining of comparator readings (Note 1).

5.3.1 The comparator for determining length changes of specimens produced in the molds shown in Fig. 1 shall provide a dial micrometer or other measuring device graduated to read in 0.002-mm units or less, accurate within 0.002 mm in any 0.020-mm range, and within 0.004 mm in any 0.200-mm range, and sufficient range (at least 8.0 mm) in the measuring device to allow for small variations in the actual length of various specimens. The terminals of the comparator shall be plane, polished and heat-treated. They shall be fitted with

collars held in place with set screws. The collars shall extend  $1.5 \pm 0.1$  mm beyond the plane face of the terminal and have an inside diameter 0.5 mm greater than the average diameter of the gage studs that must fit into the collars.

NOTE 1—One type of instrument that has been found satisfactory for use with small prisms is shown in Fig. 3. A horizontal comparator should be used with prisms with a cross section greater than 9 in.<sup>2</sup> or 58 cm<sup>2</sup>.

5.3.2 The comparator for determining length changes of specimens produced in the molds shown in Fig. 2 shall provide a dial micrometer or other measuring device graduated to read in 0.0001-in. units, accurate within 0.0001 in. in any 0.0010-in. range, and within 0.0002 in. in any 0.0100-in. range, and sufficient range (at least 0.3 in.) in the measuring device to allow for small variations in the actual length of various specimens. The terminals of the comparator shall be plane, polished and heat-treated. They shall be fitted with collars held in place with set screws. The collars shall extend  $0.062 \pm 0.003$  in. beyond the plane face of the terminal and have an inside diameter 0.02 in. greater than the average diameter of the portion of the gage studs that must fit into the collars.

5.3.3 The design shall provide a means for checking the

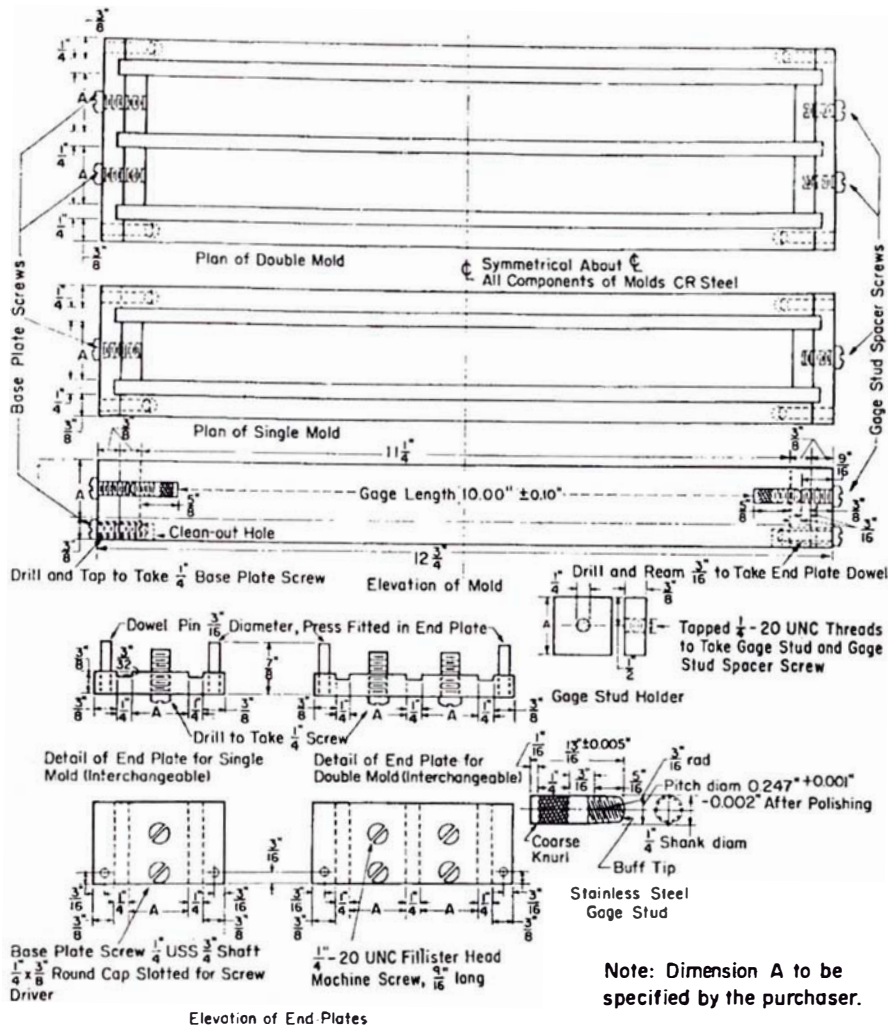


FIG. 2 Molds (Inch-Pound Units)

measuring device against a reference bar at regular intervals. The reference bar shall have an overall length of  $295 \pm 3.0$  mm or  $170 \pm 3.0$  mm [ $11\frac{5}{8} \pm \frac{1}{8}$  in. or  $6\frac{5}{8} \pm \frac{1}{8}$  in.], whichever is appropriate for the specimen in use. The bar shall be of a steel alloy having a coefficient of thermal expansion not greater than two millionths per degree Celsius. Each end of the reference bar shall be fitted with heat treated, hardened, and polished tips machined to the same shape as the contact end of the gage studs used in test specimens. Except for the tips, which are attached after heat treatment, no part of the reference bar shall be heat treated. The central 100 mm [4 in.] of the length of the reference bar shall be covered by a rubber tube with a wall at least 3 mm [ $\frac{1}{8}$  in.] thick to minimize the effect of temperature change during handling. The reference bar shall be provided near one end with a positioning mark.

NOTE 2—Alloys such as invar lose their low coefficient of thermal expansion properties when heat treated.

## 6. Procedure

6.1 *Preparation of Molds*—Prior to the molding of specimens, the outside joints of the mold and the contact lines of the mold and base plate shall be sealed to prevent loss of mixing

water from a freshly molded specimen. Thinly cover the interior surfaces of the mold with mineral oil. After this operation, set the gage studs, taking care to keep them clean, and free of oil, grease, and foreign matter.

6.2 *Use of Reference Bar*—Place the reference bar in the instrument in the same position each time a comparator reading is taken. Check the dial gage setting of the measuring device by use of the reference bar at least at the beginning and end of the readings made within a half day when the apparatus is kept in a room maintained at constant temperature. Check it more often when kept in a room where the temperature is not constant.

NOTE 3—The equation given in the section on calculation of length change contemplates that a comparator reading for the reference bar will be recorded each time the reference bar is used and a difference calculated for each test specimen reading. Alternatively, the dial gage setting can be reset, if necessary, to its original setting with the reference bar in place each time the reference bar is read. Doing so simplifies the calculation of length change by canceling the comparator reading of the reference bar from the values of  $L_s$  and  $L_r$ . If this procedure is used, care should be taken to ensure that the dial set screw is tightened adequately each time the dial is reset.



FIG. 3 Type of Suitable Apparatus for Measurement of Length Changes

6.3 *Obtaining Comparator Readings*— Rotate specimens slowly in the measuring instrument while the comparator reading is being taken. Record the minimum reading of the dial if the rotation causes a change in the dial reading. Place specimens in the instrument with the same end up each time a comparator reading is taken.

6.3.1 *Obtaining Comparator Readings of Specimens Stored Moist*—Clean the hole in the base of the comparator into which the gage stud on the lower end of the bar fits (this hole tends to collect water and sand and should be cleaned after every reading). Read and record the comparator indication of the length of the reference bar. Take one bar out of immersion, blot the pins, put the bar in the comparator, read, and record the indication. Return the bar to immersion and clean the hole in the base of the comparator. Take out the second bar and treat it in a like manner. Return the second bar to immersion, record

the reading, and clean the hole in the base of the comparator. Continue the procedure until all bars have been read, returned to immersion, and the readings recorded, cleaning the hole in the bottom of the comparator each time. After reading the last bar, clean the hole in the comparator base and read and record the reference-bar indication. Blot only around the pins (Note 4).

NOTE 4—The purpose of the minimal blotting of the pins and no blotting of the bars is to avoid drying and shrinkage of the bars. It has been observed that if the pins are blotted, and the bar placed in the comparator and the dial read, and the bar is then wiped gently with a dry cloth, the bar will shrink measurably. Therefore, drying should be minimized.

## 7. Calculation of Length Change

7.1 Calculate the length change at any age as follows:

$$L = \frac{(L_x - L_i)}{G} \times 100$$

where:

- $L$  = change in length at  $x$  age, %
- $L_x$  = comparator reading of specimen at  $x$  age minus comparator reading of reference bar at  $x$  age; in millimetres when using Fig. 1 apparatus, in inches when using Fig. 2 apparatus,
- $L_i$  = initial comparator reading of specimen minus comparator reading of reference bar at that same time; in millimetres when using Fig. 1 apparatus, in inches when using Fig. 2 apparatus, and
- $G$  = nominal gage length, 250 when using Fig. 1 apparatus, 10 when using Fig. 2 apparatus.

7.2 Calculate length change values for each specimen to the nearest 0.001 % and report averages to the nearest 0.01 %.

## 8. Temperature, Humidity, and Time

8.1 *Molding Room*—The temperature of the molding room and dry materials shall be maintained between 20 and 27.5°C [68 and 81.5°F]. The relative humidity shall be not less than 50 %. The temperature of the mixing water shall be 23.0 ± 2.0°C [73.5 ± 3.5°F].

8.2 *Moist Storage Facility*—The temperature and humidity of the air in the moist storage facility shall conform to the requirements of Specification C 511.

8.3 *Time*—Comparator readings shall be taken at specified time intervals or ages. All intervals and ages shall be met within ±2 %.

## 9. Keywords

9.1 cement paste; comparator; concrete; length change apparatus; molds; mortar

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## Standard Practice for Capping Cylindrical Concrete Specimens<sup>1</sup>

This standard is issued under the fixed designation C 617; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This practice covers apparatus, materials, and procedures for capping freshly molded concrete cylinders with neat cement and hardened cylinders and drilled concrete cores with high-strength gypsum plaster or sulfur mortar.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents of inch-pound units may be approximate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precaution statements see 4.3 and 6.2.3.1.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)<sup>2</sup>

C 150 Specification for Portland Cement<sup>2</sup>

C 472 Test Methods for Physical Testing of Gypsum, Gypsum Plasters and Gypsum Concrete<sup>2</sup>

C 595M Specification for Blended Hydraulic Cements<sup>2</sup>

C 1231 Practice for Use of Unbounded Caps in Determination of Compressive Strength of Hardened Concrete Cylinders<sup>3</sup>

#### 2.2 ANSI Standard:

B46.1 Standard for Surface Texture (Surface, Roughness, Waviness and Lay)<sup>4</sup>

### 3. Significance and Use

3.1 This practice describes procedures for providing plane surfaces on the ends of freshly molded concrete cylinders, hardened cylinders, or drilled concrete cores when the end

surfaces do not conform with the planeness and perpendicularity requirements of applicable standards. Practice C 1231 describes alternative procedures using unbonded caps or pad caps.

