PONTIFICIA UNIVERSIDAD CATÓLICA DEL PERÚ

ESCUELA DE POSGRADO

RESISTENCIA AL DESGASTE DE UN ELASTÓMERO DE POLIURETANO ELABORADO A PARTIR DE UN POLIOL DE BASE BIOLÓGICA

Tesis para optar el grado académico de:

Magíster en Ingeniería y Ciencia de los Materiales

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LIMA – PERU

2021

RESUMEN

Se elaboraron poliuretanos elastoméricos (PUE) a partir de un poliol de base biológica, el diol dimero de ácido graso (FADD), un poliol convencional (politetrametileno éter glicol-PTMEG) y el diisocianato de 4,4-difenilmetano. Se investigó la influencia del poliol FADD sobre la resistencia al desgaste y la separación de microfases de este material elastomérico. El poliuretano elastomérico fue elaborado mediante el método del prepolimero, ósea en dos etapas. En la primera etapa, se sintetizó un prepolímero de poliuretano usando diisocianato de 4,4'-difenilmetano (MDI) y politetrametileno éter glicol (PTMEG). En las formulaciones de poliuretano, el FADD reemplazó gradualmente a este último hasta alcanzar el 100% en peso del total de polioles. En la segunda etapa, los prepolímeros, conteniendo grupos isocianato terminales, se unieron usando 1,4-butanodiol (BDO) como extensor de cadena para obtener poliuretanos elastoméricos (PUE) basados en poliol de tipo poli(éter-éster). La miscibilidad entre los polioles de base éster y éter (FADD y PTMEG, respectivamente) se estudió cualitativamente con microscopía óptica y mediante espectroscopía infrarroja por transformada de Fourier (FTIR), método que también se utilizó para investigar el grado de separación de fases (DPS) de los PUE obtenidos. Los espectros FTIR resultantes mostraron posibles interacciones de tipo puentes de hidrógeno entre el grupo éster de FADD y el grupo hidroxilo de PTMEG, lo que indicaría una miscibilidad parcial entre el poliol éster FADD y el poliol éter PTMEG. Esta miscibilidad parcial fue corroborada cualitativamente por microscopía óptica. Además, el DPS de los PUE obtenidos disminuyó al aumentar el contenido de FADD en la formulación de elastómero. Sin embargo, por encima del 25% en peso de FADD, no hubo mayor disminución de este parámetro.

El rendimiento de desgaste de los PUE se determinó mediante pruebas de resistencia a la abrasión y erosión, donde las pruebas de erosión se llevaron a cabo en una máquina de erosión de fabricación propia basada en el método ASTM D-968. Los resultados mostraron que la resistencia al desgaste de los PUE aumenta al aumentar el contenido de FADD, hasta aproximadamente un 50% en peso de FADD. Esta investigación mostró que los PUEs de base biológica podrían ser adecuados para fabricar repuestos para la industria minera, donde los PUEs usados actualmente para este fin aún son elaborados con polioles a base de petróleo.

ABSTRACT

By using the fatty acid dimer diol (FADD) as the polyol in the formulation of an elastomeric polyurethane (PUE), the influence of this element on the wear resistance and microphase separation of this elastomeric material was investigated. The FADD was incorporated in the PUE by mean of two-step polymerization method. In the first step, a prepolymer was synthesized using 4,4'-diphenylmethane diisocyanate (MDI) and polytetramethylene ether glycol (PTMEG). In the formulations, FADD gradually replaced this latter up to 100 wt% of polyols. In the second stage, the prepolymers with terminal isocyanate groups were joined together using 1,4-butanediol BDO as a chain extender to obtain poly(ether-ester) based PUE. The miscibility between ester and ether polyols (FADD and PTMEG, respectively) was studied qualitatively with optical microscopy and Fourier transform infrared spectroscopy (FTIR), which was also used to investigate the degree phase separation (DPS) of obtained PUEs. The resulting FTIR spectra showed possible hydrogen bond interactions between the FADD ester group and the hydroxyl group of PTMEG, which would indicate miscibility between the FADD ester polyol and the PTMEG ether polyol. This partial miscibility was qualitatively corroborated by optical microscopy. Furthermore, the DPS of obtained PUEs decreased by increasing the content of FADD in the elastomer formulation. However, above 25wt% of FADD, that decrease showed a holding back.

