

**UNIVERSIDAD NACIONAL DE INGENIERÍA**

**FACULTAD DE INGENIERÍA QUÍMICA Y TEXTIL**



**“ESPECIFICACIONES TÉCNICAS DE ENSAYOS PARA  
REVESTIMIENTOS DE CAUCHO EN ESTRUCTURAS Y  
ACCESORIOS METÁLICOS”**

**INFORME DE SUFICIENCIA PROFESIONAL  
PARA OPTAR EL TÍTULO PROFESIONAL DE:  
INGENIERO QUÍMICO**

**POR LA MODALIDAD DE ACTUALIZACIÓN DE CONOCIMIENTOS  
PRESENTADO POR:  
JOSÉ FERNANDO CCALLUCO SALINAS**

**LIMA – PERU**

**2010**

## **RESUMEN**

Las diversas industrias requieren de información de las características de los insumos ó productos que son adquiridos por proveedores o empresas de servicio referente a su procedencia y especificaciones. Para esto se realiza un número determinado de ensayos, los cuales sirven de base para el aseguramiento de las características y propiedades en determinadas condiciones de trabajo del producto.

Muchos de estos ensayos están basados en normas internacionales. En este caso particular, el informe trata sobre las especificaciones del material, aseguramiento de las propiedades y características de las gomas para revestimiento de estructuras y accesorios utilizados en la industria minera y pesquera.

Los ensayos que se realizan en las distintas etapas de producción del producto son determinados por límites establecidos según requerimientos que deben cumplir en el campo de trabajo, por ello es indispensable de que estos tengan una hoja de especificación en donde se detalle a qué condiciones fueron producidas y que propiedades físicas presentan.

## INDICE

	<b>Pag.</b>
<b>I INTRODUCCIÓN</b>	<b>7</b>
<b>II ALCANCES DEL INFORME</b>	<b>8</b>
<b>III DESARROLLO DE LOS CONCEPTOS Y TÉCNICAS</b>	<b>9</b>
<b>3.1 Diagrama de proceso de los subproductos y productos.</b>	<b>9</b>
3.1.1 Descripción del diagrama de proceso de los subproductos.	10
3.1.2 Descripción del diagrama de proceso de los productos.	10
<b>3.2 Dosificado.</b>	<b>12</b>
3.2.1 Maquinas y herramientas.	12
3.2.2 Ingreso de materias primas al área de dosificado.	12
3.2.3 Descripción de la operación.	13
3.2.4 Disposición del dosificado hacia molienda externa.	13
3.2.5 Registros de ensayos.	13
<b>3.3 Molienda y calandrado de caucho en área de molino.</b>	<b>13</b>
3.3.1 Maquinarias.	13
3.3.2 Descripción de la operación.	14
3.3.3 Ensayos correspondientes.	16
3.3.4 Registros de ensayos correspondientes.	17
<b>3.4 Revestimientos de caucho por prensado en caliente sobre almas metálicas.</b>	<b>17</b>
3.4.1 Maquinarias.	17
3.4.2 Toma de condiciones ambientales.	19
3.4.3 Descripción de la operación.	20
3.4.3.1 Medición de la rugosidad.	20
3.4.3.2 Aplicación de capa protectora anticorrosivo en la estructura.	20
3.4.3.3 Aplicación del adhesivo.	21
3.4.3.4 Aplicación de pegamento.	21
3.4.3.5 Cargado de caucho al molde ó a la estructura metálica.	21

3.4.3.6	Prensado del molde.	21
3.4.3.7	Inspección visual.	22
3.4.3.8	Prueba de Dureza.	22
3.4.4	Identificación de parámetros a controlar.	22
3.4.5	Registro de ensayos correspondientes.	23
<b>3.5</b>	<b>Revestimiento por vulcanización en caliente (Vapor directo en autoclaves) y en frío.</b>	<b>23</b>
3.5.1	Maquinarias.	23
3.5.2	Descripción de la operación.	24
3.5.2.1	Revestimiento en caliente.	24
3.5.2.2	Revestimiento en frío.	26
3.5.3	Toma de condiciones ambientales.	27
3.5.4	Identificación de parámetros a controlar.	27
3.5.5	Registro de ensayos correspondientes.	27
<b>3.6</b>	<b>Características generales de la pulpa minera que afectan a los revestimientos de caucho.</b>	<b>28</b>
3.6.1	Clasificaciones de la pulpa minera según sus características físicas.	28
3.6.2	Parámetros físicos de la pulpa minera que influye sobre los revestimiento de caucho.	29
<b>3.7</b>	<b>Factores importantes que producen efecto en el transporte hidráulico de sólidos.</b>	<b>31</b>
3.7.1	Velocidad límite de depósito.	31
3.7.2	Pérdidas de carga en mezclas sólido-líquido.	32
3.7.3	Tasas de desgaste.	32
<b>3.8</b>	<b>Propiedades de los tipos de caucho según sus componentes.</b>	<b>33</b>
3.8.1	Elastómeros ó caucho.	33
3.8.2	Caucho Natural.	34
3.8.3	Caucho sintético.	34
3.8.4	Caucho Base.	34
3.8.5	Componentes para la aceleración del caucho.	38



<b>3.9 Características relevantes de los cauchos de acuerdo a su resistencia.</b>	<b>41</b>
3.9.1 Envejecimiento.	41
3.9.2 Resistencia a Hidrocarburos.	41
3.9.3 Temperatura de Servicio.	42
3.9.4 Adherencia al metal.	42
3.9.5 Ozono.	42
3.9.6 Permeabilidad.	42
3.9.7 Resistencia a la Llama.	42
3.9.8 Propiedades Eléctricas.	43
<b>IV DESARROLLO DEL TEMA.</b>	<b>45</b>
<b>4.1 Pruebas y ensayos antes del vulcanizado del caucho.</b>	<b>45</b>
4.1.1 Medición de la densidad relativa.	45
4.1.2 Ensayo de Reometría del caucho crudo acelerado.	48
<b>4.2 Pruebas y ensayos después del vulcanizado del caucho.</b>	<b>52</b>
4.2.1 Ensayo de Abrasión Taber.	52
4.2.2 Ensayo de Dureza.	60
4.2.3 Ensayo de Tracción y Alargamiento.	65
<b>4.3 Elaboración de la hoja de especificaciones.</b>	<b>71</b>
4.3.1 Definición de los productos.	71
4.3.2 Tipo de productos.	71
4.3.2.1 Accesorios.	71
4.3.2.2 Estructuras.	72
4.3.3 Aplicaciones de revestimientos en accesorios mineros.	72
4.3.4 Uso de productos.	74
4.3.5 Diseño del formato de especificación.	74
<b>V CONCLUSIONES Y RECOMENDACIONES</b>	<b>77</b>
<b>VI BIBLIOGRAFÍA.</b>	<b>78</b>
<b>VII GLOSARIO.</b>	<b>81</b>
<b>VIII ANEXOS.</b>	<b>83</b>
Anexo 1: Registro de control de densidad de caucho crudo.	83

<b>Anexo 2: Registro de control de densidad de masterbach.</b>	<b>84</b>
<b>Anexo 3: Registro de ensayos de reometría y dureza.</b>	<b>85</b>
<b>Anexo 4: Registro de abrasión de caucho vulcanizado.</b>	<b>86</b>
<b>Anexo 5: Registro de condiciones ambientales.</b>	<b>87</b>
<b>Anexo 6: Detalles de la ASTM D 2084-93, referente a dimensiones del espécimen, condiciones y procedimiento de ensayo.</b>	<b>88</b>
<b>Anexo 7: Detalles del equipo, condiciones de ensayo, forma del espécimen, procedimiento y cálculos de abrasión según la ASTM D 3389 – 75.</b>	<b>99</b>
<b>Anexo 8: Detalles de dimensionamiento del espécimen de prueba, tipo de indentores, procedimientos y reportes de ensayo según ASTM D 2240-02.</b>	<b>102</b>
<b>Anexo 9: Apartado de ASTM D 412-92, referente a especificaciones del equipo auxiliar.</b>	<b>112</b>

## I INTRODUCCIÓN

Las industrias proveedoras de repuestos y accesorios requieren producir los mismos en óptima calidad basadas en normas las cuales definen propiedades y características que se requiere para proteger elementos de las pulpas mineras que en su forma simple no sería posible una considerable duración en su funcionamiento.

En nuestro país muchas de estas industrias dedicadas al rubro de fabricación de revestimientos de caucho no cuentan con estas especificaciones técnicas ó la emplean parcialmente, pero es fundamental contar con esta información ya que se necesita unos procedimientos adecuados a seguir para confiar en las operaciones que se realizan para obtener productos de buena calidad. A fin de que el usuario pueda acceder a estas y contar con la información suficiente para usar el producto en las óptimas condiciones. Y así asegurar una mejor vida útil.

Los distintos ensayos que se mostrarán mas adelante se basan en normas **ASTM: American Society for Testing of Materials.** y **UNE: Unificación de Normas Españolas.** De este conjunto de normas se han considerado aquellos ensayos normados que tratan al caucho y los que son posibles de realizar dependiendo de los equipos existentes en el laboratorio de control de calidad.

El trabajo a desarrollar está dirigido a proponer una alternativa de información para el usuario.

## **II ALCANCES DEL INFORME**

El presente informe está orientado a los productos de caucho como revestimiento; que consiste de una estructura metálica que está completamente cubierta por caucho, el cual fue obtenido por medio de un prensado en caliente en un molde ó por vapor directo ó en frío. Puede también referirse a los distintos accesorios y estructuras que necesitan ser revestidas de una forma particular.

Este tipo de producto va dirigido hacia el sector minero en donde se maneja pulpas ó lodos que contienen sólidos particulados, los cuales son materiales que originan mucha abrasión (desgaste) sobre la superficie del caucho, la que puede ser muy rápida ó lenta.

El análisis de las condiciones del material, según las pruebas antes y después del vulcanizado del caucho y sus condiciones para la transformación, son importantes ya que estas aseguran si el material soportará condiciones exigidas por el cliente.

### **Objetivos**

- Desarrollo y diseño de la hoja de especificaciones técnicas de los revestimientos de estructuras y accesorios de caucho.
- Mejorar la información que se le imparte a los clientes ó usuarios sobre los productos.

### III DESARROLLO DE LOS CONCEPTOS Y TÉCNICAS

#### 3.1. Diagrama de proceso de los productos y subproductos.

En la figura N° 3.1. Se muestra el diagrama del proceso productivo del caucho para las aplicaciones que se mencionarán más adelante. Que consiste primeramente en elaborar subproductos:

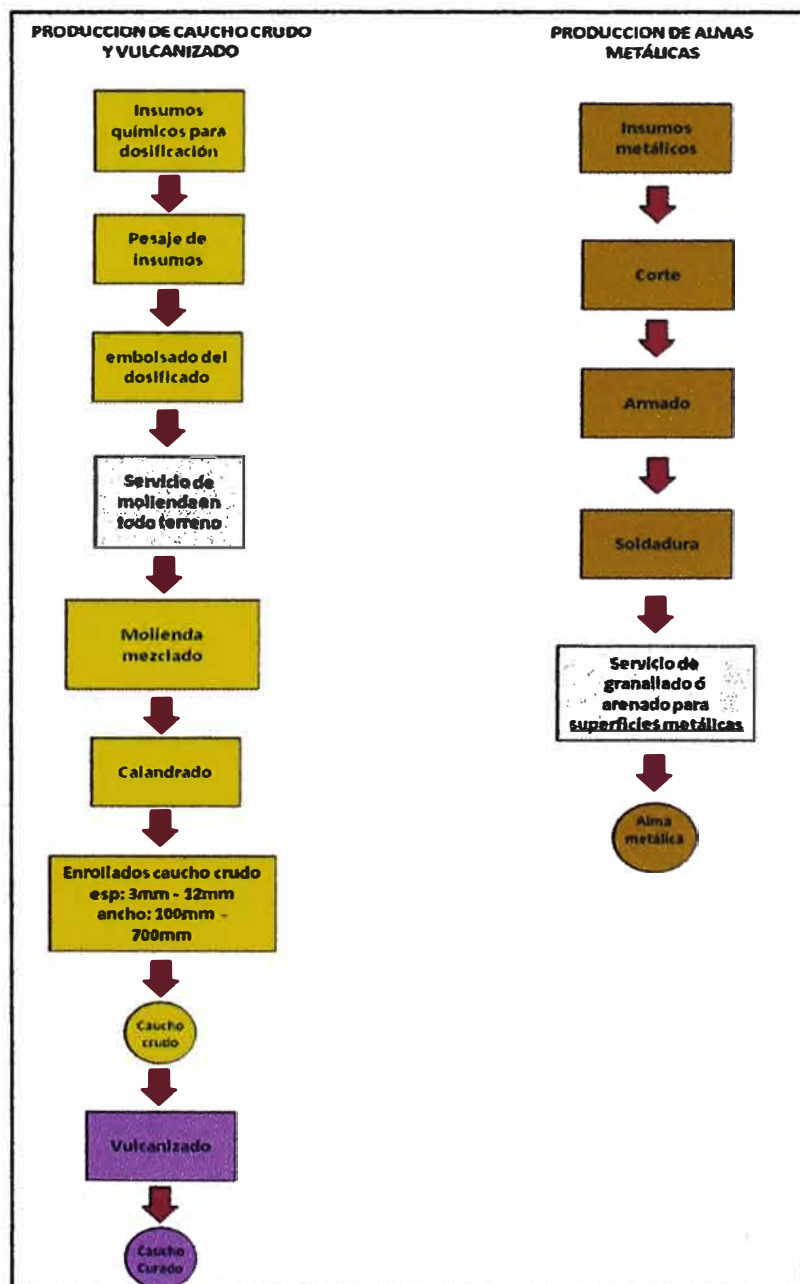


Figura N° 3.1. Diagrama de proceso de sub productos

### **3.1.1 Descripción del diagrama de proceso de los subproductos.**

Recepción de insumos químicos, dosificación, pesado y embalado de estos. Luego se envía para servicios de molienda, obteniéndose los masterbatch, posterior a este se le agrega algunos aditivos adicionales para convertirlo en una mezcla que pueda vulcanizar, obteniéndose el **caucho acelerado crudo**. Al caucho acelerado crudo se le somete a temperaturas altas en un respectivo molde de plancha obteniéndose el **caucho vulcanizado ó curado** generalmente este caucho vulcanizado se usa para revestimientos en frío.

Para las piezas metálicas que son elaboradas con el propósito de dar mas rigidez y forma definida a los respuestos de caucho, generalmente son de acero nodular, SAE1045 etc. Estos se fabrican de acuerdo a un plano se hace el respectivo corte, el armado y con uso de soldadura se une las piezas, obteniéndose el **Alma metálica**.

### **3.1.2 Descripción del diagrama de proceso de los productos.**

En la figura N° 3.2. Se detalla la utilización de los subproductos de caucho para cada línea de producción.

A la estructura se le recubre con el anticorrosivo y adhesivo se clasifica si la estructura es un alma metálica ó un accesorio (Tubería, válvula, etc.). Para un accesorio de mayor tamaño se pueden revestir en frío ó en caliente si es un accesorio de medida estándar este encaja en el interior del molde y se le hace un vulcanizado en caliente en prensa caso contrario en un autoclave.

También se detalla las operaciones de un revestimiento en frío que es casi el mismo procedimiento solo el caucho ya viene en planchas vulcanizadas.

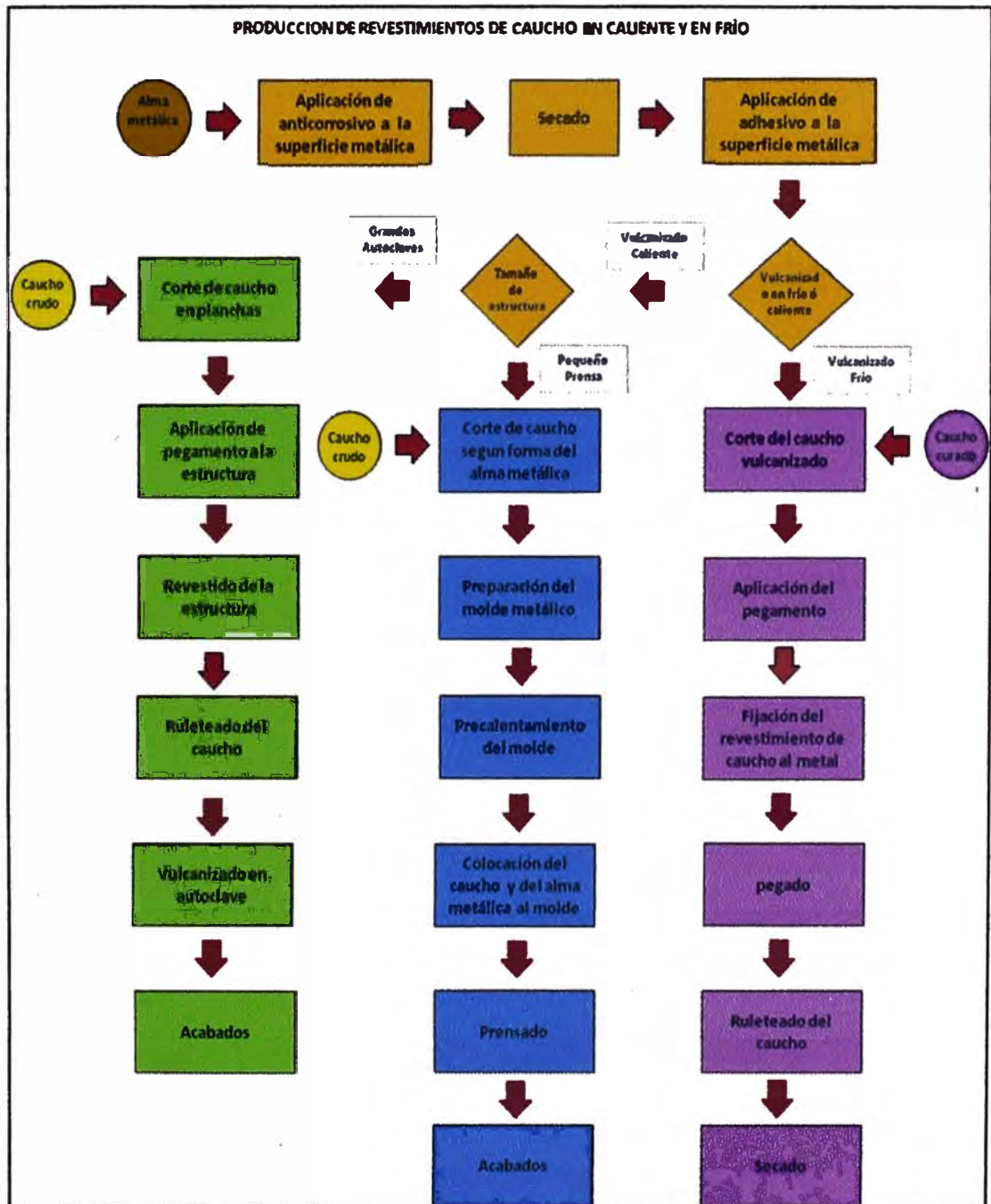


Figura N° 3.2. Diagrama de proceso de los productos.

### **3.2. Dosificado.**

#### **3.2.1 Maquinarias y herramientas.**

##### **Maquinarias**

- Guillotina, 1000 psi reductor eléctrico.
- Balanza MIYAKE, 5kg (3 cifras significativas).
- Balanza MIYAKE, 30kg (2 cifras significativas)
- Balanza ½ Tonelada.

##### **Herramientas**

- Cuchillo para corte de caucho.
- Martillo para quiebre de resinas.
- Calador de caucho.

#### **3.2.2 Ingreso de materias primas al área de dosificado.**

Cuando ingresan insumos ó materias primas para el dosificado es necesario que se tengan certificados de calidad en donde se especifique las propiedades físicas y químicas del producto, y que se pueda contrastar los datos existentes de la hoja con los datos impresos en el producto ó en el envase del mismo. El insumo se inspecciona visualmente observando las condiciones de empaque y si es posible el material, debe estar correctamente embalado con descripción de la fecha de producción, fecha de expiración y el número de lote del certificado; estos como datos son muy importantes para asegurar la buena calidad del insumo que se utiliza.



### **3.2.3 Descripción de la operación.**

Se utiliza la formulación de la composición química establecida para cada caucho en el pesado del dosificado, reúne los tipos de materiales necesarios para la producción de los Masterbach (para caucho de revestimientos) y Mezclas madre (acelerantes de los cauchos para revestimiento).

### **3.2.4 Disposición del dosificado hacia molienda externa.**

En esta etapa luego del pesaje del dosificado este se dispone hacia una molienda externa, para suministrarnos el masterbach o mezcla madre en planchas almacenables, para el trabajo en molienda de aceleración de los cauchos.

### **3.2.5 Registros de ensayo.**

De estos subproductos de molienda externa es necesario hacer la medición de la densidad utilizando densímetro el cual mide la densidad relativa con respecto al agua. Estos resultados son registrados en el registro de control de densidad (Anexo 2).

## **3.3. Molienda y calandrado de caucho en área de molino.**

### **3.3.1 Maquinarias.**

#### **Molino1:**

Marca:	Comercio Ercoles SPA.
Peso:	3000Kg.
Dimensiones:	(32 x 26 x 21) m.
Capacidad de Máxima carga:	100Kg.

**Molino Calandra:**

Modelo:	XY3 1200.
Potencia:	55kW.
Diámetro:	400mm.

**3.3.2 Descripción de la operación.****a) Molienda.**

Es la aceleración de un masterbatch para la obtención de un caucho crudo que puede vulcanizar, se tiene el masterbatch producido en la molienda externa. Para la mezcla, el masterbatch se hace pasar sobre dos cilindros posicionados en paralelo, los cuales giran en sentido contrario, separados por una apertura ó luz, y estos friccionaran con el masterbatch calentándose hasta que adquiera un estado blando, en donde se podrá agregar y mezclar con algunos componentes que no contienen los masterbatch (ver figura N° 3.3.).



Figura N° 3.3. Molino (Mezcla, Aceleración ó reprocesamiento del caucho).

**b) Calandrado.**

La calandra usada para la obtención de rollos de caucho crudo, recibe el caucho acelerado que viene del molino con una temperatura entre  $78^{\circ}\text{C}$  –  $88^{\circ}\text{C}$  en planchas, estas son pasadas a través de dos rodillos llamados polines en donde estos son calentados con vapor a una determinada temperatura entre  $95^{\circ}\text{C}$  –  $110^{\circ}\text{C}$ , dependiendo del tipo de caucho que se desea calandrar y tiene como función dar un espesor parcial adecuado al caucho para su utilización (ver Figuras N° 3.4. y N° 3.5.).



Figura N° 3.4. Calandra de Caucho.



Figura N° 3.5. Faja de la calandra.

En el segundo polín, se define el ancho de los rollos, mediante el corte que se le hace con los cuchillos giratorios, los cuales son graduados manualmente.

Luego de pasar por los dos polines, el ancho de la lámina de caucho pasa por un tercer polín, el cual solo tiene función de que el caucho tenga una determinada tensión para que pueda mantener el espesor parcial generado por los dos primeros polines y a la vez esta pueda llegar a las fajas de reposo en donde se montará capa sobre capa, obteniendo un espesor final requerido. Detalles se muestran en la figura N° 3.6.

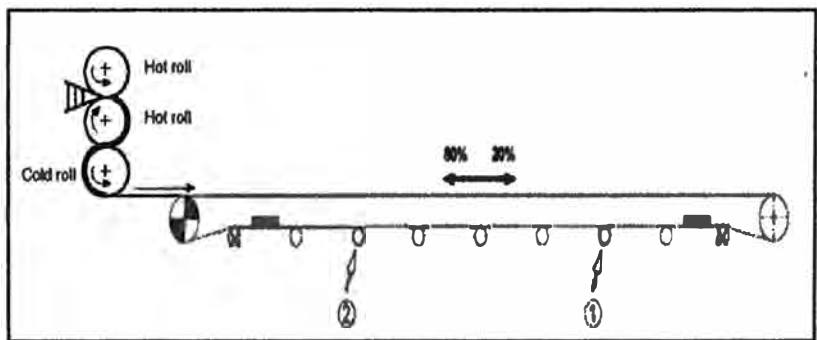


Figura N° 3.6. Esquema del enrollado del caucho en la faja de la calandra.

### 3.3.3 Ensayos correspondientes.

Entre las operaciones de aceleración de los masterbach en el molino y al momento de laminar en la calandra, se corta una probeta de caucho crudo acelerado de la zona de los rodillos de la calandra con dimensiones (10x100x200) mm aproximadamente, el cual es llevado al laboratorio para hacerle el ensayo de reometría, dureza y medición de densidad.

Tamaños de muestra para los ensayos correspondientes:

- Ensayo de reometría: (10 x 40 x 40) mm.
- Ensayo de Dureza: (10 x 70 x 70) mm.
- Ensayo de Medición de densidad: (5 x 5 x 10) mm.

De estos muestreos mediante probetas y ensayos realizados comparados con los límites establecidos se aprueba los materiales rollos de caucho crudo acelerado.

#### **3.3.4 Registros de ensayos correspondientes.**

Los resultados de estos ensayos son registrados en los formatos de:

- Registro de ensayos de reometría y dureza (Anexo 3).
- Registro de densidad de la goma cruda. (Anexo 1).

### **3.4. Revestimientos de caucho por prensado en caliente sobre almas metálicas.**

#### **3.4.1 Maquinarias.**

Para realizar el prensado de estos revestimientos es necesarios contar con prensas a vapor de características señaladas y pueden ser vistas según las Figuras N° 3.7. y N° 3.8.

**Prensa N° 1**

Presión máxima de trabajo: 1500 psi.

N° pistones: 4 de 10" y 2 de 1.1/2".

Apertura máxima: 700mm.

Superficie: 1.3m x 1.3m.

**Prensa N° 2**

Presión máxima de trabajo: 1500 psi.

N° pistones: 1 de 28"

Apertura máxima: 700mm.

Superficie: 1.2m x 1.2m.

**Prensa N° 3**

Presión máxima de trabajo: 1500 psi.

N° pistones: 4 de 24" y 2 de 1.1/2".

Apertura máxima: 785mm.

Superficie: 1.6m x 1.6m.

**Prensa N° 4**

Presión máxima de trabajo: 1500 psi.

N° pistones: 4 de 12".

Apertura máxima: 500 mm.

Superficie: 1.2m x 1.6m.

**Prensa N° 5**

Presión máxima de trabajo: 1500 psi.

N° pistones: 1 de 8".

Apertura máxima: 500 mm.

Superficie: 0.5m x 0.5m.

**Prensa N° 6**

Presión máxima de trabajo: 1500 psi.

N° pistones: 1 de 8”.

Apertura máxima: 500 mm.

Superficie: 0.5m x 0.5m.



Figura N° 3.7. Prensas grandes del 1 a 4 neumáticas a vapor.



Figura N° 3.8. Prensas pequeñas neumáticas a vapor.

**3.4.2 Toma de condiciones ambientales.**

La estructura no debe exponerse a más de 85% de humedad relativa, ya que originaría ataques de corrosión sobre la superficie de la estructura. Se mide temperatura del metal y la humedad relativa.

### **3.4.3 Descripción de la operación.**

Las almas metálicas granalladas se hacen llegar al área de prensas y se evita que estén en contacto con la humedad del ambiente, por consiguiente se efectúa la aplicación de capas de anticorrosivo y adhesión para caucho sobre la superficie metálica, ó si estas capas no son colocadas las almas metálicas se protege con laminas de plástico, para luego aplicarle los respectivos recubrimientos colocarle caucho y el prensado respectivo.

#### **3.4.3.1 Medición de la rugosidad.**

Antes de aplicar la capa protectora contra la corrosión esta estructura metálica es sometida a una medición con el rugosímetro, para asegurar si la rugosidad está dentro de lo requerido (2.5 mils de pulg.), así pueda existir un anclaje para que el revestimiento de caucho se sujete a la superficie metálica.

Por ello es importante evitar el contacto de la superficie metálica con la humedad del ambiente.

#### **3.4.3.2 Aplicación de capa protectora anticorrosivo en la estructura.**

Para evitar corrosión sobre la superficie metálica de la estructura se aplica un recubrimiento anticorrosivo (Chemlock 205), el cual mantiene la rugosidad de la superficie, así también permita la penetración del adhesivo sobre la porosidad resultante de la superficie metálica.



### **3.4.3.3 Aplicación del adhesivo.**

El propósito para la aplicación del adhesivo (Chemlock 220) es para tener un mejor anclaje metal caucho y esta pueda complementarse con la rugosidad de la superficie metálica de la estructura.

### **3.4.3.4 Aplicación de pegamento.**

Se utiliza en el momento de colocar las planchas de caucho para fijar posición en frío y sirve para ayudar al adhesivo creando un contacto del caucho con el metal, cuando el accesorio ó estructura se someta a temperaturas altas, alrededor de 140° C – 150° C, en donde empieza la vulcanización.

### **3.4.3.5 Cargado de caucho al molde ó a la estructura metálica.**

En esta operación hay dos tipos de procedimiento según la geometría del alma metálica. Uno de estos es de colocar el caucho sobre la estructura metálica para tener una buena distribución. El otro caso cuando la estructura es simple, esta se coloca dentro del molde, luego se distribuye el caucho para que cuando se someta al calor esta adquiera fluidez y se distribuya por todos los espacios libres del molde – alma metálica

### **3.4.3.6 Prensado del molde.**

Para el prensado del molde, este se somete a una temperatura de 140° C a 150° C y una presión entre 1000 psi

a 1500 psi, por un tiempo de 1.5h a 2.0h, según sea el tamaño del molde.

Luego de este tiempo la estructura metálica y el revestimiento es extraído del molde como una sola pieza, y este es dejado enfriar por espacio de 30 minutos para darle acabado.

#### **3.4.3.7 Inspección visual.**

Después del tiempo de curado en el prensado, la operación de extracción de la pieza revestida del molde es muy forzada produciéndole al revestimiento imperfecciones, por ello se le debe dar diversos acabados para eliminar los defectos originados por la mencionada operación.

#### **3.4.3.8 Prueba de Dureza.**

La prueba de Dureza se hace utilizando un durómetro de contacto que se encuentra en una escala Shore-A, esta medición se evalúa en base a tablas de especificaciones de dureza para cada tipo de Caucho).

#### **3.4.4 Identificación de parámetros a controlar.**

Los parámetros a controlar para las operaciones son:

- Rugosidad del la estructura metálica (alma metálica)
- Presión de prensado
- Temperatura de prensado
- Tiempo de prensado

- Dureza del revestimiento.

Estos son los parámetros más importantes, y generalmente dependen del tamaño y de la forma del accesorio.

#### **3.4.5 Registro de ensayos correspondientes.**

Los resultados obtenidos son llenados en los siguientes registros:

El registro de condiciones ambientales (Anexo 5).

El registro de ensayos de reometría y dureza del caucho (Anexo 3).

### **3.5. Revestimiento por vulcanización en caliente (Vapor directo Autoclaves) y en frío.**

El método de revestimiento por vulcanización usando vapor directo se aplica para estructuras que no tienen una forma geométrica definida y a la vez si tienen considerables áreas por recubrir. Esta operación se puede realizar mediante dos métodos distintos.

Para estas dos alternativas se tiene revestimientos en frío y en caliente (Vapor directo).

#### **3.5.1 Maquinarias.**

A continuación se presentan las características de los equipos y también imágenes de algunos autoclaves los cuales se puede apreciar en la figura N° 3.9.

##### **Autoclave 1:**

Fabricante: CONRAD ENGELKE.

Tipo: Horizontal.

Presión máxima de trabajo: 50psi.

Diámetro: 1.28m.

Largo: 2.5m.

### **Autoclave 3:**

Marca: Servindustria Ltda.