### 4. Capping Equipment

4.1 *Capping Plates*—Neat cement caps and high-strength gypsum-plaster caps shall be formed against a glass plate at least  $\frac{1}{4}$  in. (6 mm) thick, a machined metal plate at least 0.45 in. (11 mm) thick, or a polished plate of granite or diabase at least 3 in. (76 mm) thick. Sulfur mortar caps shall be formed against similar metal or stone plates except that the recessed area which receives molten sulfur shall not be deeper than  $\frac{1}{2}$  in. (12 mm). In all cases, plates shall be at least 1 in. (25 mm) greater in diameter than the test specimen and the working surfaces shall not depart from a plane by more than 0.002 in. (0.05 mm) in 6 in. (152 mm). The surface roughness of newly finished metal plates shall not exceed that set forth in Table 4 of American National Standard B46.1, or 125  $\mu$ m (3.2  $\mu$ m) for any type of surface and direction of lay. The surface, when new, shall be free of gouges, grooves, or indentations beyond those caused by the finishing operation. Metal plates that have been in use shall be free of gouges, grooves, and indentations greater than 0.010 in. (0.25 mm) deep or greater than 0.05 in.<sup>2</sup> (32 mm<sup>2</sup>) in surface area.

NOTE 1—A Rockwell hardness of 48 HRC is suggested for capping plates of devices used to form sulfur mortar caps.

4.2 *Alignment Devices*—Suitable alignment devices, such as guide bars or bull's-eye levels, shall be used in conjunction with capping plates to ensure that no single cap will depart from perpendicularity to the axis of a cylindrical specimen by more than 0.5° (approximately equivalent to  $\frac{1}{8}$  in. in 12 in. (3.2 mm in 305 mm)). The same requirement is applicable to the relationship between the axis of the alignment device and the surface of a capping plate when guide bars are used. In addition, the location of each bar with respect to its plate must be such that no cap will be off-centered on a test specimen by more than  $\frac{1}{16}$  in. (2 mm).

4.3 *Melting Pots for Sulfur Mortars*—Pots used for melting sulfur mortars shall be equipped with automatic temperature controls and shall be made of metal or lined with a material that is nonreactive with molten sulfur.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.61 on Testing Concrete for Strength.

Current edition approved August 10, 1998. Published December 1998. Originally published as C 617 – 68. Last previous edition C 617 – 94.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>4</sup> Available from American Society of Mechanical Engineers, 345 E. 47th Street, New York, NY 10017.

4.3.1 **Caution:** Melting pots equipped with peripheral heating will ensure against accidents during reheating of cooled sulfur mixture that have a crusted-over surface. When using melting pots not so equipped, a build-up of pressure under the hardened surface crust on subsequent reheating may be avoided by use of a metal rod that contacts the bottom of the pot and projects above the surface of the fluid sulfur mix as it cools. The rod should be of sufficient size to conduct enough heat to the top on reheating to melt a ring around the rod first and thus avoid the development of pressure. A large metal ladle can be substituted for the rod.

4.3.1.1 Use sulfur melting pots in a hood to exhaust the fumes to outdoors. Heating over an open flame is dangerous because the flash point of sulfur is approximately 440°F (227°C) and the mixture can ignite due to overheating. Should the mixture start to burn, covering will snuff out the flame. The pot should be recharged with fresh material after the flame has been extinguished.

**5. Capping Materials**

5.1 The strength of the capping material and the thickness of the caps shall conform to the requirements of Table 1.

5.1.1 If sulfur mortar, high strength gypsum plaster and other materials except neat cement paste are to be used to test concrete with a strength greater than 7000 psi (50 MPa), the manufacturer or the user of the material must provide documentation:

5.1.1.1 That the average strength of 15 cylinders capped with the material is not less than 98 percent of the average strength of 15 companion cylinders capped with neat cement paste or 15 cylinders ground plane to within 0.002 in. (0.05 mm).

5.1.1.2 That the standard deviation of the strengths of the capped cylinders is not greater than 1.57 times that of the standard deviation of the reference cylinders.

5.1.1.3 That the cap thickness requirements were met in the qualification tests, and

5.1.1.4 Of the hardening time of the caps used in the qualification tests.

5.1.2 Additionally, the qualification test report must include the compressive strength of 2 in. cubes of the material qualified and of neat cement paste cubes, if used. Capping materials conforming to these requirements is permitted to be used for cylinders with strengths up to 20 percent greater than the concrete tested in these qualification tests. The manufacturer must requalify lots of material manufactured on an annual basis

or whenever there is a change in the formulation or the raw materials. The user of the material must retain a copy of the qualification results, and the dates of manufacture of material qualified and of the material currently being used. See Table 2.

5.1.3 The compressive strength of capping materials shall be determined by testing 2 in. cubes following the procedure described in Test Method C 109. Except for sulfur mortars, molding procedures shall be as in Test Method C 109 unless other procedures are required to eliminate large entrapped air voids. See Test Methods C 472 for alternative compaction procedures. Cure cubes in the same environment for the same length of time as the material used to cap specimens.

5.1.4 The strength of the capping material shall be determined on receipt of a new lot and at intervals not exceeding three months. If a given lot of the capping material fails to conform to the strength requirements, it shall not be used, and strength tests of the replacement material shall be made weekly until four consecutive determinations conform to specification requirements.

*5.2 Neat Hydraulic Cement Paste:*

5.2.1 Make qualification tests of the neat hydraulic cement paste prior to use for capping to establish the effects of water-cement ratio and age on compressive strength of 2 in. (50 mm) cubes.

NOTE 2—The cements used generally conform to Specification C 150 Types I, II or III; however, Specification C 595 blended cements, calcium aluminate or other hydraulic cements producing acceptable strength may be used.

5.2.2 Mix the neat cement paste to the desired consistency at a water-cement ratio equal to or less than that required to produce the required strength, generally 2 to 4 h before the paste is to be used (Note 3). Remix as necessary to maintain acceptable consistency (Note 4). Some retempering of the paste is acceptable if the required water-cement ratio is not exceeded. Optimum consistency is generally produced at water-cement ratios of 0.32 to 0.36 by mass for Type I and Type II cements and 0.35 to 0.39 by mass for Type III cements.

NOTE 3—Freshly mixed pastes tend to bleed, shrink, and make unacceptable caps. The 2 to 4 h period is generally appropriate for portland cements.

NOTE 4—The required consistency of the paste is determined by the appearance of the cap when it is stripped. Fluid paste results in streaks in the cap. Stiff paste results in thick caps.

*5.3 High-Strength Gypsum Cement Paste:*

5.3.1 No fillers or extenders may be added to neat high-strength gypsum cement paste subsequent to the manufacture of the cement. (Note 5) Qualification tests shall be made to determine the effects of water-cement ratio and age on compressive strength of 2 in. (50 mm) cubes. Retarders may be used to extend working time, but their effects on required water-cement ratio and strength must be determined. (Note 6)

NOTE 5—Low-strength molding plaster, plaster of paris, or mixtures of plaster of paris and portland cement are unsuitable for capping.

NOTE 6—The water-gypsum cement ratio should be between 0.26 and 0.30. Use of low water-cement ratios and vigorous mixing will usually permit development of 5000 psi (35 MPa) at ages of 1 or 2 h. Higher water-gypsum cement ratios extend working time, but reduce strength.

5.3.2 Mix the neat gypsum cement paste at the desired

**TABLE 1 Compressive Strength and Maximum Thickness of Capping Materials**

Cylinder Compressive Strength psi (MPa)	Minimum Strength of Capping Material	Maximum Average Thickness of Cap	Maximum Thickness Any Part of Cap
500 to 7000 psi (3.5 to 50 MPa)	5000 psi (35 MPa) or cylinder strength whichever is greater	¼ in. (6 mm)	⅝ in. (8 mm)
greater than 7000 psi (50 MPa)	Compressive strength not less than cylinder strength, except as provided in 5.1.1	⅙ in. (3 mm)	⅜ in. (5 mm)

**TABLE 2 Sample Report of Qualification of a Capping Material**

NOTE—Manufacturer: Testing Supplies Co.

Capping Material: Super Strong AAA-Sulfur mortar

Lot: 12a45 Date Tested: 11/3/98

Signed by: \_\_\_\_\_ (testing agency and responsible official)

Item	Capping Material	Control Cylinders	Ratio	Criteria	Pass/Fail
Type of capping material	Sulfur	Concrete Cylinder Test Data			
Average Concrete Strength, psi [MPa]	11 061 (76.2)	Ground 11 008 (75.9)	1.005	>0.98 X <sub>c</sub>	Pass
Standard Deviation, psi [MPa]	376 (2.59)	250 (1.72)	1.504	≤1.57 C	Pass
Number of cylinders tested	15	15			
Cap age when cylinders tested	7 days	na			
Average cap thickness, in. [mm]	0.11 (2.8)	Capping Material Test Data			
Compressive strength of 2 in. [50 mm] cubes, psi (MPa)	12 195 (91)	na			
Cube age when tested.	7 days				
Maximum concrete strength qualified, psi (MPa)				1.2 Av. Str = 13 273 (91.5) <sup>A</sup>	

<sup>A</sup> Nominally a specified strength of 11 000 psi (75 MPa) and perhaps somewhat higher.

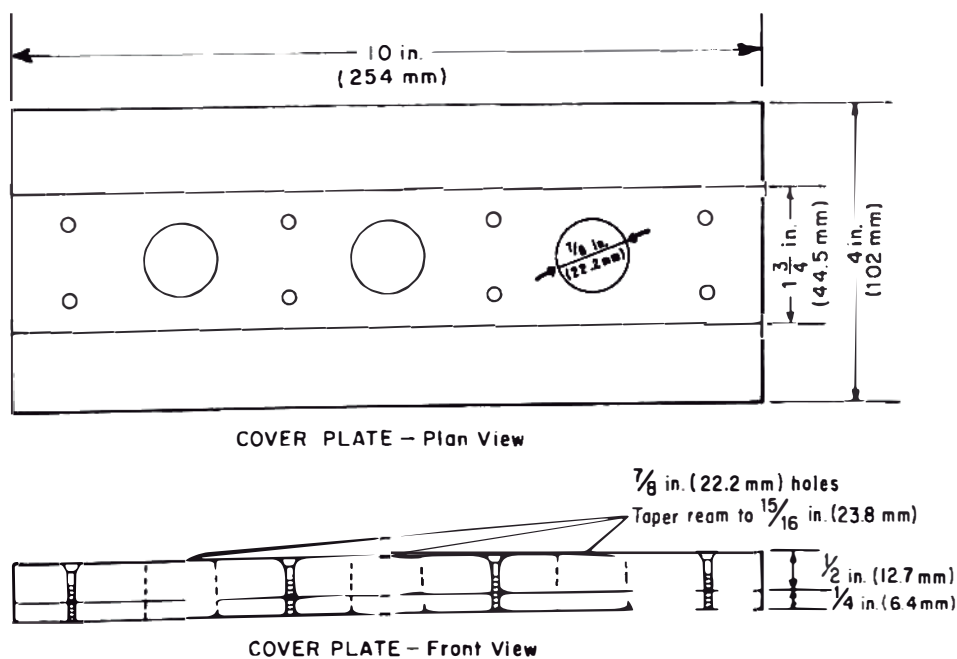
water-cement ratio and use it promptly since it sets rapidly.

**5.4 Sulfur Mortar:**

5.4.1 Proprietary or laboratory prepared sulfur mortars are permitted if allowed to harden a minimum of 2 h before testing concrete with strength less than 5000 psi (35 MPa). For concrete strengths of 5000 psi or greater, sulfur mortar caps must be allowed to harden at least 16 h before testing, unless a shorter time has been shown to be suitable as specified in 5.1.1.