The wear performance of PUEs was determined by abrasion and erosion resistance tests, where the erosion tests were carried out on a self-made erosion machine based on the ASTM D968 method. The results showed that the wear resistance of PUEs increase by increasing FADD content, at least up to 50 wt% of FADD. This research showed that bio-based PUEs could be suitable for manufacturing spare parts for the mining industry, where the PUEs currently used for this purpose are still made with petroleum-based polyols.

AGRADECIMIENTOS

Detrás de la realización de este trabajo esta una innumerable cantidad de eventos que construyeron una historia de aprendizaje con un final que ciertamente es un comienzo tangible de un sueño, y quiero expresar mi profunda gratitud al principal actor de esta historia, y es Dios, quien me fortalece para trabajar en mi propósito.

Agradezco a mis padres Rufino y Sofia por establecer en mi sus valores y principios, a mi hermana Sara y su esposo Mariano por su amor e inapreciable apoyo para culminar mis estudios, a mi esposa Karen y mi hija Frances por ser mi fuente de motivación.

Este trabajo de investigación fue realizado con el apoyo financiero de la Empresa Peruana Poliuretanos S.A. y el Programa Nacional de Innovación para la Competitividad y Productividad del Perú, y quiero agradecer su apoyo para realizar esta investigación.

Quiero expresar mi profundo aprecio y gratitud a mi asesor Dr. Juan Carlos Rueda Sánchez por sus consejos y orientación que hicieron posible terminar este trabajo.

Agradezco enormemente al Ing. Santiago Devoto por poner a mi disposición las instalaciones de la compañía Poliuretanos S.A. para realizar este trabajo de investigación, también quiero agradecer a todos los colaboradores y compañeros de trabajo en Poliuretanos S.A. por su apoyo en todas las etapas de esta investigación.

Finalmente quiero agradecer a los profesores de la maestría por compartir sus conocimientos y por brindarme sus consejos que contribuyeron para realizar este trabajo de investigación