Año Fabricación: Abril 2008.

Presión máxima de trabajo: 50psi.

Diámetro útil: 1.97m.

Largo útil: 12m.



Figura N° 3.9. Autoclaves para vulcanizado de estructuras.

## **3.5.2 Descripción de la Operación.**

### **3.5.2.1 Revestimiento en caliente.**

En esta operación se prepara la estructura a revestir con un arenado ó granallado y se hace la medición de la rugosidad con una lámina pelicular, con el objetivo de tener 2.5 mils de pulgada de rugosidad; como mínimo después se fijan las

condiciones de operación donde la humedad del ambiente toma un papel determinante sobre la estructura a revestir, debe ser menor a 85%.

Luego de esto se procede a colocarle el anticorrosivo el cual servirá de protección contra la humedad y la corrosión, este tendrá que colocarse sobre toda la superficie del alma metálica.

De igual forma se aplica el adhesivo que se coloca por todo el área que se desea revestir, este recubrimiento facilita la unión del Caucho con la superficie metálica en el momento que se le someterá al calor.

Para fijar el caucho crudo sobre las zonas de la estructura a revestir, se le coloca pegamento, previamente secado al ambiente por 30 minutos se hace el respectivo ruleteo (ver figura N° 3.10.).



**Figura N° 3.10. Ruleteo de plancha de caucho sobre las superficies metálicas.**

Estas estructuras son llevadas a la autoclave según el tamaño que este tenga y estas autoclaves son calentadas con vapor de agua directo con la estructura a revestir.

### **3.5.2.2 Revestimiento en frío**

Para este tipo de revestimiento previamente se hace el vulcanizado de una plancha de caucho de espesor según el espesor y área requerida. Aquí se prepara la superficie metálica a revestir con un proceso de granallado ó arenado y que la rugosidad en esta sea como mínimo a 2.5 mils.

Sobre la superficie de la estructura se hace la aplicación de anticorrosivo y adhesivo previamente arenada o granallada para evitar formación de capas de oxido que pueden perjudicar el anclaje de las capas protectoras y del caucho.

Se prepara la plancha de caucho vulcanizado, previamente cortada según el tipo del revestido, con la aplicación de pegamento en frío de igual forma sobre la superficie metálica que se desea revestir, se deja secar por espacio de 1 hora. Previo a esto se debe cuidar tanto las superficies de la estructura como la superficie de caucho que contienen el pegamento.

Transcurrido el tiempo de secado el revestimiento es colocado, presentado una vez que se fija todas las zonas, y se procede a incidirle presión entre la cara del caucho y la superficie metálica que contienen el pegamento, para que este pueda juntarse lo necesario así el pegamento actúe como medio de anclaje en la unión de caucho metal.

Después de esta tarea se espera 24h como mínimo para el secado.

### **3.5.3 Toma de condiciones ambientales.**

Estas mediciones se hacen 3 veces por día, al inicio de la jornada, a mitad de la jornada y casi al finalizar la jornada, para saber cómo es el comportamiento del ambiente si este trae mucha humedad o si se encuentra en condiciones optimas de trabajo humedad como máximo 85%.

### **3.5.4 Identificación de parámetros a controlar.**

Los parámetros a controlar en la operación son:

- Humedad de ambiente de trabajo.
- Dureza del caucho vulcanizado.
- Acabado superficial e inspección visual.

### **3.5.5 Registro de ensayos correspondientes.**

Los datos obtenidos se llenan en los formatos siguientes:

- El registro de condiciones ambientales. (Anexo 5)
- El registro de ensayos de reometría y dureza del caucho (Anexo 3)

### **3.6. Características generales de la pulpa minera que afectan a los revestimientos de caucho.**

#### **3.6.1 Clasificaciones de la pulpa minera según sus características físicas.**

La mezcla que está en contacto con los revestimientos se clasificar en tres categorías: mezcla ligera, media y pesada.

##### **a) Mezcla ligera:**

Son generalmente las mezclas de pulpas mineras que no están destinados a transportar sólidos. La presencia de los sólidos ocurre accidentalmente ya que el diseño del elemento para el transporte (tubería) no lo contempla.

- Tamaño de los sólidos menores o iguales a 200 micrones.
- Presencia de sólidos es principalmente por accidente
- La gravedad específica es de 1.05
- Sólidos menos del 5% en peso.

##### **b) Mezcla media:**

Son los lodos mineros que se encuentran en el intermedio de un lodo ligero y pesado. En general, el porcentaje de sólidos en una mezcla media oscilará entre 5% a 20% en peso.

- Los sólidos se clasifican desde 200 micrones a 1/4 pulgada (6.4 mm)
- La gravedad específica de la mezcla es de 1.15



- El porcentaje de sólidos es del 5% a del 20% en peso.

**c) Mezcla Pesada:**

Son lodos pesados que están diseñados para transportar el material de un lugar a otro y son los casos más reales en donde el fluido tiene una consistencia pastosa con partículas no homogéneas. Muy a menudo el fluido portador en una mezcla pesada es muy necesario para ayudar a transportar el material deseado.

- El tamaño de los sólidos es mayor que 1/4 pulgada (6.4 mm).
- El de la gravedad específica de la mezcla es de 1.15.
- El porcentaje de los sólidos mayor del 20% en peso.

**3.6.2 Parámetros físicos de la pulpa minera que influyen sobre los revestimientos de caucho.**

La pulpa minera genera efectos que depende de las características y propiedades físicas que presenta el fluido de trabajo:

**a) Del solido a transportar.**

- Granulometría.
- Densidad.
- Forma.
- Dureza.

**b) Del fluido como medio de transporte.**

- Densidad.
- Viscosidad.

**c) De la instalación.**

- Diámetro interno de la tubería.
- Longitud.
- Desnivel.
- Rugosidad interna.
- Ángulos de inclinación de la tubería.
- Singularidades (estrechamiento, codos, etc.).

**d) Del régimen sistema.**

- Tonelaje de sólidos a transportar.
- Velocidad de flujo
- Pérdida de carga.

**e) De la mezcla.**

- Concentración de sólidos en volumen y en peso
- Densidad de la mezcla.

### **3.7. Factores importantes que producen efecto en el transporte hidráulico de sólidos.**

En forma paralela al desarrollo teórico, se realizaron estudios experimentales que permitieron conocer las características de funcionamiento del transporte hidráulico de sólidos.

Debido a la carencia de una teoría bien desarrollada para el transporte hidráulico de sólidos, los primeros análisis experimentales, fundamentalmente para flujo en tuberías a presión, se caracterizaron por su aleatoriedad en la fijación de las variables de estudio.

Los estudios aludidos se centraron en el análisis de los tres parámetros más importantes del transporte hidráulico de sólidos desde el punto de vista industrial.

#### **3.7.1 Velocidad límite de depósito.**

Como su nombre lo indica, la velocidad límite es la mínima velocidad de flujo para que no exista riesgo de depósito y obstrucción en la tubería.

La definición más usada y de fácil determinación experimental es aquella que identifica como la velocidad a la cual los sólidos gruesos permanecen detenidos por periodos importantes en el fondo de la tubería (formación de dunas móviles y/o lecho fijo de fondo).

La velocidad límite en transporte hidráulico de sólidos depende fundamentalmente de las siguientes variables.

- Granulometría de las partículas sólidas.

- Densidad relativa de las partículas sólidas.
- Diámetro de la tubería.
- Concentración de sólidos en la mezcla.
- Inclinación de la tubería o pendiente del canal.

En menor grado, la velocidad límite también depende de:

- Factor de forma de las partículas sólidas.
- Temperatura de la mezcla.

### **3.7.2 Pérdida de carga en mezclas sólido-líquido.**

La resistencia al flujo en una mezcla sólido – líquido, que fluye por una tubería puede ser considerable mayor que la resistencia en el caso de un líquido puro.

### **3.7.3 Tasa de Desgaste.**

El desgaste que sufren inevitablemente las instalaciones de transporte hidráulico de sólidos tiene dos causas principales.

La abrasión mecánica de las tuberías, tiene su origen en la formación de tensiones locales altas en la pared, causadas por el incesante impacto sobre esta por parte de las partículas que contienen gran energía cinética, la repetición de estas tensiones fatigan el metal u otro material de uso como protección erosionando la superficie.

Las variables que influyen en la abrasividad de un flujo sólido líquido son múltiples: tamaño, dureza, densidad y forma de las partículas, concentración de sólidos, velocidad y características geométricas y mecánicas de las líneas. De todas estas variables. Las

más importantes de controlar para un sistema dado son: la **velocidad media de la mezcla y los cambios bruscos en la dirección del flujo.**

Se ha demostrado que en la generalidad de los casos la tasa de abrasión depende de la velocidad en la siguiente razón (según ecuación 1).

$$\text{Tasa abrasión} = V^{2.0 \rightarrow 3.0} \dots(\text{ecuación 1})$$

La abrasión local por los cambios de dirección puede controlarse diseñando las curvas con radio amplio (superiores a 50 diámetros ó instalando protecciones antiabrasivas en codos y curvas

Aunque las características de abrasividad de una pulpa dada deben ser obtenidas desde pruebas en planta piloto, a veces es posible extrapolar condiciones de abrasividad para la distinta granulometría del material.

### **3.8. Propiedades de los tipos de caucho según sus componentes.**

Los elastómeros son macromoléculas que parten de un determinado monómero principal acompañado de otros compuestos los cuales favorecen a que el compuesto premezclado obtenga propiedades definidas después de la vulcanización.

#### **3.8.1 Elastómeros o Cauchos.**

Los elastómeros se caracterizan por su gran elasticidad, capacidad de estiramiento y rebote, recuperando su forma primitiva una vez que se retira la fuerza que los deformaba.

Sustancia elástica, impermeable, resistente a la abrasión y a la corriente eléctrica. Se obtiene del látex de diversas plantas y especialmente de la *Hevea brasiliensis*, de la familia euforbiáceas u obtenida también por métodos sintéticos.

### **3.8.2 Caucho natural.**

El **Caucho natural** procede del látex, el líquido lechoso que se extrae de diversas plantas tropicales. El **Caucho natural** vulcanizado es el producto de base y complemento indispensable para conseguir cauchos de síntesis de gran valor. Se distingue por su enorme elasticidad, la resistencia al estiramiento y su capacidad para conservar la flexibilidad incluso en condiciones de frío extremo.

### **3.8.3 Caucho sintético.**

El **Caucho sintético** es menos extensible y resistente que el **Caucho natural**, aventaja a éste con su mayor resistencia a los solventes orgánicos, aceites, petróleos y sus derivados; su menor oxidación y envejecimiento originado por el calor o los productos oxidantes y su menor permeabilidad a los gases.

### **3.8.4 Cauchos Base.**

Los cauchos bases más conocidos en la industria por su uso son:

#### **a) Poliisopreno Natural (NR) y Sintético (IR).**

Los Poliisoprenos, tanto el natural como el sintético, se caracterizan por una resiliencia excepcional, una buena

resistencia al desgarro, a la abrasión, a la fatiga por flexión y una excelente elasticidad. Poseen asimismo excelentes características de resistencia a la tracción y pueden operar a bajas temperaturas ( $-54^{\circ}\text{C}$ ). No es recomendable su uso para altas temperaturas, ozono, luz solar, petróleo e hidrocarburos. El caucho natural, en comparación con los sintéticos, posee propiedades ligeramente mejores en resistencia a la tracción, resistencia al desgarro, compresión, flexión y resistencia a la fatiga.

**b) Butadieno – Estireno (SBR).**

El SBR es un elastómero de uso general de bajo coste económico. Conocido como Buna-S, fue desarrollado originalmente para sustituir al caucho natural en la fabricación de neumáticos. El SBR posee muy buena resistencia a la fatiga y es resistente a muchos productos químicos de tipo polar, tales como alcoholes y cetonas. Asimismo, se considera apto para su uso en contacto con líquido de frenos para automóviles; sin embargo, no es resistente a los fluidos a base de petróleo.

**c) Polibutadieno (BR).**

El Polibutadieno posee excelente flexibilidad a bajas temperaturas ( $-62^{\circ}\text{C}$ ) y una excepcionalmente elevada resiliencia. Resistencia a la abrasión y a la flexión son también características sobresalientes de este elastómero. No se recomienda su uso con aceites, gasolinas y disolventes a base de hidrocarburos.

**d) Poliisobutileno (IIR)/BUTYL.**

Es conocido por su excelente resistencia al agua, vapor, álcalis y disolventes oxigenados. Otra de su característica más sobresaliente es su baja permeabilidad a los gases. El butilo es capaz de proporcionar una alta absorción de energía (amortiguación) y una buena resistencia al desgarro. Su buena resistencia al calor, la abrasión, el oxígeno, el ozono y la luz solar dependen del nivel de saturación del polímero.

Muy buena resistencia a los ácidos fluidos y a los detergentes, así como los ácidos fuertes. El Butilo sin embargo, muestra una pobre resistencia a aceites a base de petróleo, gasolinas y disolventes a base de hidrocarburos.

**e) Etileno - Propileno (EPDM)/BUNA-EP.**

Los elastómeros de EPDM poseen excelente resistencia al calor, al agua, al vapor, al ozono y a los rayos UV (estabilidad de color) a la vez que tienen muy buenas propiedades de flexibilidad a bajas temperaturas. Soportan los efectos de líquidos de frenos, medios alcalinos, ambientes levemente ácidos y disolventes oxigenados. Poseen un inmejorable comportamiento frente al envejecimiento en su uso a la intemperie a largo plazo. Los elastómeros de EPDM son asimismo muy adecuados para su uso con agua caliente y vapor. Son especialmente adecuados para trabajar con líquidos de freno a altas temperaturas. Los compuestos de caucho EPDM, no se recomienda su uso con gasolinas, aceites y grasas a base de petróleo y con disolventes a base de hidrocarburos.



**f) Polietileno Clorosulfonado (CSM) HYPALON.**

Los compuestos de Hypalon, proporcionan una excelente resistencia al ozono, a la oxidación, a la luz solar (degradación de color) y a la intemperie. Poseen asimismo una excelente resistencia a los ácidos y los álcalis y una buena resistencia a gran variedad de productos químicos con excepción de combustibles y disolventes. Poseen una muy buena resistencia mecánica.

**g) Policloropreno (CR)/ Neopreno.**

Las características físicas generales del neopreno lo sitúan como un elastómero de uso de amplio espectro. Sus excelentes características de envejecimiento frente al ozono y los agentes atmosféricos a la vez que su buena resistencia a la abrasión y a la flexión, le otorgan la categoría de caucho de uso general. El neopreno es resistente a los ácidos y álcalis, retardador de llama y adecuado para su uso con aceites con base de petróleo. Las grasas animales y vegetales también proporcionan un entorno muy estable para este polímero. Se caracteriza por una buena resistencia a la flexión, excelente resistencia a la fatiga y amplia resistencia a la intemperie y el ozono. Su excelente adherencia a los metales lo hace ideal para el moldeo con insertos metálicos. El Neopreno no es eficaz en contacto con disolventes aromáticos y oxigenados.

**h) Butadieno Acrilo Nitrilo (NBR).**

El NBR, conocido también como Buna-N y Nitrilo es un elastómero basado en un copolímero de acrilonitrilo butadieno. El NBR es inherentemente resistente a los fluidos hidráulicos, aceites lubricantes, fluidos de transmisión y otros productos a base de petróleo no polar, debido a la estructura polar de este elastómero. Los nitrilos también son resistentes a los agentes atmosféricos y al agua. Con la utilización de la variedad de polímeros de nitrilo y de otros ingredientes en su composición, se pueden obtener compuestos de caucho nitrilo para resistir entornos que requieren baja compresión, resistencia a la abrasión, baja temperatura de flexión, resistencia a la penetración del gas y resistencia al ozono. Por hidrogenación (HNBR), adición de ácidos carboxílicos (XNBR) o mezcla de PVC (NBR/PVC), los polímeros de nitrilo pueden satisfacer una gama más amplia de necesidades físicas o químicas.

**3.8.5 Componentes para la aceleración del caucho.**

Los principales insumos que componen al caucho son:

**a) Masterbach.**

Un producto sólido (normalmente de plástico, hule o elastómero) en la que los pigmentos o aditivos son dispersados de manera óptima en una concentración elevada contenido en un material de soporte. El material de soporte es compatible con el caucho principal en los que se mezclan durante el moldeo, mediante el cual el producto

elastomérico final obtiene el color o las propiedades del masterbatch.

**b) Mezcla Madre:**

Es un tipo de insumo que se fabrica con el propósito de obtener un menor tiempo de molienda en la aceleración del caucho, y por ello al acelerar con masterbatch se evitará de estar adicionando los insumos uno por uno. Un material premezclado es estable químicamente y este pueda agilizar el mezclado final del caucho acelerado.

**c) Aceleradores de la vulcanización:**

Son sustancias que añadidas en cantidades pequeñas a la mezcla de caucho aumentan considerablemente la rapidez de vulcanización a la vez que mejoran notablemente la calidad del producto y disminuyen la cantidad de azufre empleada.

**d) Activadores y retardantes:**

Un retardante es una sustancia que prolonga el comienzo de la vulcanización, no afecta el curso subsiguiente de la vulcanización y ayuda a que los acelerantes ejerzan por completo su efecto. Un retardante verdaderamente eficaz debe aumentar el tiempo requerido para el comienzo de la vulcanización (a cualquier temperatura), pero no debe retardar la subsiguiente velocidad de vulcanización.

**e) Agentes vulcanizadores:**

Un agente vulcanizador es aquel que efectúa la vulcanización después de ser expuesto a temperatura conveniente. Este agente vulcanizador debe ser soluble en el caucho o estar dividido en partículas finas para que pueda dispersarse con facilidad y uniformidad en el caucho.

**f) Antioxidantes:**

Son sustancias que retardan el deterioro del caucho natural, ya sea bruto o vulcanizado, causado por la oxidación también se conocen con el nombre de antioxidantes.

**g) Cargas:**

Existen dos tipos de cargas las reforzantes y las diluyentes. Las cargas reforzantes se emplean por razones preferentemente técnicas (aunque con frecuencia también den lugar a un menor costo del material), para aumentar la resistencia mecánica del vulcanizado, en especial su resistencia a la abrasión y al desgarro, y en muchas ocasiones también la resistencia a la tracción. Por el contrario las cargas diluyentes se usan por razones puramente económicas para abaratar el material, pero a costa de un empeoramiento de las características mecánicas.

Una carga que le da gran particularidad de dureza al caucho es el negro de humo. Es la carga por excelencia en la industria del caucho, consta de finísimas partículas de carbón, obtenidas por combustión parcial de gas natural o

aumenta el poder reforzante de la mezcla y por consiguiente se pueden obtener vulcanizados con mayor resistencia a la tracción, al desgarro y a la abrasión. También aumentan la dureza y la rigidez de las gomas, y disminuye el alargamiento en la rotura.

#### **h) Plastificantes.**

Son sustancias que aceleran la reducción de la viscosidad del caucho durante la masticación. Se usan principalmente con el caucho natural, el cual en estado bruto es demasiado viscoso para su empleo inmediato y requiere el reblandecimiento previo.

### **3.9. Características relevantes de los cauchos de acuerdo a su resistencia.**

Los agentes más comunes que afectan a las superficies expuestas de los cauchos ya sea en su operación ó almacenamiento son:

#### **3.9.1 Envejecimiento.**

Se dice que el caucho vulcanizado experimenta envejecimiento cuando está expuesto a la luz del sol en una atmósfera de hidrógeno, dióxido de carbono, aire, oxígeno, y en el vacío.

#### **3.9.2 Resistencia a Hidrocarburos.**

Son propiedades innatas de los cauchos sintéticos más resistentes a los hidrocarburos, tanto alifáticos como aromáticos.

### **3.9.3 Temperatura de Servicio.**

Es la temperatura de trabajo a la cual el revestimiento de caucho es sometido en operación normal.

### **3.9.4 Adherencia al metal.**

Es la unión que existe entre el caucho y el metal después de la vulcanización, los diferentes tipos de caucho tienen mayor ó menor anclaje al metal.

### **3.9.5 Ozono.**

El enemigo mayor del caucho es el ozono. Las parafinas añadidas al caucho evitan el agrietamiento debido a la acción del ozono sobre el caucho estirado.

### **3.9.6 Permeabilidad.**

Las diferencias en la permeabilidad de los elastómeros de diferentes gases son causadas principalmente por diferencias en la velocidad de difusión y sólo en un grado muy menor a las diferencias en la solubilidad.

### **3.9.7 Resistencia a la Llama.**

Es la capacidad que tiene el elastómero para soportar temperaturas altas con incidencia directa a una llama.

### **3.9.8 Propiedades Eléctricas.**

Algunos cauchos son eléctricamente continuos, semicontinuos ó nulos según los materiales que este tenga en su composición. En la industria del caucho se utilizan básicamente dos tipos de caucho: el natural y el sintético. Este último, obtenido a través de diferentes polímeros, sirve para la fabricación de una gran variedad de productos (según la tabla N° 3.1.).

Tabla N° 3.1. Propiedades de los tipos de caucho.

Tipo de caucho Elastómero	Producción (en miles de toneladas en 2000)		Propiedades	Usos Comunes
	Origen	Cantidad		
Caucho Natural	Tailandia	1,501	Usos generales; no resisten al aceite, se hincha con los disolventes; no resisten al oxígeno, ozono y luz UV	Neumáticos, soportes elásticos, burlletes, acoplamientos soportes de puentes y para la construcción, calzado, mangueras, correas transportadoras, productos moldeados, revestimientos, rodillos, guantes, preservativos, dispositivos sanitarios, pegamentos, tejidos de fondo para alfombras, hilos, espuma.
	Indonesia	1,353		
	Malasia	923		
	India	426		
Polisopreno(IR)	EE.UU.	47	Usos generales; no resisten al aceite, se hincha con los disolventes; no resisten al oxígeno, ozono y luz UV	Véase Caucho natural (párrafo anterior).
	Europa occidental	15		
	Japón	52		
EstirenoButadieno(SBR)	EE.UU.	920	Usos generales; sustituyó al caucho natural durante la segunda guerra mundial; baja resistencia al aceite y disolventes	Neumáticos(75%), correas transportadoras, esponjos productos moldeados, calzado, mangueras, recubrimientos de rodillos, pegamentos, productos impermeables, forros de alfombra de latex, productos de espuma.
	Europa occidental	1,117		
	Japón	620		
Polibutadieno(BR)	EE.UU.	465	Baja resistencia al aceite y disolventes; no resistente a la intemperie, alta resiliencia resistencia a la abrasión y flexibilidad a baja temperatura.	Neumáticos, calzado, correas transportadoras, correas de transmisión, pelotas de juguete.
	Europa occidental	297		
	Japón	215		
	Europa oriental	62(1996)		
Butilo(IIR)	EE.UU.	130	Baja permeabilidad al gas; resistente a calor acidos y líquidos polares; no resiste al aceite y disolventes; excelentes propiedades eléctricas	Interior de tubos, cámaras de vulcanización de neumáticos, calefateo y selladores, aislamiento de cables, aisladores vibracionales, revestimiento protector de estanques y membranas para tejados, correas transportadoras y mangueras para alta temperatura.
	Europa occidental	168		
	Europa oriental	90		
	Japón	83		
Etilenpropileno / Etilenpropilendieno	EE.UU.	261	Flexibilidad a baja temperatura, resistente a la intemperie y al calor pero no al aceite, o a los disolventes; excelentes propiedades eléctricas.	Recubrimientos de cables; desfibradores y burlletes extruidos; productos moldeados, juntas aislantes; recubrimientos para silos, tejados, estanques, zanjas y vertederos controlados.
	Europa occidental	201		
	Japón	124		
Policloropreno(CR) (Neopreno)	EE.UU.	105	Resistente al aceite, llamas, calor e intemperie	Recubrimientos de cables, mangueras, correas transportadoras, calzado, ropa impermeable, tejidos recubiertos y productos inflable, extruidos pegamentos, soportes de puente y rail, revestimiento, juntas de esponja, productos de espuma de latex.
	Europa occidental	102		
	Japón	74		
Nitrilo(NBR)	EE.UU.	64	Resistente al aceite, disolventes y aceite vegetal; se hincha con disolventes polares como cetonas	Sustancias taponadoras, recubrimientos y juntas para mangueras resistentes a combustibles, camisas de rodillo correas transportadoras suela de zapato, guantes, pegamentos, equipos de perforación para pozos petrolíferos
	Europa occidental	108		
	Japón	70		
	Europa oriental	30		
Silicona(MQ)	EE.UU.	95	Estable a temperatura altas y bajas; resistentes al aceite, disolventes e intemperie fisiología y químicamente está inerta.	Aislamiento de cables, burlletes, pegamentos, juntos productos productos moldeados y extruidos, mascarillas de gas y respiradores, tubos para alimentación e implantes quirúrgicos.
	Europa occidental	107		
	Japón	59(1990)		
Polisulfuros(OT)	EE.UU.	20	Resistente al aceite, disolventes, bajas temperaturas e intemperie; baja permeabilidad del gas	Camisas de rodillos, revestimientos de mangueras juntas, productos moldeados, taponadores, diafragmas para gasómetros, selladores de vidrio, aglomerante, sólido de combustible para cohetes
	Europa occidental	0		
	Japón	3		
Caucho Reciclado	-	-	cadena polímeros mas cortos; mayor facilidad de procesamiento; menor tiempo de mezclado y menor consumo de energía menor resistencia a la tracción y menor coste.	Neumáticos, interior de tubos, felpudos, productos mecánicos, pegamentos, asfalto couchutado



## **IV DESARROLLO DEL TEMA**

Los ensayos realizados al caucho se realizan con probetas crudas aceleradas y probetas vulcanizadas ó curadas así podemos tener un análisis más completo del material que se va emplear en los revestimientos.

### **4.1 Pruebas y ensayos antes del vulcanizado del caucho.**

Estas pruebas se caracterizan debido a que el caucho para el análisis se encuentra en estado crudo sin vulcanizar en donde se somete a pruebas que definen su estructura interna y el comportamiento al cambio de estado molecular aleatorio a un estado molecular ordenado, Este estado molecular aleatorio debe ser homogéneo en todo el material producido y así asegurar que toda la masa llegará a comportarse de manera similar en cualquier punto ó zona.

#### **4.1.1 Medición de la densidad relativa**

La densidad está relacionada con el grado de acumulación de materia (un cuerpo compacto es, por lo general, más denso que otro más disperso), pero también lo está con el peso. Así, un cuerpo pequeño que es mucho más pesado que otro más grande es también mucho más denso.

La densidad relativa es el cociente entre las densidades de una determinada sustancia ó compuesto con respecto a otra sustancia o compuesto que se toma como referencia o patrón.

Para esto se usa un densímetro que tiene el principio de Arquímedes. La prueba consiste en obtener dos muestras de dimensiones 15mm x 15 mm x 8mm aproximadamente del material que se desea analizar,

luego realizamos un peso seco en la plataforma del densímetro, después el peso de la misma muestra sumergida en el recipiente con agua, esta muestra se colocará sobre la plataforma que se encuentra sumergida en el recipiente con agua (ver figura N° 4.1.), finalizando el equipo nos devuelve la densidad relativa respecto al fluido (Agua).



Figura N° 4.1. Densímetro Electrónico.

Se hace una muestra por duplicado, para asegurar que el valor conseguido sea repetitivo y así se pueda decir que la densidad obtenida es representativa.

**a) Norma de ensayo:**

**UNE 53526:2001 - Método A. Determinación de la densidad. UNE 53526:1970**

**b) Procedimiento de ensayo.**

- i. Se extrae una pequeña muestra de caucho crudo acelerado, vulcanizado ó masterbach de una probeta previamente muestreada (Ver figuras N° 4.2. y N° 4.3.), esta se enjuaga y se seca.



Figura N° 4.2. Muestra de caucho 228S acelerado.



Figura N° 4.3. Masterbach de goma 228S.

- ii. Se coloca en la plataforma superior la muestra para el peso en seco y se guarda en la memoria del instrumento.
- iii. Luego se sumerge la muestra y se coloca en la plataforma que se encuentra inmersa en el agua, se procede a grabar el resultado y automáticamente el instrumento devuelve la densidad relativa.

- iv. Se llena el registro de control, Fecha, temperatura de medición, densidad relativa, responsable de ensayo según el formato (Anexo 2 y 3).

#### **4.1.2 Ensayo de reometría del caucho crudo acelerado.**

Es el comportamiento reológico de un compuesto durante la vulcanización, cuando una muestra de caucho se mantiene en una cámara regulada a temperatura y presión constante, esta somete a esfuerzos de cizallamiento alternantes de poca amplitud según un ciclo sinusoidal; se determina la resistencia ofrecida por tales esfuerzos (torques) y su variación con respecto al tiempo del ensayo. El resultado de todas estas variaciones se obtiene en un reograma el cual muestra el comportamiento del material.

El ensayo reométrico se basa en someter una muestra colocada en dos elementos mecánicos, que consisten en dos platos paralelos cilíndricos coaxiales. En esta operación el caucho experimenta una deformación de cizalla en forma de torsión, oscilando por medio de un disco móvil giratorio a  $\pm 1^\circ$  contra un cavidad existente en el plato superior. Esta deformación puede ser impuesta a velocidad de deformación constante, tensión constante o incluso de manera oscilatoria. El disco del reómetro que se encuentra a una temperatura de  $190^\circ\text{C}$ , tienen cavidades circulares con pequeñas ranuras los cuales ayudaran a que el caucho tenga adherencia al disco y esta pueda experimentar un torque, estos valores serán detectados por pequeñas variaciones de voltaje que se producen en el interior del reómetro y estas serán traducidas en señales digitales para ser graficadas sobre el reograma.

**a) Norma de ensayo:**

**ASTM D 2084 Propiedades del caucho – Vulcanización con uso de disco medidor de curado oscilante.**

**b) Consideraciones del espécimen de ensayo:**

Un espécimen circular se toma de una muestra, debe tener un diámetro de 30 +/- 2 mm (1.2 +/- 0.1 in) y un espesor de 11.5 +/- 1.5 mm (0.45 +/- 0.05 in) ó un equivalente de 9 cm<sup>3</sup> (0.5 in<sup>3</sup>).

El espécimen de la prueba se considera de un tamaño apropiado, cuando un pequeño trozo del compuesto se saca uniformemente alrededor de la periferia del dado mientras que este está cerrado.

Se alcanza esto cuando el volumen del espécimen está entre 8 y 11 cm<sup>3</sup> (9 a 13g del compuesto de caucho con una gravedad específica de 1.15). Los especímenes de tamaño insuficiente pueden causar la presión baja de la cavidad y lecturas bajas del esfuerzo de torsión. Los especímenes de gran tamaño refrescan los dados excesivamente durante la parte anterior del ciclo de la prueba que afecta a las características de la vulcanización.

**c) Equipo: Reómetro Monsanto 100S.**

El sistema estándar de platos y disco rotor para el uso del Reómetro Monsanto 100S es el MPC (Control de Micro Producción). Como especificación por ASTM D 2084 – 79,

BS1673 Pt 10 e ISO 3417 1977, el pequeño volumen de muestra empleado hace los datos del M.P.C. altamente convenientes para los usos de prueba múltiples y rápidos, tales como: control de producción, este sistema de platos es igualmente aplicable a todos los tipos de prueba de investigación y desarrollo. Los M.P.C., los sistemas de platos utilizan un sello superficial de teflón alrededor del eje del rotor.(Ver figura 14)



Figura N° 4.4. Reómetro de platos Monsanto 100S

**d) Procedimiento del ensayo:**

1. Se hace una limpieza respectiva del plato, cavidades y disco rotor.
- ii. Se verifica el torque cero menor o igual que 5dN.m

- iii. Se toma una muestra de  $\frac{1}{4}$ "x1"x1", de caucho crudo aproximadamente.
  - iv. Luego se ingresan los datos para identificar la muestra a analizar en el software, como Tipo de goma, Orden de fabricación, N° Batch tipo de análisis.
  - v. Una vez que está identificada la muestra y elegido el ensayo se coge la muestra y se coloca en el rotor e iniciamos la prueba accionando la entrada de aire para cerrar los platos.
  - vi. Al cerrar los platos se activa el sensor de medición y comienza a graficarse el reograma del caucho por un espacio de 4 minutos.
  - vii. Se acciona la apertura de platos y se extrae el material vulcanizado.
  - viii. Se extrae los datos y se reporta en el formato de parámetros de reometría, datos como: fecha, torque máximo, torque mínimo, tiempo de torque máximo, tiempo de torque mínimo, etc. (Anexo 3).
- e) **Explicación de la grafica reométrica del ensayo.**

**Tramo 1:** El caucho presenta un torque ascendente que es debido al caucho crudo que está originando un esfuerzo de oposición, el cual se encuentra en un estado sólido. Esto se manifiesta con una gráfica de pendiente (0)  $\rightarrow$  (+).