5.4.2 *Determination of Compressive Strength*—Prepare test specimens using a cube mold and base plate conforming to the requirements of Test Method C 109 and a metal cover plate conforming in principle to the design shown in Fig. 1 (Note 7). Bring the various parts of the apparatus to a temperature of 68 to 86°F (20 to 30°C), lightly coat the surfaces that will be in

contact with the sulfur mortar with mineral oil, and assemble near the melting pot. Bring the temperature of the molten-sulfur mortar in the pot within a range of 265 to 290°F (129 to 143°C), stir thoroughly, and begin casting cubes. Using a ladle, or other suitable pouring device, quickly fill each of the three compartments until the molten material reaches the top of the filling hole. Allow sufficient time for maximum shrinkage, due to cooling, and solidification to occur (approximately 15 min) and refill each hole with molten material (Note 8). After solidification is complete, remove the cubes from the mold without breaking off the knob formed by the filling hole in the cover plate. Remove oil, sharp edges, and fins from the cubes and check the planeness of the bearing surfaces in the manner described in Test Method C 109. After storage at room temperature to the desired age, but not less than 2 h, test cubes in



**FIG. 1 Sketch of Cover Plate for 2-in. (50-mm) Cube Mold**

compression following the procedure described in Test Method C 109, and calculate the compressive strength.

NOTE 7—If desired, a plane phenol formaldehyde (bakelite) plate of 1/4-in. (3-mm) thickness, provided with three appropriately spaced filling holes, may be inserted between the cover plate and the mold to slow the rate of cooling of test specimens.

NOTE 8—The second filling helps to prevent the formation of a large void or shrinkage pipe in the body of a cube. However, such defects may occur no matter how much care is exercised, and it therefore is advisable to inspect the interior of tested sulfur mortar cubes for homogeneity whenever the strength values obtained are significantly lower than anticipated.

## 6. Capping Procedures

6.1 *Freshly Molded Cylinders*—Use only neat portland cement pastes (Note 9) to cap freshly molded cylinders. Make caps as thin as practicable. Do not apply the neat paste to the exposed end until the concrete has ceased settling in the molds, generally from 2 to 4 h after molding. During the molding of the cylinder, strike off the upper end even with or slightly below the plane of the rim of the mold. Remove free water and laitance from the top of the specimen immediately before capping. Form the cap by placing a conical mound of paste on the specimen and then gently pressing a freshly oiled capping plate on the conical mound until the plate contacts the rim of the mold. A very slight twisting motion may be required to extrude excess paste and minimize air voids in the paste. The capping plate must not rock during this operation. Carefully cover the capping plate and mold with a double layer of damp burlap and a polyethylene sheet to prevent drying. Removal of the capping plate after hardening may be accomplished by tapping the edge with a rawhide hammer in a direction parallel to the plane of the cap.

NOTE 9—Type I neat cement caps generally require at least 6 days to develop acceptable strength and Type III neat cement caps at least 2 days. Dry concrete specimens will absorb water from freshly mixed neat cement paste and produce unsatisfactory caps. Neat cement paste caps will shrink and crack on drying and, therefore, should be used only for specimens that are to be moist cured continuously until time of testing.

NOTE 10—High-strength gypsum caps soften and deteriorate on contact with water and cannot be used on freshly mixed concrete or stored in a moist room for more than very brief periods.

### 6.2 *Hardened Concrete Specimens:*

6.2.1 *General*—If an end of a specimen has a coating or deposit of oily or waxy materials that would interfere with the bond of the cap, remove such coatings or deposits. If necessary, the ends of a specimen may be slightly roughened with a steel file or wire brush to produce proper adhesion of the cap. If desired, capping plates may be coated with a thin layer of mineral oil or grease to prevent the capping material from adhering to the surface of the plate.

6.2.2 *End Condition*—The distance of any point on an uncapped end from a plane that passes through the highest point of the end surface and is perpendicular to the axis of the cylinder shall not exceed 1/8 in. (3 mm) (Note 11). If the end exceeds this limit, the end of the cylinder shall be cut, lapped or ground prior to capping.

NOTE 11—This provision is to control the difference between the thickest and thinnest parts of a cap. The distance may be checked using a square with one blade touching the cylinder parallel to the cylinder axis

and the other blade touching the highest point on the end of the cylinder. The distance between the blade of the square and the lowest point on the end of the cylinder is measured.

6.2.3 *Capping with High-Strength Gypsum Plaster or Neat Cement Paste*—Mix the paste as described in Section 2. Do not exceed the water-cement ratio determined in qualification tests. Form the caps as described in 6.1 using capping plates described in 4.1 to achieve the alignment required in 4.2 (Note 12). Generally, capping plates may be removed within 45 min with gypsum cement pastes and after 12 h with neat cement paste, without visibly damaging the cap.

NOTE 12—A number of methods have been used to obtain the desired perpendicularity of the cap to the axis of the cylinder. A mound of paste can be placed on a capping plate and the specimen lowered into it. A bull's-eye level on the top of the cylinder helps obtain alignment. A mound of paste can be placed on top of the cylinder and a capping plate pressed into it, again using the bull's-eye level. A better system is to make a half-height mold with a vertical split so that it can be slipped over the hardened cylinder. A clamp is used to position the mold and to ensure the required cap thickness. The mound of paste can then be placed either on a capping plate or on top of the cylinder and pressed until the plate contacts the mold. As noted earlier, very stiff paste may require excessive pressure and produce thick or defective caps.

6.2.4 *Capping with Sulfur Mortar*—Prepare sulfur mortar for use by heating to about 265°F (130°C) as determined by an all-metal thermometer inserted near the center of the mass. Check the temperature at approximately hourly intervals during capping. Empty the pot and recharge with fresh material at intervals to ensure that the oldest material in the pot has not been used more than five times. When capping concrete cylinders with a compressive strength of 5000 psi (35 MPa) or greater, it is not permitted to reuse compound recovered from the capping operation or old caps. Fresh sulfur mortar must be dry at the time it is placed in the pot as dampness may cause foaming. Keep water away from molten sulfur mortar for the same reason. The capping plate or device should be warmed before use to slow the rate of hardening and permit the production of thin caps. Oil the capping plate lightly and stir the molten sulfur mortar immediately prior to pouring each cap. The ends of moist cured specimens shall be dry enough at the time of capping to preclude the formation of steam or foam pockets under or in the cap larger than 1/4 in. (6 mm) in diameter. Replace caps with steam pockets or voids larger than 1/4 in. (6 mm) (Note 13). To ensure that the cap is bonded to the surface of the specimen, the end of the specimen shall not be oiled prior to the application of the cap. When using a vertical device, pour the mortar onto the surface of the capping plate, lift the cylinder above the plate and contact the cylinder sides with the guides, slide the cylinder down the guides onto the capping plate while keeping constant contact with the alignment guides. The cylinder end should continue to rest on the capping plate with cylinder sides in positive contact with the alignment guides until the mortar has hardened. Use sufficient material to cover the cylinder end after the sulfur mortar solidifies.

NOTE 13—Periodically, the sulfur mortar cap should be examined after testing for air or steam pockets in the cap. Before testing, the cap can be tapped with a coin or rubbed with a light metal implement to see if a hollow sound can be detected. Caps with hollow areas should be removed and recapped.



6.2.4.1 **Caution:** Hydrogen sulfide gas may be produced during capping when sulfur mortar is contaminated with organic materials such as paraffin or oil. The gas is colorless and has a notoriously bad odor of rotten eggs; however, the odor should not be relied upon as a warning sign, since the sensitivity to the odor disappears rapidly on exposure. High concentrations are lethal and less concentrated dosages may produce nausea, stomach distress, dizziness, headache, or irritation of the eyes. For this and other reasons, the melting pot must be located under a hood with an exhaust fan and that capping area must be well ventilated.

6.2.5 *Daily Check:*

6.2.5.1 During each day's capping operation, check the planeness of the caps prior to compression testing on at least three specimens, selected at random, representing the start, middle, and end of the run. Check planeness with a straight-edge and feeler gage, making a minimum of three measurements on different diameters to ensure that the surface of the caps do not depart from a plane by more than 0.002 in. (0.05 mm). Check also for hollow areas (Note 13). Record the results of these determinations in the quality control documentation for the laboratory. If caps fail to satisfy the planeness requirement or have hollow areas, remove and reapply the caps.

6.2.5.2 During each day's compressive strength testing operation, check the thickness of caps on at least three specimens, selected at random, from the start, middle, and end of that day's operation. After completing the compression test, recover at

least six pieces of capping material from the top of the selected specimen (Note 14). The pieces shall be selected at random and be distributed over the entire area of the cap. The selected pieces shall have debonded completely from the concrete. Measure and record the thicknesses of the pieces to the nearest 0.01 in. (0.2 mm) using a micrometer, caliper or other thickness measurement device. Compare the average and maximum thicknesses with the values in Table 1. Record the results of the thickness determinations in the quality control documentation for the laboratory.

**NOTE 14**—Caps may be removed by using a hammer and sharp chisel. Place the chisel tip at the bond line and nearly parallel with the plane of the cap so as to create a wedging action when the chisel is struck with the hammer. Recovery of the entire cap may be simplified by placing duct tape over the cap prior to attempting its removal. The tape will keep the pieces of capping material from being dispersed during removal and will simplify the selection of pieces uniformly distributed over the cap area.

## 7. Protection of Specimens After Capping

7.1 Maintain moist cured specimens in a moist condition between the completion of capping and the time of testing by returning them to moist storage or wrapping them with a double layer of wet burlap. Do not store specimens with gypsum plaster caps immersed in water or for more than 4 h in a moist room. Protect plaster caps from dripping water.

7.2 Do not test capped specimens before the capping material has sufficient time to develop the strength required in 5.1.

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# Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete<sup>1</sup>

This standard is issued under the fixed designation C 618; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope \*

1.1 This specification covers coal fly ash and raw or calcined natural pozzolan for use as a mineral admixture in concrete where cementitious or pozzolanic action, or both, is desired, or where other properties normally attributed to finely divided mineral admixtures may be desired, or where both objectives are to be achieved.

NOTE 1—Finely divided materials may tend to reduce the entrained air content of concrete. Hence, if a mineral admixture is added to any concrete for which entrainment of air is specified, provision should be made to ensure that the specified air content is maintained by air content tests and by use of additional air-entraining admixture or use of an air-entraining admixture in combination with air-entraining hydraulic cement.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 The text of this standard references notes and footnotes, which provide explanatory information. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 125 Standard Terminology Relating to Concrete and Concrete Aggregates<sup>2</sup>

C 311 Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete<sup>2</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 The terms used in this specification are defined in Terminology C 125.

3.1.2 *fly ash*—the finely divided residue that results from

the combustion of ground or powdered coal and that is transported by flue gasses.

NOTE 2—This definition of fly ash does not include, among other things, the residue resulting from: (1) the burning of municipal garbage or any other refuse with coal; (2) the injection of lime directly into the boiler for sulfur removal; or (3) the burning of industrial or municipal garbage in incinerators commonly known as “incinerator ash.”

## 4. Classification

4.1 *Class N*—Raw or calcined natural pozzolans that comply with the applicable requirements for the class as given herein, such as some diatomaceous earths; opaline cherts and shales; tuffs and volcanic ashes or pumicites, calcined or uncalcined; and various materials requiring calcination to induce satisfactory properties, such as some clays and shales.

4.2 *Class F*—Fly ash normally produced from burning anthracite or bituminous coal that meets the applicable requirements for this class as given herein. This class fly ash has pozzolanic properties.

4.3 *Class C*—Fly ash normally produced from lignite or subbituminous coal that meets the applicable requirements for this class as given herein. This class of fly ash, in addition to having pozzolanic properties, also has some cementitious properties.

NOTE 3—Some Class C fly ashes may contain lime contents higher than 10 %.

## 5. Ordering Information

5.1 The purchaser shall specify any supplementary optional physical requirements.

5.2 The purchaser shall indicate which procedure, A or B, shall be used when specifying requirements for effectiveness in contribution to sulfate resistance under Table 3.

## 6. Chemical Composition

6.1 Fly ash and natural pozzolans shall conform to the requirements as to chemical composition prescribed in Table 1.

## 7. Physical Properties

7.1 Fly ash and natural pozzolans shall conform to the physical requirements prescribed in Table 2. Supplementary

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.24 on Ground Slag and Pozzolanic Admixtures.

Current edition approved Aug. 10, 2001. Published September 2001. Originally published as C618–68 T to replace C350 and C402. Last previous edition C618–00.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

\*A Summary of Changes section appears at the end of this standard.

**TABLE 1 Chemical Requirements**

	Mineral Admixture Class		
	N	F	C
Silicon dioxide (SiO <sub>2</sub> ) plus aluminum oxide (Al <sub>2</sub> O <sub>3</sub> ) plus iron oxide (Fe <sub>2</sub> O <sub>3</sub> ), min, %	70.0	70.0	50.0
Sulfur trioxide (SO <sub>3</sub> ), max, %	4.0	5.0	5.0
Moisture content, max, %	3.0	3.0	3.0
Loss on ignition, max, %	10.0	6.0 <sup>A</sup>	6.0

<sup>A</sup>The use of Class F pozzolan containing up to 12.0 % loss on ignition may be approved by the user if either acceptable performance records or laboratory test results are made available.

**TABLE 2 Physical Requirements**

	Mineral Admixture Class		
	N	F	C
<i>Fineness:</i>			
Amount retained when wet-sieved on 45 µm (No. 325) sieve, max, % <sup>A</sup>	34	34	34
<i>Strength activity index:</i> <sup>B</sup>			
With portland cement, at 7 days, min, percent of control	75 <sup>C</sup>	75 <sup>C</sup>	75 <sup>C</sup>
With portland cement, at 28 days, min, percent of control	75 <sup>C</sup>	75 <sup>C</sup>	75 <sup>C</sup>
Water requirement, max, percent of control	115	105	105
<i>Soundness:</i> <sup>D</sup>			
Autoclave expansion or contraction, max, %	0.8	0.8	0.8
<i>Uniformity requirements:</i>			
The density and fineness of individual samples shall not vary from the average established by the ten preceding tests, or by all preceding tests if the number is less than ten, by more than:			
Density, max variation from average, %	5	5	5
Percent retained on 45-µm (No. 325), max variation, percentage points from average	5	5	5

<sup>A</sup>Care should be taken to avoid the retaining of agglomerations of extremely fine material.

<sup>B</sup>The *strength activity index* with portland cement is not to be considered a measure of the compressive strength of concrete containing the mineral admixture. The mass of mineral admixture specified for the test to determine the *strength activity index* with portland cement is not considered to be the proportion recommended for the concrete to be used in the work. The optimum amount of mineral admixture for any specific project is determined by the required properties of the concrete and other constituents of the concrete and is to be established by testing. *Strength activity index* with portland cement is a measure of reactivity with a given cement and may vary as to the source of both the mineral admixture and the cement.

<sup>C</sup>Meeting the 7 day or 28 day *strength activity index* will indicate specification compliance.

<sup>D</sup>If the mineral admixture will constitute more than 20 % by weight of the cementitious material in the project mix design, the test specimens for autoclave expansion shall contain that anticipated percentage. Excessive autoclave expansion is highly significant in cases where water to mineral admixture and cement ratios are low, for example, in block or shotcrete mixes.

optional physical requirements are shown in Table 3.

## 8. Methods of Sampling and Testing

8.1 Sample and test the mineral admixture in accordance with the requirements of Test Methods C 311.

8.2 Use cement of the type proposed for use in the work and, if available, from the mill proposed as the source of the cement, in all tests requiring the use of hydraulic cement.

## 9. Storage and Inspection

9.1 The mineral admixture shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment.

9.2 Inspection of the material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

## 10. Rejection

10.1 The purchaser has the right to reject material that fails to conform to the requirements of this specification. Rejection shall be reported to the producer or supplier promptly and in writing.

10.2 The purchaser has the right to reject packages varying

more than 5 % from the stated weight. The purchaser also has the right to reject the entire shipment if the average weight of the packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified.

10.3 The purchaser has the right to require that mineral admixture in storage prior to shipment for a period longer than 6 months after testing be retested. The purchaser has the right to reject such material if it fails to meet the fineness requirements.

## 11. Packaging and Package Marking

11.1 When the mineral admixture is delivered in packages, the class, name, and brand of the producer, and the weight of the material contained therein, shall be plainly marked on each package. Similar information shall be provided in the shipping invoices accompanying the shipment of packaged or bulk mineral admixture.

## 12. Keywords

12.1 fly ash; mineral admixtures; natural pozzolan; pozzolans

**TABLE 3 Supplementary Optional Physical Requirements**

NOTE 1—These optional requirements apply only when specifically requested.

	Mineral Admixture Class		
	N	F	C
Multiple factor, calculated as the product of loss on ignition and fineness, amount retained when wet-sieved on 45- $\mu\text{m}$ (No. 325) sieve, max, % <sup>A</sup>	...	255	...
Increase of drying shrinkage of mortar bars at 28 days, max, difference, in %, over control <sup>B</sup>	0.03	0.03	0.03
Uniformity Requirements: In addition, when air-entraining concrete is specified, the quantity of air-entraining agent required to produce an air content of 18.0 vol % of mortar shall not vary from the average established by the ten preceding tests or by all preceding tests if less than ten, by more than, %	20	20	20
Effectiveness in Controlling Alkali-Silica Reaction: <sup>C</sup> Expansion of test mixture as percentage of low-alkali cement control, at 14 days, max, %	100	100	100
Effectiveness in Contributing to Sulfate Resistance: <sup>D</sup> Procedure A: Expansion of test mixture: For moderate sulfate exposure after 6 months exposure, max, % For high sulfate exposure after 6 months exposure, max, %	0.10 0.05	0.10 0.05	0.10 0.05
Procedure B: Expansion of test mixture as a percentage of sulfate resistance cement control after at least 6 months exposure, max,%	100	100	100

<sup>A</sup>Applicable only for Class F mineral admixtures since the loss on ignition limitations predominate for Class C.

<sup>B</sup>Determination of compliance or noncompliance with the requirement relating to increase in drying shrinkage will be made only at the request of the purchaser.

<sup>C</sup>Mineral admixtures meeting this requirement are considered as effective in controlling alkali aggregate reactions as the use of the low-alkali control cement used in the evaluation. However, the mineral admixture shall be considered effective only when the mineral admixture is used at percentages by mass of the total cementitious material equal to or exceeding that used in the tests and when the alkali content of the cement to be used with the mineral admixture does not exceed that used in the tests by more than 0.05 %. See Appendix XI, Test Methods C 311.

<sup>D</sup>Fly ash or natural pozzolan shall be considered effective only when the fly ash or natural pozzolan is used at percentages, by mass, of the total cementitious material within 2 % of those that are successful in the test mixtures or between two percentages that are successful, and when the  $C_3A$  content of the project cement is less than, or equal to, that which was used in the test mixtures. See Appendix X2 of Test Method C 311.

### SUMMARY OF CHANGES

This section identifies the location of changes to this specification that have been incorporated since the last issue.

(1) Revised sections 5 and 6.

Chemical Requirements" was deleted.

(2) The Table previously titled "Supplementary Optional



Designation: C 1012 – 04

## Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution<sup>1</sup>

This standard is issued under the fixed designation C 1012; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of length change of mortar bars immersed in a sulfate solution. Mortar bars made using mortar described in Test Method C 109/ C 109M are cured until they attain a compressive strength of  $20.0 \pm 1.0$  MPa ( $3000 \pm 150$  psi), as measured using cubes made of the same mortar, before the bars are immersed.

1.2 The values stated in SI units are to be regarded as the standard. The values shown in parentheses are for information purposes only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50mm] Cube Specimens)
- C 114 Test Method for Chemical Analysis of Hydraulic Cement
- C 150 Specification for Portland Cement
- C 157/C 157M Test Method for Length Change of Hardened Hydraulic-Cement, Mortar, and Concrete
- C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens
- C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
- C 348 Test Method for Flexural Strength of Hydraulic Cement Mortars
- C 349 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure)

- C 452 Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate
  - C 490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete
  - C 511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
  - C 595 Specification for Blended Hydraulic Cements
  - C 597 Test Method for Pulse Velocity Through Concrete
  - C 618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete
  - C 684 Test Method for Making, Accelerated Curing, and Testing Concrete Compression Test Specimens
  - C 778 Specification for Standard Sand
  - C 917 Test Method for Evaluation of Cement Strength Uniformity From a Single Source
  - C 989 Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars
  - D 1193 Specification for Reagent Water
  - E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
- 2.2 *American Concrete Institute Reports:*  
ACI C201-2R-01 Guide to Durable Concrete<sup>3</sup>

### 3. Significance and Use

3.1 This test method provides a means of assessing the sulfate resistance of mortars made using portland cement, blends of portland cement with pozzolans or slags, and blended hydraulic cements. Method C 452 is suitable for evaluating portland cements but not blended cements or blends of portland cement with pozzolans or slags.

3.2 The standard exposure solution used in this test method, unless otherwise directed, contains 352 moles of  $\text{Na}_2\text{SO}_4$  per  $\text{m}^3$  (50 g/L). Other sulfate concentrations or other sulfates such as  $\text{MgSO}_4$  may be used to simulate the environmental exposure of interest. Further discussion of these and other technical issues is given in the Appendix.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.29 on Sulfate Resistance.

Current edition approved Jan. 1, 2004. Published February 2004. Originally approved in 1984. Last previous edition approved in 2003 as C 1012 – 03.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333.

#### 4. Apparatus

4.1 *Mixer*, conforming to the requirements of Practice C 305.

4.2 *Cube Molds*, conforming to the requirements of Test Method C 109/C 109M.

4.3 *Bar Molds*, conforming to the requirements of Specification C 490.

4.4 *Comparator*, conforming to the requirements of Specification C 490.

4.5 *Containers*—The containers in which the bars are immersed shall be corrosion resistant such as plastic, glass, or ceramic. Support the bars so that no end or side of a bar rests against the container. Seal the container with a lid so that the sulfate solution cannot evaporate.

4.6 *Curing Tank*, conforming to the requirements of Test Method C 684.

#### 5. Reagents and Materials

5.1 *Purity of Reagents*—USP or technical grade chemicals may be used, provided it is established that any reagent used is of sufficiently high purity to permit its use without lessening the accuracy of the determination. When tests are made that are expected to produce results that are close to an acceptance-rejection value, it is recommended that reagent grade chemicals be used. Such chemicals shall conform to the specifications of the Committee on Analytical Reagents for the American Chemical Society where such specifications are available.<sup>4</sup>

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

5.3 *Sodium Sulfate* ( $\text{Na}_2\text{SO}_4$ )—Check the water content by loss on ignition each time the solution is prepared. Any anhydrous or hydrated sodium sulfate may be used if the water content of the salt is checked by loss on ignition and proper corrections made to account for the specified sulfate concentration.