**Tramo 2:** En esta zona el caucho crudo comienza a derretirse ó fluidizarse por acción de la temperatura y la viscosidad empieza a disminuir. En la gráfica esto se manifiesta por el cambio en la pendiente de (+)  $\rightarrow$  (0)

**Tramo 3:** El caucho fluidizado empieza a experimentar un reordenamiento molecular el cual se manifiesta por un incremento del torque en la grafica a medida que este se vulcaniza y aumenta hasta un cierto límite dependiendo de la naturaleza del caucho. El cambio en la pendiente es de (0)  $\rightarrow$  (+)

**Tramo 4:** El Caucho adquiere su máximo torque y luego de esto la temperatura comienza a actuar sobre el caucho vulcanizado de forma desfavorable (degradación del caucho) y se manifiesta en la gráfica con un decrecimiento en la gráfica con pendiente (-).

Todas estas características de la curva reométrica se pueden notar en el siguiente reograma (Ver Figura N° 4.5.).

## **4.2 Pruebas y ensayos después del vulcanizado del caucho.**

### **4.2.1 Ensayo de Abrasión Taber.**

La abrasión es el efecto de desgaste por causa del fraccionamiento de partículas que se encuentran colisionando sobre la superficie del material afectado. El desgaste es más eficiente cuando hay una gran diferencia de dureza entre abrasivo y sustrato.



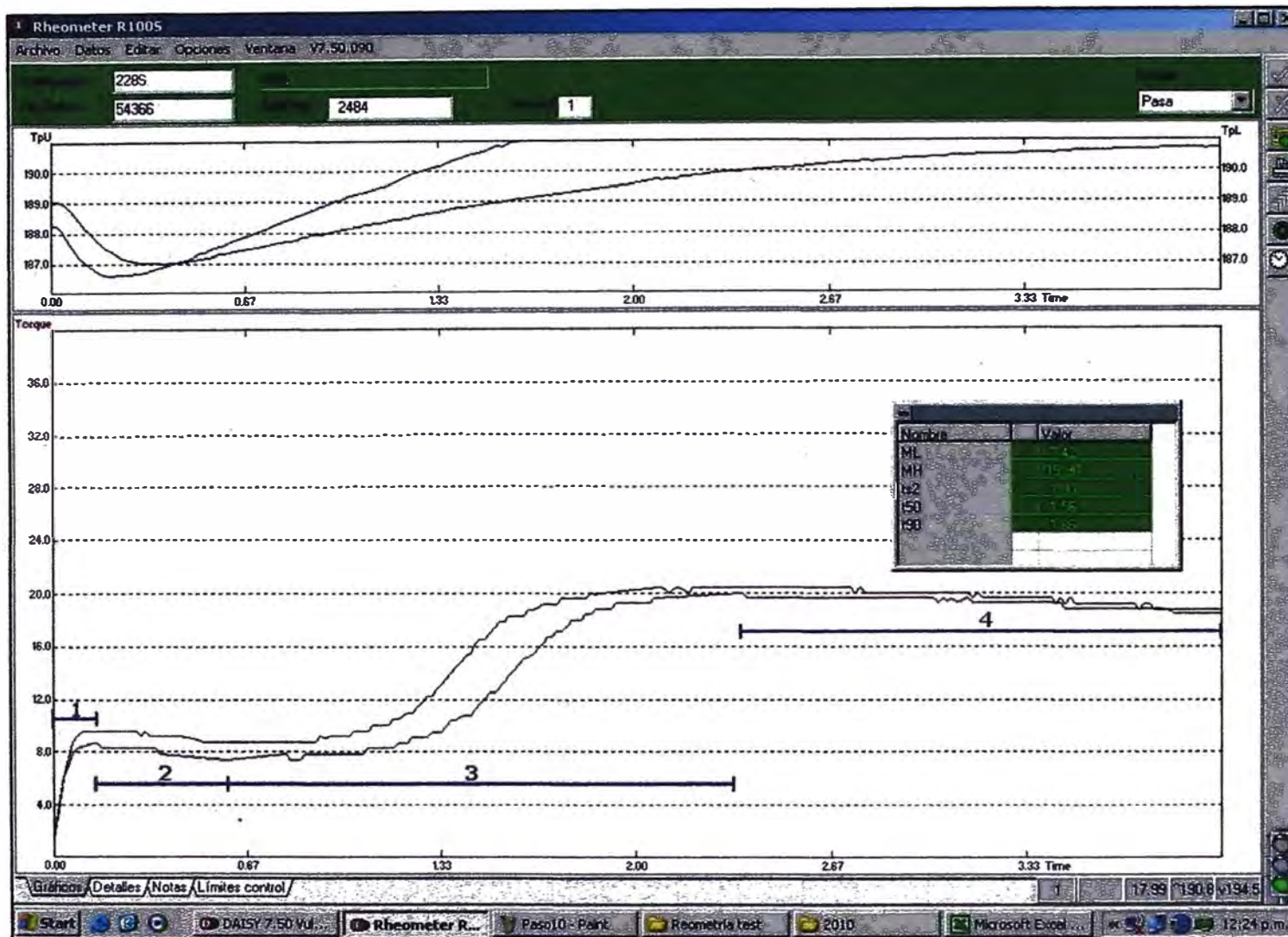


Figura N° 4.5. Reograma de la vulcanización del caucho 228S caucho base Natural.

a) **Tamaño de partículas abrasivas:**

Expresado en micrómetros. Se dividen en:

- Finas (0 - 10  $\mu\text{m}$ ).
- Medias (10 - 100  $\mu\text{m}$ ).
- Gruesas (100 - 500  $\mu\text{m}$ ).

b) **Forma de la partícula del abrasivo:**

Las partículas irregulares abrasionan con mayor rapidez que aquellas redondeadas, pero también producirán rayas más profundas.

La velocidad de abrasión baja por el redondeamiento de las partículas y por contaminación con partículas del sustrato (ver Figura N° 4.6.).

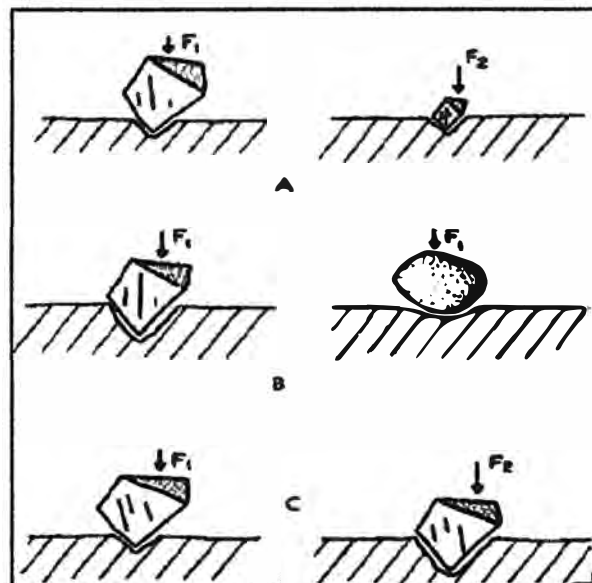


Figura N° 4.6. Forma de las partículas abrasivas.

**c) Factores que modifican la velocidad de abrasión:**

Los factores que modifican la velocidad de abrasión son:

- **Velocidad de los sólidos.**
- **Presión.**

A mayor velocidad, mayor desgaste y mayor producción de calor. A mayor presión se produce el mismo fenómeno.

Los discos de los especímenes de la prueba se hacen girar en una plataforma circular giratoria y son desgastados por un par de muelas de desbastar para un número especificado de ciclos bajo carga especificada. El método de la prueba especifica que el cambio en el peso del espécimen de la prueba esté determinado como medida de resistencia de abrasión. Es más común, sin embargo, ver la resistencia de abrasión divulgada como el cambio en la masa del espécimen de la prueba o el cambiar en masa por el número de ciclos. El cambio total es debido a la pérdida material de la abrasión.

**d) Norma de ensayo:**

**ASTM D 3389 -75 Resistencia a la abrasión (Plataforma rotatoria, Doble cabeza abrasiva).**

**e) Equipo:**

Consta de un plano circular para colocar el espécimen, un par de brazos movibles cada uno de estos contiene discos

abrasivos, motor para la rotación de la plataforma donde se colocará el espécimen, un ventilador para el enfriamiento del motor y un contador indicador de revoluciones (ver Figura N° 4.7.).



Figura N° 4.7. Abrasímetro Taber de doble rueda

1. El espécimen deberá ser apoyado por un adaptador, el cual está impulsado por el motor quien le proporciona movimiento para el circuito cíclico del espécimen.
- ii. El anillo de fijación de abrazadera deberá ser usado para asegurar que se sostiene al espécimen.
- iii. Los discos abrasivos deberán ser tipo S-35 rueda de acero y estarán colocados independientemente sobre los brazos giratorios libre equilibrio en la acción compensatoria por alguna desigualdad en el espécimen y asegura una presión uniforme de los discos de abrasión contra el espécimen en todo el tiempo mediante unas pesas.

- iv. El aparato debe estar provisto de una fuerza vertical ajustable (Pesos), para variar la fuerza vertical de los discos abrasivos sobre el espécimen.
- v. Los brazos de la rueda de desgaste sin masas o contrapesos auxiliares, aplican una fuerza vertical contra el espécimen 2.45N (2.45 gf) por disco. En un comienzo en el extremo posterior del brazo de abrasión se puede utilizar un contrapeso cuando se desea, para reducir la carga de la rueda de 2.45N (250 gf) a 1.23N (125gf) cuando los materiales son delicados a la prueba.

**f) Equipo auxiliar:**

Este equipo es considerado como un cepillo duro que sirve para remover las partículas sueltas de las superficies de los discos un pequeño aspirador para remover las partículas sueltas del espécimen durante el ensayo. Aire comprimido el cual deberá estar libre de humedad y aceite, deberá ser usado para limpiar la superficie del espécimen, El aire es enviado hacia una tobera donde la presión deberá ser mantenida en 200 +/- 35kPa (30 +/- 5psi). El aspirador de aire deberá estar funcionando durante toda la prueba.

**g) Posición de la plataforma:**

La distancia vertical del centro de punto de giro del brazo abrasivo hacia el punto esperado del espécimen deberá ser 25mm, este promedio deberá ser especificado para prevenir la posibilidad de errores incurridos por la instalación del

rodamiento de empuje como apoyarse en la plataforma del espécimen.

**h) Velocidad de la plataforma**

La velocidad de giro de la plataforma deberá de ser  $7.0 \pm 0.11$  rad/s ( $70 \pm 1$  rpm).

**i) Especímen de ensayo**

1. Hacer cinco pruebas de muestra. A menos que se especifique el número de pruebas.
- ii. Corte los especímenes aproximadamente 110m m ( $4.1/2$  de la prueba circular adentro) en diámetro. corte un agujero de 6m m ( $1/4$  adentro) en el centro del espécimen. Utilice la mejor porción de la muestra que se probará. Debe estar libre de agujeros, de ampollas, o de otras imperfecciones.
- iii. Para los ensayos de especímenes condicionados a la atmósfera a una temperatura  $20 \pm 2.0$  °C y humedad relativa de 64 del  $\pm 2\%$ .

**j) Procedimiento de ensayo**

1. Instale las ruedas S-35 en sus respectivos sostenedores ensanchados según lo indicado por la impresión en el lado de la rueda.

11. Se necesita de una probeta circular de diámetro 100mm previamente curada.
111. La superficie debe estar completamente limpia o en su defecto debe de ser limpiada.
- 1v. Se hace la medición de la masa inicial de la probeta.
- v. Se coloca la probeta en la plataforma de giro del abrasímetro y este se asegura con sus respectivos elementos.
- vi. Se coloca las dos piedras y las respectivas pesas de 250g cada una.
- vii. Para el ensayo debe estar definido el número de ciclos a la cual se desea hacer el ensayo entre 500 a 1000 ciclos.
- viii. Se empieza la prueba y se coloca el peso seleccionado sobre la superficie en giro y se activa el aspirador para coleccionar los residuos.
- ix. Luego de completar todos los ciclos, este se saca, se somete a una limpieza y luego se coloca en una balanza para obtener el peso final.
- x. Se hace los cálculos para hallar el índice de abrasión (según ecuación 2) y se registra todos los datos como: fecha de análisis, Orden de fabricación, tipo de goma, N° de prueba, Fuerza normal aplicada, masa inicial y final, N° ciclos, índice de abrasión, tipo de rueda, etc.

En el registro de abrasión de caucho vulcanizado (Anexo 4).

**k) Cálculos**

$$MP = (MAE - MDE) / NR \dots \text{ecuación 2}$$

- MP: Masa perdida por revoluciones.
- MAE: Masa de la muestra antes del ensayo.
- MDE: Masa de la muestra después del ensayo.
- NR: Numero de revoluciones.

**4.2.2 Ensayo de Dureza.**

El propósito de este ensayo será el de poder medir la dureza basada en la penetración de un indentor de un tipo determinado sobre las muestras de caucho a analizar. Teóricamente, los resultados de la dureza de indentación deberán ser inversamente proporcionales a la hendidura realizada y directamente proporcional al módulo de elasticidad y a la viscosidad del material. Es importante mencionar que los resultados de este ensayo dependerán en gran medida de la geometría del indentor y la fuerza aplicada sobre el material objeto de análisis; sin embargo, no existe una relación entre los valores de dureza determinados por diferentes durómetros sobre un mismo material. Las unidades de dureza a utilizar en los ensayos será la de Shore A.

Para la medición de la dureza Shore se utilizan varias escalas: Shore A, B, C, D, 0 y 00. La escala Shore A es la más conveniente para medir la dureza de los cauchos blandos, hasta 90 grados, mientras que la escala Shore D se usa para la medición de dureza de cauchos



más duros que 90 grados Shore A. Aproximadamente 40 grados Shore D corresponden a 90 Shore A. En todas las escalas se fabrican durómetros digitales y análogos, así como portátiles y de mesa, siendo estos últimos más precisos. En los durómetros portátiles la medición depende de la influencia del operario, unida a la variabilidad de un instrumento a otro, aunque ambos se encuentren calibrados. En Shore A, por ejemplo, es necesario admitir una variación de +/-5 puntos (ver Tabla N° 4.2.).

**a) Norma de ensayo:**

**ASTM D 2240 - 02 Propiedad del caucho – medición de dureza con uso de durómetro.**

El método de ensayo es basado en la penetración de un tipo específico de indentor. El valor de la dureza del penetrador está inversamente relacionado con la penetración al material y es dependiente en el módulo de elástico y visco elástico del mismo (más detalles en Anexo 8).

**b) Condiciones del espécimen para el ensayo.**

El espécimen de la prueba, debe tener por lo menos 6.0 mm (0.24 pulg.) en espesor, a menos que se sepa que los resultados equivalentes a los valores de 6.0 milímetros (0.24 pulg.) están obtenidos con un espécimen más fino.

Para cualquier material cubierto, el pie del penetrador está en contacto con el espécimen, por ejemplo, el recorrido inicial del penetrador ha cesado, la lectura indicada será

registrada dentro de  $1 \pm 0.1s$ , o después de cualquier periodo de tiempo, convenido entre algunos laboratorios.

**c) Selección del durómetro.**

La guía de la selección del durómetro se diseña para asistir a la selección del tipo apropiado del durómetro para los varios usos.

Se reconoce generalmente que la determinación de la dureza con el durómetro debajo de 20 y sobre 90 es no fiable. Se recomienda que el tipo más bajo o más alto (referente a la escala), esté utilizado en estas situaciones. También se recomienda siempre que sea posible, un soporte del funcionamiento se emplee en la ejecución de la medición de la dureza. A continuación se muestra la Tabla N° 4.2. donde detalla los tipos de durómetros y sus rangos de empleo.

**d) Equipo.**

Tipos de pie del durómetro A, con un orificio (para permitir la protrusión del penetrador) de un diámetro tal como se especifica en la Figura. 20, con el centro de un mínimo de 6,0 mm (0.24in) de cualquier borde del pie, el pie del durómetro no es de un diseño plano circular (ver figura N° 4.8.).

Tabla N° 4.2. Selección de tipo de durómetro según el rango de dureza.

<b>Selección de durómetro: Usos típicos</b>		
<b>Tipo de Escala</b>	<b>Ejemplos típicos de los materiales a prueba</b>	<b>Dureza durómetro (Usos típicos)</b>
A	Caucho flexible vulcanizado, caucho natural, nitrilos, elastómeros termoplásticos, termoestables poliacrílicos flexible, cera, fieltro y cueros.	20-90A
B	Moderadamente materiales de goma dura, elastómeros termoplásticos, productos de papel y textiles.	Por encima de: 90 A Por debajo de: 20 D
C	Caucho de dureza media, elastómeros termoplásticos, plásticos de dureza media, y termoplásticos.	Por encima de: 90 B Por debajo de: 20 D
D	Caucho endurecido, elastómeros termoplásticos, más plásticos y termoplásticos rígidos.	Por encima de: 90 A
DO	Caucho endurecido moderadamente, elastómero termoplástico, y las bobinas textiles muy denso	Por encima de: 90 C Por debajo de: 20 D
M	Delgada forma irregular de caucho, elastómeros termoplásticos, así como muestras de plástico.	20 - 85 A
O	Caucho flexible, elastómeros termoplásticos, plásticos y termoplásticos muy suave, bobinados de densidad media textil.	Por debajo de: 20 DO
OO	Extremadamente goma suave, elastómeros termoplásticos, esponja, plásticos extremadamente suave y termoplásticos, espumas, devanados de baja densidad de materia textil, tejidos humanos y animales.	Por debajo de: 20 O
CF	Los materiales compuestos de espuma, como cojines de seguridad, asientos de vehículos, cuadros de mando, reposacabezas, apoyabrazos, y paneles de las puertas.	Método de ensayo Véase el F 1957

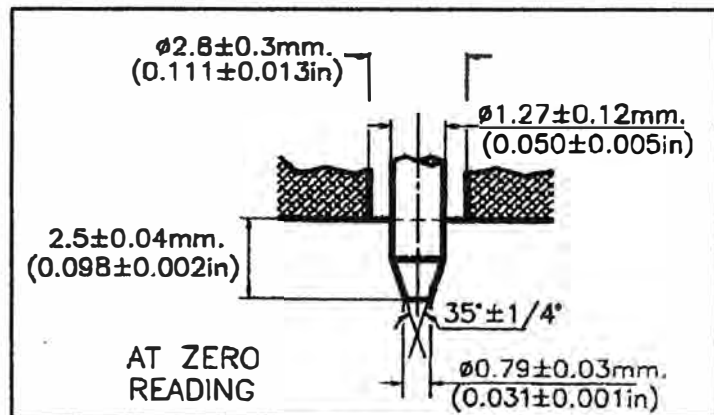


Figura N° 4.8. Tipo A y C de penetrador

e) **Procedimiento.**

1. Se dispone de una muestra de caucho crudo acelerado de cualquier tipo.
- ii. Luego se corta en medidas 50mm x 50 mm y se colocan en molde de prueba para su prensado en caliente 6 probetas por espacio de 15 min.
- iii. Verificar la lectura del durómetro Tipo A con probetas vulcanizadas estándares de dureza definida para asegurar la medición de esta.
- iv. Después del enfriamiento de las probetas vulcanizadas de prueba se dispone para la medición de dureza por medio del durómetro (ver figura N° 4.9).



Figura N° 4.9. Medición de dureza

- v. Esta medición se compara en la tabla de comparación de gomas por su dureza con una tolerancia de  $\pm 5$  Shore-A (ver Tabla N° 4.3.).
- vi. Estos Datos son registrados en el formato del Reometría y dureza del caucho (Anexo 3).

#### 4.2.3 Ensayo de Tracción y Alargamiento.

La resistencia a la tracción, que es la medida de la capacidad de un polímero a resistir los esfuerzos de estiramiento, normalmente se mide aplicando un esfuerzo a una probeta, tal como se muestra en la figura N° 4.10.

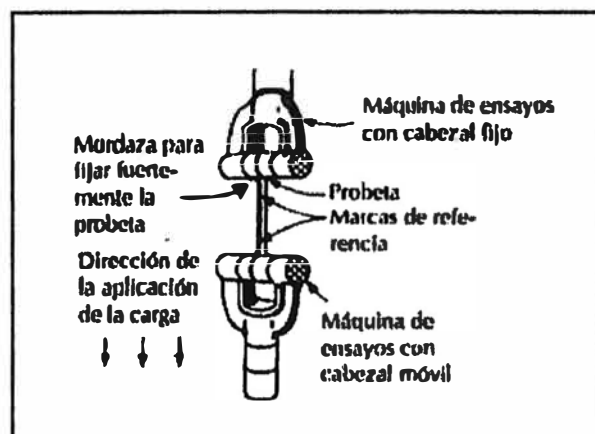


Figura N° 4.10. Forma de probeta y ensayo de tracción.

Tabla N° 4.3. Dureza Shore-A y Shore-D de algunos cauchos.

<b>Goma</b>	<b>Caucho base</b>	<b>Color</b>	<b>Dureza Shore (± 5) (A)</b>
<b>101</b>	<b>Natural</b>	<b>Rojo</b>	<b>40</b>
<b>121D</b>	<b>Natural-Estireno-B.</b>	<b>Negro</b>	<b>65</b>
<b>121M</b>	<b>Natural-Estireno-B.</b>	<b>Negro</b>	<b>65</b>
<b>121S</b>	<b>Natural-Estireno-B.</b>	<b>Negro</b>	<b>60</b>
<b>121SK</b>	<b>Natural</b>	<b>Negro</b>	<b>65</b>
<b>127</b>	<b>Estireno-ButadienoB</b>	<b>Negro</b>	<b>70</b>
<b>129</b>	<b>Estireno-Butadieno</b>	<b>Negro</b>	<b>55</b>
<b>130B</b>	<b>Natural-Neopreno</b>	<b>Negro</b>	<b>60</b>
<b>134</b>	<b>Natural</b>	<b>Negro</b>	<b>40</b>
<b>138F</b>	<b>Natural</b>	<b>Negro</b>	<b>60</b>
<b>177</b>	<b>Natural</b>	<b>Negro</b>	<b>65</b>
<b>200</b>	<b>Natural</b>	<b>Negro</b>	<b>45</b>
<b>222X</b>	<b>Natural</b>	<b>Negro</b>	<b>70 (D)</b>
<b>228A</b>	<b>Natural</b>	<b>Rojo</b>	<b>45</b>
<b>228C</b>	<b>Natural</b>	<b>Rojo</b>	<b>35</b>
<b>228E</b>	<b>Natural</b>	<b>Rojo</b>	<b>45</b>
<b>228S</b>	<b>Natural</b>	<b>Rojo</b>	<b>45</b>
<b>228SK</b>	<b>Natural</b>	<b>Rojo</b>	<b>45</b>
<b>228SS</b>	<b>Natural</b>	<b>Rojo</b>	<b>45</b>
<b>228SW</b>	<b>Natural</b>	<b>Negro</b>	<b>45 ± 5</b>
<b>249I</b>	<b>Estireno-Butadieno</b>	<b>Negro</b>	<b>60 (D)</b>
<b>26180</b>	<b>Acrilo-Nitrilo</b>	<b>Verde</b>	<b>80</b>
<b>284</b>	<b>Natural</b>	<b>Negro</b>	<b>55</b>
<b>285</b>	<b>Natural</b>	<b>Negro</b>	<b>70</b>
<b>291</b>	<b>Natural</b>	<b>Negro</b>	<b>60</b>
<b>545</b>	<b>Neopreno</b>	<b>Negro</b>	<b>55</b>
<b>562</b>	<b>Butilo</b>	<b>Negro</b>	<b>50</b>
<b>608</b>	<b>Neopreno</b>	<b>Negro</b>	<b>65</b>
<b>629B</b>	<b>Neopreno</b>	<b>Negro</b>	<b>60</b>
<b>667S</b>	<b>Neopreno</b>	<b>Negro</b>	<b>55</b>
<b>670</b>	<b>Neopreno</b>	<b>Negro</b>	<b>65</b>
<b>673S</b>	<b>Neopreno</b>	<b>Negro</b>	<b>60</b>
<b>690</b>	<b>Neopreno</b>	<b>Negro</b>	<b>65</b>
<b>924</b>	<b>Poliuretano</b>	<b>Azul</b>	<b>82</b>

Se define como la fuerza por unidad de área de la sección transversal necesaria para romper por estiramiento una probeta de características establecidas normativamente (ver ecuación 3), que corresponde al material de ensayo, las unidades pueden darse en: Kg/cm<sup>2</sup>, N/m<sup>2</sup>, etc.

$$R_t = (F_r / A_t) \dots (\text{ecuación 3}).$$

- **R<sub>t</sub>** : Resistencia a la tracción (Pascales).
- **F<sub>r</sub>** : Fuerza necesaria para romper la muestra (N).
- **A<sub>t</sub>** : Área de la sección transversal (m<sup>2</sup>)

**Un alargamiento o deformación recuperable** se llama deformación elástica. En este caso, las moléculas tensionadas vuelven a ocupar sus posiciones relativas originales después de la desaparición de la fuerza aplicada.

El alargamiento también puede ser consecuencia del movimiento global de unas cadenas respecto de otras.

El alargamiento se mide mediante la una relación la cual se muestra en la (ecuación 4). El porcentaje de alargamiento es igual a la variación dimensional dividida por la longitud original de la muestra y multiplicado por 100.

$$\%El = (\Delta l / l) \times 100 \dots \text{ecuación 4}$$

- **%El**: Porcentaje de alargamiento.
- **Δl**: Variación de Longitud
- **l**: Longitud inicial

**a) Norma de ensayo:**

**ASTM D 412 - 92 Tensión de Cauchos vulcanizados, cauchos termoplásticos y elastómeros termoplásticos. Method A: Especímenes moldeados con troqueladora**

**b) Equipo:**

La prueba de tensión será hecha en una máquina accionada por motor, equipada para producir un índice uniforme de separación del apretón de 500 +/- 50mm/min (20 +/- 2in/min) en una distancia de por lo menos 750mm (30pulg). La máquina de prueba tendrá un dinamómetro conveniente y un sistema de la indicación o de grabación para medir la fuerza aplicada dentro +/-2%. Si la gama de la capacidad no se puede cambiar para una prueba (como en el caso de los dinamómetros del péndulo) la rotura aplicada de la fuerza será medida dentro +/-2% del valor completo, y la fuerza extensible más pequeña medida será exacta dentro del 10%.

**c) Selección de los especímenes de ensayo:**

A menos que se indicare en forma diferente el caucho termoplástico o los especímenes termoplásticos del elastómero, o ambos, deben ser cortados de las hojas moldeadas inyección o las placas con un espesor del espécimen de 1.3mm a 3.3mm del otro grueso no darán necesariamente resultados comparables. Los especímenes deben ser probados en las direcciones paralelas y perpendiculares a la dirección del flujo en el molde. Las dimensiones de la hoja o de la placa deben ser lo



suficientemente homogéneo. Para la obtención de la forma de las probetas de ensayo se debe utilizar un troquel cuya cuchilla deberá reproducir la probeta elegida (véase Figura N° 4.11.).

**d) Equipo Auxiliar.**

La forma y dimensiones de las cavidades del molde para la preparación de muestras de tensión (Troquel), se ajustará a los que se muestran en la sección reducida será perpendicular al plano formado por los filos de corte y pulido para la distancia de al menos 5 mm (0,2 pulg), estos datos se detallan en el anexo 9.

**e) Procedimiento de ensayo**

- i. Se tiene una probeta de 3.2 mm de espesor vulcanizada.
- ii. Se ingresa la probeta a una troqueladora obteniéndose una probeta troquelada de la siguiente forma ASTM – 412 – C UL – 62 – C (véase figura N° 4.11.).
- iii. Se hace la identificación de la probeta en el software de análisis, datos como: tipo de caucho y número de batch, se fija la distancia de análisis.
- iv. Se hace la medición del espesor de la probeta y el ancho (w) de la zona de esfuerzo datos que el software de ensayo lo pide.

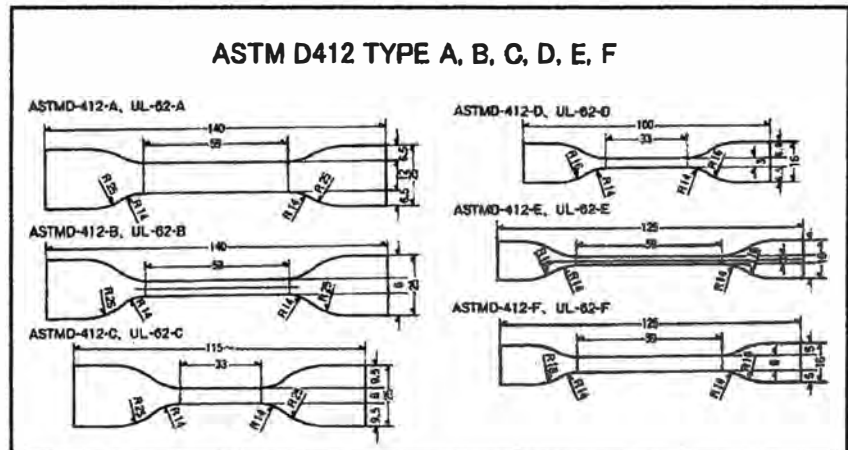


Figura N° 4.11. Probetas de ensayo de Tensión y  
alargamiento máximo

- v. Esta probeta es sujeta de sus extremos por las mordazas del tensiómetro, se procede a colocar las pinzas en los extremos de la zona de esfuerzo de la probeta.
- vi. Se pone en marcha la prueba en donde la mordaza inferior está estática y la superior que se encuentra en la cruceta móvil, se estira la muestra hasta que esta colapsa por ruptura en la zona de entre las pinzas de la probeta.
- vii. Se hace la lectura de las variables y son registradas en los formatos de control.
- viii. A continuación se detalla algunos resultados de material caucho 121S, ensayado con el equipo tensiómetro T2000 con software de Alpha Technology en la figura N° 4.12.