5.4 *Sulfate Solution*—Each litre of solution shall contain 50.0 g of  $\text{Na}_2\text{SO}_4$  dissolved in 900 mL of water, and shall be diluted with additional distilled or deionized water to obtain 1.0 L of solution. Mix the solution on the day before use, cover, and store at  $23.0 \pm 2.0^\circ\text{C}$  ( $73.5 \pm 3.5^\circ\text{F}$ ). Determine the pH of the solution before use; reject the solution if the pH range is outside 6.0 to 8.0. Maintain the volume proportion of sulfate solution to mortar bars in a storage container at  $4.0 \pm 0.5$  volumes of solution to 1 volume of mortar bars. For mortar bars 1 by 1 by 1 1/4 in. (volume of 184 mL or 11.25 in.<sup>3</sup>), this is 645 to 830 mL of solution per mortar bar in the storage container. For mortar bars 25 by 25 by 285 mm (volume 178 mL), this is 625 to 800 mL of solution per mortar bar in the storage container.

5.5 *Materials*:

5.5.1 *Graded Standard Sand*, as specified in Specification C 778.

5.5.2 *Stainless Steel Gage Studs*, as specified in Specification C 490.

#### 6. Hazards

6.1 **Warning**—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.<sup>5</sup>

#### 7. Preparing Mortars

7.1 Make mortars as described in Test Method C 109/C 109M, that is, 1 part cement to 2.75 parts of sand by mass. Use a water-cement ratio by mass of 0.485 for all non-air-entraining portland cements and 0.460 for all air-entraining portland cements. Use a water-cement ratio by mass of 0.485 for non-air-entraining portland-pozzolan (IP) and portland-blast furnace slag (IS) cements. For blends of portland cement with a pozzolan or slag, use a water-cement ratio that develops a flow within  $\pm 5$  of that of the portland-cement mortar at a water-cement ratio of 0.485.

#### 8. Specimen Molds

8.1 Prepare the specimen molds in accordance with the requirements of Specification C 490 except the interior surfaces of the mold shall be covered with a release agent. A release agent will be acceptable if it serves as a parting agent without affecting the setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen.

NOTE 1—TFE-fluorocarbon tape complies with the requirements for a mold release agent.

#### 9. Procedure

9.1 *Molding and Initial Curing of Specimens*—Mold the test bars in accordance with Test Method C 157/C 157M. Mold the cubes in accordance with Test Method C 109/C 109M. A set of specimens to test one cement consists of 6 bars and up to 21 cubes (Note 2). Immediately after molding, cover the molds with a rigid steel, glass, or plastic plate, seal the plate to the mold so as to be watertight, and place the mold in the curing tank in water at  $35 \pm 3^\circ\text{C}$  ( $95 \pm 5^\circ\text{F}$ ) for  $23\frac{1}{2} \text{ h} \pm 30 \text{ min}$ , as in Test Method C 684, Procedure A (Warm-Water Method). Place the sealed molds in the curing tank with the bottom of the bars as cast down, that is, in the same relative position in which the bars were cast. At  $23\frac{1}{2} \text{ h} \pm 30 \text{ min}$ , remove molds from tank and demold the specimens.

NOTE 2—The set of cubes consists of 21 cubes to be tested as described herein when significant information on the strength development rate is not available. When information is available (as for example, from the use of the procedures of Test Method C 917) that would justify making fewer cubes, only those needed to confirm the time the mortar achieves 20.0 – 1.0 MPa ( $3000 \pm 150 \text{ psi}$ ) are needed.

9.2 *Subsequent Curing and Preparation for Test*—After demolding, store all bars and cubes, except the two to be

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>5</sup> See *Manual of Cement Testing*, Section on Safety, *Annual Book of ASTM Standards*, Vol 04.01.

broken, in a curing tank of saturated limewater at  $23.0 \pm 2.0^\circ\text{C}$  ( $73.5 \pm 3.5^\circ\text{F}$ ). Break two cubes in compression in accordance with Test Method C 109/C 109M after demolding when the specimens have cooled to ambient temperature under moist cloths. If the mean strength of the two cubes is 20 MPa (2850 psi) or more, observe and record comparator readings in accordance with Specification C 490 and as prescribed in the section on Measurements of Length Change and place all the bars in the sulfate solution. If 20 MPa (2850 psi) is not achieved, store the demolded cubes and mortar bars in the curing tank and test additional cubes. Predict from the first two cubes when a compressive strength of at least 20 MPa (2850 psi) will be reached. Verify the prediction, and at that time observe and record comparator readings and place all the bars in the sulfate solution (Note 3). This measurement is designated as the initial length. The storage temperature and test temperature shall be  $23.0 \pm 2.0^\circ\text{C}$  ( $73.5 \pm 3.5^\circ\text{F}$ ).

NOTE 3—If the value for strength at 24 h is less than 20.0 MPa (2850 psi) and additional testing on the same day is not possible, or, is unlikely to yield a value over 20.0 MPa (2850 psi) and the strength is over 21 MPa (3150 psi) when tested early the next day, it is not necessary to remake the batch.

**9.3 Storage of Test Bars during Exposure to Test Solution—**Cover the container of the bars and test solution, and seal it to prevent evaporation from the inside, or dilution with water from the outside. (See Note 4.) The storage temperature and test temperature shall be  $23.0 \pm 2.0^\circ\text{C}$  ( $73.5 \pm 3.5^\circ\text{C}$ ). (See Note 5.)

NOTE 4—Gaffers tape or duct tape has been found to be suitable for sealing the container.

NOTE 5—This is the same temperature and temperature range as that specified for moist rooms in Specification C 511.

**9.4 Measurements of Length Change—**At 1, 2, 3, 4, 8, 13, and 15 weeks after the bars are placed in the sulfate solution, test them for length change using the length comparator in accordance with Specification C 490. Review the data at 15 weeks. Make the subsequent measurements at a minimum of 4, 6, 9, and 12 months. If at any time the rate of change between readings is great, insert other readings as needed to ensure the intervals are short enough to permit observing and reporting the behavior of the bars. If compliance with ACI C201-2R-01 Class 3 exposure is required, measure the bars at 4, 6, 9, 12, 15, and 18 months (Note 6).

NOTE 6—ACI C201-2R-01 requires an 18-month limit for Class 3 exposure.

**9.4.1 Details of Measurement of Bars for Length Change:**

9.4.1.1 Clean the hole in the base of the comparator into which the gage stud on the lower end of the bar fits (this hole tends to collect water and sand and should be cleaned after every reading). Read and record the comparator indication of the length of the reference bar. Take one bar out of immersion, blot the pins, put the bar in the comparator, read, and record the indication. Return the bar to immersion and clean the hole in the base of the comparator. Take out the second bar and treat it in a like manner. Return the second bar to immersion, record the reading, and clean the hole in the base of the comparator. Continue the procedure until all bars have been read, returned

to immersion, and the readings recorded, cleaning the hole in the bottom of the comparator each time. After reading the last bar, clean the hole in the comparator base and read and record the reference-bar indication.

9.4.1.2 When the required strength of the mortar is achieved in accordance with 9.2, store the bars in fresh sulfate solution. At subsequent readings for length change, proceed as described in 9.4.1.1; cleaning the socket in the base of the comparator before reading the reference bar initially and after reading each mortar bar. Record reference bar and mortar bar readings. Read and record the reference bar again after measurement of the last bar. Blot only around the pins (Note 7). Return each bar to the used sulfate solution after reading. When all the bars have been measured, discard the used solution. Rinse the container once with water, pouring out water and debris. Replace the frame holding the bars in the container, fill the container with enough new sulfate solution to immerse bars, and secure the lid on the container.

NOTE 7—The purpose of the minimal blotting of the pins and no blotting of the bars is to avoid drying and shrinkage of the bars. It has been observed that if the pins are blotted, and the bar placed in the comparator and the dial read, and the bar is then wiped gently with a dry cloth, the bar will shrink measurably. Therefore, drying should be minimized.

**9.4.2 Examination of Specimens After Measuring Length Change—**When the bars seem to have behaved in an unusual way or when the test is part of a research study, test the specimens for warping by placing them on a plane surface so that the ends are curved down and the maximum bowing measured. Note cracking (presence, location, type); also note surface deposits, mottling, exudations (nature, thickness, type).

**9.5 Tolerance on Time—**All references to elapsed time in 9.4 are intended to have a tolerance of  $\pm 2\%$ .

**TABLE 1 Maximum Permissible Range of Values**

Remaining No. of Specimens	Blended Cements	Portland Cement
3	0.034	0.010
4	0.037	0.011
5	0.039	0.012
6	0.041	0.012

**10. Calculation**

10.1 Calculate the length change at any age as follows:

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100 \quad (1)$$

where:

- $\Delta L$  = change in length at  $x$  age, %
- $L_x$  = comparator reading of specimen at  $x$  age—reference bar comparator reading at  $x$  age, and
- $L_i$  = initial comparator reading of specimen—reference bar comparator reading, at the same time
- $L_g$  = nominal gage length, or 250 mm (10 in.) as applicable. (See C 490).

10.2 Calculate length change values for each bar to the nearest 0.001 % and report averages to the nearest 0.01 %.

## 11. Report

11.1 Report type of cement, and, if blending material is used, its identification and amount and whether cement and blending material, if used, meet the applicable specifications. Report the initial comparator reading of each bar at the time of immersion in sulfate solution; this is the base value for calculation of length changes. Report the subsequent length changes in percent of base value to the nearest 0.001 % for individual bars and the nearest 0.01 % for averages. Comment on the visual condition of bars at end of the test. With each report of mean length change of bars at a particular age, report number of bars (*n*), standard deviation of length change, and coefficient of variation of length change. Data from at least three bars must be available at any age to constitute a valid test at that age. Depending upon how many remaining specimens there are, the maximum permissible range of the values must not exceed the values of length change in percent in Table 1.

11.2 Report any variation from the procedure outlined in the text as it relates to solution composition, concentration, or temperature, mortar proportions, age, or maturity, and so forth.

## 12. Precision and Bias

12.1 The precision of this test method has been evaluated by cooperative testing and found to vary with the type of cement studied, hence separate precision statements are given.<sup>6</sup>

12.1.1 *Blended Cements*—The single-operator standard deviation has been found to be 0.010 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same blended cement Type IP or IS, should not differ from each other by more than

0.028 %. The multi-laboratory standard deviation has been found to be 0.020 % for expansions between 0.04 %, and 0.07 %. Therefore, results of two properly conducted tests on the same blended cement Type IP or IS in two different laboratories, should not differ from each other by more than 0.056 %.

12.1.2 *Type II Cement*—The single-operator standard deviation has been found to be 0.005 %, for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same cement, should not differ from each other by more than 0.014 %. The multi-laboratory standard deviation has been found to be 0.020 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests on the same cement in two different laboratories, should not differ from each other by more than 0.056 %.

12.1.3 *Type V Cement*—The single-operator standard deviation has been found to be 0.003 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests by the same operator on the same cement, should not differ from each other by more than 0.009 %. The multi-laboratory standard deviation has been found to be 0.010 % for expansions between 0.04 % and 0.07 %. Therefore, results of two properly conducted tests on the same cement in two different laboratories should not differ from each other by more than 0.028 %.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, no statement on bias is being made.

## 13. Keywords

13.1 acceptability; expansion; fly ash; mortars; pozzolans; precision; search report; slag; sulfate attack; sulfate-resisting cements; tests

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C01 – 1003.

## ANNEX

### (Mandatory Information)

#### A1. Determination of Water Content of Sodium Sulfate

##### A1.1 Apparatus

A1.1.1 Porcelain crucible, 15mL,

A1.1.2 Oven, drying capable of maintaining  $110 \pm 5^\circ\text{C}$

A1.1.3 Desiccator with a good desiccant, such as magnesium perchlorate, or activated alumina. Calcium sulfate treated with a color-change indicator that shows when it has lost its effectiveness is also suitable.

A1.1.4 Balance as described in Section 4 of Test Method C 114.

##### A1.2 Determining the water content

Determine the mass of approximately 1 g of the sodium sulfate in a tared porcelain crucible to the nearest 0.0001 g. Dry the crucible and its contents for  $1 \pm 0.25$  hour in an oven at a temperature of  $110 \pm 5^\circ\text{C}$ . Remove the crucible and its contents from the oven and cool in a desiccator until they are

at room temperature. (See Note A1.1) Record the mass of the crucible and sodium sulfate after heating.

NOTE A1.1—15-30 minutes are usually sufficient. A small Pyrex beaker could be used as long as the mass can accurately be weighed (e.g. 50ml beaker).

##### A1.3 Calculation

Calculate the water content of the sodium sulfate to the nearest 0.1%:

$$\% \text{ WC} = \frac{(M_i - M_f) \times 100}{M_{cs}} \quad (\text{A1.1})$$

where:

% WC = water content, %

$M_i$  = mass of sodium sulfate + crucible before heating, g



$M_f$  = mass of sodium sulfate + crucible after heating, g  
 $M_{ss}$  = mass of sodium sulfate before heating, g

Use the % WC to correct the mass of sodium sulfate used in Section 5.4 (see Note A1.2).

NOTE A1.2—Example: Water content was determined to be 5.0 %. Thus, the corrected mass of anhydrous sodium sulfate present would be:

$$\frac{\text{Mass sodium sulfate uncorrected} \times (100 - \% \text{ WC})}{100} \quad (\text{A1.2})$$

or:

$$\frac{50 \text{ g Na}_2\text{SO}_4 \cdot x \text{ H}_2\text{O} \times (100 - 5)}{100} = 47.5 \text{ g} \quad (\text{A1.3})$$

or in reverse:

$$\frac{50 \text{ g Na}_2\text{SO}_4 \times 100}{(100 - 5)} = 52.6 \text{ g Na}_2\text{SO}_4 \cdot x \text{ H}_2\text{O} \quad (\text{A1.4})$$

## APPENDIX

### (Nonmandatory Information)

#### X1. Technical Background

X1.1 Test Method C 1012 was developed to explore the suitability of a test method for sulfate resistance based on length-change measurements of molded prisms immersed in sulfate solution. This approach was taken after it was established that Test Method C 452, in which the sulfate is added to the mortar as it is mixed, and the prisms stored in fresh water, was not able properly to evaluate sulfate resistance of blended cements and blends of portland cement with pozzolan or slag.

X1.2 This test method has been subjected to two rounds of cooperative testing. The first program involved five cements (Types I, II, and V of Specification C 150, and Types IS and IP of Specification C 595). The test solution contained both sodium sulfate and magnesium sulfate, each in the amount of 0.176 mol/L. A report of this work is available. A paper giving results using this test method has been published (1).<sup>7</sup> The second program involved eight blends using a Type I and a Type II cement with 50 and 70 % slag, 25 % Class F, and 35 % Class C fly ash (Specification C 618). Two test solutions were used: one contained 0.303 mol/L sodium sulfate and 0.049 % magnesium sulfate; the other contained 0.352 mol/L sodium sulfate only.

X1.3 This test method involves length change of hydraulic-cement mortar bars immersed in a sulfate solution. Mortar bars are placed in the sulfate solution after companion mortar cubes have reached a compressive strength of  $20.0 \pm 1.0$  MPa ( $3000 \pm 150$  psi). Attempts to use Test Method C 452 for blended cements and blends of portland cement and slag (Specification C 989) or pozzolan (Specification C 618) with portland cement failed to yield results that correlate with field and laboratory experience where the mortar or concrete developed some maturity prior to being exposed to external sulfate solution (2). Since some blended cements and some blends of slag and pozzolan with portland cement have been successfully used where sulfate resistance was needed, they are tested under circumstances which permit comparisons to be based on results obtained when all the sulfate exposures are begun at equivalent

strength since, in practice, it is likely that the concrete will be at about the same strength when sulfate attack begins regardless of the type of cementitious medium employed. This test method is also applicable for use in evaluating portland cements.

X1.4 The solution of 0.176 mol/L of each of the two sulfates (magnesium and sodium) studied in the first cooperative program, is believed likely to adversely affect blends or blended cements containing slag, due to a magnesium ion (3, 4) attack, this attack being unrelated to sulfate attack. Therefore, this solution should not be used to evaluate such systems unless they are to be exposed in service to attack in which the magnesium ion ( $\text{Mg}^{++}$ ) is present in amounts approaching that of the test solution (4200 ppm).

X1.5 This test method does not simulate the mechanism of sulfate attack by solutions of sulfate compositions other than that used. If evaluation of behavior due to exposure to a given sulfate solution is desired, that solution should be used.

X1.6 The user may therefore modify this test method to use any relevant compositions or concentrations of aggressive solutions, other ages or degrees of maturity as the basis for beginning the exposure, mortars of different proportions, or different or additional means of assessing the influence of the aggressive solution on the specimens. Much work was done using mortar bars of lower strength (higher porosity) in previous cooperative tests sponsored by Committee C01 (5-7). A procedure in which sodium sulfate is kept at constant alkalinity/acidity has been proposed (8). Other methods of assessing the influence of the aggressive solutions that have been used or proposed include (a) change in pulse velocity (Test Method C 597), (b) change in resonant frequency (Test Method C 215), (c) change in compressive strength (Test Methods C 109/C 109M and C 349), (d) change in flexural strength (Test Method C 348), (e) change in mass, and (f) change in hardness (Test Methods E 18).

X1.7 Work reported by Polivka and Brown (9) in 1958 and by Mehta and Polivka in 1975 (10) included tests of concrete exposed to a mixed sulfate solution containing 5 % each of

<sup>7</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

sodium and magnesium sulfate. Other relevant work is cited in the references (1-19).

X1.8 The second cooperative study used both a mixed sodium/magnesium sulfate solution and a sodium sulfate

solution. Precision of the results and the ranking of the cements did not differ between the solutions. Hence, the simpler test, using sodium sulfate, was selected as the standard.

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## Standard Test Method for Temperature of Freshly Mixed Portland Cement Concrete<sup>1</sup>

This standard is issued under the fixed designation C 1064/C 1064M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This test method covers the determination of temperature of freshly mixed portland cement concrete.

1.2 The values stated in inch-pound or SI units are to be regarded separately as standard. Within the text, SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the specification.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*

C 172 Practice for Sampling Freshly Mixed Concrete<sup>2</sup>

### 3. Significance and Use

3.1 This test method provides a means for measuring the temperature of freshly mixed concrete. It may be used to verify conformance to a specified requirement for temperature of concrete.

3.2 Concrete containing aggregate of a nominal maximum size greater than 3 in. [75 mm] may require up to 20 min for the transfer of heat from aggregate to mortar. (See ACI Committee 207.1R Report.<sup>3</sup>)

### 4. Apparatus

4.1 *Container*, shall be made of nonabsorptive material and large enough to provide at least 3 in. [75 mm] of concrete in all directions around the sensor of the temperature measuring device; concrete cover must also be at least three times the nominal maximum size of the coarse aggregate.

4.2 *Temperature Measuring Device*, shall be capable of accurately measuring the temperature of the freshly mixed concrete to  $\pm 1^\circ\text{F}$  [ $\pm 0.5^\circ\text{C}$ ] throughout a range of 30° to 120°F [0° to 50°C]. The temperature measuring device shall require immersion of 3 in. [75 mm] or less during operation.

4.3 Partial immersion liquid-in-glass thermometers (and possibly other types) shall have a permanent mark to which the device must be immersed without applying a correction factor.

4.4 *Reference Temperature Measuring Device*, shall be readable and accurate to  $\pm 0.5^\circ\text{F}$  [0.2°C] at the verification points in 5.1. A certificate or report that verifies the accuracy shall be available in the laboratory for review. Accuracy of liquid-in-glass reference temperature measuring devices shall be verified once. Verification of direct-reading resistance reference temperature measuring device shall be performed every twelve months. The certificate or report shall provide documentation that the reference standard used in the verification is traceable to NIST.

### 5. Calibration of Temperature Measuring Device

5.1 Each temperature measuring device used for determining temperature of freshly mixed concrete shall be calibrated annually, or whenever there is a question of accuracy. This calibration shall be performed by comparing the readings of the temperature measuring device at two temperatures at least 30°F [15°C] apart.

5.2 Calibration of the temperature measuring devices may be made in oil or other suitable baths having uniform density if provision is made to:

5.2.1 Maintain the bath temperature constant within 0.5°F [0.2°C] during the period of the test.

5.2.2 Have both the temperature and reference temperature measuring devices maintained in the bath for a minimum of 5 min before reading temperatures.

5.2.3 Continuously circulate the bath liquid to provide a uniform temperature.

5.2.4 Slightly tap thermometers containing liquid to avoid adhesion of the liquid to the glass if the temperature exposure is being reduced.

### 6. Sampling Concrete

6.1 The temperature of freshly mixed concrete may be measured in the transporting equipment provided the sensor of

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.60 on Fresh Concrete Testing.

Current edition approved Nov. 10, 2001. Published January 2002. Originally published as C 1064 – 86. Last previous edition C 1064 – 99.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> Available from American Concrete Institute, Box 9094, Farmington Hills, MI 48333.

temperature measuring device has at least 3 in. [75 mm] of concrete cover in all directions around it.

5.2 Temperature of the freshly mixed concrete may be obtained following concrete placement using the forms as the container.

5.3 If the transporting equipment or placement forms are not used as the container, a sample shall be prepared as follows:

6.3.1 Immediately, prior to sampling the freshly mixed concrete, dampen (with water) the sample container.

6.3.2 Sample the freshly mixed concrete in accordance with practice C 172, except that composite samples are not required the only purpose for obtaining the sample is to determine temperature.

6.3.3 Place the freshly mixed concrete into the container.

6.3.4 When concrete contains a nominal maximum size of aggregate greater than 3 in. [75 mm], it may require 20 min before the temperature is stabilized after mixing.

### Procedure

7.1 Place the temperature measuring device in the freshly mixed concrete so that the temperature sensing portion is

submerged a minimum of 3 in. [75 mm]. Gently press the concrete around the temperature measuring device at the surface of the concrete so that ambient air temperature does not affect the reading.

7.2 Leave the temperature measuring device in the freshly mixed concrete for a minimum period of 2 min or until the temperature reading stabilizes, then read and record the temperature.