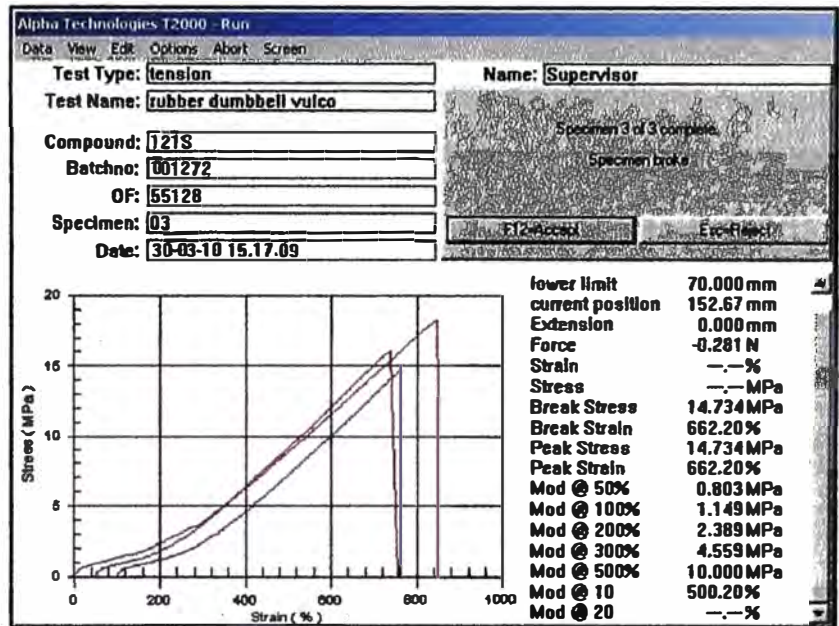


Figura N° 4.12. Ensayo de tensiómetro para el caucho 121S.

### 4.3 Elaboración de la hoja de especificaciones.

#### 4.3.1 Definición de los productos.

Los revestimientos como accesorios y estructuras son elementos de uso en un determinado punto de un circuito de la minería por tal estos deben ser capaces de soportar alta abrasividad debido al desgaste que ocasiona dependiendo de las condiciones ya antes mencionadas al producto se le designa un determinado tipo de caucho para un buen desenvolvimiento del accesorio ó estructura

#### 4.3.2 Tipo de productos.

##### 4.3.2.1 Accesorios:

Estos constan de Alma metálica de fierro dulce en su interior y cubierta por caucho vulcanizado. Estas piezas

sirven para reemplazar distintos repuestos de bombas de pulpa minera, lifter de molinos SAG, hidrociclones, etc.

#### **4.3.2.2 Estructuras:**

Estos constan de ser elementos grandes cuya característica es que no se producen con recurrencia debido a que son elementos de diseño a pedido y es considerado como servicio, por ejemplo tuberías desde diámetro de 4" a 42", distribuidores de batería de ciclones, revestimiento de tanques de alimentación, válvulas de cambio etc.

#### **4.3.3 Aplicación de revestimientos en accesorios mineros.**

Los repuestos de las bombas como un impulsor BGV 1.5", el cual puede verse a continuación (véase figura N° 4.13.), está producido por el prensado en caliente usando un molde en donde dentro de esta se coloca el alma metálica en las respectivas guías y luego se procede a colocar la goma cruda se prensa en caliente por espacio de 1 hora y 30min y se obtiene un repuesto que presenta propiedades mezcladas, por ejemplo consistencia uniforme y resistente a fluidos corrosivos y abrasivos.

Se muestra en la figura N° 4.14. Una tubería de 30" de diámetro y una longitud de 4 metros, en la cual el vulcanizado es al vapor directo en autoclave, el curado del caucho en esta tubería se realizara a una temperatura bordeando los 120 °C y 4 horas de tiempo.



Figura N° 4.13. Impulsor revestido para bombas de pulpa minera.



Figura N° 4.14. Tuberías matrices de pulpa minera.

En la figura N° 4.15, se tiene un tanque de alimentación para bomba de descarga, en donde este ha sido revestido con un caucho negro que puede soportar la acidez de la pulpa y la abrasión que se origina al circular por las superficies expuestas del tanque.



Figura N° 4.15. Tanques de Alimentación.

#### 4.3.4 Uso de productos

Los tipos de accesorio y estructuras que son producidos utilizando el caucho son destinados a sustituir ó reemplazar piezas y estructuras respectivamente que están en contacto con lodos mineros los cuales son deteriorados por el desgaste que origina la pulpa minera ya que el recubrimiento cumple un papel determinante en la protección de los mencionados elementos. Por las características explicadas anteriormente estos productos tienen un tiempo de vida variable que va a depender de los parámetros de interacción de la pulpa mineral los cuales pueden variar de despreciable hasta muy crítico.

#### 4.3.5 Diseño de la hoja de especificaciones.

Se toma en cuenta seis puntos importantes, estos son:

- **Datos de productos:** Se especifica el tipo de producto, Cliente, área de producción, orden de fabricación y tipo de revestido.

- **Identificación del tipo de caucho:** Se tiene Tipo de caucho, caucho base, número de batch de análisis y Color de caucho.
  
- **Condiciones de fabricación:** Se especifica la temperatura de vulcanizado, presión proceso, tiempo de vulcanizado y rugosidad del alma metálica si es que tuviera.
  
- **Propiedades especiales generales del caucho:** La clasificación según su afinidad para resistir pueden ser E: excelente; B: bueno; P: pobre; C: uso en casos especiales; N: no usar; S/D: sin determinar.
  
- **Propiedades reológicas:** Valores máximos y mínimos de torque y valores obtenidos.
  
- **Propiedades físicas del caucho:** Valores máximos y mínimos de densidad relativa, Dureza, Abrasión, Elongación y Resistencia a la tracción.

Por lo tanto la tabla N° 4.1. tiene como resultado la siguiente forma:



**Tabla N° 4.1. Hoja de especificaciones técnicas del revestimiento**

**Especificaciones Técnicas de productos con revestimiento de caucho**

**Datos de productos :**

Producto : Revestimiento succion Warman 5 x 4  
 Cliente : Southern Copper Corporation  
 Area de producción : Prensas  
 Orden de fabricación : 56520  
 Tipo de Revestido: En caliente prensado

**Identificación del tipo de caucho:**

Tipo de Caucho : 228S  
 Caucho Base : Natural  
 Color : Rojo

Numero de Batch: 2751

**Condiciones de Fabricación:**

Temp. de Vulcanizado : 154 °C  
 Presión de proceso : 1450 Psi  
 Tiempo de vulcanizado : 1.5 horas

**Estructura metálica interna:**

Si:   
 No:

Rugosidad superficial: 2.5 mils

**Propiedades especiales generales del caucho**

Envejecimiento: P-B  
 Resistencia a los hidrocarburos: N  
 Permeabilidad: P  
 Temperatura de servicio: -20°C a 70°C  
 Resistencia a la llama: N  
 Propiedades electricas: N-P  
 Ozono: N  
 Adhesión a metal: E

**E: excelente; B: bueno; P: pobre; C: uso de casos especiales; N: No usar; S/D: Sin Determinar**

**Propiedades Reológicas**

	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Torque Max(dN/m):	20	18.0	22	21.54
	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Torque Min(dN/m):	9	7.0	11.0	7.56
Norma :	ASTM D 2084			

**Propiedades físicas del Caucho**

	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Densidad (rC/rA):	1.05	1.04	1.06	1.0569
Norma :	UNE 53526:2001			
	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Dureza (Shore-A):	45	40	50	46
Norma :	ASTM D 2240			
	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Abrasion (mg/ciclo):	0.045	0.04	0.05	0.051
Norma :	ASTM D 3389			
	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Elongación (%) :	700	665	735	712
Norma :	ASTM D 412			
	Valor Nom.	Valor Min	Valor Max	Valor obtenido
Resist Tracc (Kg/cm2):	196	186.2	205.8	189.5
Norma :	ASTM D 412 - 92			



## **V CONCLUSIONES Y RECOMENDACIONES**

- Las estructuras o repuestos de equipos para la minería pueden ser protegidos haciendo una elección adecuada del caucho los cuales va depender de las características del fluido y condiciones de operación a los cuales estarán sometidos.
- Los revestimientos de caucho obtendrán un tiempo de vida más prolongado ó considerado en comparación si estas estructuras ó repuestos operarían sin protección alguna.
- El ensayo de reometría para los cauchos crudos acelerados, es la prueba más importante y predominante de todas, debido a que en esta prueba muestra como el caucho va cambiando internamente en su estructura molecular debido a su propiedad viscoelástica.
- El ensayo de abrasión nos puede dar una referencia cuantitativa cercana del continuo desgaste de los revestimientos de caucho de las estructuras y repuestos que experimentan en su respectiva operación.
- Es importante que estos repuestos sean almacenados correctamente en un lugar libre de los rayos solares, dióxido de carbono, aire, oxígeno, debido a que estos anteriores son agentes de envejecimiento los cuales pueden aminorar las propiedades del caucho.
- Cumpliendo los ensayos basados en la norma se puede asegurar la calidad del producto que se suministra al cliente.
- Es recomendable colocar un termómetro infrarrojo permanente en la calandra para evitar sobrecalentamiento del caucho.

## **VI. BIBLIOGRAFIA**

- 6.1 **Ciencia y tecnología de los materiales**  
Donald R. Askeland, Pradeep P. Phulé, International Thomson, 1998
- 6.2 **Rubber Technology Handbook**  
**Hofmann. Hanser Publishers, 1989**
- 6.3 **Introducción a la Química de los Polímeros**  
**Raimond B. Seymour, Charles E. Carraher, Editorial Reverte, 2002**
- 6.4 **Rubber Compounding, Principles, Materials and Techniques**  
**Barlow, F. 1988**
- 6.5 **Rubber Technology**  
**Morton, M. 1989**
- 6.6 **ASTM D 2084 -93 Standard Test Methods for Rubber Property –  
Vulcanization Using Oscillating Disk Cure Meter1.**
- 6.7 **ASTM D 412 -92 Standard Test Methods for Vulcanized Rubber and  
Thermoplastic Rubbers and Thermoplastic Elastomers – Tensión1.**
- 6.8 **ASTM D 3389 -75 Standard Test Methods for Testing Coated  
Fabrics–Abrasion Resistance (Rotary Platform, Double-Head  
Abrader).**
- 6.9 **ASTM D 2240 -02 Standard Test Methods for Rubber Property –  
Durometer Hardness1.**

- 6.10 Especificaciones Cualitativas del caucho en neumáticos  
[http://www.goodyear.com.ve/tireschool/read\\_sidewall.html](http://www.goodyear.com.ve/tireschool/read_sidewall.html)
- 6.11 Características de los Elastómeros  
<http://www.mc.usb.ve/Programas/MC2516.pdf>
- 6.12 Rubber Hardness  
<http://www.materials.co.uk/rubber.htm>
- 6.13 Cidra Minerals Processing  
[http://www.cidra.com/document\\_library/BI0401sp\\_Phosphate\\_Minig\\_App\\_note.pdf](http://www.cidra.com/document_library/BI0401sp_Phosphate_Minig_App_note.pdf)
- 6.14 Transporte Hidráulico de Sólidos  
<http://www.metalurgia.uda.cl/apuntes/ptapia/mecanica%20II/transpo rte%20hidraulico%20de%20s%C3%B3lidos.pdf>
- 6.15 Biblioteca Virtual de INDECOPI  
[http://www.bvindicopl.gob.pe/wcircu/query.exe?cod\\_user=wwwcircu &key\\_user=wwwcircu&base=02&perlo=1&fmt=01&nreg=20&ldio ma=all&boolexp=ABRACION&trunca=%24%2F%2876%2C77%29](http://www.bvindicopl.gob.pe/wcircu/query.exe?cod_user=wwwcircu &key_user=wwwcircu&base=02&perlo=1&fmt=01&nreg=20&ldio ma=all&boolexp=ABRACION&trunca=%24%2F%2876%2C77%29)
- 6.16 Manual der caucho STRUKOL  
[http://www.struktol.com/pdfs/Manual\\_del\\_caucho.pdf](http://www.struktol.com/pdfs/Manual_del_caucho.pdf)
- 6.17 Capítulo 3 / Norma ASTM D-2000  
[www.ryrasociados.com/imagenes/capitulo3.ppt](http://www.ryrasociados.com/imagenes/capitulo3.ppt)
- 6.18 Manufactures Cusell S.L.  
<http://www.arandelasyjuntas.com/es/19069/Materiales/Cauchos.html>

6.19 The Permeability of Different Rubbers to Gases and Its Relation to Diffusivity and Solubility.

[http://jap.aip.org/japiau/v17/i11/p972\\_s1?isAuthorized=no](http://jap.aip.org/japiau/v17/i11/p972_s1?isAuthorized=no)

## **VII. GLOSARIO**

**Reometría:** Es la ciencia que describe tanto los métodos de medida como los instrumentos que permiten obtener datos reológicos de un material (ver reología). Determina las relaciones cuantitativas y cualitativas entre la deformación y la tensión mecánica y sus derivadas.

**Alma metálica:** Estructura metálica interna que se coloca a un revestimiento para que adquiera más compactación y rigidez.

**Vulcanización:** Es un proceso mediante el cual se calienta el caucho crudo en presencia de azufre, con el fin de transformar el material con propiedades específicas elasticidad dureza y resistencia química y mecánica.

**Mils:** milésimas de pulgada ( $10^{-3}$  pulgadas)

**Reograma:** Es un diagrama reológico que indica qué tipo de comportamiento tiene un determinado material.

**Modulo elástico:** Velocidad del cambio de deformación como función del esfuerzo que se ejerce sobre un material

**Modulo visco elástico:** referido a la viscosidad que presenta un material elástico.

**Protrusión:** Deformación de una zona del material por aumento de volumen debido a esfuerzo externo.

- **Indentor:** aguja del durómetro que entra en contacto con el material el cual se dispone medir su dureza.



**Anexo 2: Registro de control de densidad de masterbatch.**

<b>REGISTRO DE CONTROL DE DENSIDAD</b>					
<b>MATERIAL: MASTERBATCH 121-S</b>					
<b>Los ensayos se realizan según recomendaciones de la norma UNE 83628:2001. Método A.</b>					
<b>FECHA</b>	<b>Temperatura de ensayo °C</b>	<b>Probeta 1 g/cm3</b>	<b>Probeta 2 g/cm3</b>	<b>DENSIDAD MEDIA g/cm3</b>	<b>CONTROLADO POR:</b>
<b>COMENTARIOS / OBSERVACIONES:</b>					
SGI F 1016	Version 00	14/08/2008	pag 1		
REPORTE DE CONTROL DE DENSIDAD					



**Anexo 3: Registro de ensayos de reometría y dureza.**

CONTROL DE LOTES - TRAZABILIDAD DE GOMA										
FECHA	TIPO	OPERARIO	NUMERO BATCH	DUREZA (Shore-A)	REOMETRIA					OBSERVACIONES
					ML (dNm)	tml (s/min)	MH (dNm)	tmh (min)	t (min)	
04/01/2010	228E	Carpio	647	45	< 1.94	1.94	< 13.82	2.19	1.71	52980
04/01/2010	228E	Carpio	648	41	< 1.94	1.79	< 13.82	2.04	1.59	52980
05/01/2010	228E	Carpio	649	42	< 1.94	1.86	< 13.38	2.09	1.64	52980
05/01/2010	228E	Carpio	650	41	< 1.94	1.64	< 13.82	1.91	1.43	52980
12/01/2010	121M	Villaverde	566	60	5.31	1.67	20.53	2.14	1.21	53447
12/01/2010	121M	Villaverde	567	61	6.16	1.42	19.68	1.91	1.11	53447
12/01/2010	121M	Villaverde	568	60	5.73	1.56	19.68	2.06	1.21	53447
12/01/2010	121M	Villaverde	569	60	6.38	1.51	19.68	1.95	1.16	53447
12/01/2010	121M	Carpio	570	59	8.27	1.63	20.1	2.16	0	51833
12/01/2010	121M	Carpio	571	58	6.38	1.55	19.26	2.13	1.25	51833
12/01/2010	121M	Carpio	572	60	6.38	1.57	19.26	2.15	1.25	51833
12/01/2010	177	Carpio	102	60	8.27	1.22	30.67	1.53	0.71	54193
12/01/2010	177	Carpio	103	59	8.27	1.25	28.56	1.69	0.88	54193
12/01/2010	177	Carpio	104	61	8.27	1.76	< 19.68	2.6	1.28	54193
12/01/2010	177	Carpio	105	59	7.42	1.37	27.71	1.75	0.94	54191
12/01/2010	177	Carpio	106	59	7.85	1.33	27.71	1.78	0.95	54194
12/01/2010	177	Carpio	107	61	7.85	1.27	27.71	1.7	0.9	54194
12/01/2010	177	Carpio	108	60	9.11	1.33	30.25	1.7	0.95	54195
12/01/2010	177	Carpio	109	60	8.97	1.25	30.25	1.6	0.87	54195
12/01/2010	177	Carpio	110	61	8.69	1.45	28.98	1.85	0.98	54195
13/01/2010	228S W	Villaverde	441	42	< 7.85	1.69	< 19.26	1.92	1.36	54077
13/01/2010	228S W	Villaverde	442	43	< 7.85	1.51	< 19.26	1.77	1.21	54077
14/01/2010	121S	Carpio	1233	60	8.97	1.5	26.87	1.93	1.09	54207
14/01/2010	121S	Carpio	1234	59	9.11	1.27	26.87	1.66	0.88	54207
14/01/2010	121S	Carpio	1235	60	8.69	1.29	24.33	1.76	0.9	54207
14/01/2010	121S	Carpio	1236	59	8.69	1.39	24.33	1.86	0.98	54207
14/01/2010	121S	Carpio	1237	59	8.69	1.33	24.33	1.79	0.94	54207
14/01/2010	150S	Villaverde	54	52	6.3	2.15	19.68	2.55	1.68	54014

**Anexo 4: Registro de abrasión de caucho vulcanizado.**

TEST ABRASION RESISTANCE											
Norma: ASTM D 3389 -75 Abrasion Resistance (Rotary Platform, Double-Head Abrader)¹											
Fecha	O.F.	Goma	Nº Prueba	Fuerza g	Masa Inicial (g)	Masa Final (g)	Nº Revol (ciclos)	Índice Abrasión (mg/ciclos)	Promedio Índice Abrasión (máximo)	Tipo Rueda	Observaciones
14/01/2010	51833	1308	1	1000	33.3003	33.2976	750	0.0036	0.0045	Taber H-18	
			2	1000	32.1052	32.1011	750	0.0055			
			3	1000	33.2631	33.2597	750	0.0045			
29/01/2010	54486	228S	1	1000	29.2964	29.2433	1000	0.0531	0.0457	Taber H-18	
			2	1000	31.5898	31.5608	750	0.0387			
			3	1000	30.2826	30.2487	750	0.0452			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			
			1					#DIV/0!			
			2					#DIV/0!			
			3					#DIV/0!			



## Anexo 6: Detalles de la ASTM D 2084-93, referente a dimensiones del espécimen, condiciones y procedimiento de ensayo.



Designation: D 2084 - 93

### Standard Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter<sup>1</sup>

This standard is issued under the fixed designation D 2084; 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 test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

#### 1. Scope

1.1 This test method describes the use of the oscillating disk cure meter for determining selected vulcanization characteristics of vulcanizable rubber compounds.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 ISO 3417 is very similar to this test method. It has minor technical differences that are not considered to be significant.

1.4 This standard does not purport to address all of the safety problems, 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:

- D 1349 Practice for Rubber—Standard Temperatures for Testing<sup>2</sup>
- D 3185 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil<sup>2</sup>
- D 3186 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil<sup>2</sup>
- D 3187 Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)<sup>2</sup>
- D 3190 Test Methods for Rubber—Evaluation of General-Purpose CR (Chloroprene Rubber)<sup>2</sup>
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2</sup>

##### 2.2 ISO Standard:

- ISO 3417 Rubber—Measurement of Vulcanization Characteristics With the Oscillating Disk Rheometer<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.12 on Processability Tests.

Current edition approved March 15, 1993. Published May 1993. Originally published as D 2084 - 71 T, replacing Methods D 2705 and D 2706. Last previous edition D 2084 - 92a.

<sup>2</sup> Annual Book of ASTM Standards, Vol 09.01.

<sup>3</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

#### 3. Terminology

##### 3.1 Descriptions of Terms Specific to This Standard:

3.1.1 The following measurements may be taken from the curve of torque versus time (see Fig. 1).

3.1.1.1 *cure rate index*—measure of rate of vulcanization based on the difference between optimum vulcanization and incipient scorch time.

3.1.1.2 *maximum, plateau, or highest torque*—measure of stiffness or shear modulus of the fully vulcanized test specimen at the vulcanization temperature.

3.1.1.3 *minimum torque*—measure of the stiffness of the unvulcanized test specimen taken at the lowest point of the curve.

3.1.1.4 *time to incipient cure (scorch time)*—measure of the time at which vulcanization begins.

3.1.1.5 *time to a percentage of full cure*—measure of optimum cure based on the time to develop some percentage of the highest torque or difference in torque from the minimum.

3.1.2 *torque*—maximum amplitude torque.

#### 4. Summary of Test Method

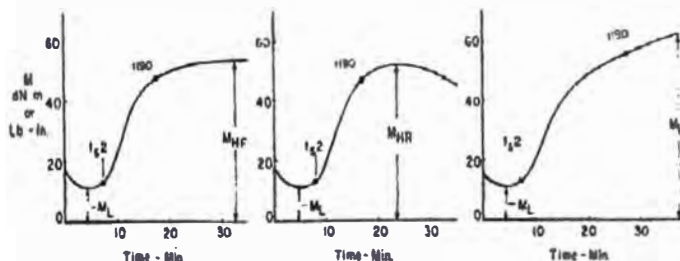
4.1 A test specimen of vulcanizable rubber compound is inserted into the cure meter test cavity and after a closure action is contained in a sealed cavity under positive pressure. The cavity is maintained at some elevated vulcanization temperature. The rubber totally surrounds a biconical disk after the dies are closed (see Fig. 2). The disk is oscillated through a small rotational amplitude (1° or 3°) and this action exerts a shear strain on the test specimen. The force required to oscillate or rotate the disk to maximum amplitude is continuously recorded as a function of time, with the force being proportional to the shear modulus (stiffness) of the test specimen at the test temperature. This stiffness first decreases as it warms up; then it increases due to vulcanization. The test is completed when the recorded torque either rises to an equilibrium or maximum value, or when a predetermined time has elapsed. The time required to obtain a cure curve is a function of the characteristics of the rubber compound and of the test temperature (see Fig. 1 for typical cure curves).

#### 5. Significance and Use

5.1 This test method is used to determine the vulcanization characteristics of (vulcanizable) rubber compounds.

5.2 This test method may be used for quality control in rubber manufacturing processes, for research and develop-

## D 2084



Left Curve: Cure to Equilibrium Torque.  
 Middle Curve: Cure to a Maximum Torque with Reversion.  
 Right Curve: Cure to No Equilibrium in Maximum Torque.

FIG. 1 Types of Cure Curve

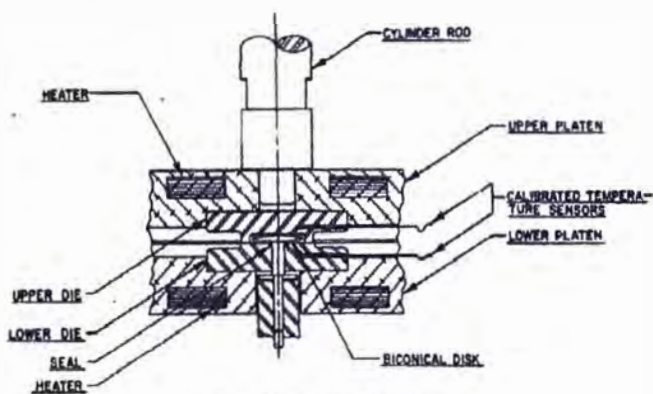


FIG. 2 Cure Meter Assembly

ment testing of raw-rubber compounded in an evaluation formulation, and for evaluating various raw materials used in preparing (vulcanizable) rubber compounds.

## 6. Apparatus

6.1 *Cure meter*, consists of the following major components: specimen chamber and closure mechanism, temperature control system, rotor drive and torque measuring system (see Fig. 2 for a detailed drawing of cure meter assembly).

6.2 *Specimen Chamber*—Consists of platens, dies, and a biconical disk.

6.2.1 *Platens*—Two platens made of aluminum alloy, each containing an electric heater, and each having in the center, a cavity to accommodate a die and from the side, a well for inserting a temperature sensor.

6.2.2 *Dies*—Two which form a cavity when closed and which shall be fabricated from tool steel having a minimum Rockwell Hardness HRC 50. The geometry of the standard dies is shown in Figs. 3 and 4 with dimensions and tolerances (see Table 1). The top and bottom surfaces of the die cavity shall contain rectangularly shaped grooves arranged radially about the center and spaced at 20° intervals. Each die shall have a well or hole drilled from the side to accommodate a temperature sensor inserted through the platen. The lower dies shall have a hole in the center to allow for the insertion

of the disk shaft. A suitable low-friction seal shall be provided in this hole to prevent material leaking from the cavity.

6.2.3 *Disk*—The biconical disk (see Fig. 5) shall be fabricated from tool steel having a minimum Rockwell Hardness of HRC 50. The disk shall be fitted with a stem that fits into the torque shaft. The disk is shown in Fig. 5 (see Table 2).

6.2.3.1 Disk wear will affect test results. A disk worn to such an extent that the disk diameter is less than the minimum diameter shown in this procedure shall not be used.

6.2.3.2 The standard frequency of the rotary oscillation of the disk shall be constant at 1.7 Hz (100 cpm). Other frequencies may be used, if required.

6.2.3.3 A rotary drive system shall be provided for oscillatory rotation of the disk. The amplitude of oscillation of the unloaded disk shall be constant at  $\pm 1.00^\circ$  with a tolerance of  $\pm 0.03^\circ$  about the center position, that is, a total amplitude of  $2^\circ$ . Other amplitudes may be used, if specified.

NOTE 1—Where the effect of surface contamination on the disk is not a problem, greater sensitivity in determining curing characteristics may be obtained using  $\pm 3^\circ$  angle of oscillation.

6.2.4 *Die Clamping Mechanism*—A pneumatic cylinder or



D 2084

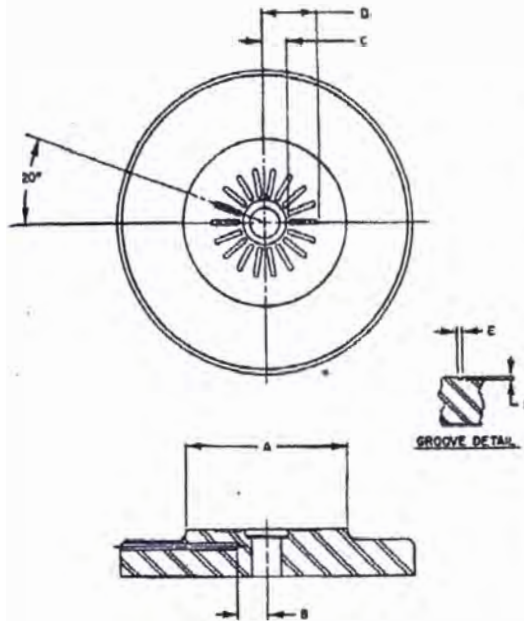


FIG. 3 Lower Die

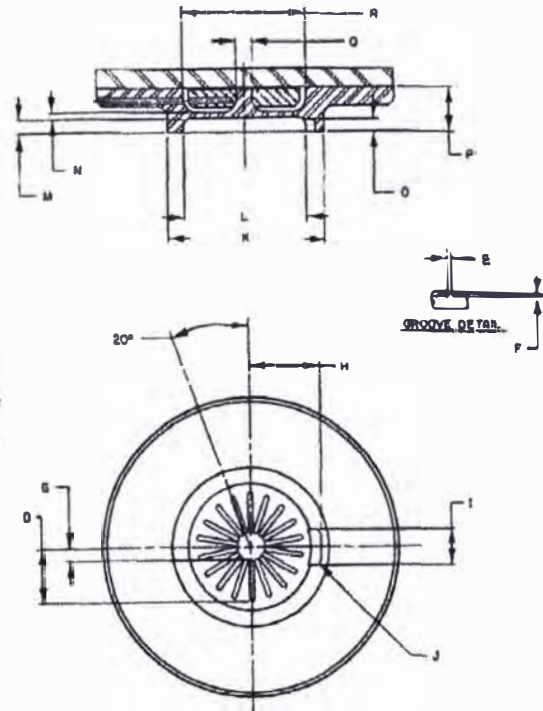


FIG. 4 Upper Die

TABLE 1 Die Dimensions

Code	Dimension, mm	Tolerance, mm
A	65.88	±0.13
B	10.64	±0.25
C	7.94	±0.13
D	18.28	±0.13
E	1.57	±0.13
F	0.8	±0.1
G	4.76	±0.13
H	24.07	±0.08
I	12.70	±0.40
J	2.38	±0.40
K	64.61	±0.05
L	41.91	±0.03
M	5.35	±0.01
N	1.65	±0.03
O	4.57	±0.13
P	15.38	±0.03
Q	5.08	±0.05
R	41.91	±0.03

other device shall close the dies and hold them closed during the test with a force of 11.0 ± 0.5 kN (2500 ± 100 lbf).

NOTE 2—The manufacturer recommends the source air pressure be adjusted to 345 kPa (50 psi). Provisions are made for this adjustment in the instrument. Therefore, 345 kPa acting on the instrument's 203-mm (8-in.) diameter air cylinder will produce a force of 11 kN (2500 lbf) on the die per the following equation:

$$F = P \left( \frac{\pi D^2}{4} \right)$$

where:

F = closure force on die.  
P = source air pressure, and

D = diameter of piston in pneumatic cylinder.

To calculate maximum cavity pressure, the effect of this force acting on the surface area of the upper die may be calculated per the following equation:

$$P_c = \frac{4F}{\pi d^2}$$

where:

P<sub>c</sub> = pressure on sample in upper die cavity, and  
d = diameter of upper die cavity (55.9 mm (2.2 in.)).

(For example, P<sub>c</sub> =

$$\left( P_c = \frac{(4)(2500)}{(3.14)(2.2)^2} = \frac{10\,000}{(3.14)(4.84)} = \frac{10\,000}{12.06} = 829 \text{ psi.} \right)$$

6.3 Temperature Controlling System—A temperature controller shall be provided for maintaining the dies within ±0.5°C (±1°F) of the specified test temperature.

6.4 Torque Measuring System—The torque measuring system shall consist of a device, such as a torque transducer, producing a signal that is directly proportional to the torque required to oscillate the disk. A recorder to record the maximum amplitude signal from the torque transducer shall be provided. The recorder shall have a full-scale deflection response on the torque scale of 1 s or less and be capable of recording the torque with accuracy of ±0.5 % of the torque range. Four torque ranges shall be provided; 0 to 25, 0 to 50, 0 to 100, and 0 to 200 dN·m (or 0 to 25, 0 to 50, 0 to 100, and 0 to 200 lbf·in.)

## D 2084

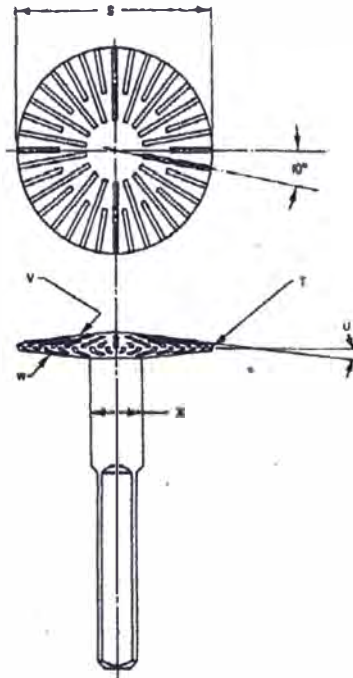


FIG. 5 Biconical Disk

TABLE 2 Disk Dimensions

Code	Dimension, mm	Tolerance, mm
S	Diameter	±0.01
T	Radius	±0.03
U	Groove depth	±0.08*
V <sup>A</sup>	Groove width	±0.05
	Groove depth	±0.1
	Groove length, min	7.5
W <sup>A</sup>	Groove width	±0.05
	Groove depth	±0.1
	Groove length, min	9.5
X	Diameter	±0.02
		-0.00

\* Grooves on top and bottom surfaces should be displaced 5°.

NOTE 3—The term “recorder” as used in this test method implies the use of any suitable data collection device, including printers, plotters, and computers.

NOTE 4—Direct proportionality between torque and stiffness cannot be expected under all test conditions, particularly in higher torque ranges, because elastic deformation of the disk shaft and driving device must be taken into account. However, for routine quality control test purposes corrections are not necessary.

### 7. Sampling

7.1 The sample shall be taken from a vulcanizable rubber compound as required by the mixing method or other sampling instructions.

7.2 The sample shall be in sheeted form, at room temperature, and as free of air as possible.

### 8. Test Specimen

8.1 A circular test specimen taken from a sample shall be  $30 \pm 2$  mm ( $1.2 \pm 0.1$  in.) in diameter and  $11.5 \pm 1.5$  mm ( $0.45 \pm 0.05$  in.) in thickness or equivalent to a volume of  $9 \text{ cm}^3$  ( $0.5 \text{ in.}^3$ ).

8.2 The test specimen is considered to be of proper size when a small bead of compound is extruded uniformly around the periphery of the die as it is closed. This is achieved when the specimen volume is between 8 and  $11 \text{ cm}^3$  (9 to 13 g of rubber compound with a specific gravity of 1.15). Undersized specimens can cause low cavity pressure and low torque readings. Oversized specimens cool the dies excessively during the early part of the test cycle affecting the vulcanization characteristics.

### 9. Test Temperatures

9.1 The standard test temperature shall be  $160^\circ\text{C}$  ( $320^\circ\text{F}$ ).

9.2 The test temperature tolerance shall be  $\pm 0.5^\circ\text{C}$  ( $\pm 1.0^\circ\text{F}$ ).

9.3 Tests may be carried out at other temperatures, if required. They should be selected in accordance with Practice D 1349.

### 10. Calibration

10.1 The cure meter shall be calibrated mechanically in accordance with the manufacturer's instructions.

10.2 Provisions shall be made for electronic verification of the recorder and for torque transducer calibration by means of a resistor incorporated in the torque measuring circuit that simulates an applied torque of specified value.

10.3 The cure meter shall be calibrated with the torque standard supplied by the manufacturer any time the results are suspected of being inaccurate, after any repairs, any change in arc, or frequently enough to ensure the maintenance of proper calibration. The cure meter shall read zero when running empty and read the certified value with the torque standard inserted.

### 11. Procedure

#### 11.1 Preparation for Test:

11.1.1 Bring the temperature of both dies to the temperature of test with the disk in place and the dies in the closed position. Set recorder range to zero and adjust the recorder pen to zero torque and zero time position on the chart. Select the correct running time and choose the torque range to give maximum torque in the upper half of the recorder chart.

11.1.2 “Running Zero” may be checked at this point and should be off no more than  $0.5 \text{ dN}\cdot\text{m}$  (or  $0.5 \text{ lbf}\cdot\text{in.}$ ). If in excess of this, check the cure meter for frictional drag that could be caused either by bad bearings, by improper friction seal (6.2.2), by rotor misalignment, or by sample “build-up” around the rotor shaft. If the error persists, consult the manufacturers manual.

#### 11.2 Loading the Cure Meter:

11.2.1 Open the dies, place the test specimen (Notes 5 and 6) on top of the disk and close the dies. This operation must be completed within 20 s.

11.2.2 Start the recorder at the instant the dies are closed.

## D 2084

TABLE 3 Precision

Note— $S_w$  = within laboratory standard deviation,  $r$  = repeatability (in measurement units),  $(r)^A$  = repeatability (in percent),  $S_b$  = between laboratory standard deviation,  $R$  = reproducibility (in measurement units),  $(R)^A$  = reproducibility (in percent).

Test Parameter	Range of Values	Mean Value	Within Laboratory			Between Laboratory		
			$S_w$	$r$	$(r)^A$	$S_b$	$R$	$(R)^A$
<b>Type 1 Precision:</b>								
$M_L$ (dN·m)	7.1 to 9.7	8.4	0.158	0.45	5.4	1.36	3.85	45.8
$M_H$ (dN·m)	28.4 to 38.9	33.7	0.181	0.51	1.6	1.98	5.80	16.6
$t_{1/2}$ (min)	2.3 to 6.3	3.8	0.12	0.34	9.0	0.25	0.71	18.7
$t'$ 50 (min)	3.9 to 8.6	6.4	0.15	0.43	8.7	0.34	0.98	15.0
$t'$ 90 (min)	6.4 to 14.0	10.2	0.20	0.57	5.8	0.60	1.70	18.7
<b>Type 2 Precision:</b>								
$M_L$ (dN·m)	7.8 to 8.8	8.7	0.215	0.61	7.0	0.735	2.08	23.9
$M_H$ (dN·m)	32.0 to 47.7	39.9	0.35	0.99	2.5	2.60	7.30	18.4
$t_{1/2}$ (min)	4.4 to 6.8	5.1	0.12	0.34	6.7	0.37	1.05	20.6
$t'$ 50 (min)	7.7 to 9.9	8.6	0.18	0.61	5.8	0.50	2.55	29.0
$t'$ 90 (min)	15.9 to 18.0	17.0	0.19	0.54	3.2	1.50	4.25	25.0

<sup>A</sup> These are estimated values, using the mid-point of the range for the parameter mean value.

Type 1 precision is obtained from fully prepared test specimens (compounds mixed in one laboratory); these are circulated to all participating laboratories.

Type 2 precision is obtained by circulating all compounding materials (drawn from a common source) to each participating laboratory. The mixing to prepare the compound is done in each laboratory and therefore mixing variation is part of the "total test" variation or test precision.

The disk may be oscillating at zero time or oscillation may be started not later than 1 min after the dies are closed. In the latter case, report preheat as required in 12.1.8.

**NOTE 5**—When testing sticky rubber compounds, cellophane, or other thin film that will not melt at the test temperature, may be inserted below and above the test specimen, but not against the rotor. This prevents the rubber from sticking to the dies. Suitable film materials are Dupont 600 PD/001,<sup>4</sup> Avisco 300P1,<sup>5</sup> or Polyester Film Mylar Type A-100.<sup>6</sup>

**NOTE 6**—A deposit of material from the rubber compounds under test may build up on the disk and dies. This may affect the final torque values. It is suggested that stable vulcanizable rubber compound be tested daily to detect this occurrence. If such contamination develops, it may be removed by cleaning with a noncorrosive compound or solution that does not degrade the aluminum insert contained in the upper die of most cure meters. After solvent cleaning one or two runs on a nonabrasive rubber compound are required to eliminate solvent or residue completely. Abrasive cleaning may be used with caution. The recommended cleaner is 220 grit aluminum oxide.

## 12. Report

12.1 Report the following information on the sample and instrument used:

- 12.1.1 Sample or specimen identification, or both,
- 12.1.2 Method of specimen preparation (for example, amount of milling),
- 12.1.3 Make and model of the cure meter,
- 12.1.4 Temperature of the dies,
- 12.1.5 Amplitude of oscillation used, reported as half of total amplitude,
- 12.1.6 Frequency of oscillation, Hz (or cpm),
- 12.1.7 Recorder range,
- 12.1.8 Full-scale recorder time, and
- 12.1.9 Preheat time, if not zero.

12.2 Test results reported are normally chosen from the following parameters (refer to Fig. 1 for guidance). The conversion from dN·m to lbf·in. is: 1.13 (dN·m) = 1.00 (lbf·in.).

12.2.1  $M_L$ —Minimum torque, dN·m (lbf·in.).

12.2.2 **Maximum torque**—All in dN·m (or lbf·in.).

12.2.2.1  $M_{HP}$ —Maximum torque where curve plateaus.

12.2.2.2  $M_{HR}$ —Maximum torque of reverting curve.

12.2.2.3  $M_H$ —Highest torque attained during specified period of time when no plateau or maximum torque is obtained.

12.2.3 Scorch time, min.

12.2.3.1  $t_{51}$  is equal to the time to 1 dN·m (or lbf·in.) rise above  $M_L$ ; is used with 1° amplitude.

12.2.3.2  $t_{52}$  is equal to the time to 2 dN·m (or 2 lbf·in.) rise above  $M_L$ ; is used with 3° (and 5°) amplitudes.

12.2.4 Cure time, min.

12.2.4.1  $t'x$  is equal to the time to x % of torque increase or  $t'x$  = minutes to  $M_L + x(M_H - M_L)/100$  torque.

**NOTE 7**—This test method of determining the cure times is considered the standard test method.

12.2.4.2  $rx$  is equal to the time to x % of maximum torque, or  $rx$  = minutes to  $x M_H/100$  torque.

**NOTE 8**—This is an alternative test method for cure time determination. The most commonly used values of x are 50 and 90.

12.2.5 Cure Rate Index = 100/(cure time - scorch time).

## 13. Precision and Bias

13.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

13.2 All precision data were obtained using the diaphragm upper die shown in Fig. 4. The use of the alternative solid die shown in Fig. A 1.1 may cause different results.

13.3 Both Type 1 and Type 2 precision results are given in Table 3. For both types of precision repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the test value, as specified by this test method, obtained on one determination or measurement of the property or parameter in question.

13.4 For the Type 1 precision, four compounds (or materials) were used; these were tested in eleven laboratories on two different days (see Table 3).

13.5 For the Type 2 precision, the precision results reported in Table 3 represent pooled average values obtained from four (other) rubber evaluation standards; Test Methods

<sup>4</sup> Dupont 600 PD/001 is suitable for this purpose.

<sup>5</sup> Avisco 300P1 is suitable for this purpose.

<sup>6</sup> Polyester Film Mylar Type A-100 is suitable for this purpose.



## D 2084

D 3185 (SBR, OE-SBR) and Methods D 3186 (SBR-BMB), D 3187 (NBR), and D 3190 (CR). These precision values are derived from interlaboratory programs with two different types of materials (for each rubber as listed above), in seven laboratories with the mixing and testing both conducted on two different days essentially one week apart.

13.6 The precision of this test method may be expressed in the format of the following statements that use what is called an "appropriate value" of  $r$ ,  $R$ , ( $r$ ), or ( $R$ ), that is, that value obtained from Table 3, to be used in decisions about test results (obtained with the test method).

13.7 *Repeatability*—The repeatability,  $r$ , of this test method has been established as the appropriate value for any parameter as tabulated in Table 3. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated  $r$  must be considered as derived from different or nonidentical sample populations.

13.8 *Reproducibility*—The reproducibility,  $R$ , of this test method has been established as the appropriate value for any parameter as tabulated in Table 3. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated  $R$  must be considered to have come from different or nonidentical sample populations.

13.9 Repeatability and reproducibility expressed as a percentage of the mean level, ( $r$ ) and ( $R$ ), have equivalent application statements as 13.7 and 13.8 for  $r$  and  $R$ . For the ( $r$ ) and ( $R$ ) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

13.10 *Precision (International Interlaboratory Testing)*—The Appendix gives precision results obtained in an international interlaboratory program conducted in ISO Technical Committee 45 on Rubber/Rubber Products in 1984/1985. These results are given for additional background on a broad-based comprehensive interlaboratory program. Please refer to the appendix for details and a full report on the precision results.

13.11 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

## 14. Keywords

14.1 compounds; ODR oscillating disk cure meter; vulcanization characteristics

## ANNEX

## (Mandatory Information)

## A1. ALTERNATIVE UPPER DIE

A1.1 This annex describes an alternative upper die for use in the oscillating disk cure meter (ODC). This die does not contain a diaphragm as shown in the die in Fig. 4.

A1.2 Some manufacturers of ODC instruments furnish upper dies with a diaphragm while others do not.

A1.3 The stated purpose of the diaphragm die is to allow

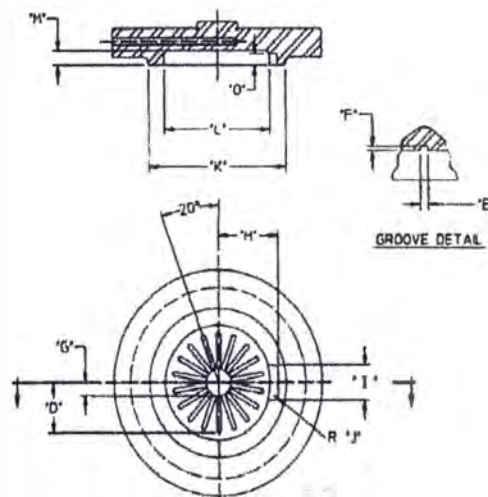


FIG. A1.1 Alternative Upper Die

 D 2084

the diaphragm to be flexed when the dies are closed on a specimen and then to maintain essentially constant pressure on the specimen as it shrinks slightly in volume during vulcanization.

A1.4 The precision and bias observed with the alternative

solid upper die as well as any differences in observed results have not been determined.

A1.5 The solid upper die is shown in Fig. A1.1. Dimensions are listed in Table 1.

## APPENDICES

(Nonmandatory Information)

## XI. INTERNATIONAL INTERLABORATORY TESTING

X1.1 This appendix gives the precision results of an international interlaboratory Oscillating Disc Cure Meter (ODC) test program conducted in ISO TC-45 in 1984 and 1985. It is added as an appendix to this test method to give additional information on the precision of ODC testing. The ISO/TC45 standard that is analogous to Test Method D 2084 is ISO 3417.

X1.2 The practice for analysis and expression of precision results for ISO TC45 is equivalent in its basic fundamentals and format to Practice D 4483.

## X1.3 Test Details:

X1.3.1 An interlaboratory test program (ITP) was organized in late 1984 to obtain precision results. Four compounds with a range of cure properties were mixed and prepared in one laboratory, sealed in metal foil packets, and distributed to laboratories located in 19 countries in Europe, Asia, and North and South America. Tests were conducted in late January and early February 1985 according to the following schedule:

X1.3.1.1 *Part I of 160C*—One test (determination) on each of two days, one week apart, for all four compounds.

X1.3.1.2 *Part II of 150C*—One test on each of two days, one week apart, for all four compounds.

X1.3.2 The formulations for the four compounds are listed in Table X1.1. Compound A has a moderate black level with a non-free sulfur (TMTD) cure system. Compounds B and C are relatively high black with conventional

cure systems. Compound D is a gum compound with a conventional cure system.

X1.3.3 A *Type 1* precision was measured in the ITP (no processing operations required on the circulated materials in any given laboratory). The time period for repeatability and reproducibility is on a scale of days.

X1.3.4 A test result is (the test value) obtained from one measurement or determination with the ODC at any temperature.

X1.3.5 A total of 50 laboratories participated in Part I, and 45 laboratories participated in Part II in addition to their participation in Part I.

## X1.4 Precision Results:

X1.4.1 The precision results for both Parts I and II are given for five ODC cure parameters. These five parameters are as follows:

X1.4.1.1  $M_L$ —Minimum torque (N·m).

X1.4.1.2  $M_{HF}$ —Maximum torque (N·m).

X1.4.1.3  $t_s$ —Scorch time, (minutes).

X1.4.1.4  $t_{50}^1$ —50 % cure time, (minutes).

X1.4.1.5  $t_{90}^1$ —90 % cure time, (minutes).

X1.4.2 For Part I of 160C, the precision results are given in Table X1.2.

X1.4.3 For Part II of 150C, the precision results are given in Table X1.3.

## X1.5 Use of Precision Results:

X1.5.1 The general procedure for using precision results is with the symbol  $|x_1 - x_2|$  designating a positive difference, that is, without regard to sign. The symbol  $x$  refers to any parameter value.

X1.5.1.1 Select the ODC parameter on which decisions are to be made. Find in Table X1.2 (160C) or Table X1.3 (150C) the Summary section for this parameter. If the tests and data under consideration are not at 150 or 160C, select the summary results for the temperature nearest to the actual test temperature under consideration.

X1.5.1.2 Enter the Summary Precision Result section of either Table X1.2 or X1.3 at an average material value nearest to the test data average under consideration. This will give the applicable  $r$ , ( $r$ ),  $R$ , and ( $R$ ) for use in the decision process.

X1.5.1.3 With these  $r$  and ( $r$ ) values the following general repeatability statements may be used to make decisions. For an absolute difference, the difference,  $|x_1 - x_2|$ , between two measured ODC parameters values, found on (presumed) identical material samples under normal and correct ODC operation conditions, will exceed the tabulated  $r$ , on average not more than once in 20 cases. For a percentage difference, the percent difference,  $|x_1 - x_2| / (x_1 + x_2) / 2 \times 100$ , between

TABLE X1.1 Compound Formulations (ISO 3417-ITP)

Material	Formulations Used			
	A	B	C	D
SBR 1502	100.0	...	...	100.0
SBR 1712 <sup>a</sup>	...	68.8	137.5	...
BR (CB441) <sup>b</sup>	...	68.8	...	...
Zinc oxide	5.0	5.0	5.0	5.0
Stearic acid	1.0	1.5	1.5	1.5
RB Number 5 <sup>c</sup>	...	80.0	60.0	...
N330	45.0	...	...	...
Process Oil <sup>d</sup>	...	8.8	5.0	5.0
DPPD <sup>e</sup>	...	1.5	1.5	1.5
Antioxidant <sup>f</sup>	...	1.5	1.5	1.5
TBBS <sup>g</sup>	...	1.2	1.0	1.0
TMTD <sup>h</sup>	3.0	...	...	...
Sulfur	...	2.0	2.0	2.0
Specific Gravity	1.13	1.16	1.16	0.98

<sup>a</sup> 37.5 (phr) oil extended SBR.

<sup>b</sup> 37.5 (phr) oil extended, BR rubber.

<sup>c</sup> ASTM Committee D-24 Industry Reference Carbon Black Number 8.

<sup>d</sup> Sundex 7250T or equivalent.

<sup>e</sup> Dimethyl-butylphenyl-phenylene diamine.

<sup>f</sup> Trimethyl-dihydroquinoline.

<sup>g</sup> N-tert-butyl-2-benzothiazole-sulfenamide.

<sup>h</sup> Tetramethylthiuram disulfide.



D 2084

TABLE X1.2 ISO 3417: Type 1—Precision at 180 °C

Parameter 1—Min torque, ML (N-M) 180 °C Final Summary Table: Precision Values Averages given in increasing order							
Material	Average	Within Laboratories			Between Laboratories		
		Sr	r	(r)	SR	R	(R)
4. Compound D	0.38	0.0315	0.0692	23.040	0.1404	0.3973	102.649
3. Compound C	0.70	0.0318	0.0693	12.700	0.1997	0.5652	60.390
2. Compound B	0.99	0.0339	0.0690	9.638	0.4369	1.2448	125.147
1. Compound A	1.65	0.0856	0.1657	11.249	0.1442	0.4081	24.715
Pooled values	0.92	0.0432	0.1223	13.298	0.2645	0.7466	81.422

Parameter 2—Max torque, MHF (N-M) 180 °C Final Summary Table: Precision Values Averages given in increasing order							
Material	Average	Within Laboratories			Between Laboratories		
		Sr	r	(r)	SR	R	(R)
4. Compound D	2.21	0.0582	0.1646	7.447	0.1996	0.5648	25.560
3. Compound C	2.71	0.0511	0.1446	5.328	0.2207	0.6246	23.007
2. Compound B	3.26	0.0750	0.2123	6.381	0.2625	0.7430	22.820
1. Compound A	4.26	0.0548	0.1644	3.621	0.3628	1.0266	24.073
Pooled Values	3.11	0.0606	0.1713	6.506	0.2689	0.7902	24.432

Parameter 3—Search time, (min) 180 °C Final Summary Table: Precision Values Averages given in increasing order							
Material	Average	Within Laboratories			Between Laboratories		
		Sr	r	(r)	SR	R	(R)
1. Compound A	1.41	0.1028	0.2910	20.680	0.1647	0.4377	30.967
4. Compound D	5.04	0.2017	0.5708	11.394	0.7011	1.9842	36.364
2. Compound B	6.08	0.1836	0.5197	10.222	0.7278	2.0698	40.612
3. Compound C	6.57	0.2302	0.6515	11.689	0.6762	1.9109	34.284
Pooled Values	4.32	0.1864	0.6247	12.140	0.6234	1.7842	40.622

two measured ODC parameter values, found on (presumed) identical material samples under normal and correct ODC operation conditions, will exceed the tabulated  $(r)$ , on average not more than once in 20 cases.

X1.5.1.4 With these  $R$  and  $(R)$  values the following general reproducibility statements may be used to make decisions. For an absolute difference, the difference  $|x_1 - x_2|$  between two independently measured ODC parameter values, found in two laboratories using normal and

correct ODC procedures on identical test material samples, will exceed the tabulated reproducibility  $R$ , not more than once in 20 cases. For a percentage difference, the percent difference,  $[|x_1 - x_2| / (x_1 + x_2) / 2] \times 100$ , between two independently measured ODC parameter values, found in two laboratories using normal and correct ODC procedures on identical material samples, will exceed the tabulated reproducibility  $(R)$ , not more than once in 20 cases.

## D 2084

TABLE X1.3 ISO 3417: Type 1—Precision of 150 °C

NOTE— $S_r$  = repeatability standard deviation,  $r$  = repeatability = 2.83 (square root of the repeatability variance),  $(r)$  = repeatability (as percentage of material average),  $S_R$  = reproducibility standard deviation,  $R$  = reproducibility = 2.83 (square root of the reproducibility variance),  $(R)$  = reproducibility (as percentage of material average).

Parameter 1—Min torque, ML (N-M) 150 °C  
Final Summary Table: Precision Values  
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
4. Compound D	0.40	0.0300	0.0848	21.000	0.1288	0.3639	90.135
3. Compound C	0.69	0.0403	0.1141	18.448	0.1025	0.2901	41.798
2. Compound B	0.77	0.0258	0.0730	9.511	0.0743	0.2101	27.376
1. Compound A	1.74	0.0517	0.1463	8.399	0.1816	0.4574	26.230
Pooled Values	0.90	0.0384	0.1066	12.098	0.1221	0.3457	38.505

Parameter 2—Max torque, MHF (N-M) 150 °C  
Final Summary Table: Precision Values  
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
4. Compound D	2.31	0.0324	0.0916	3.960	0.1942	0.5495	23.768
3. Compound C	2.82	0.0684	0.1937	6.861	0.2431	0.6876	24.384
2. Compound B	3.38	0.0642	0.1816	5.354	0.2654	0.7511	22.124
1. Compound A	4.34	0.0787	0.2228	5.134	0.2464	0.6972	16.066
Pooled Values	3.20	0.0633	0.1793	5.584	0.2397	0.6783	21.169

Parameter 3—Scorch time, (min) 150 °C  
Final Summary Table: Precision Values  
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
1. Compound A	1.99	0.1141	0.3230	18.285	0.2471	0.6992	35.214
2. Compound B	6.61	0.4932	1.3958	16.213	1.2572	3.5577	41.325
4. Compound D	8.77	0.2524	0.7143	8.149	0.6697	1.8951	21.621
3. Compound C	9.73	0.3233	0.9150	9.406	1.3838	3.9181	40.257
Pooled Values	7.31	0.3266	0.9243	12.650	0.9867	2.7924	38.217

Parameter 4—50 % cure time, (min) 150 °C  
Final Summary Table: Precision Values  
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
1. Compound A	5.58	0.2100	0.5943	10.691	0.4854	1.3172	23.698
2. Compound B	13.34	0.5802	1.5832	11.662	0.8906	2.8032	21.011
3. Compound C	17.20	0.3975	1.1250	6.539	1.1619	3.2863	19.113
4. Compound D	17.56	0.3393	0.9603	5.470	1.1049	3.1289	17.812
Pooled Values	13.46	0.3963	1.1272	6.374	0.9726	2.7526	20.448

Parameter 5—90 % cure time, (min) 150 °C  
Final Summary Table: Precision Values  
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
1. Compound A	11.91	0.6954	1.9680	16.521	1.2883	3.6894	30.133
2. Compound B	19.80	0.7055	1.9944	10.031	1.4111	3.9934	20.065
3. Compound C	30.07	0.8367	1.8075	6.010	2.1003	5.9436	19.763
4. Compound D	33.52	0.9787	2.7699	8.284	2.5473	7.2088	21.507
Pooled Values	23.91	0.7971	2.1708	9.080	1.9092	5.4030	22.599



## D 2084

## X2. HISTORY OF THE OSCILLATING DISK CURE METER

X2.1 Oscillating disk cure meters were first made commercially available in 1963. The first units oscillated at a frequency of three cycles per minute, typically at  $\pm 3^\circ$  of arc. The dies (SCD) for these early cure meters were commonly a 2-in. square cavity 0.4 in. high, with a biconical rotor centered in the cavity. A typical rubber sample of 1.15 specific gravity weighed 22 g, and was loaded in two pieces, above and below the rotor. A 20 to 60-s preheat was required after closure before collecting data. The strain on the sample at  $3^\circ$  arc was 21 %.

X2.2 Frequencies of oscillation of 10, 100, and 900 cpm were made available over the next five years. These created different curve shapes due to the heat energy added to the cavity in working the rubber, breakdown of polymer structure when curing under dynamic conditions, and the shear rate dependence of the rubber flow resistance. Figure X2.1 compares the cure meter curves for the various speeds of oscillation using the square dies and an arc of  $\pm 3^\circ$ .

X2.3 When the oscillating disk cure meter was first proposed as an ASTM standard in 1968, a smaller, production-sized table model of the cure meter was introduced along with a new die that was 2 in. in diameter (LPC dies). This circular die had the same height and used the same rotor as the square dies. The LPC die produced similar torque values for minimum and maximum torques as the SCD die. The sample could be loaded as one piece on top of the rotor for most stocks. The practice of adding a preheat, as commonly used with the SCD die, was eliminated. The elimination of the preheat time led to slightly faster cures for the LPC die. Another advantage of this die was the flat lower die surface which allowed easier removal of the cured sample for most stocks.

X2.4 In 1971, a smaller version of the LPC die, called the MPC die, was adopted in conformance with Test Method D 2084. The MPC die used a rotor with a different conical angle. At  $3^\circ$  of arc, the strain is 48 % versus 21 % for the

larger dies and rotor. Higher strain leads to higher torques, and the smaller specimen (10 to 12 g for a stock with a specific gravity of 1.15) gives a cure curve with a significantly different shape, as shown in Fig. X2.2. Higher torques have been shown to cause slippage at the surface of the rotor for many stocks, and that slippage is a potential source of variation in test results. Studies conducted with the smaller dies and rotor indicated slippage due to rotor contamination was common above 50 in.-lbs of torque. As a result, Test Method D 2084 specifies a  $1^\circ$  arc of oscillation as standard with a 16 % shear strain.

X2.5 The curves obtained with MPC dies at  $1^\circ$  arc were used as standards until 1987, when further reductions in the temperature recovery time became possible due to improved temperature controllers.

X2.6 Another improvement introduced at this time was the reduction of the mechanical compliance of the rotor drive system for improved reproducibility between instruments. The improved mechanical design increased maximum torques, but lowered minimum torques by reducing friction. Faster temperature recovery has two advantages: first, the cure is closer to the desired cure temperature of the test for more accurate results; second, the potential for variation in results due to cooling of the rotor during loading and unloading is greatly reduced. Table X2.1 illustrates the potential operator effects.

X2.7 As part of the continuing evolution in cure meter design, a number of manufacturers have introduced rotorless cure meters. These cure meters use a sample of 3 to 5 g, with a thinner cross section to obtain more rapid temperature recovery and more uniform temperature throughout the specimen. The dies are usually directly heated, and smaller in mass than for the oscillating disk cure meters, so that faster temperature recovery can be achieved. By eliminating the rotor, the surface area under load is reduced so that smaller torque values are achieved. Faster temperature recovery leads to faster cure times. Figure X2.3 compares typical cure curves for an SBR stock from the rotorless and oscillating disk cure meters.

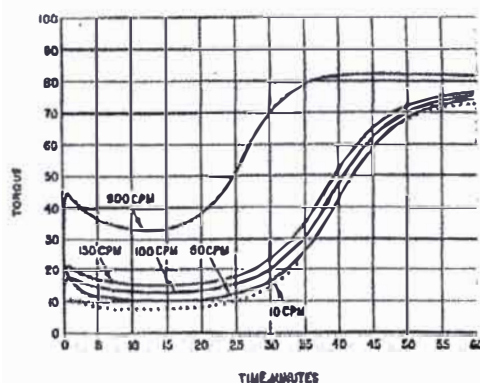


FIG. X2.1 Comparison of Oscillation Frequencies Using the Square Die and  $\pm 3^\circ$  Arc

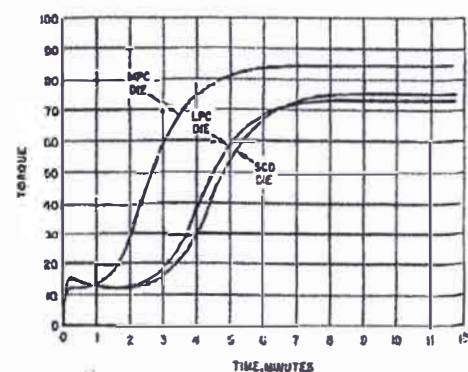


FIG. X2.2 Comparison of Cure Meter Dies

ASTM D 2084

TABLE X2.1 Operator Effects on ODR Cure Meter Tests

Sample Loading Variable	Cure Times, min		
	T'S2	T'60	T'80
<i>Slow Temperature Recovery:</i>			
Optimum loading time (20 s)	2.01	3.51	5.86
2-min loading time	2.33	3.90	6.33
% change with loading time	15.9	11.1	6.0
<i>Rapid Temperature Recovery:</i>			
Optimum loading time (20 s)	1.71	3.06	5.40
2-min loading time	1.86	3.23	5.60
% change with loading time	8.8	6.6	3.7

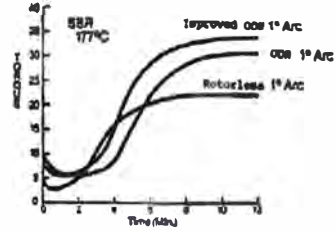


FIG. X2.3 Comparison of Cure Meters Using SBR Stock

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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, 1916 Race St., Philadelphia, PA 19103.

**Anexo7: Detalles del equipo, condiciones de ensayo, forma del espécimen,  
procedimiento y cálculos de abrasión según la ASTM D 3389 – 75.**



Designation: D 3389 – 75

**Standard Method for Testing,  
COATED FABRICS—ABRASION RESISTANCE  
(ROTARY PLATFORM, DOUBLE-HEAD  
ABRADER)<sup>1</sup>**

This Standard is issued under the fixed designation D 3389; 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.

**1. Scope**

1.1 This method covers the determination of the resistance to abrasion of fabrics coated with rubber or plastics. The abrasion is measured by mass loss.

**2. Summary of Method**

2.1 Abrasion resistance of fabrics coated with rubber or plastics is measured by subjecting the specimen to the rotary-rubbing action of two abrasive wheels under controlled conditions of pressure by the use of the revolving platform, double-head (RPDH) abrader<sup>2</sup> (Fig. 1). This action is maintained by the use of steel Type S-35 abrasive wheels.

**3. Time Lapse Between Manufacturing and Testing**

3.1 For all test purposes, the minimum time between manufacturing and testing should be 16 h.

3.2 For non-product tests the maximum time between manufacturing and testing should be 4 weeks. For evaluation intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

3.3 For product tests, whenever possible, the time between manufacturing and testing should not exceed 3 months. In all other cases, tests should be made within 2 months of the date of receipt by the customer.

**4. Apparatus**

4.1 *Abrasion Apparatus* (RPDH), comprised of a removable flat circular specimen holder, a pair of pivoted arms to which the

abrasive wheels are attached, a motor for rotating the platform and specimen, a fan for cooling the motor, and a counter to indicate the revolutions of the specimen holder. The specimen holder shall be mounted to produce a circular surface travel of an essentially flat specimen in the plane of its surface at a uniform angular velocity. The abrasive wheels, which are attached to the free end of the pivoted arms, shall rotate and have, when resting on the specimen, a peripheral engagement with the surface of the specimen, the direction of travel of the periphery of the wheels and of the specimen at the contacting portions being at acute angles, and the angle of travel of one wheel periphery being opposite to that of the other. Motion of the abrasive wheels, in opposite directions, shall be provided by rotation of the specimen and the associated friction therefrom.