7.3 Complete the temperature measurement of the freshly mixed concrete within 5 min after obtaining the sample.

### 8. Report

8.1 Record the measured temperature of the freshly mixed concrete to the nearest 1°F [0.5°C].

### 9. Precision and Bias

9.1 The precision and bias of this test method have not been determined. A precision and bias statement will be included when sufficient test data have been obtained and analyzed.

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# Standard Specification for Use of Silica Fume as a Mineral Admixture in Hydraulic- Cement Concrete, Mortar, and Grout<sup>1</sup>

This standard is issued under the fixed designation C 1240; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope \*

1.1 This specification covers silica fume for use in concrete and other systems containing hydraulic cement.

1.2 In the cases of slurried or densified silica fume, perform the tests on the raw silica fume from which these products have been made.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 The following safety hazards caveat pertains only to the test methods portions, Sections 10-19, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Read the material safety data sheets for materials used.

1.5 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes (excluding those in tables) shall not be considered as requirements of this standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)<sup>2</sup>

C 114 Test Methods for Chemical Analysis of Hydraulic Cement<sup>2</sup>

C 125 Terminology Relating to Concrete and Concrete Aggregates<sup>3</sup>

C 157 Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete<sup>3</sup>

C 183 Practice for Sampling and the Amount of Testing of Hydraulic Cement<sup>2</sup>

C 185 Test Method for Air Content of Hydraulic Cement Mortar<sup>2</sup>

C 219 Terminology Relating to Hydraulic Cement<sup>2</sup>

C 311 Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete<sup>3</sup>

C 430 Test Method for Fineness of Hydraulic Cement by the 45- $\mu$ m (No. 325) Sieve<sup>2</sup>

C 441 Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction<sup>3</sup>

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>3</sup>

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements<sup>2</sup>

C 1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution<sup>2</sup>

C 1069 Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption<sup>4</sup>

C 1437 Test Method for Flow of Hydraulic Cement Mortar<sup>2</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *silica fume*—very fine pozzolanic material, composed mostly of amorphous silica produced by electric arc furnaces as a byproduct of the production of elemental silicon or ferrosilicon alloys (also known as condensed silica fume and microsilica).

3.1.2 Other terms in this specification are defined in Terminologies C 125 and C 219.

## 4. Ordering Information

4.1 The purchaser shall specify any optional chemical or physical requirements.

## 5. Chemical Composition

5.1 Silica fume shall conform to the requirements for chemical composition prescribed in Table 1.

## 6. Physical Requirements

6.1 Silica fume shall conform to the physical requirements prescribed in Table 2. Optional physical requirements are given in Table 3.

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.24 on Ground Slag and Pozzolanic Admixtures.

Current edition approved Aug. 10, 2001. Published September 2001. Originally published as C 1240 – 93. Last previous edition C 1240 – 00<sup>1</sup>.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 15.02.

\*A Summary of Changes section appears at the end of this standard.

**TABLE 1 Chemical Requirements**

SiO <sub>2</sub> , min, %	85.0
Moisture content, max, %	3.0
Loss on ignition, max, %	6.0

**TABLE 2 Physical Requirements**

Oversize:	
Percent retained on 45- $\mu$ m (No. 325), max, % <sup>A</sup>	10
Percent retained on 45- $\mu$ m (No. 325), max variation from average, percentage points <sup>B</sup>	5
Accelerated pozzolanic strength activity index: <sup>C</sup>	
With portland cement at 7 days, min percent of control	105
Specific surface, min, m <sup>2</sup> /g	15

<sup>A</sup> Exercise care to avoid retaining agglomerations of extremely fine material.  
<sup>B</sup> The average shall consist of the ten preceding tests or all of the preceding tests if the number is less than ten.  
<sup>C</sup> Accelerated pozzolanic strength activity index is not to be considered a measure of the compressive strength of concrete containing the silica fume. This is a measure of the reactivity of a given silica fume with a given cement and may vary with the source of both the silica fume and the cement.

**TABLE 3 Optional Physical Requirements<sup>A</sup>**

Uniformity requirements:	
When air-entraining concrete is specified, the quantity of air-entraining agent required to produce air content of 18.0 vol % of mortar shall not vary from the average established by the ten preceding tests or by all preceding tests if less than ten, by more than, %	20
Reactivity with cement alkalis: <sup>B</sup>	
Reduction of mortar expansion at 14 days, min, %	80
Sulfate resistance expansion, <sup>C</sup>	
(moderate resistance) 6 months, max, %	0.10
(high resistance) 6 months, max, %	0.05
(very high resistance) 1 year, max, %	0.05

<sup>A</sup> Will be made only at the request of the purchaser.  
<sup>B</sup> The indicated tests for reactivity with cement alkalis shall not be requested unless the material is to be used with an aggregate that is regarded as deleteriously reactive with alkalis in hydraulic cement. The test for reduction of mortar expansion may be made using any high-alkali cement in accordance with Test Methods C 311, if the cement to be used in the work is not known or is not available at the time of the test. The test for mortar expansion should be performed by each of the high-alkali cements to be used in the work.  
<sup>C</sup> Only one limit shall be specified.

**7. Sampling**

7.1 When the purchaser desires that the silica fume be sampled and tested to verify compliance with this specification, perform the sampling and testing in accordance with Practice C 183, modified as described in 7.3.

NOTE 1—Exercise caution in the interpretation of Practice C 183, since there is a difference between the continuous manufacture of hydraulic cement and the generation and collection of silica fume. To a great extent, storage is dictated by the design of the silica-fume collection system. The design of silica-fume collection systems may not have provided for sampling points and practices.

7.2 Practice C 183, as modified, is not designed for manufacturing quality control and is not required for manufacturer’s certification.

7.3 The following modification of Practice C 183 is necessary to render it applicable to silica fume.

7.3.1 Replace the words “hydraulic cement” and “cement” with the words “silica fume” every time that they appear in the text.

7.3.2 All samples, whether grab or composite, shall have a mass of at least 1 kg (2 lb).

7.3.3 When compliance verification tests of silica fume are required to be made at a laboratory other than that of the silica-fume manufacturer or marketer, coordinate the silica-fume sampling schedule, sample transportation time, and sample testing schedule among the purchaser, manufacturer, and testing laboratory so that the test results will be available when the decision to accept or reject the silica fume must be made.

7.3.4 The section entitled “Sampling” is modified as follows:

7.3.4.1 Take two grab samples or two composite samples for the first 100 Mg (110 tons) of silica fume. Take a grab sample or a composite sample for each subsequent 100 Mg (110 tons) of silica fume, but not less than two samples shall be taken in any sampling program.

7.3.4.2 *From Bulk Storage at Points of Discharge*—Withdraw silica fume from the discharge openings in a steady stream until sampling is completed. In sampling bulk storage at points of discharge, while the silica fume is flowing through the openings, take samples at such intervals so that, at a minimum, the sampling requirements of 7.3.4.1 are met.

7.3.5 The section entitled “Amount of Testing” is modified by deleting the first paragraph, “General.”

**8. Frequency of Tests**

8.1 Make all chemical determinations and physical tests on composite samples representing no more than 400 Mg (440 tons) each. Prepare each composite sample by combining portions from the samples representing each 100 Mg (110 tons), so that each 100 Mg is represented equally.

8.2 Test specific surface samples and accelerated pozzolanic strength activity index composite every 3000 Mg (3300 tons) or 3 months, whichever gives the highest frequency. Prepare each composite sample by combining portions from the samples representing each 1000 Mg (1100 tons) or 1 month, whichever gives the highest frequency, so that each sample is represented equally.

**9. Preparation of Sample**

9.1 Prepare composite samples for tests, as required in Section 8, by arranging all test samples in groups, with each group representing the number of megagrams required by the test or tests for which the composite sample is intended. From each of the samples in a group, take equal portions, sufficient in amount to form a composite sample large enough to permit making the required physical or chemical determinations.

9.2 Prior to testing, mix grab samples and composite samples thoroughly. A clean and dry laboratory concrete drum mixer provides adequate mixing for this purpose. Take care to limit the volume of silica fume in the drum mixer to the range of 10 to 50 % of the drum’s total capacity. If necessary, secure a sheet of polyethylene film on the drum with an elastic tiedown to keep the material in the drum. Limit the mixing action to 5 ± 1 min.

9.2.1 When a small sample size precludes the use of a concrete mixer, use a heavy plastic bag, of a capacity at least five times larger than the sample volume, to mix the sample thoroughly. After placing the sample in the bag, close the bag by tying the bag opening tightly, and mix the material by

rolling the bag around for  $5 \pm 1$  min.

9.3 Take material for specific tests from a thoroughly mixed sample by using a sampling device (sampling tube, scoop, etc.) of appropriate size to make a test specimen. Make this test specimen from at least six random subsamples.

**TEST METHODS—CHEMICAL ANALYSIS**

**10. Silicon Dioxide**

10.1 *Reference Method*—Use the reference method in Test Methods C 114 for cements with insoluble residue greater than 1 %.

**11. Moisture Content, Loss on Ignition, and Available Alkalies**

11.1 Follow the applicable provisions of Test Methods C 311.

**TEST METHODS—PHYSICAL TESTS**

**12. Density**

12.1 *Equipment:*

- 12.1.1 *Two 500-mL Volumetric Flasks*, Class A.
- 12.1.2 *Balance*, with an accuracy of at least 0.01 g.
- 12.1.3 *Constant Temperature Bath*, capable of being regulated within  $\pm 0.5^\circ\text{C}$  ( $1.0^\circ\text{F}$ ).

12.2 *Deionized Water.*

12.3 *Procedure:*

12.3.1 Determine the density of the material as received, unless otherwise specified, as follows. If density determination on an ignited sample is required, first ignite the sample as described in the test for loss on ignition in the applicable section given in Test Methods C 114.

12.3.2 Determine the mass ( $W_p$ ), of a 500-mL volumetric flask, to an accuracy of 0.01 g. Add 30 g of silica fume. Determine the mass of the flask and the contents ( $W_a$ ) to the nearest 0.01 g. Add water to the flask to fill it one-half full, and shake it to ensure thorough wetting of the material. Fill to the mark with water. Remove air bubbles by shaking the flask at 15-min intervals until the liquid is free of air or by applying a vacuum to the flask. After all of the air bubbles are removed, place the flask in a constant temperature bath at  $23 \pm 0.5^\circ\text{C}$  until the flask and its contents reach a constant temperature. Remove the flask from the water bath; immediately add or remove water, at the same temperature, to the flask to get the meniscus on the mark. Wipe dry the exterior of the flask and determine the mass of the flask and its contents ( $W_s$ ).

12.3.3 Empty, clean, and determine the mass of the 500-mL volumetric flask, used above, filled to the mark with water ( $W_w$ ) stabilized at  $23 \pm 0.5^\circ\text{C}$ .

12.4 *Calculation:*

$$D_{sf} = \frac{(W_a - W_p)}{500 \text{ mL} - [(W_s - W_a)/D_w]} \quad (1)$$

where:

- $D_{sf}$  = density of silica fume,  $\text{Mg}/\text{m}^3$ ,
- $W_p$  = mass of 500-mL volumetric flask, g,
- $W_a$  = mass of 500-mL volumetric flask plus approximately 30 g of silica fume, g,

$W_s$  = mass of 500-mL volumetric flask plus silica fume plus water to the mark, g,

$W_r$  = mass of 500-mL volumetric flask plus water to the mark, g, and

$D_w = (W_r - W_p)/500\text{-mL}$ ,  $\text{Mg}/\text{m}^3$ .