4.1.1 The specimen holder shall be supported by an adapter that is motor-driven and that provides motion for the circular travel of the specimen holder.

4.1.2 A clamping ring shall be used to secure the specimen to the specimen holder.

4.1.3 The abrasive wheels shall be Type S-35 steel wheels and are mounted on independently pivoted arms, which provide free-floating ac-

<sup>1</sup> This method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.37 on Coated Fabrics and Rubber Thread.

<sup>2</sup> Current edition approved May 30, 1975. Published June 1975.

<sup>3</sup> The Laber Abrader and S-35 wheels, manufactured by the Laber Instrument Co., North Tonawanda, N. Y., meets these requirements.

## D 3389

tion to compensate for any minor unevenness in the specimen and ensure uniform pressure of the abrasion wheels against the specimen at all times.

4.1.4 The apparatus shall be provided with a vertical-force adjustment (weights) for varying the vertical force of the abrader wheels on the specimen. The pivoted abrader arms without auxiliary masses or counterweights apply a vertical force against the specimen of 2.45 N (250-gf) per wheel. A start on the rear end of the abrading arm may be used to carry a counterweight when it is desired to reduce the wheel load from 2.45 N (250 gf) to 1.23 N (125 gf) when testing delicate materials.

4.2 *Auxiliary Apparatus*—A stiff brush shall be provided for removal of loose particles from the surface of the wheels and a small vacuum cleaner attachment to remove the loose particles from the specimen during the test. Compressed air, which shall be free of moisture and oil, should be used for cleaning the surface of the specimen. The air is delivered to a manifold or nozzle where the pressure shall be maintained at 200 ± 35 kPa (30 ± 5 psi). The vacuum cleaner and air should be turned on and used throughout the test.

4.3 *Boluses*, suitable for weighing to the nearest 1 mg.

4.4 *Wheel Bearings*—The abrader wheel bearings, that is, the two pairs of bearings installed in the free end of the pivoting arms to support the abrader wheels, should not stick when caused to spin rapidly by a quick driving motion of the motor. The degree of freedom of rotation of these bearings, however, is not critical.

4.5 *Platform Position*—The vertical distance from the center of the pivot point of the abrader arms to the top of the specimen holder shall be approximately 25 mm (1.0 in.). This measurement is specified to prevent possibility of errors incurred by installing a thrust bearing or the like to support the specimen platform. Adjustments shall be made such that the platform will remain at the above specified level. The specimen platform shall rotate in the plane of its surface. If it fails to do so and exhibits a tendency to wobble, the holder and adapter shall be replaced or a thrust bearing installed to support the specimen holder.

4.6 *Platform Speed*—The speed of rotation

of the platform shall be  $7.0 \pm 0.11$  rad/s ( $70 \pm 1$  rpm).

## 5. Test Specimens

5.1 Unless otherwise specified, make five tests on each sample of coated fabrics.

5.2 Cut circular test specimens approximately 110 mm (4 1/4 in.) in diameter. Cut a 6-mm (1/4-in.) hole in the center of the specimen. Take care in cutting out specimens. Use the best portion of the sample to be tested. It should be free of holes, blisters, or other imperfections.

## 6. Procedure

6.1 Test the conditioned specimens in the standard atmosphere for testing,  $20 \pm 2.0^\circ\text{C}$  and  $65 \pm 3\%$  relative humidity, unless otherwise specified. On thin flexible materials that cannot be clamped to the specimen holder, it will be necessary to cement (Note 1) these specimens to some other substrate. A 10-ply white cardboard has been found satisfactory.

Note 1—A good rubber cement will be satisfactory, however, ensure that the cement used does not have any adverse effect on the fabric or coating. If a solvent-base cement is used, allow the assembly to condition at least overnight or until the assembly maintains constant mass.

6.2 Install the S-35 wheels in their respective flanged holders as indicated by the printing on the side of the wheel.

6.3 Determine the original mass of the specimen or the assembly, or both. Place the test specimen with its coated side up over the rubber mat on the specimen holder. Secure the washer and knurled nut in place to hold the center of the specimen. Place the ring clamp over the specimen and tighten the screw of the ring clamp.

6.4 The tester is equipped with a counter that operates in conjunction with the turntable. Set the counter at zero.

6.5 Start the abrader and run to the end point. The end point shall be defined as that point just before abrading through the coating to the fabric. First decide number of revolutions and the vertical force to be used by testing a specimen from each sample. The quality and the thickness of the coating will indicate the required vertical force and the number of revolutions needed to measure the abrasion resistance of the coating. After establishing the





## D 3389

required vertical force and number of revolutions, test the specified number of specimens for each sample. Do not abrade through the coating. This method is for testing the abrasion resistance of the coating only.

6.6 *Cleaning of Specimen*—Clean the specimen of abrasive particles on a scheduled basis. The vacuum cleaner, compressed air, and a brush should be used for this purpose. Wipe the rubber mat clean after each test.

6.7 At the conclusion of the test, weigh the specimen and report the mass loss as grams loss per revolution.

## 7. Calculation

7.1 Calculate the loss in mass as follows:

Mass loss per revolution, g -

$$\frac{\text{original mass (before test)} - \text{final mass (after test)}}{\text{number of revolutions}}$$

Example:

12.3596 Original Mass, g

12.2829 Final Mass, g

0.0767 Mass loss, g, for 500 revolutions

$$g = \frac{0.0767 \times 1000}{500} = 0.153 \text{ mg per revolution}$$

## 8. Report

8.1 The report shall include the following:

8.1.1 Test conditions,

8.1.2 Number of specimens tested,

8.1.3 Type of wheels,

8.1.4 Total revolutions and vertical force used, and

8.1.5 Mass loss per revolutions, mg

## 9. Precision

9.1 *Precision*—In Table 1 the coefficients of variation have been estimated for the loss in mass with the S-35 wheels over the ranges of test conditions, which denote machine loading and both rate and duration of abrasion, respectively. The maximum differences (ranges) should be considered acceptable for two results.

Note 2—These precision data are approximations based on limited data. Five specimens of each of six materials were tested over a 2-day period, by five laboratories, covering the range of test conditions and property levels tabulated above. Three of the materials, abraded at nearly identical rates, were utilized to estimate single operator precision at 10 degrees of freshness. Multilaboratory precision has been estimated at 17 degrees of freshness.

TABLE 1 Applicable Test Variable Ranges

	Single Operator (Repeatability)	Multilaboratory (Reproducibility)
Added load, g	500 to 1000	0 to 1000
Abrasion rate, mg per revolution	0.15	0.03 to 0.33
Total revolutions	500 to 2000	100 to 5000
Coefficient of variation, percent of mean, S %	17	20
Acceptable range of two results, percent of mean, D 25%	54	63

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, is entirely their own responsibility.

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 approved 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, 1916 Race St., Philadelphia, Pa. 19103, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.

## Anexo 8: Detalles de dimensionamiento del espécimen de prueba, tipo de indentores, procedimientos y reportes de ensayo según ASTM D 2240-02.



Designation: D 2240 – 02

### Standard Test Method for Rubber Property—Durometer Hardness<sup>1</sup>

This standard is issued under the fixed designation D 2240; 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 describes eight types of rubber hardness measurement devices known as durometers; types A, B, C, D, DQ, O, OO and M. The procedure for determining indentation hardness of substances classified as thermoplastic elastomers, vulcanized (thermoset) rubber, elastomeric materials, cellular materials, and some plastics is also described.

1.2 This test method is not equivalent to other indentation hardness methods and instrument types, specifically those described in Test Method D 1415.

1.3 This test method is not applicable to the testing of coated fabrics.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only. Many of the stated dimensions in SI are direct conversions from the U. S. Customary System to accommodate the instrumentation, practices and procedures that existed prior to the Metric Conversion Act of 1975.

1.5 All materials, instruments, or equipment used for the determination of mass, force or dimension shall have traceability to the National Institute for Standards and Technology, or other internationally recognized organization parallel in nature.

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:

D 374 Test Methods for Thickness of Solid Electrical Insulation<sup>2</sup>

D 638 Practice for Conditioning Plastics for Testing<sup>3</sup>

D 785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials<sup>3</sup>

D 1349 Practice for Rubber—Standard Temperatures For Testing<sup>4</sup>

D 1415 Test Method for Rubber Property—International Hardness<sup>4</sup>

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>4</sup>

F 1957 Test Method for Composite Foam Hardness—Durometer Hardness<sup>5</sup>

#### 3. Summary of Test Method

3.1 This test method permits hardness measurements based on either initial indentation or indentation after a specified period of time, or both. Durometers with maximum reading indicators used to determine maximum hardness values of a material may yield lower hardness when the maximum indicator is used.

3.2 The procedures for Type M, or micro hardness durometers, accommodate specimens that are, by their dimensions or configuration, ordinarily unable to have their durometer hardness determined by the other durometer types described. Type M durometers are intended for the testing of specimens having a thickness or cross sectional diameter of 1.25 mm (0.050 in.) or greater, although specimens of lesser dimensions may be successfully accommodated under the conditions specified in Section 6, and have a Type M durometer hardness range of between 20 and 90. Those specimens which have a durometer hardness range other than specified shall use another suitable procedure for determining durometer hardness.

#### 4. Significance and Use

4.1 This test method is based on the penetration of a specific type of indenter when forced into the material under specified conditions. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behavior of the material. *The geometry of the indenter and the applied force influence the measurements such that no simple relationship exists between the measurements obtained with one type of durometer and those obtained with another type of durometer or other instruments used for measuring hardness.* This test method is an empirical test

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing. Current edition approved Jan 10, 2002. Published March 2002. Originally published as D 2240 - 64 T. Last previous edition D 2240 - 00.

<sup>2</sup> Annual Book of ASTM Standards, Vol 10.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 09.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 15.07.

ASME D 2240

intended primarily for control purposes. No simple relationship exists between indentation hardness determined by this test method and any fundamental property of the material tested. For specification purposes it is recommended that Test Method D 785 be used for materials other than those described in 1.1.

5. Apparatus

5.1 Hardness measuring apparatus, or durometer, and an operating stand, Type 1, Type 2, or Type 3, refer to 5.1.2, consisting of the following components:

5.1.1 Durometer:

5.1.1.1 *Presser Foot*, with an orifice (to allow for the protrusion of the indenter) having a diameter as specified in Fig. 1a, Fig. 1b, or Fig. 1c, with the center a minimum of 6.0 mm (0.24 in.) from any edge of the foot.

5.1.1.2 *Presser Foot, Type M*, with an orifice (to allow for the protrusion of the indenter) having a diameter as specified in Fig. 1d, with the center a minimum of 1.60 mm (0.063 in.) from any edge of the flat circular presser foot.

5.1.1.3 *Indenter*, formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1a, Fig. 1b, or Fig.

1c, polished over the contact area so that no flaws are visible under 20x magnification, with an indenter extension of  $2.50 \pm 0.04$  mm ( $0.098 \pm 0.002$  in.).

5.1.1.4 *Indenter, Type M*, formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1d, polished over the contact area so that no flaws are visible under 50x magnification, with an indenter extension of  $1.25 \pm 0.02$  mm ( $0.049 \pm 0.001$  in.).

5.1.1.5 *Indenter Extension Indicator*, (analog or digital electronic), having a display that is an inverse function of the indenter extension. The display shall indicate from 0 to 100 with equal divisions throughout the range at a rate of one hardness point for each 0.025 mm (0.001 in.) of indenter movement, for Type M durometers, the display shall indicate from 0 to 100 with no less than 100 equal divisions at a rate of one hardness point for each 0.0125 mm (0.0005 in.) of indenter movement.

5.1.1.6 *Timing Device (optional)*, capable of being set to a desired elapsed time, signaling the operator or holding the hardness reading when the desired elapsed time has been reached. The timer shall be automatically activated when the

Fig 1d

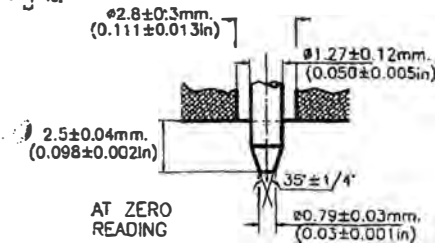


Figure 1a Type A and C Indentor

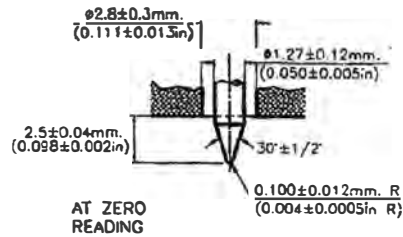


Figure 1b Type B and D Indentor

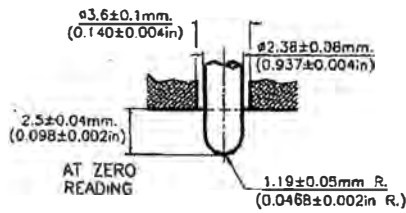


Figure 1c O, DO, and oo Indentor

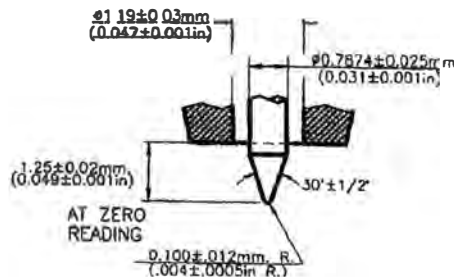


Figure 1d Type M Indentor

FIG. 1 Durometers

D 2240

presser foot is in contact with the specimen being tested, for example, the initial indenter travel has ceased. Digital electronic durometers may be equipped with electronic timing devices that shall not affect the indicated reading or determinations attained by more than one half the calibration tolerance stated in Table 1.

5.1.1.7 *Maximum Indicators (optional)*, maximum indicating pointers are auxiliary analog indicating hands designed to remain at the maximum hardness value attained until reset by the operator. Electronic maximum indicators are digital displays electronically indicating and maintaining the maximum value hardness value achieved, until reset by the operator.

5.1.1.8 Analog maximum indicating pointers have been shown to have a nominal effect on the values attained, however this effect is greater on durometers of lesser total mainspring loads, for example, the effect of a maximum indicating pointer on Type D durometer determinations will be less than those determinations achieved using a Type A durometer. Analog style durometers may be equipped with maximum indicating pointers. The effect of a maximum indicating pointer shall be noted at the time of calibration in the calibration report, refer to 10.1.4, and when reporting hardness determinations, refer to 10.2.4. Analog Type M durometers shall not be equipped with maximum indicating pointers.

5.1.1.9 Digital electronic durometers may be equipped with electronic maximum indicators that shall not affect the indicated reading or determinations attained by more than one half of the spring calibration tolerance stated in Table 1.

5.1.1.10 *Calibrated Spring*, for applying force to the indenter, in accordance with Fig. 1a through Fig. 1d and capable of applying the forces as specified in Table 1.

5.1.2 *Operating Stand:*

5.1.2.1 Type 1, Type 2, and Type 3, shall be capable of supporting the durometer presser foot surface parallel to the specimen support table (Fig. 2) throughout the travel of each. The durometer presser foot to specimen support table parallelism shall be verified each time the test specimen support table is adjusted to accommodate specimens of varying dimensions. This may be accomplished by applying the durometer presser foot to the point of contact with the specimen support table and making adjustments by way of the durometer mounting assembly or as specified by the manufacturer.

5.1.2.2 *Operating Stand, Type 1* (specimen to indenter type), shall be capable of applying the specimen to the indenter

TABLE 1 Durometer Spring Force Calibration  
All Values are in N

Indicated Value	Type M	Type A, B, O	Type C, D, DO	Type OO
0	0.324	0.55		0.203
10	0.368	1.3	4.445	0.294
20	0.412	2.05	8.89	0.385
30	0.456	2.8	13.335	0.478
40	0.5	3.55	17.78	0.568
50	0.544	4.3	22.225	0.657
60	0.588	5.05	26.67	0.748
70	0.633	5.8	31.115	0.839
80	0.677	6.55	35.56	0.93
90	0.721	7.3	40.005	1.02
100	0.766	8.05	44.45	1.111
Durometer unit	0.0044	0.075	0.4445	0.00908
Spring Calibration Tolerance	±0.0176 N	±0.075 N	±0.4445 N	±0.0182 N

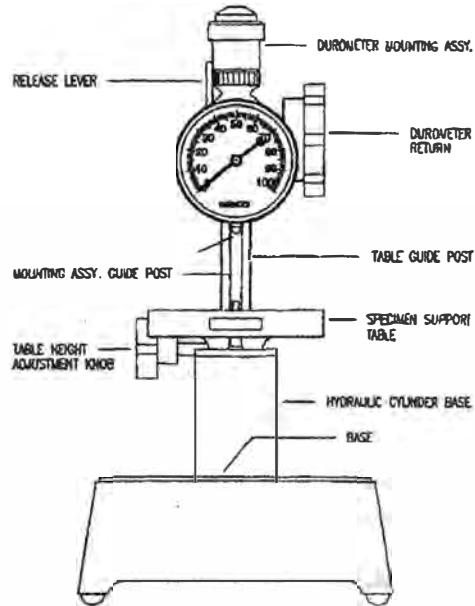


FIG. 2 Durometer Operating Stand

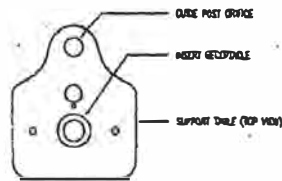
in a manner that minimizes shock.

5.1.2.3 *Operating Stand, Type 2* (indenter to specimen type), shall be capable of controlling the rate of descent of the indenter to the specimen at a maximum of 3.20 mm/s (0.125 in/s) and applying a force sufficient to overcome the calibrated spring force as shown in Table 1.

5.1.2.4 *Operating Stand, Type 3* (indenter to specimen type), hydraulic dampening, pneumatic dampening or electro-mechanical (required for the operation of Type M durometers) shall be capable of controlling the rate of descent of the indenter to the specimen at a maximum of 3.2 mm/s (0.125 in/s) and applying a force sufficient to overcome the calibrated spring force as shown in Table 1. Manual application, Type 1 or Type 2 operating stands are not acceptable for Type M durometer operation.

5.1.2.5 The entire instrument should be plumb and level, and resting on a surface that will minimize vibration. Operating the instrument under adverse conditions will negatively affect the determinations attained.

5.1.2.6 *Specimen Support Table*, (Fig. 2) integral to the operating stand, and having a solid flat surface. The specimen support platform may have orifices designed to accept various inserts or support fixtures (Fig. 3) to provide for the support of irregularly configured specimens. When inserts are used to support test specimens, care must be taken to align the indenter to the center of the insert, or the point at which the indenter is to contact the specimen. Care should be exercised to assure that the indenter does not abruptly contact the specimen support

 D 2240


TYPICAL TABLE FIXTURE USED FOR POSITIONING TUBES, O-RINGS AND SMALL SPECIMENS

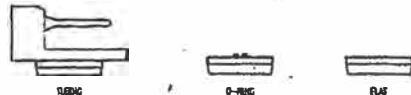


FIG. 3 Small Specimen Support Table

table as damage to the indenter may result.

#### 6. Test Specimen

6.1 The test specimen, herein referred to as "specimen" or "test specimen" interchangeably, shall be at least 6.0 mm (0.24 in.) in thickness unless it is known that results equivalent to the 6.0 mm (0.24 in.) values are obtained with a thinner specimen.

6.1.1 A specimen may be composed of plied pieces to obtain the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens as the surfaces of the plied specimens may not be in complete contact. The lateral dimensions of the specimen shall be sufficient to permit measurements at least 12.0 mm (0.48 in.) from any edge unless it is known that identical results are obtained when measurements are made at a lesser distance from an edge.

6.1.2 The surfaces of the specimen shall be flat and parallel over an area to permit the presser foot to contact the specimen over an area having a radius of at least 6.0 mm (0.24 in.) from the indenter point. The specimen shall be suitably supported to provide for positioning and stability. *A suitable hardness determination cannot be made on an uneven or rough point of contact with the indenter.*

6.2 Type M test specimens should be at least 1.25 mm (0.05 in.) in thickness unless it is known that results equivalent to the 1.25 mm (0.05 in.) values are obtained with a thinner specimen.

6.2.1 A Type M specimen that is not of a configuration described in 6.2.2 may be composed of plied pieces to obtain the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens because the surfaces of the plied specimens may not be in complete contact. The lateral dimensions of the specimen should be sufficient to permit measurements at least 2.50 mm (0.10 in.) from any edge unless it is known that identical results are obtained when measurements are made at lesser distance from an edge. *A suitable hardness determination cannot be made on an uneven or rough point of contact with the indenter.*

6.2.2 The Type M specimen, when configured as an o-ring, circular band, or other irregular shape shall be at least 1.25 mm (0.05 in.) in cross sectional diameter, unless it is known that results equivalent to the 1.25 mm (0.05 in.) values are obtained with a thinner specimen. The specimen shall be suitably supported in a fixture (Fig. 3) to provide for positioning and stability.

6.3 The minimum requirement for the thickness of the specimen is dependent on the extent of penetration of the indenter into the specimen; for example, thinner specimens may be used for materials having higher hardness values. The minimum distance from the edge at which measurements may be made likewise decreases as the hardness increases.

#### 7. Calibration

##### 7.1 Indenter Extension Adjustment Procedure:

7.1.1 Place precision ground dimensional blocks (Grade B or better) on the support table and beneath the durometer presser foot and indenter. Arrange the blocks so that the durometer presser foot contacts the larger block(s) and the indenter tip just contacts the smaller block (Fig. 4). It is necessary to observe the arrangement of the blocks and the presser foot/indenter under a minimum of 20X magnification to assure proper alignment.

7.1.2 Indenter extension and shape must be in accordance with 5.1.1.3 or 5.1.1.4 respective to durometer type (Fig. 1a through Fig. 1d). Examination of the indenter under 20X magnification, 50X for Type M indentors, is required to examine the indenter condition. Misshapen or damaged indentors shall be replaced.

7.1.3 A combination of dimensional gage blocks shall be used to achieve a difference of 2.54 mm +0.00 -0.0254 mm (0.100 in +0.00 -0.001 in.) between them. For Type M durometers the gage block dimensions are 1.27 mm +0.0 -0.0127 mm (0.050 in +0.00 -0.0005 in.) between them (Fig. 4).

7.1.4 Carefully lower the durometer presser foot until it contacts the largest dimensional block(s), the indenter tip should just contact the smaller block, verifying full indenter extension.

7.1.5 Adjust the indenter extension to 2.50 ± 0.04 mm (0.098 ± 0.002 in.). For Type M durometers, adjust the indenter extension to 1.25 ± 0.02 mm (0.049 ± 0.001 in.), following the manufacturer's recommended procedure.

7.1.5.1 When performing the procedures in 7.1, care should be used so as not to cause damage to the indenter tip. Fig. 4

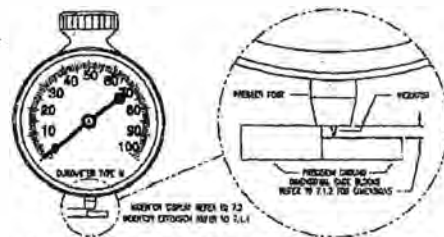


FIG. 4 Detail of Indenter Extension & Display Adjustment




**D 2240**

between laboratories or between supplier and user and are in accordance with alternative procedures identified in Practice D 618.

8.5 No conclusive evaluation has been made on durometers at temperatures other than  $23.0 \pm 2.0^\circ\text{C}$  ( $73.4 \pm 3.6^\circ\text{F}$ ). Conditioning at temperatures other than the above may show changes in calibration. Durometer use at temperatures other than the above should be decided locally (see Practice D 1349).

## 9. Procedure

### 9.1 Operating Stand Operation (Type 3 Operating Stand Required for Type M):

9.1.1 Care shall be exercised to minimize the exposure of the instrument to environmental conditions that are adverse to the performance of the instrument, or adversely affect test results.

9.1.2 Adjust the presser foot to support table parallelism as described in 5.1.2.1. It is necessary to make this adjustment each time the support table is moved to accommodate specimens of varying dimensions.

9.1.3 Prior to conducting a test, adjust the vertical distance from the presser foot to the contact surface of the test specimen to  $25.4 \pm 2.5$  mm ( $1.00 \pm 0.100$  in.), unless it is known that identical results are obtained with presser foot at a greater or lesser vertical distance from the test specimen contact surface, or if otherwise stipulated by the manufacturer.

9.1.4 Place the specimen on the specimen support table, in a manner that the contact point of the indenter is in accordance with Section 6, unless it is known that identical results obtained when measurements are made with the indenter lesser distance from the edge of the test specimen.

9.1.5 Actuate the release lever (Fig. 2) of the operating stand or activate the electromechanical device, allowing the durometer to descend at a controlled rate and apply the presser foot to the specimen in accordance with 5.1.2. In the case of "specimen to indenter" type operating stands, operate the lever or other mechanism to apply the specimen to the indenter in a manner that assures parallel contact of the specimen to the durometer presser foot without shock and with just sufficient force to overcome the calibrated spring force as shown in Table 1.

9.1.6 An operating stand that applies the mass at a controlled rate of descent, without shock is mandatory for Type M durometers. Hand held application or the use of a Type 1 or Type 2 operating stands for the Type M durometer is not an acceptable practice, refer to 5.1.2.4.

9.1.7 For any material covered in 1.1, once the presser foot is in contact with the specimen, for example, the initial indenter travel has ceased, the indicated reading shall be recorded within  $1 \pm 0.1$  s, or after any period of time agreed upon among laboratories or between supplier and user. If the durometer is equipped with a maximum indicator, the maximum indicated reading shall be recorded within  $1 \pm 0.1$  s of the cessation of indenter travel. The indicated hardness reading may change with time.

9.1.8 Make five determinations of hardness at different positions on the specimen at least 6.0 mm (0.24 in.) apart, 0.80 mm (0.030 in.) apart for Type M; and calculate the arithmetic mean, or alternatively calculate the median. The means of

calculating the determinations shall be reported according to 10.2.8

### 9.2 Manual (Hand Held) Operation of Durometer:

9.2.1 Care shall be exercised to minimize the exposure of the instrument to environmental conditions that are adverse to the performance of the instrument, or adversely affect test results.

9.2.2 Place the specimen on a flat, hard, horizontal surface. Hold the durometer in a vertical position with the indenter tip at a distance from any edge of the specimen as described in Section 6, unless it is known that identical results are obtained when measurements are made with the indenter at a lesser distance.

9.2.3 Apply the presser foot to the specimen, maintaining it in a vertical position keeping the presser foot parallel to the specimen, with a firm smooth downward action that will avoid shock, rolling of the presser foot over the specimen, or the application of lateral force. Apply sufficient pressure to assure firm contact between the presser foot and the specimen.

9.2.4 For any material covered in 1.1, after the presser foot is in contact with the specimen, the indicated reading shall be recorded within  $1 \pm 0.1$  s, or after any period of time agreed upon among laboratories or between supplier and user. If the durometer is equipped with a maximum indicator, the maximum indicated reading shall be recorded within  $1 \pm 0.1$  s of the cessation of initial indenter travel. The indicated hardness reading may change with time.

9.2.5 Make five determinations of hardness at different positions on the specimen at least 6.0 mm (0.24 in.) apart and calculate the arithmetic mean, or alternatively calculate the median. The means of calculating the determinations shall be reported according to Section 10.2.8.

9.3 It is acknowledged that durometer readings below 20 or above 90 are not considered reliable. It is suggested that readings in these ranges not be recorded.

9.4 Manual operation (hand held) of a durometer will cause variations in the results attained. Improved repeatability may be obtained by using a mass, securely affixed to the durometer and centered on the axis of the indenter. Recommended masses are 1 kg for Type A, B and O durometers, 5 kg for Type C, D and DO durometers, and 400 g for Type OO durometers. Further improvement may be achieved by the use of a durometer operating stand which controls the rate of descent of the durometer presser foot to the test specimen and incorporates the masses described above.

## 10. Report

### 10.1 Instrument Calibration Report (Durometer or Operating Stand):

10.1.1 Date of calibration.

10.1.2 Date of last calibration.

10.1.3 Manufacturer, type, model, and serial number of the instrument, and a notation when a maximum indicator or timing device is present.

10.1.4 Values obtained (pre- and post calibration results), including a notation of the affect of a maximum indicator, if present. The method of reporting the calibrated value shall be by attaining the arithmetic mean of the determinations.

10.1.5 Ambient temperature.

## D 2240

TABLE 2 Type 1 Precision—Type M Durometer Method

Material	Within Laboratories			Between Laboratories			
	MEAN	Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	31.8	1.26	3.68	11.24	3.76	10.83	38.41
2	40.8	1.14	3.23	7.90	2.47	7.00	17.13
3	54.0	0.975	2.78	6.11	2.35	6.73	12.48
4	62.5	0.782	2.21	3.62	2.24	6.34	10.10
5	70.9	0.709	2.01	2.83	0.974	2.78	3.89
6	80.6	1.888	4.77	5.92	1.81	4.56	5.65
7	87.7	1.15	3.25	3.71	2.63	7.45	8.50
8	32.4	0.947	2.68	8.28	3.64	10.29	31.73
9	41.8	0.797	2.26	5.40	2.23	6.31	15.11
10	53.3	0.669	1.89	3.55	2.29	6.49	12.17
11	63.2	0.485	1.37	2.17	2.19	6.20	9.80
12	89.8	0.737	2.08	3.00	0.89	2.80	4.02
13	78.3	0.784	2.22	2.64	1.04	2.84	3.76
14	87.6	1.121	3.17	3.62	2.65	7.49	8.55
15	34.1	0.85	2.40	7.05	1.84	5.20	15.25
16	42.3	0.635	1.80	4.25	1.20	3.39	8.01
17	54.6	0.86	1.69	2.60	2.18	6.09	11.16
18	62.9	1.12	3.17	5.04	1.47	4.18	6.81
19	70.3	0.669	1.95	2.77	0.944	2.57	3.80
20	81.7	0.463	1.37	1.67	1.10	3.10	3.80
21	67.9	0.679	2.49	2.83	2.07	5.88	6.67
AVERAGE	61.4						
POOLED VALUES		0.924	2.62	4.26	2.146	6.07	9.89

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

TABLE 3 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	51.4	0.640	1.83	3.66	1.36	4.41	8.59
2	65.3	0.878	2.48	3.61	2.21	6.06	9.27
3	68.0	0.433	1.23	1.80	2.28	6.45	9.49
Pooled	61.8	0.877	1.92	3.11	2.018	5.72	9.28

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

TABLE 4 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	42.6	0.316	0.894	2.10	2.62	7.98	16.7
2	54.5	0.797	2.24	4.11	3.64	10.0	16.4
3	82.3	1.01	2.86	3.47	3.54	10.0	12.2
Pooled	69.8	0.782	2.16	3.61	3.32	8.40	15.7

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

## 10.1.6 Relative humidity.

## 10.1.7 Technician identification.

## 10.1.8 Applicable standards to which the instrument is calibrated.

10.1.9 Calibrating instrument information to include type, serial number, manufacturer, date of last calibration, and a statement of traceability of standards used to NIST or other acceptable organization. See 1.5.

## 10.2 Hardness Measurement Report.

## 10.2.1 Date of test.

## 10.2.2 Relative humidity.

## 10.2.3 Ambient temperature.

10.2.4 Manufacturer, type and serial number of the durometer or operating stand, or both, including a notation when a maximum indicator or timing device is present, and date of last calibration.

10.2.5 Means of testing, whether manual (hand held), Type 1 operating stand (specimen to indenter), Type 2 operating stand (indenter to specimen type), or Type 3 operating stand (electromechanical or hydraulically dampened).

10.2.6 Description of test specimen, including thickness, number of pieces plied if less than the thickness indicated in Section 6, including the vulcanization date.

## 10.2.7 Complete identification of material tested.

10.2.8 Hardness value obtained and method of calculation, either arithmetic mean or alternatively, the median.

10.2.9 Indentation hardness time interval at which determination was made. Readings may be reported in the form: M/60/l where M is the type of durometer, 60 the reading, and l the time in seconds that the presser foot is in contact with the specimen or from an electronic timing device.

## 11. Precision and Bias

11.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Refer to this Practice for terminology and other testing and statistical concepts.

11.2 The Type 1 precision for the Type M method was determined from an interlaboratory program with 21 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for the Type M testing program. All materials were supplied from a single source, being those commonly supplied as reference materials with the instruments from the manufacturer.

11.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described above. The precision parameters should not be used for acceptance or rejection testing, or both, of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

11.4 The Type 1 precision for both Type A and D methods was determined from an interlaboratory program with 3 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for both A and D testing programs. All materials were supplied from a single source.

11.5 A test result for hardness, for Types A, D, and M, was the median of five individual hardness readings on each day in each laboratory.

11.6 Table 2 shows the precision results for Type M method<sup>6</sup>, Table 3 shows the precision results for Type A

<sup>6</sup> Supporting data are available from ASTM International Headquarters. Request RR: D11-1091.





D 2240

TABLE 2 Type 1 Precision—Type M Durometer Method

Material	Within Laboratories			Between Laboratories			
	MEAN	Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	31.0	1.28	3.68	11.24	3.76	10.83	33.41
2	40.0	1.14	3.23	7.80	2.47	7.00	17.13
3	54.0	0.975	2.76	5.11	2.38	6.73	12.46
4	62.0	0.782	2.21	3.62	2.24	6.34	10.10
5	70.9	0.700	2.01	2.83	0.974	2.76	3.89
6	80.8	1.888	4.77	6.92	1.81	4.66	5.85
7	87.7	1.15	3.25	3.71	2.63	7.46	6.60
8	32.4	0.947	2.68	6.26	3.64	10.29	31.73
9	41.6	0.797	2.26	5.40	2.23	6.31	15.11
10	53.3	0.669	1.89	3.55	2.29	6.49	12.17
11	63.2	0.485	1.37	2.17	2.49	6.20	9.80
12	89.8	0.737	2.08	3.00	0.88	2.80	4.02
13	78.3	0.784	2.22	2.64	1.04	2.94	3.75
14	87.6	1.121	3.17	3.62	2.65	7.49	8.55
15	34.1	0.85	2.40	7.05	1.84	5.20	15.25
16	42.3	0.885	1.80	4.25	1.20	3.39	6.01
17	54.8	0.56	1.59	2.90	2.15	6.09	11.15
18	62.9	1.12	3.17	6.04	1.47	4.18	6.61
19	70.3	0.888	1.95	2.77	0.944	2.67	3.80
20	81.7	0.483	1.37	1.67	1.10	3.10	3.80
21	87.9	0.879	2.49	2.83	2.07	5.66	6.67
AVERAGE	61.4						
POOLED VALUES		0.924	2.62	4.26	2.146	6.07	9.89

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

TABLE 3 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	51.4	0.646	1.83	3.56	1.56	4.41	6.69
2	65.3	0.878	2.48	3.81	2.21	6.06	9.27
3	68.0	0.433	1.23	1.80	2.28	6.45	9.49
Pooled	61.8	0.677	1.92	3.11	2.018	5.72	9.28

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

TABLE 4 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr <sup>a</sup>	r <sup>b</sup>	(r) <sup>c</sup>	SR <sup>d</sup>	R <sup>e</sup>	(R) <sup>f</sup>
1	42.8	0.316	0.894	2.10	2.82	7.98	18.7
2	54.5	0.791	2.24	4.11	3.54	10.0	18.4
3	62.3	1.01	2.88	3.47	3.54	10.0	12.2
Pooled	60.8	0.762	2.16	3.61	3.32	9.40	16.7

- <sup>a</sup> Sr = repeatability standard deviation, measurement units.  
<sup>b</sup> r = repeatability = 2.83 × Sr, measurement units.  
<sup>c</sup> (r) = repeatability, relative, (that is, in percent).  
<sup>d</sup> SR = reproducibility standard deviation, measurement units.  
<sup>e</sup> R = reproducibility = 2.83 × SR, measurement units.  
<sup>f</sup> (R) = reproducibility, relative, (that is, in percent).

10.1.6 Relative humidity.

10.1.7 Technician identification.

10.1.8 Applicable standards to which the instrument is calibrated.

10.1.9 Calibrating instrument information to include type, serial number, manufacturer, date of last calibration, and a statement of traceability of standards used to NIST or other acceptable organization. See 1.5.

## 10.2 Hardness Measurement Report:

10.2.1 Date of test.

10.2.2 Relative humidity.

10.2.3 Ambient temperature.

10.2.4 Manufacturer, type and serial number of the durometer or operating stand, or both, including a notation when a maximum indicator or timing device is present, and date of last calibration.

10.2.5 Means of testing, whether manual (hand held), Type 1 operating stand (specimen to indenter), Type 2 operating stand (indenter to specimen type), or Type 3 operating stand (electromechanical or hydraulically damped).

10.2.6 Description of test specimen, including thickness, number of pieces plied if less than the thickness indicated in Section 6, including the vulcanization date.

10.2.7 Complete identification of material tested.

10.2.8 Hardness value obtained and method of calculation, either arithmetic mean or alternatively, the median.

10.2.9 Indentation hardness time interval at which determination was made. Readings may be reported in the form: M/60/l where M is the type of durometer, 60 the reading, and l the time in seconds that the presser foot is in contact with the specimen or from an electronic timing device.

## 11. Precision and Bias

11.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Refer to this Practice for terminology and other testing and statistical concepts.

11.2 The Type 1 precision for the Type M method was determined from an interlaboratory program with 21 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for the Type M testing program. All materials were supplied from a single source, being those commonly supplied as reference materials with the instruments from the manufacturer.

11.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described above. The precision parameters should not be used for acceptance or rejection testing, or both, of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

11.4 The Type 1 precision for both Type A and D methods was determined from an interlaboratory program with 3 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for both A and D testing programs. All materials were supplied from a single source.

11.5 A test result for hardness, for Types A, D, and M, was the median of five individual hardness readings on each day in each laboratory.

11.6 Table 2 shows the precision results for Type M method<sup>5</sup>, Table 3 shows the precision results for Type A<sup>5</sup> Supporting data are available from ASTM International Headquarters. Request RR: D11-1091.

 D 2240

method<sup>7</sup>, and Table 4 gives the precision results for Type D method<sup>7</sup>.

11.7 **Precision**—The precision of this test method may be expressed in the format of the following statements which use as appropriate value  $r$ ,  $R$ , ( $r$ ), or ( $R$ ), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of  $r$  or  $R$  associated with a mean level in Table 1 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

11.7.1 **Repeatability**—The repeatability,  $r$ , of these test methods has been established as the appropriate value tabulated in Tables 2-4. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated  $r$  (for any given level) must be considered as derived from different or non-identical sample populations.

11.7.2 **Reproducibility**—The reproducibility,  $R$ , of these test methods has been established as the appropriate value tabu-

<sup>7</sup> Supporting data are available from ASTM International Headquarters. Request RR: D11-1029.

lated in Tables 2-4. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated  $R$  (for any given level) must be considered to have come from different or non-identical sample populations.

11.7.3 Repeatability and reproducibility are expressed as a percentage of the mean level, ( $r$ ) and ( $R$ ), have equivalent application statements as above for  $r$  and  $R$ . For the ( $r$ ) and ( $R$ ) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.8 **Bias**—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by this test method. Bias, therefore cannot be determined.

## 12. Keywords

12.1 durometer; durometer hardness; hardness; indentation hardness; micro durometer hardness

## APPENDICES

### (Nonmandatory Information)

#### X1. DUROMETER SELECTION GUIDE

X1.1 The durometer selection guide is designed to assist in the selection of the proper durometer type for various applications.

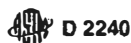
X1.2 It is generally recognized that durometer hardness determination below 20 and above 90 are unreliable. It is

recommended that the next lower or higher type (scale) be used in these situations.

X1.3 It is also recommended that, whenever possible, an operating stand be employed in performing durometer hardness tests.

TABLE X1.1 Durometer Selection: Typical Uses

Type (Scale)	Typical Examples of Materials Tested	Durometer Hardness (Typical Uses)
A	Soft vulcanized rubber, natural rubber, nitriles, thermoplastic elastomers, flexible polycrylics and thermosets, wax, felt, and leathers	20-90 A
B	Moderately hard rubber, thermoplastic elastomers, paper products, and fibrous materials	Above 90 A Below 20 D
C	Medium-hard rubber, thermoplastic elastomers, medium-hard plastics and thermoplastics	Above 90 B Below 20 D
D	Hard rubber, thermoplastic elastomers, harder plastics, and rigid thermoplastics	Above 90 A
DO	Moderately hard rubber, thermoplastic elastomers, and very dense textile windings	Above 90 C Below 20 D
M	Thin, irregularly shaped rubber, thermoplastic elastomer, and plastic specimens	20-85 A
O	Soft rubber, thermoplastic elastomers, very soft plastics and thermoplastics, medium-density textile windings	Below 20 DO
OO	Extremely soft rubber, thermoplastic elastomers, sponge, extremely soft plastics and thermoplastics, foams, low-density textile windings, human and animal tissue	Below 20 O
CF	Composite foam materials such as amusement ride safety cushions, vehicle seats, dashboards, headrests, armrests, and door panels	See Test Method F 1967



D 2240

## X2. RELATED TEST METHODS

C 367 Test Methods for Strength Properties of Prefabricated Architectural Acoustical Tile or Lay-In Ceiling Panels<sup>8</sup>

C 473 Test Methods for Physical Testing of Gypsum Panel Product<sup>9</sup>

C 581 Practice for Determining Chemical Resistance of Thermosetting Resins Used in Glass-Fiber-Reinforced Structures Intended for Liquid Service<sup>10</sup>

C 661 Test Method for Indentation Hardness of Elastomeric-Type Sealants by Means of a Durometer<sup>11</sup>

C 836 Specification for High Solids Content, Cold Liquid-Applied Elastomeric Waterproofing Membrane for Use with Separate Wearing Course<sup>11</sup>

D 461 Test Methods for Felt<sup>12</sup>

D 531 Test Method for Rubber Property—Pusey and Jones Indentation<sup>4</sup>

D 619 Test Methods for Vulcanized Fibre Used for Electrical Insulation<sup>2</sup>

D 1037 Test Methods for Evaluating Properties of Wood-Base Fiber and Particle Panel Materials<sup>13</sup>

D 1054 Test Method for Rubber Property—Resiliance Using a Rebound Pendulum<sup>4</sup>

D 1414 Test Methods for Rubber O-Rings<sup>14</sup>

D 1474 Test Methods for Indentation Hardness of Organic Coatings<sup>15</sup>

D 2134 Test Method for Determining the Hardness of Organic Coatings with a Sward-Type Hardness Rocker<sup>15</sup>

D 2287 Specification for Nonrigid Vinyl Chloride Polymer and Copolymer Molding and Extrusion Compounds<sup>3</sup>

D 2583 Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor<sup>16</sup>

D 2632 Test Method for Rubber Property—Resiliance by Vertical Rebound<sup>4</sup>

D 4289 Test Method for Elastomer Compatibility of Lubricating Greases and Fluids<sup>17</sup>

D 5672 Test Method for Testing Flexible Cellular Materials Measurement of Indentation Force Deflection Using a 25 mm (1 in.) Deflection Technique<sup>18</sup>

D 6546 Test Methods for and Suggested Limits for Determining Compatibility of Elastomer Seals for Industrial Hydraulic Fluid Applications<sup>19</sup>

F 1151 Test Method for Determining Variations in Hardness of Film Ribbon Pancakes<sup>20</sup>

Note X2.1—The hardness testing of other nonmetallic materials may be under the jurisdiction of one or more ASTM committees; the respective committee should be contacted for specific information.

<sup>8</sup> Annual Book of ASTM Standards, Vol 04.06.

<sup>9</sup> Annual Book of ASTM Standards, Vol 04.01.

<sup>10</sup> Annual Book of ASTM Standards, Vol 08.04.

<sup>11</sup> Annual Book of ASTM Standards, Vol 04.07.

<sup>12</sup> Annual Book of ASTM Standards, Vol 07.01.

<sup>13</sup> Annual Book of ASTM Standards, Vol 04.10.

<sup>14</sup> Annual Book of ASTM Standards, Vol 09.02.

<sup>15</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>16</sup> Annual Book of ASTM Standards, Vol 08.02.

<sup>17</sup> Annual Book of ASTM Standards, Vol 05.02.

<sup>18</sup> Annual Book of ASTM Standards, Vol 08.03.

<sup>19</sup> Annual Book of ASTM Standards, Vol 05.04.

<sup>20</sup> Annual Book of ASTM Standards, Vol 15.09.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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 International 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, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19380-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9586 (phone), 610-832-9555 (fax), or service@astm.org (e-mail), or through the ASTM website (www.astm.org).

## Anexo 9: Apartado de ASTM D 412-92, referente a especificaciones del equipo auxiliar.



Designation: D 412 - 92

### Standard Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension<sup>1</sup>

This standard is issued under the fixed designation D 412; 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.

*These test methods have been approved for use by agencies of the Department of Defense to replace methods 4001, 4116, 4121, and 4111 of Federal Test Method Standard No. 601 and for listing in the DoD Index of Specifications and Standards.*

#### 1. Scope

1.1 These test methods describe procedures used to evaluate the tensile (tension) properties of vulcanized rubbers and thermoplastic rubbers and thermoplastic elastomers. The test methods are not applicable to ebonite and similar hard, low elongation materials. The methods appear as follows:

Test Method A—Dumbbell and Straight Section Specimens  
Test Method B—Cut Ring Specimens

1.2 The values stated in either SI or non-SI units shall be regarded separately as normative for this standard. The values in each system may not be exact equivalents; therefore each system must be used independently, without combining values.

1.3 This standard does not purport to address all of the safety problems, 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:

- D 1349 Practice for Rubber—Standard Temperatures for Testing<sup>2</sup>
  - D 1566 Terminology Relating to Rubber<sup>2</sup>
  - D 3182 Practice for Rubber—Materials, Equipment and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets<sup>3</sup>
  - D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products<sup>3</sup>
  - D 3767 Practice for Rubber—Measurement of Dimensions<sup>3</sup>
  - D 4483 Practice for Rubber—Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2</sup>
  - E 4 Practices for Load Verification of Testing Machines<sup>4</sup>
- 2.2 ASTM Adjunct:

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Nov. 15, 1992. Published February 1993. Originally published as D 412 - 35 T. Last previous edition D 412 - 87.

<sup>2</sup> Annual Book of ASTM Standards, Vols 09.01 and 09.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 09.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 03.01.

#### Cut Ring Specimens, Method B (D 412)<sup>5</sup>

##### 2.3 ISO Standards:

ISO 37 Rubber, Vulcanized and Thermoplastic Determination of Tensile Stress-Strain Properties<sup>6</sup>

#### 3. Terminology

##### 3.1 Definitions:

3.1.1 *tensile set*—the extension remaining after a specimen has been stretched and allowed to retract in a specified manner, expressed as a percentage of the original length. (D 1566)

3.1.2 *tensile set-after-break*—the tensile set measured by fitting the two broken dumbbell pieces together at the point of rupture.

3.1.3 *tensile strength*—the maximum tensile stress applied in stretching a specimen to rupture. (D 1566)

3.1.4 *tensile stress*—a stress applied to stretch a test piece (specimen). (D 1566)

3.1.5 *tensile stress at-given-elongation*—the stress required to stretch the uniform cross section of a test specimen to a given elongation. (D 1566)

3.1.6 *thermoplastic elastomers*—a diverse family of rubberlike materials that unlike conventional vulcanized rubbers can be processed and recycled like thermoplastic materials.

3.1.7 *ultimate elongation*—the elongation at which rupture occurs in the application of continued tensile stress.

3.1.8 *yield point*—that point on the stress-strain curve, short of ultimate failure, where the rate of stress with respect to strain, goes through a zero value and may become negative. (D 1566)

3.1.9 *yield strain*—the level of strain at the yield point. (D 1566)

3.1.10 *yield stress*—the level of stress at the yield point. (D 1566)

#### 4. Summary of Test Methods

4.1 The determination of tensile properties starts with test pieces taken from the sample material and includes the preparation of the specimens and the testing of the specimens. Specimens may be in the shape of dumbbells, rings or straight pieces of uniform cross-sectional area.

4.2 Measurements for tensile stress, tensile stress at a

<sup>5</sup> Detailed drawings are available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. PCN 12-404121-20.

<sup>6</sup> Available from American National Standards Institute, 11 W. 42nd St., 15th Floor, New York, NY 10036.

## D 412

given elongation, tensile strength, yield point, and ultimate elongation are made on specimens that have not been prestressed. Tensile stress, yield point, and tensile strength are based on the original cross-sectional area of a uniform cross-section of the specimen.

4.3 Measurement of tensile set is made after a previously unstressed specimen has been extended and allowed to retract by a prescribed procedure. Measurement of "set after break" is also described.

### 5. Significance and Use

5.1 All materials and products covered by these test methods must withstand tensile forces for adequate performance in certain applications. These test methods allow for the measurement of such tensile properties. However, tensile properties alone may not directly relate to the total end use performance of the product because of the wide range of potential performance requirements in actual use.

5.2 Tensile properties depend both on the material and the conditions of test (extension rate, temperature, humidity, specimen geometry, pretest conditioning, etc.); therefore materials should be compared only when tested under the same conditions.

5.3 Temperature and rate of extension may have substantial effects on tensile properties and therefore should be controlled. These effects will vary depending on the type of material being tested.

5.4 Tensile set represents residual deformation which is partly permanent and partly recoverable after stretching and retraction. For this reason, the periods of extension and recovery (and other conditions of test) must be controlled to obtain comparable results.

### 6. Apparatus

6.1 *Testing Machine*—Tension tests shall be made on a power driven machine equipped to produce a uniform rate of grip separation of  $500 \pm 50$  mm/min ( $20 \pm 2$  in./min) for a distance of at least 75 mm (30 in.) (see Note 1). The testing machine shall have both a suitable dynamometer and an indicating or recording system for measuring the applied force within  $\pm 2\%$ . If the capacity range cannot be changed for a test (as in the case of pendulum dynamometers) the applied force at break shall be measured within  $\pm 2\%$  of the full scale value, and the smallest tensile force measured shall be accurate to within 10%. If the dynamometer is of the compensating type for measuring tensile stress directly, means shall be provided to adjust for the cross-sectional area of the specimen. The response of the recorder shall be sufficiently rapid that the applied force is measured with the requisite accuracy during the extension of the specimen to rupture. If the testing machine is not equipped with a recorder, a device shall be provided that indicates, after rupture, the maximum force applied during extension. Testing machine systems shall be capable of measuring elongation of the test specimen in minimum increments of 10%.

NOTE 1—A rate of elongation of  $1000 \pm 100$  mm/min ( $40 \pm 4$  in./min) may be used and notation of the speed made in the report. In case of dispute, the test shall be repeated and the rate of elongation shall be  $500 \pm 50$  mm/min ( $20 \pm 2$  in./min).

#### 6.2 Test Chamber for Elevated and Low Temperatures—

The test chamber shall conform with the following requirements:

6.2.1 Air shall be circulated through the chamber at a velocity of 1 to 2 m/s (3.3 to 6.6 ft/s) at the location of the grips or spindles and specimens maintained within  $2^\circ\text{C}$  ( $3.6^\circ\text{F}$ ) of the specified temperature.

6.2.2 A calibrated sensing device shall be located near the grips or spindles for measuring the actual temperature.

6.2.3 The chamber shall be vented to an exhaust system or to the outside atmosphere to remove fumes liberated at high temperatures.

6.2.4 Provisions shall be made for suspending specimens vertically near the grips or spindles for conditioning prior to test. The specimens shall not touch each other or the sides of the chamber except for momentary contact when agitated by the circulating air.

6.2.5 Fast acting grips suitable for manipulation at high or low temperatures may be provided to permit placing dumbbells or straight specimens in the grips in the shortest time possible to minimize any change in temperature of the chamber.

6.2.6 The dynamometer shall be suitable for use at the temperature of test or it shall be thermally insulated from the chamber.

6.2.7 Provision shall be made for measuring the elongation of specimens in the chamber. If a scale is used to measure the extension between the bench-marks, the scale shall be located parallel and close to the grip path during specimen extension and shall be controlled from outside the chamber.

6.3 *Dial Micrometer*—The dial micrometer shall conform to the requirements of Practice D 3767 (Method A). For ring specimens, see 17.10 of these test methods.

6.4 *Apparatus for Tensile Set Test*—The testing machine described in 6.1 or an apparatus similar to that shown in Fig. 1 may be used. A stop watch or other suitable timing device measuring in minute intervals for at least 30 min, shall be provided. A scale or other device shall be provided for measuring tensile set to within 1%.

### 7. Test Temperature

7.1 Unless otherwise specified, the standard temperature for testing shall be  $23 \pm 2^\circ\text{C}$  ( $73.4 \pm 3.6^\circ\text{F}$ ). Specimens shall be conditioned for at least 3 h when the test temperature is  $23^\circ\text{C}$  ( $73.4^\circ\text{F}$ ). If the material is affected by moisture, maintain the relative humidity at  $50 \pm 5\%$  and condition the specimens for at least 24 h prior to testing. When testing at any other temperature is required use one of the temperatures listed in Practice D 1349.

7.2 For testing at temperatures above  $23^\circ\text{C}$  ( $73.4^\circ\text{F}$ ) preheat specimens for  $10 \pm 2$  min for Method A and for  $6 \pm 2$  min for Method B (see Note 2). Place each specimen in the test chamber at intervals ahead of testing so that all specimens of a series will be in the chamber the same length of time. The preheat time at elevated temperatures must be limited to avoid additional vulcanization or thermal aging.

NOTE 2: Precaution—In addition to other precautions, suitable heat or cold resistant gloves should be worn for arm and hand protection when testing at other than  $23^\circ\text{C}$  ( $73.4^\circ\text{F}$ ). A mask for the face is very desirable for high temperature testing to prevent the inhalation of toxic fumes when the door of the chamber is open.





## D 412

by the material, test equipment and the sample or piece available for test. A longer specimen may be used for rubbers having low ultimate elongation to improve precision of elongation measurement.

### 9. Calibration of the Testing Machine

9.1 Calibrate the testing machine in accordance with Procedure A of Practice E 4. If the dynamometer is of the strain-gage type, calibrate the tester at one or more forces in addition to the requirements in Sections 7 and 18 of Practice E 4. Testers having pendulum dynamometers may be calibrated as follows:

9.1.1 Place one end of a dumbbell specimen in the upper grip of the testing machine.

9.1.2 Remove the lower grip from the machine and attach it, by means of the gripping mechanism to the dumbbell specimen in the upper grip.

9.1.3 Attach a hook to the lower end of the lower specimen grip mechanism.

9.1.4 Suspend a known mass from the hook of the lower specimen grip mechanism in such a way as to permit the mass assembly to temporarily rest on the lower testing machine grip framework or holder (see Note 3).

9.1.5 Start the grip separation motor or mechanism, as in normal testing, and allow it to run until the mass is freely suspended by the specimen in the upper grip.

9.1.6 If the dial or scale does not indicate the force applied (or its equivalent in stress for a compensating type tester) within specified tolerance, thoroughly inspect the testing machine for malfunction (for example, excess friction in bearings and other moving parts). Ensure that the mass of the lower grip mechanism and the hook are included as part of the known mass.

9.1.7 After machine friction or other malfunction has been removed, recalibrate the testing machine at a minimum of three points using known masses to produce forces of approximately 10, 20 and 50 % of capacity. If pawls or ratchets are used during routine testing, use them for calibration. Check for friction in the head by calibrating with the pawls up.

NOTE 3—It is advisable to provide a means for preventing the known mass from falling to the floor in case the dumbbell should break.

9.2 A rapid approximate calibration of the testing machine may be obtained by using a spring calibration device.

### 10. Report

10.1 Report the following information:

10.1.1 Results calculated in accordance with Section 16 or 20, whichever is applicable,

10.1.2 Type or description of test specimen,

10.1.3 Date of test,

10.1.4 Rate of extension if not as specified,

10.1.5 Temperature and humidity of test room if not as specified,

10.1.6 Temperature of test if at other than  $23 \pm 2^\circ\text{C}$  ( $73.4 \pm 3.6^\circ\text{F}$ ) and

10.1.7 Date of vulcanization, preparation of the rubber, or both, if known.

### 11. Precision and Bias

11.1 This precision and bias section has been prepared in

accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical details.

11.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that the parameters are applicable to those particular materials and the specific testing protocols that include these test methods.

#### 11.3 Test Method A (Dumbbells):

11.3.1 For the main interlaboratory program a Type 1 precision was evaluated in 1986. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. A test result is the median value, as specified by this test method, obtained on three determination(s) or measurement(s) of the property or parameter in question.

11.3.2 Three different materials were used in this interlaboratory program, these were tested in 10 laboratories on two different days.

11.3.3 For the main interlaboratory program cured sheets of each of the three compounds were circulated to each laboratory and stress-strain (dumbbell) specimens were cut, gaged, and tested. A secondary interlaboratory test was conducted for one of the compounds (R19160). For this testing, uncured compound was circulated and sheets were cured at a specified time and temperature (10 min at  $157^\circ\text{C}$ ) in each laboratory. From these individually cured sheets, test specimens were cut and tested on each of two days one week apart as in the main program. The main program results are referred to as "Test Only" and the secondary program results are referred to as "Cure and Test."

11.3.4 The results of the precision calculations for repeatability and reproducibility are given in Tables 1 and 2, in ascending order of material average or level, for each of the materials evaluated and for each of the three properties evaluated.

11.3.5 The precision of this test method may be expressed in the format of the following statements that use what is called an "appropriate value" of  $r$ ,  $R$ , ( $r$ ), or ( $R$ ), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of  $r$  or  $R$  associated with a mean level in Tables 1 through 4 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

11.3.6 *Repeatability*—The repeatability,  $r$ , of this test method has been established as the appropriate value tabulated in Tables 1 and 2. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated  $r$  (for any given level) must be considered as derived from different or nonidentical sample populations.

11.3.7 *Reproducibility*—The reproducibility,  $R$ , of this test method has been established as the appropriate value tabulated in Tables 1 and 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated  $R$  (for any given level) must be considered to have come from different

## D 412

TABLE 1 Type 1 (Test Only) Precision on Method A Die C Dumbbell Test Specimens

NOTE:  
 Sr = repeatability standard deviation.  
 r = repeatability = 2.83 times the square root of the repeatability variance.  
 (r) = repeatability (as percentage of material average).  
 SR = reproducibility standard deviation.  
 R = reproducibility = 2.83 times the square root of the reproducibility variance.  
 (R) = reproducibility (as percentage of material average).

Part 1 Tensile Strength, MPa:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
1. N18081	9.88	0.200	0.568	5.75	0.293	0.829	8.40	
3. E17074	15.36	0.467	1.323	8.60	0.482	1.368	8.88	
2. R19160	25.70	0.438	1.235	4.80	1.890	5.351	20.82	
Pooled Values <sup>a</sup>	18.99	0.385	1.090	6.42	1.102	3.120	18.37	
Part 2 Percent Elongation:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
3. E17074	158.3	6.304	17.842	11.41	11.461	32.492	20.78	
2. R19160	510.4	11.471	32.484	6.36	21.243	60.120	11.77	
1. N18081	591.8	17.810	50.402	8.52	27.198	76.972	13.01	
Pooled Values <sup>a</sup>	419.4	12.761	35.114	6.81	20.999	59.427	14.18	
Part 3 Stress at 100% Elongation, MPa:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
1. N18081	1.17	0.053	0.151	12.98	0.081	0.1744	14.92	
2. R19160	2.01	0.050	0.142	7.10	0.274	0.7755	38.62	
3. E17074	9.08	0.489	1.385	15.25	0.738	2.0910	23.02	
Pooled Values <sup>a</sup>	4.09	0.265	0.808	19.79	0.458	1.2915	31.60	

<sup>a</sup> No values omitted.TABLE 2 Type 1 (Cure and Test) Precision on Method A Die C Dumbbell Specimens<sup>a</sup>

NOTE 1:  
 Sr = repeatability standard deviation.  
 r = repeatability = 2.83 times the square root of the repeatability variance.  
 (r) = repeatability (as percentage of material average).  
 SR = reproducibility standard deviation.  
 R = reproducibility = 2.83 times the square root of the reproducibility variance.  
 (R) = reproducibility (as percentage of material average).

NOTE 2:

N18081—highly extended, low durometer CR (Neoprene).  
 R19160—high tensile NR.  
 E17047—moderately filled EPDM.

Part 1 Tensile Strength, MPa:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
1. R19160	26.0	0.613	1.73	6.66	1.74	4.95	19.0	
Part 2 Percent Elongation:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
1. R19160	626.9	13.32	37.7	7.15	19.8	55.70	10.5	
Part 3 Stress at 100% Elongation, MPa:								
Material	Average	Within Laboratories			Between Laboratories			
		Sr	r	(r)	SR	R	(R)	
1. R19160	1.88	0.072	0.205	11.21	0.226	0.641	34.6	

<sup>a</sup> Seven laboratories participated in this cure and test program.

or nonidentical sample populations.

11.3.8 Repeatability and reproducibility expressed as a percentage of the mean level, (r) and (R), have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.3.9 Bias—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

## 11.4 Test Method B (Rings):



## D 412

TABLE 3 Type 1 Precision—Test Method B (Rings)

## Note:

- $S_r$  = repeatability standard deviation.  
 $r$  = repeatability = 2.83 times the square root of the repeatability variance.  
 $(r)$  = repeatability (as percentage of material average).  
 $S_R$  = reproducibility standard deviation.  
 $R$  = reproducibility = 2.83 times the square root of the reproducibility variance.  
 $(R)$  = reproducibility (as percentage of material average).

Material	Average	Tensile Strength (MPa)					
		Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
6. MATL 5	11.5	0.666	1.885	16.3	1.49	4.06	35.3
6. MATL 6	12.7	0.274	0.775	6.0	0.83	2.35	18.5
1. MATL 1	14.6	0.367	1.040	7.1	0.40	1.15	7.9
4. MATL 4	15.0	0.553	1.585	10.4	3.03	8.59	57.2
2. MATL 2	20.3	1.293	3.660	18.0	2.47	6.99	34.4
3. MATL 3	22.3	1.556	4.405	19.6	1.55	4.40	19.6
Pooled Values <sup>a</sup>	15.9	0.942	2.688	16.7	1.97	5.31	33.2

<sup>a</sup> No values omitted.

TABLE 4 Type 1 Precision—Test Method B (Rings)

## Note:

- $S_r$  = repeatability standard deviation.  
 $r$  = repeatability = 2.83 times the square root of the repeatability variance.  
 $(r)$  = repeatability (as percentage of material average).  
 $S_R$  = reproducibility standard deviation.  
 $R$  = reproducibility = 2.83 times the square root of the reproducibility variance.  
 $(R)$  = reproducibility (as percentage of material average).