12.5 Report the average of two density determinations.

**13. Oversize, Amount Retained When Wet-Sieved on a 45- $\mu\text{m}$  (No. 325) Sieve**

13.1 Use Test Method C 430.

NOTE 2—Oversize is used to determine the amount of contaminating material retained on the 45- $\mu\text{m}$  sieve. See Appendix X2.

**14. Specific Surface**

14.1 Determine the specific surface by the BET, nitrogen adsorption method, in accordance with Test Method C 1069.

NOTE 3—Manufacturers and examples of nitrogen adsorption instrumentation include Horiba Instruments, Inc., Irvine, CA, 5A-9600; Micromeritics Instrument Corporation, Norcross Georgia, FlowSorb-II 2300; Quantachrome Corporation, Boynton Beach, FL, Quantasorb Jr.; and JUWE Laborgerate Service GmbH, Korschbroich, Germany, Stroehlein AREAmeter II.

**15. Air Entrainment of Mortar**

15.1 Follow the applicable provisions of Test Methods C 311, except use the following test mixture and equation for  $W_c$ :

	Test Mixture
Portland cement, g	300
Silica fume, g	30
20–30 Standard Ottawa sand, g	1170
Water, mL, sufficient to give a flow of 80 to 95 %	Y
Neutralized Vinsol resin solution, mL, sufficient to produce an air content of $18 \pm 3$ %	Z

$$W_c = \frac{300 + 1170 + 30 + (300 \times P \times 0.01)}{300/3.15 + 1170/2.65 + (30/D) + [(300 \times P \times 0.01)/1]} \quad (2)$$

Then calculate:

$$\text{Air content, volume \%} = 100[1 - (W_d/W_c)]W_a = W/400 \quad (3)$$

where:

- $W_a$  = actual mass per unit of volume of mortar as determined by Test Method C 185,  $\text{g}/\text{mL}$ ,
- $W$  = mass of the specified 400 mL of mortar (see Test Method C 185), g,
- $W_c$  = theoretical mass per unit volume, calculated on an air-free basis and using the values for density and quantities of the materials in the mixture,  $\text{g}/\text{mL}$ ,
- $P$  = percent of mixing water plus Vinsol resin solution based on mass of cement, and
- $D$  = density of silica fume used in the mixture,  $\text{Mg}/\text{m}^3$ .

15.2 Determine the flow in accordance with the applicable provisions of Test Method C 109/C 109M.

**16. Accelerated Pozzolanic Strength Activity Index with Portland Cement**

16.1 Use the applicable section on strength activity index with portland cement of Test Methods C 311, except change to reflect testing at constant water to cementitious materials ratio. Prepare test specimens from the batch proportions below,

molding three cubes from both the control mix and the test mix.

16.1.1 *Control Mixture:*

- 16.1.1.1 500 g of portland cement,
- 16.1.1.2 1375 g of graded standard sand, and
- 16.1.1.3 242 mL of water.

16.1.2 *Test Mixture:*

- 16.1.2.1 450 g of portland cement,
- 16.1.2.2 50 g of silica fume,
- 16.1.2.3 1375 g of graded standard sand, and
- 16.1.2.4 242 mL of water.
- 16.1.2.5 *X* grams of dry high-range water reducer, meeting ASTM C 494 Type F, required to produce a flow of 100 to 115 %. Add the high-range water reducer directly to the mixing water in the mixing bowl. Then add the cement or the cement-silica fume mixture and start the mixing cycle.

16.1.3 Determine the flow in accordance with the applicable provisions of Test Method C 1437.

16.1.4 *Storage of Specimens*—After 24 h of initial curing in the moist room ( $23 \pm 2^\circ\text{C}$  and relative humidity of not less than 95 %), place the cubes in airtight glass containers and store at  $65 \pm 2^\circ\text{C}$  for six days.

16.1.5 Determine the compressive strength, as specified in Test Method C 109/C 109M, of the three specimens of the control mixture and the three specimens of the test mixture at 7 days after molding.

## 17. Reactivity with Cement Alkalies

17.1 Determine the reduction of mortar expansion in accordance with Test Method C 441, except that the amount of silica fume in the test mixture shall be 10 % by mass of cementitious material.

## 18. Sulfate Resistance

18.1 Determine sulfate resistance according to Test Method C 1012, except that the amount of silica fume used in the test mixture is 10 % by mass of cementitious material.

## 19. Bulk Density

19.1 The bulk density of silica fume is defined as the mass of a unit volume of loose silica fume.

19.2 This test method covers determination of the bulk density of silica fume, as silica fume is transferred from one container to another with controlled minimum compaction. Its particular usefulness is in connection with identifying material form (as produced or densified), silo or truck storage capacity, material handling and transportation characteristics.

19.3 *Equipment:*

19.3.1 *Balance*, meeting Specification C 1005, with a sensitivity of 0.1 g.

19.3.2 *Vibrating Table*<sup>5</sup>, Table top, electromagnetic vibrating table, with a controlled low-amplitude that does not exceed 1 mm linear vibration. Approximate deck size is 175 × 250 mm with a 5 kg capacity. The amplitude of the vibration shall be capable of being regulated to suit the characteristics of the material being handled.

19.3.3 *Stainless Steel Beaker*, of known volume, not less than 1 L calibrated to the nearest  $\pm 1$  mL. Without a spout.

19.4 *Procedure:*

19.4.1 Determine the mass of the clean dry beaker to the nearest 1 g.

19.4.2 Fill the beaker with silica fume and compact by use of the vibrating table at a mid-range setting for 15 s, adding material as needed.

19.4.3 Screed or strike off the measure, with a straight edge or spatula, to produce a flat, even surface, that is level with rim or edge of the beaker. Wipe off any excess silica fume that may adhere to the sides.

19.4.4 Place the filled measure on the balance and determine the mass of the silica fume to the nearest 1 g.

19.5 *Calculation:*

19.5.1 Divide the net mass of the silica fume in grams by the volume of the container in milliliters. Multiply by 1000 to express the density in kilograms per cubic meter. To convert the value in kilograms per cubic meter to pounds per cubic foot, divide by 16.01846.

## 20. Report

20.1 Report the following information:

- 20.1.1 SiO<sub>2</sub> content, %,
- 20.1.2 Moisture content, %,
- 20.1.3 Loss on ignition, %,
- 20.1.4 Oversize, %,
- 20.1.5 Bulk density, kg/m<sup>3</sup>,
- 20.1.6 Density, Mg/m<sup>3</sup>,
- 20.1.7 Name of manufacturer and brand, if applicable,
- 20.1.8 Accelerated Pozzolanic Strength Activity Index,
- 20.1.9 Specific surface, m<sup>2</sup>/g, and
- 20.1.10 Available alkalies, as equivalent Na<sub>2</sub>O, %.

20.2 Report the following information when specifically requested by the purchaser:

- 20.2.1 The quantity of air-entraining agent compared to the 10 preceding tests, %,
- 20.2.2 Reduction of mortar expansion, %, and
- 20.2.3 Sulfate resistance expansion, %.

## 21. Precision and Bias

21.1 *Precision:*

21.1.1 *Accelerated Pozzolanic Strength Activity Index Test:*

21.1.1.1 *Single-Operator Precision*—The precision of this test will be evaluated using Practice C 670.

21.1.1.2 *Multilaboratory Precision*—The precision of this test will be evaluated using Practice C 670.

21.1.2 *Density Test:*

21.1.2.1 *Single-Operator Precision*—The single-operator standard deviation among single test results (a test result is defined in this specification as the average of two separate measurements) has been found to be 0.035 Mg/m<sup>3</sup>.<sup>6</sup> Therefore, results of two properly conducted tests by the same operator should not differ by more than 0.099 Mg/m<sup>3</sup> on the same silica fume.

<sup>5</sup> A suitable vibrating table is the Syntron Paper Jogger, Model J-1, manufactured by F.M.C. Corp., 57 Cooper Ave., Homer City, PA 15748.

<sup>6</sup> These measurements represent, respectively, the (1s) and (d2s) limits in accordance with Practice C 670.



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21.1.2.2 *Multilaboratory Precision*—The multilaboratory standard deviation among single test results (a test result is defined in this specification as the average of two separate measurements) has been found to be  $0.047 \text{ Mg/m}^3$ .<sup>6</sup> Therefore, results of two properly conducted tests in different laboratories on the same silica fume should not differ by more than  $0.132 \text{ Mg/m}^3$  of their average.<sup>6</sup>

### 21.1.3 *Bulk Density*:

21.1.3.1 *Precision*—The precision of this test method will be evaluated using Practice C 670.

21.1.3.2 *Bias*—Since there is no accepted reference material suitable for determining any bias that might be associated with this test method, no statement on bias is being made.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures for measuring the accelerated pozzolanic strength activity index and the density, no statement on bias is being made.

## 22. Rejection and Retesting

22.1 The purchaser has the right to reject material that fails to conform to the requirements of this specification. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the tests, the producer or supplier is not prohibited from making a claim for retesting.

## 23. Certification

23.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples have been tested as directed in this specification and the specified requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

## 24. Packaging and Package Marking

24.1 When silica fume is delivered in packages, the name, and brand, if applicable, of the manufacturer or distributor and the mass of the silica fume contained therein shall be marked plainly on each package. Similar information shall be provided in the shipping invoices accompanying the shipment of packaged or bulk silica fume in dry or slurried forms. All packages shall be in good condition at the time of inspection.

## 25. Storage and Inspection

25.1 Silica fume shall be stored in such a manner as to permit easy access for the proper inspection and identification of each shipment. Facilities for inspection and sampling shall be provided at the point from which the material is to be shipped.

## APPENDIXES

### (Nonmandatory Information)

#### X1. SILICA CONTENT

X1.1 Since the quantity of silica in the amorphous state is one of the primary characteristics that determines the amount of activity of silica fume, the chemical analysis for silica content is important. At the present time, there are no National Institute of Standards and Technology (NIST) SRMs of silica fume, and reference silicon dioxides ( $\text{SiO}_2$ ) are therefore the only materials available for instrumental standards. Since silica in this specification is limited to 85 %  $\text{SiO}_2$  or higher, silica flour (99.9 %), or silica brick (93.94 %), etc. is adequate for flame atomic absorption (AA) analysis. At the present time, there are problems using inductively coupled plasma (ICP) for the analysis of solutions with high percentages of silica as well

as problems with borate clogging the nebulizer. Another problem is that when a lithium borate fusion is dissolved in HCl, some of the silica returns to a solid phase and can be filtered out. This will affect the total silica analysis by any instrumental method that uses this method of fusion. Energy dispersive X-ray (EDX) is the technique most likely to be used with the X-ray based methods. This technique needs a similar material, a silica fume, for comparison. The “wet method,” sodium carbonate fusion, in which silica is recovered gravimetrically as  $\text{SiO}_2$ , is the only method, at the present, that requires no standard and yields accurate results.

#### X2. OVERSIZE

X2.1 The  $45\text{-}\mu\text{m}$  (No. 325) sieve specification is to be used to determine the amount of foreign material present. Since silica fume is much finer than cement or fly ash, the particles will all pass through the sieve except for foreign material.

Extremely fine materials tend to form agglomerations; good judgment must be exercised to differentiate between easily dispersible agglomerates and foreign materials.