Material	Average	Ultimate Elongation, %					
		Within Laboratories			Between Laboratories		
		$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
1. MATL 1	322.1	15.25	43.18	13.40	33.4	94.7	29.4
2. MATL 2	445.4	11.35	32.12	7.21	34.1	96.6	21.7
4. MATL 4	509.4	27.44	77.65	15.24	51.1	144.9	28.4
5. MATL 5	545.0	2.91	8.25	1.51	58.3	169.5	28.2
6. MATL 6	599.7	12.91	36.55	6.09	14.0	39.5	6.50
3. MATL 3	815.9	16.25	45.99	5.83	90.6	255.5	31.4
Pooled Values <sup>a</sup>	539.8	16.54	46.82	8.67	48.2	136.4	23.2

<sup>a</sup> No values omitted.

11.4.1 A Type 1 precision was evaluated in 1985. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. A test result is the mean value, as specified by this test method, obtained on three determinations or measurements of the property or parameter in question.

11.4.2 Six different materials were used in the interlaboratory program, these were tested in four laboratories on two different days.

11.4.3 The results of the precision calculations for repeatability and reproducibility are given in Tables 3 and 4, in ascending order of material average or level, for each of the materials evaluated.

11.4.4 Repeatability,  $r$ , varies over the range of material levels as evaluated. Reproducibility,  $R$ , varies over the range of material levels as evaluated.

11.4.5 The precision of this test method may be expressed in the format of the following statements that use what is called an "appropriate value" of  $r$ ,  $R$ ,  $(r)$ , or  $(R)$ , that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of  $r$  or  $R$  associated with a mean level in Tables 1 through 4 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

11.4.6 *Repeatability*—The repeatability,  $r$ , of this test method has been established as the appropriate value tabulated in Tables 3 and 4. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated  $r$  (for any given level) must be considered as derived from different or nonidentical sample populations.

11.4.7 *Reproducibility*—The reproducibility,  $R$ , of this test method has been established as the appropriate value tabulated in Tables 3 and 4. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated  $R$  (for any given level) must be considered to have come from different or nonidentical sample populations.

11.4.8 Repeatability and reproducibility expressed as a percentage of the mean level,  $(r)$  and  $(R)$ , have equivalent application statements as 11.3.6 and 11.3.7 for  $r$  and  $R$ . For the  $(r)$  and  $(R)$  statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.4.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is

D 412

exclusively defined by the test method. Bias, therefore, cannot be determined.

12. Keywords

12.1 elongation; set after break; tensile properties; tensile set; tensile strength; tensile stress; yield point

TEST METHOD A—DUMBBELL AND STRAIGHT SPECIMENS

13. Apparatus

13.1 Die—The shape and dimensions of the die for preparing dumbbell specimens shall conform with those shown in Fig. 2. The inside faces in the reduced section shall be perpendicular to the plane formed by the cutting edges and polished for a distance of at least 5 mm (0.2 in.) from the

cutting edge. The die shall at all times be sharp and free of nicks (see Note 4).

NOTE 4—Careful maintenance of die cutting edges is of extreme importance. The cutting edges can be maintained by a daily procedure of light touching up of the edges with a jeweler's honing stone. The condition of the die may be determined by investigating the rupture point on any series of broken (ruptured) specimens. Remove such specimens from the grips of the testing machine, stack the joined-together specimens on top of each other, and note if there is any tendency for tensile breaks to occur at the same position on each of the specimens. Rupture consistently at the same place indicates that the die may be dull, nicked, or bent at that location.

13.2 Bench Marker—The two marks placed on the specimen and used to measure elongation or strain are called "bench marks" (see Note 5). The bench marker shall consist

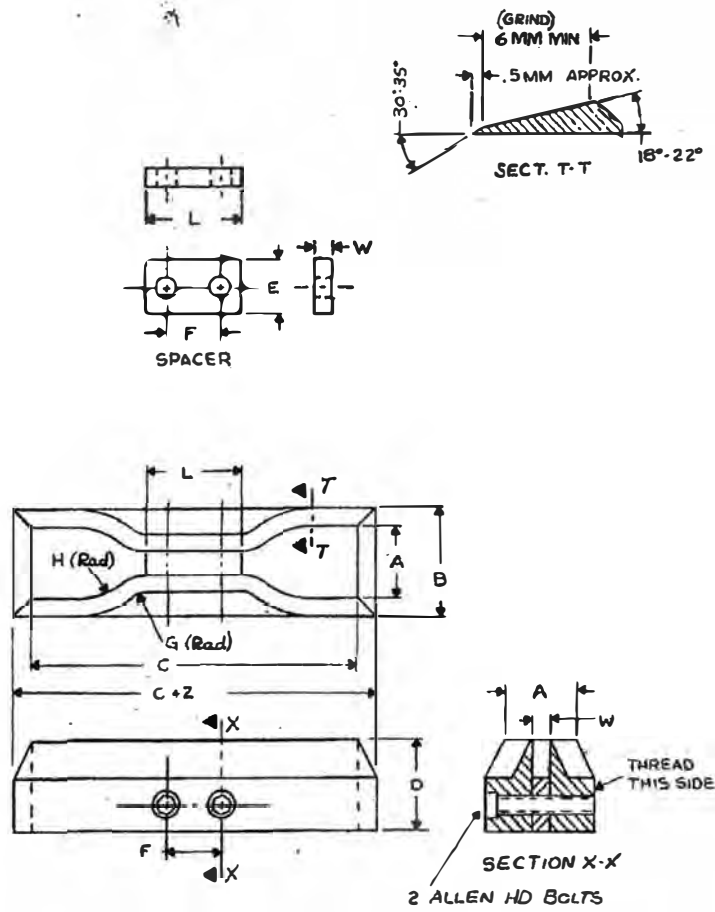


FIG. 2 Standard Dies for Cutting Dumbbell Specimens (continued)

## D 412

Dimensions of Standard Dumbbell Dies<sup>a</sup>

Dimension	Units	Tolerance	Die A	Die B	Die C	Die D	Die E	Die F
A	mm	±1	26	26	25	18	18	16
	in.	±0.04	1	1	1	0.62	0.62	0.62
B	mm	max	40	40	40	30	30	30
	in.	max	1.6	1.6	1.6	1.2	1.2	1.2
C	mm	min	140	140	115	100	125	125
	in.	min	5.5	5.5	4.5	4	5	5
D	mm	±6 <sup>b</sup>	32	32	32	32	32	32
	in.	±0.25 <sup>b</sup>	1.25	1.25	1.25	1.25	1.25	1.25
D-E	mm	±1	13	13	13	13	13	13
	in.	±0.04	0.5	0.5	0.5	0.5	0.5	0.5
F	mm	±2	38	38	19	19	38	38
	in.	±0.08	1.5	1.5	0.75	0.75	1.5	1.5
G	mm	±1	14	14	14	14	14	14
	in.	±0.04	0.56	0.56	0.56	0.56	0.56	0.56
H	mm	±2	25	25	25	16	16	16
	in.	±0.08	1	1	1	0.63	0.63	0.63
L	mm	±2	59	59	33	33	59	59
	in.	±0.08	2.32	2.32	1.31	1.31	2.32	2.32
W	mm	+0.05, -0.00	12	6	6	3	3	6
	in.	+0.002, -0.000	0.500	0.250	0.250	0.125	0.125	0.250
Z	mm	±1	13	13	13	13	13	13
	in.	±0.04	0.6	0.5	0.5	0.5	0.6	0.5

<sup>a</sup> Dies whose dimensions are expressed in metric units are not exactly the same as dies whose dimensions are expressed in U.S. customary units. However, equivalent results may be expected from either die. Dies dimensioned in metric units are intended for use with apparatus calibrated in metric units.

<sup>b</sup> For dies used in sliding machines it is preferable that this tolerance be ±0.5 mm or ±0.02 in.

FIG. 2 Continued

of a base plate containing two raised parallel projections. The surfaces of the raised projections (parallel to the plane of the base plate) are ground smooth in the same plane. The raised projection marking surfaces shall be between 0.05 and 0.08 mm (0.002 and 0.003 in.) wide and at least 15 mm (0.6 in.) long. The angles between the parallel marking surfaces and the sides of the projections shall be at least 75°. The distance between the centers of the two parallel projections or marking surfaces shall be within 1 % of the required or target bench mark distance. A handle attached to the back or top of the bench marker base plate is normally a part of the bench marker.

NOTE 5—If an automatic extensometer is used to measure elongation, bench marks are not necessary.

13.3 *Ink Applicator*—A flat unyielding surface (hardwood, metal, or plastic) shall be used to apply either ink or powder to the bench marker. The ink or powder shall adhere to the specimen, have no deteriorating effect on the specimen and be of contrasting color to that of the specimen.

13.4 *Grips*—The testing machine shall have two grips, one of which shall be connected to the dynamometer.

13.4.1 *Grips for testing dumbbell specimens* shall tighten automatically and exert a uniform pressure across the gripping surfaces, increasing as the tension increases in order to prevent slippage and to favor failure of the specimen in the straight reduced section. Constant pressure pneumatic type grips also are satisfactory. At the end of each grip a positioning device is recommended for inserting specimens to the same depth in the grip and for alignment with the direction of pull.

13.4.2 *Grips for testing straight specimens* shall be constant pressure pneumatic, wedged, or toggle type designed to transmit the applied gripping force over the entire width of the gripped specimen.

#### 14. Specimens

14.1 *Dumbbell Specimens*—Whenever possible, the test

specimens shall be injection molded or cut from a flat sheet not less than 1.3 mm (0.05 in.) nor more than 3.3 mm (0.13 in.) thick and of a size which will permit cutting a specimen by one of the standard methods (see Practice D 3182). Sheets may be prepared directly by processing or from finished articles by cutting and buffing. If obtained from a manufactured article, the specimen shall be free of surface roughness, fabric layers, etc. in accordance with the procedure described in Practice D 3183. All specimens shall be cut so that the lengthwise portion of the specimens is parallel to the grain unless otherwise specified. In the case of sheets prepared in accordance with Practice D 3182, the specimen shall be 2.0 ± 0.2 mm (0.08 ± 0.008 in.) thick died out in the direction of the grain. Use Die C, Fig. 2 (unless otherwise noted) to cut the specimens from the sheet with a single impact stroke (hand or machine) to ensure smooth cut surfaces.

14.1.1 *Marking Dumbbell Specimens*—Dumbbell specimens shall be marked with the bench marker described in 13.2, with no tension on the specimens at the time of marking. Marks shall be placed on the reduced section, equidistant from its center and perpendicular to the longitudinal axis. The between bench mark distance shall be as follows: for Die C or Die D of Fig. 2, 25.00 ± 0.25 mm (1.00 ± 0.01 in.); for any other Die of Fig. 2, 50.00 ± 0.5 mm (2.00 ± 0.02 in.).

14.1.2 *Measuring Thickness of Dumbbell Specimens*—Three measurements shall be made for the thickness, one at the center and one at each end of the reduced section. The median of the three measurements shall be used as the thickness in calculating the cross sectional area. Specimens with a difference between the maximum and the minimum thickness exceeding 0.08 mm (0.003 in.), shall be discarded. The width of the specimen shall be taken as the distance between the cutting edges of the die in the restricted section.

14.2 *Straight Specimens*—Straight specimens may be prepared if it is not practical to cut either a dumbbell or a ring specimen as in the case of a narrow strip, small tubing or

## D 412

narrow electrical insulation material. These specimens shall be of sufficient length to permit their insertion in the grips used for the test. Bench marks shall be placed on the specimens as described for dumbbell specimens in 14.1.1. To determine the cross sectional area of straight specimens in the form of tubes, the mass, length, and density of the specimen may be required. The cross sectional area shall be calculated from these measurements as follows:

$$A = M/DL \quad (1)$$

where:

$A$  = cross-sectional area,  $\text{cm}^2$ ,

$M$  = mass, g,

$D$  = density,  $\text{g}/\text{cm}^3$ , and

$L$  = length, cm.

NOTE 6— $A$  in square inches =  $A (\text{cm}^2) \times 0.155$ .

### 15. Procedure

**15.1 Determination of Tensile Stress, Tensile Strength and Yield Point**—Place the dumbbell or straight specimen in the grips of the testing machine, using care to adjust the specimen symmetrically to distribute tension uniformly over the cross section. This avoids complications that prevent the maximum strength of the material from being evaluated. Unless otherwise specified, the rate of grip separation shall be  $500 \pm 50 \text{ mm}/\text{min}$  ( $20 \pm 2 \text{ in.}/\text{min}$ ) (see Note 7). Start the machine and note the distance between the bench marks, taking care to avoid parallax. Record the force at the elongation(s) specified for the test and at the time of rupture. The elongation measurement is made preferably through the use of an extensometer, an autographic mechanism or a spark mechanism. At rupture, measure and record the elongation to the nearest 10%. See Section 16 for calculations.

NOTE 7—For materials having a yield point (yield strain) under 20% elongation when tested at  $500 \pm 50 \text{ mm}/\text{min}$  ( $20 \pm 2 \text{ in.}/\text{min}$ ), the rate of elongation shall be reduced to  $50 \pm 5 \text{ mm}/\text{min}$  ( $2.0 \pm 0.2 \text{ in.}/\text{min}$ ). If the material still has a yield point (strain) under 20% elongation, the rate shall be reduced to  $5 \pm 0.5 \text{ mm}/\text{min}$  ( $0.2 \pm 0.002 \text{ in.}/\text{min}$ ). The actual rate of separation shall be reported.

**15.2 Determination of Tensile Set**—Place the specimen in the grips of the testing machine described in 6.1 or the apparatus shown in Fig. 1, and adjust symmetrically so as to distribute the tension uniformly over the cross section. Separate the grips at a rate of speed as uniformly as possible, that requires 15 s to reach the specified elongation. Hold the specimen at the specified elongation for 10 min, release quickly without allowing it to snap back and allow the specimen to rest for 10 min. At the end of the 10 min rest period, measure the distance between the bench marks to the nearest 1% of the original between bench mark distance. Use a stop watch for the timing operations. See Section 16 for calculations.

**15.3 Determination of Set-After-Break**—Ten minutes after a specimen is broken in a normal tensile strength test, carefully fit the two pieces together so that they are in good contact over the full area of the break. Measure the distance between the bench marks. See Section 16 for calculations.

### 16. Calculation

**16.1** Calculate the tensile stress at any specified elongation as follows:

$$T_{(xxx)} = F_{(xxx)}/A \quad (2)$$

where:

$T_{(xxx)}$  = tensile stress at (xxx) % elongation, MPa (lbf/in.<sup>2</sup>),

$F_{(xxx)}$  = force at specified elongation, MN or (lbf), and

$A$  = cross-sectional area of unstrained specimen,  $\text{m}^2$  (in.<sup>2</sup>).

**16.2** Calculate the yield stress as follows:

$$Y_{(stress)} = F_{(y)}/A \quad (3)$$

where:

$Y_{(stress)}$  = yield stress, that stress level where the yield point occurs, MPa (lbf/in.<sup>2</sup>),

$F_{(y)}$  = magnitude of force at the yield point, MN (lbf), and

$A$  = cross-sectional area of unstrained specimen,  $\text{m}^2$  (in.<sup>2</sup>).

**16.3** Evaluate the yield strain as that strain or elongation magnitude, where the rate of change of stress with respect to strain, goes through a zero value.

**16.4** Calculate the tensile strength as follows:

$$TS = F_{(RB)}/A \quad (4)$$

where:

$TS$  = tensile strength, the stress at rupture, MPa (lbf/in.<sup>2</sup>),

$F_{(RB)}$  = the force magnitude at rupture, MN (lbf), and

$A$  = cross-sectional area of unstrained specimen,  $\text{m}^2$  (in.<sup>2</sup>).

**16.5** Calculate the elongation (at any degree of extension) as follows:

$$E = 100[L - L_{(0)}]/L_{(0)} \quad (5)$$

where:

$E$  = the elongation in percent (of original bench mark distance),

$L$  = observed distance between bench marks on the extended specimen, and

$L_{(0)}$  = original distance between bench marks (use same units for  $L$  and  $L_{(0)}$ ).

**16.6** The breaking or ultimate elongation is evaluated when  $L$  is equal to the distance between bench marks at the point of specimen rupture.

**16.7** Calculate the tensile set, by using Eq (5), where  $L$  is equal to the distance between bench marks after the 10 min retraction period.

**16.8 Test Result**—A test result is the median of three individual test measurement values for any of the measured properties as described above, for routine testing. There are two exceptions to this and for these exceptions a total of five specimens (measurements) shall be tested and the test result reported as the median of five.

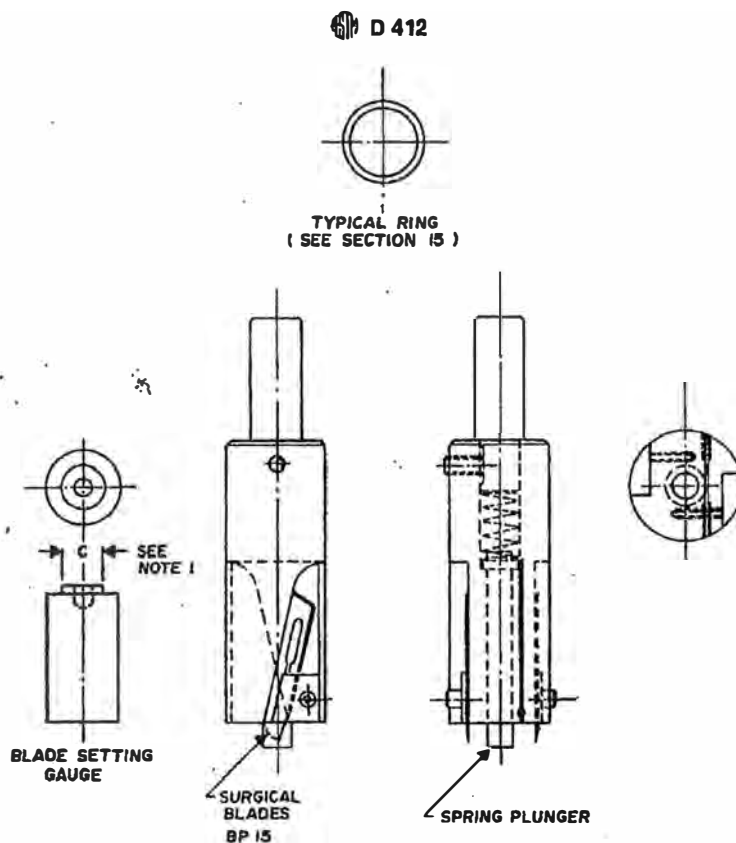
**16.8.1 Exception 1**—If one or two of the three measured values do not meet specified requirement values when testing for compliance with specifications.

**16.8.2 Exception 2**—If referee tests are being conducted.

### TEST METHOD B—CUT RING SPECIMENS

#### 17. Apparatus

**17.1 Cutter**—A typical ring cutter assembly is illustrated in Fig. 3. This is used for cutting rings from flat sheets by mounting the upper shaft portion of the cutter in a rotating housing that can be lowered onto a sheet held by the rubber holding plate as shown in Fig. 4.



NOTE—Dimension C to be 2 mm (0.08 in.) less than the inside diameter of the ring.

FIG. 3 Typical Ring Cutter Assembly

17.1.1 *Blade Depth Gage*—This gage consists of a cylindrical disk having a thickness of at least 0.5 mm (0.02 in.) greater than the thickness of the rubber to be cut and a diameter less than the inside diameter of the specimen used for adjusting the protrusion of the blades from the body of the cutter. See Fig. 3.

17.2 *Rubber Holding Plate*—The apparatus for holding the sheet during cutting shall have plane parallel upper and lower surfaces and shall be a rigid polymeric material (hard rubber, polyurethane, polymethylmethacrylate) with holes approximately 1.5 mm (0.06 in.) in diameter spaced 6 or 7 mm (0.24 or 0.32 in.) apart across the central region of the plate. All the holes shall connect to a central internal cavity which can be maintained at a reduced pressure for holding the sheet in place due to atmospheric pressure. Figure 4 illustrates the design of an apparatus for holding standard sheets (approximately 150 × 150 × 2 mm) during cutting.

17.3 *Source of Reduced Pressure*—Any device such as a vacuum pump that can maintain an absolute pressure below 10 kPa (0.1 atm) in the holding plate central cavity.

17.4 *Soap Solution*—A mild soap solution shall be used

on the specimen sheet to lubricate the cutting blades.

17.5 *Cutter Rotator*—A precision drill press or other suitable machine capable of rotating the cutter at an angular speed of at least 30 rad/s (approximately 300 r/min) during cutting shall be used. The cutter rotator device shall be mounted on a horizontal base and have a vertical support orientation for the shaft that rotates the spindle and cutter. The run-out of the rotating spindle shall not exceed 0.01 mm (0.004 in.).

17.6 *Indexing Table*—A milling table or other device with typical x-y motions shall be provided for positioning the sheet and holder with respect to the spindle of the cutter rotating device.

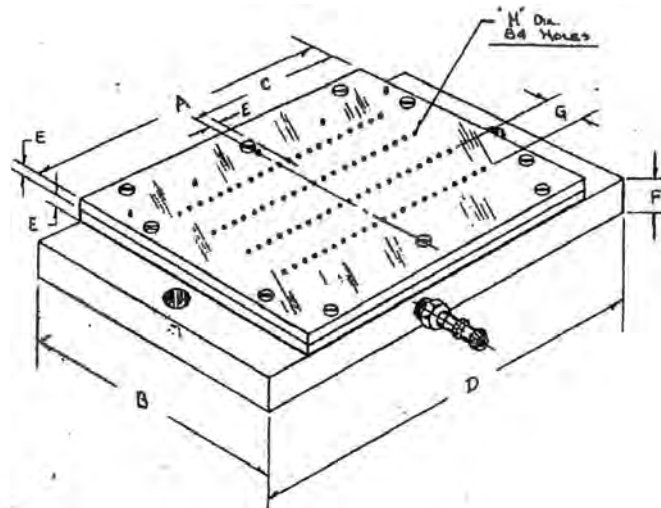
17.7 *Tensile Testing Machine*—A machine as specified in 6.1 shall be provided.

17.8 *Test Fixture*—A test fixture as shown in Fig. 5 shall be provided for testing the ring specimens. The test machine shall be calibrated as outlined in Section 9.

17.9 *Test Chamber*—A chamber for testing at high and low temperatures shall be provided as specified in 6.2.

17.9.1 The fixtures specified in 17.8 are satisfactory for

## D 412



Dimension	mm	in.	Dimension	mm	in.
A	178	7.0	F	19	0.75
B	162	6.0	G	23	0.90
C	89	3.5	H	1.5	0.062
D	229	9.0			
E	6	0.25			

FIG. 4 Rubber Holding Plate

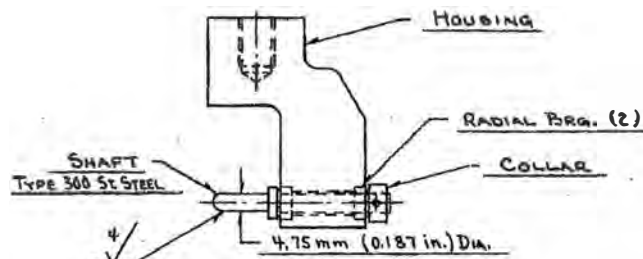


FIG. 5 Assembly, Ring Tensile Test Fixture

testing at other than room temperature. However at extreme temperatures, a suitable lubricant shall be used to lubricate the spindle bearings.

17.9.2 The dynamometer shall be suitable for use at the temperature of test or thermally insulated from the chamber.

17.10 *Dial Micrometer*—A dial micrometer shall be provided that conforms to the requirements of Practice D 3767.

17.10.1 The base of the micrometer used to measure the radial width shall consist of an upper cylindrical surface

(with its axis oriented in a horizontal direction) at least 12 mm (0.5 in.) long and  $15.5 \pm 0.5$  mm ( $0.61 \pm 0.02$  in.) in diameter. To accommodate small diameter rings that approach the 15.5 mm (0.61 in.) diameter of the base and to avoid any ring extension in placing the ring on the base, the bottom half of the cylindrical surface may be truncated at the cylinder centerline, that is, a half cylinder shape. This permits placing small rings on the upper cylindrical surface without interference fit problems. Curved feet on the end of

## D 412

the dial micrometer shaft to fit the curvature of the ring(s), may be used.

## 18. Ring Specimen

18.1 *ASTM Cut Rings*—Two types of cut ring specimens may be used. Unless otherwise specified, the Type 1 ring specimen shall be used.

### 18.1.1 Ring Dimensions:

	mm	in.
<b>Type 1</b>		
Circumference (inside)	50.0 ± 0.01	2.0 ± 0.004
Diameter (inside)	15.92 ± 0.003	0.637 ± 0.001
Radial width	1.0 ± 0.01	0.040 ± 0.0004
Thickness, minimum	1.0	0.040
maximum	3.3	0.13
<b>Type 2</b>		
Circumference mean	100.0 ± 0.2	4.0 ± 0.0004
Diameter (inside)	29.8 ± 0.06	1.19 ± 0.001
Radial width	2.0 ± 0.02	0.08 ± 0.0008
Thickness, minimum	1.0	0.04
maximum	3.3	0.13

18.2 *ISO Cut Rings*—The normal size and the small size ring specimens in ISO 37 have the following dimensions given in mm. See ISO 37 for specific testing procedures for these rings.

	Normal	Small
Diameter, inside	44.6 ± 0.2 mm	8.0 ± 0.1 mm
Diameter, outside	52.6 ± 0.2 mm	10.0 ± 0.1 mm
Thickness	4.0 ± 0.2 mm	1.0 ± 0.1 mm

18.3 *Rings Cut from Tubing*—The dimensions of the ring specimen(s) depend on the diameter and wall thickness of the tubing and should be specified in the product specification.

18.4 *Preparation of Cut Ring Specimens*—Place the blades in the slots of the cutter and adjust the blade depth using the blade depth gage. Place the cutter in the drill press and adjust the spindle or table so that the bottom of the blade holder is about 13 mm (0.5 in.) above the surface of the holding plate. Set the stop on the vertical travel of the spindle so that the tips of the cutting blades just penetrate the surface of the plate. Place the sheet on the holding plate and reduce the pressure in the cavity to 10 kPa (0.1 atm) or less. Lubricate the sheet with mild soap solution. Lower the cutter at a steady rate until it reaches the stop. Be sure that the blade holder does not contact the sheet. If necessary, readjust the blade depth. Return the spindle to its original position and repeat the operation on another sheet.

18.5 *Preparation of Ring Specimens from Tubing*—Place the tubing on a mandrel preferably slightly larger than the inner diameter of the tubing. Rotate the mandrel and tubing in a lathe. Cut ring specimens to the desired axial length by means of a knife or razor blade held in the tool post of the lathe. Lay thin wall tubing flat and cut ring specimens with a die or cutting mechanism having two parallel blades.

### 18.6 Ring Dimension Measurements:

18.6.1 *Circumference*—The inside circumference can be determined by a stepped cone or by "go-no go" gages. Do not use any stress in excess of that needed to overcome any ellipticity of the ring specimen. The mean circumference is obtained by adding to the value for the inside circumference, the product of the radial width and  $\pi$  (3.14).

18.6.2 *Radial Width*—The radial width is measured at

three locations distributed around the circumference using the micrometer described in 17.10.

18.6.3 *Thickness*—For cut rings, the thickness of the disk cut from the inside of the ring is measured with a micrometer described in Practice D 3767.

18.6.4 *Cross-Sectional Area*—The cross-sectional area is calculated from the median of three measurements of radial width and thickness. For thin wall tubing, the area is calculated from the axial length of the cut section and wall thickness.

## 19. Procedure

19.1 *Determination of Tensile Stress, Tensile Strength, Breaking (Ultimate) Elongation and Yield Point*—In testing ring specimens, lubricate the surface of the spindle with a suitable lubricant, such as mineral oil or silicone oil. Select one with documented assurance that it does not interact or affect the material being tested. The initial setting of the distance between the spindle centers may be calculated and adjusted according to the following equation:

$$IS = [C_{(TS)} - C_{(SP)}] / 2 \quad (6)$$

where:

IS = initial separation of spindle centers, mm (in.),

$C_{(TS)}$  = circumference of test specimen, inside circumference for Type 1 rings, mean circumference for Type 2 rings, mm (in.), and

$C_{(SP)}$  = circumference of either (one) spindle, mm (in.).

Unless otherwise specified the rate of spindle separation shall be 500 ± 50 mm/min (20 ± 2 in./min) (see Notes 7 and 8). Start the test machine and record the force and corresponding distance between the spindles. At rupture, measure and record the ultimate (breaking) elongation and the tensile (force) strength. See Section 20 for calculations.

NOTE 8—When using the small ISO ring, the rate of spindle separation shall be 100 ± 10 mm/min (4 ± 0.4 in./min).

19.2 *Tests at Temperatures Other than Standard*—Use the test chamber described in 6.2 and observe the precautionary statement in Note 2. For tests at temperatures above 23°C (73.4°F), preheat the specimens 6 ± 2 min at the test temperature. For below room temperature tests cool the specimens at the test temperature for at least 10 min prior to test. Use test temperatures prescribed in Practice D 1349. Place each specimen in the test chamber at intervals such that the recommendations of 7.2 are followed.

## 20. Calculation

20.1 *Stress-strain properties* for ring specimens are, in general calculated in the same manner as for dumbbell and straight specimens with one important exception. Extending a ring specimen generates a nonuniform stress (or strain) field across the width (as viewed from left to right) of each leg of the ring. The initial inside dimension (circumference) is less than the outside dimension (circumference), therefore for any extension of the grips, the inside strain (or stress) is greater than the outside strain (or stress) because of the differences in the initial (unstrained) dimensions.

20.2 The following options are used to calculate stress at a specified elongation (strain) and breaking or ultimate elongation.

20.2.1 *Stress at a Specified Elongation*—The mean circumference of the ring is used for determining the elongation.

## D 412

tion. The rationale for this choice is that the mean circumference best represents the average strain in each leg of the ring.

20.2.2 *Ultimate (Breaking) Elongation*—This is calculated on the basis of the inside circumference since this represents the maximum strain (stress) in each leg of the ring. This location is the most probable site for the initiation of the rupture process that occurs at break.

20.3 Calculate the tensile stress at any specified elongation by using Eq 2 in 16.1.

20.3.1 The elongation to be used to evaluate the force as specified in Eq 2 (16.1), is calculated as follows:

$$E = 200[L/MC_{(TS)}] \quad (7)$$

where:

$E$  = elongation (specified), percent,  
 $L$  = increase in grip separation at specified elongation, mm (in.), and

$MC_{(TS)}$  = mean circumference of test specimen, mm (in.).

20.3.2 The grip separation for any specified elongation can be found by rearranging Eq 7, as given below:

$$L = E \times MC_{(TS)} / 200 \quad (8)$$

20.4 Calculate the yield stress by using Eq 3 in 16.2.

20.5 Evaluate the yield strain as given in 16.3. Since yield strain may be considered to be an average bulk property of any material, use the mean circumference for this evaluation.

20.6 Calculate the tensile strength by using Eq 4 in 16.4.

20.7 Calculate the breaking or ultimate elongation as follows (see Notes 9 and 10):

$$E = 200[L/IC_{(TS)}] \quad (9)$$

where:

$E$  = breaking or ultimate elongation, percent,  
 $L$  = increase in grip separation at break, mm (in.), and  
 $IC_{(TS)}$  = inside circumference of ring test specimen, mm (in.).

20.8 The inside circumference is used for both types of rings, see 18.1.1 for dimensions. Use the inside diameter to calculate the inside circumference for Type 2 rings.

NOTE 9—Equations 8, 9, and 10 are applicable only if the initial setting of the spindle centers is adjusted in accordance with Eq 7.

NOTE 10—The user of these test method should be aware that because of the different dimensions used in calculating (1) stress at a specified elongation (less than the ultimate elongation) and (2) the ultimate (breaking) elongation (see 20.1 and 20.2), it is possible that a stress at a specified elongation, slightly less (4 to 5 %) than the ultimate elongation cannot be measured (calculated).

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*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, 1916 Race St., Philadelphia, PA 19103.*