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CAPÍTULO I

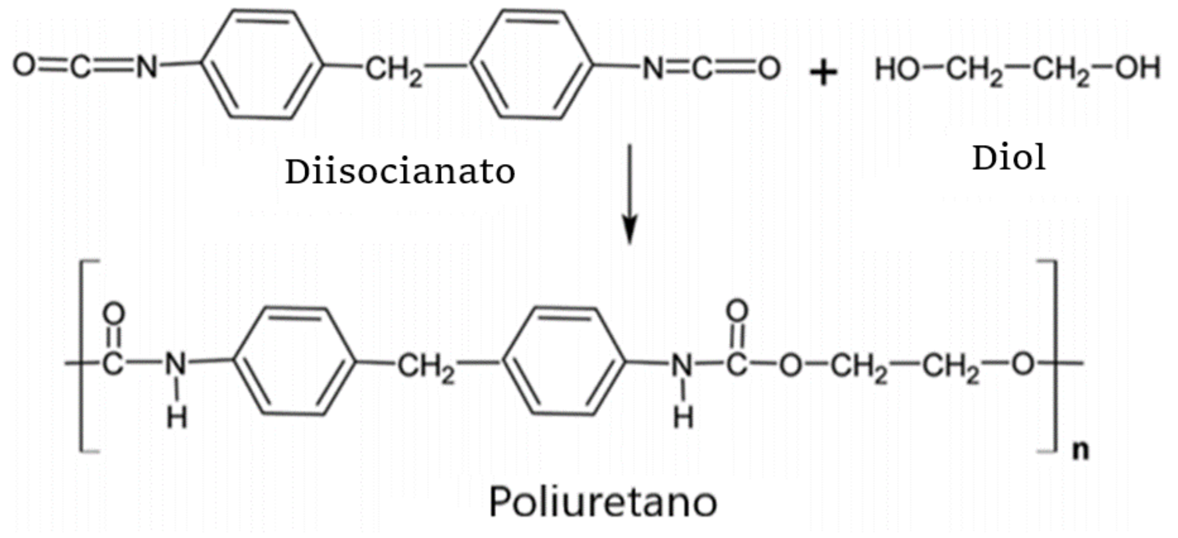
MARCO TEÓRICO

1.1 Teoría general de los poliuretanos

1.1.1 Los poliuretanos

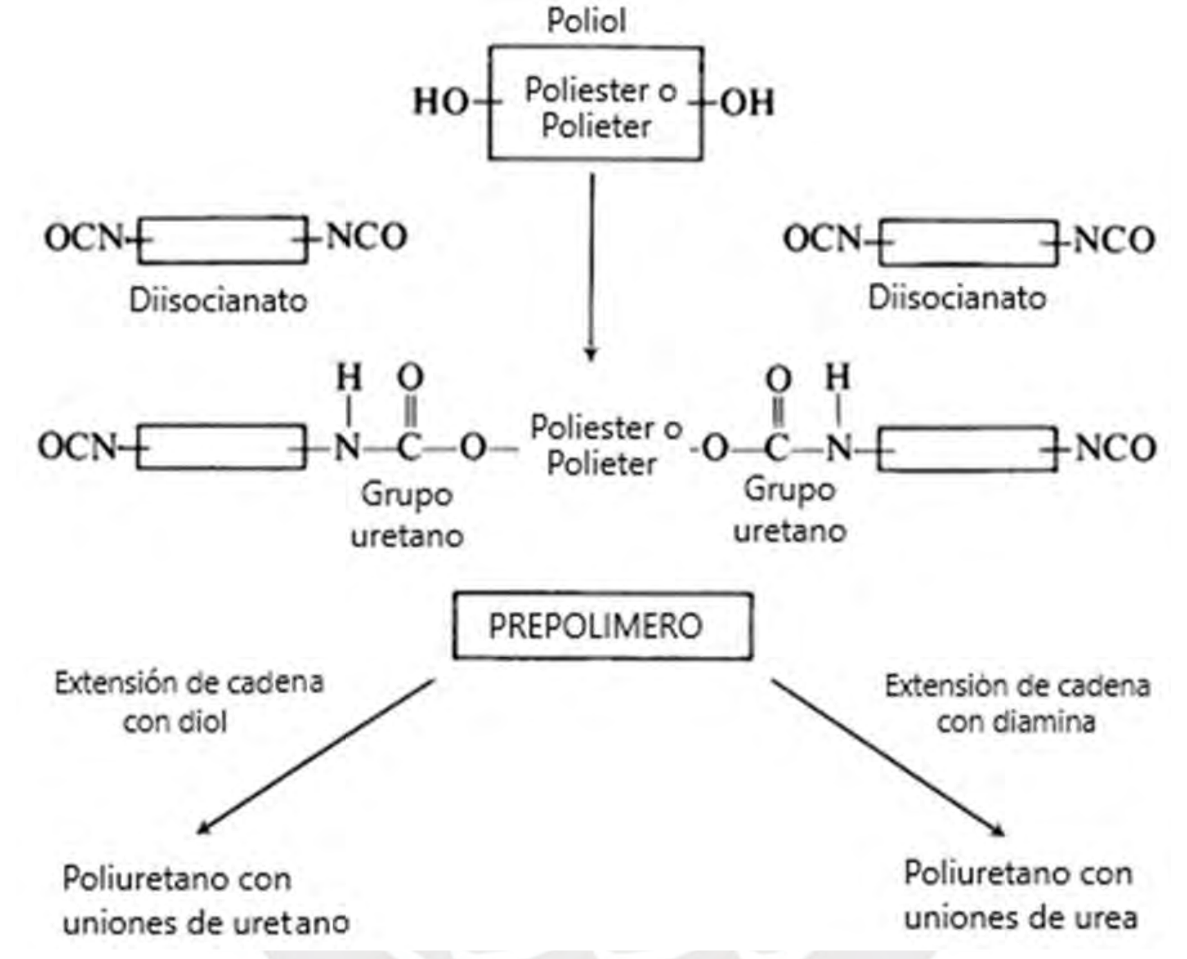
Los poliuretanos son polímeros orgánicos que contienen grupos uretanos en su estructura polimérica, la química y desarrollo de los poliuretanos comenzó en el año 1937 en los laboratorios Farben I.G. una subdivisión de la corporación Bayer en Leverkusen, Alemania, donde el Profesor Otto Bayer, con el propósito de encontrar una fibra polimérica capaz de competir con el Nylon, descubrió como fabricar un polímero usando diisocianatos por medio de la técnica de polimerización por adición [1].

Los poliuretanos pueden ser producidos por diferentes métodos, una reacción típica para la obtención de los poliuretanos es a través de la reacción entre los diisocianatos y polioles (Esquema 1).



Esquema 1.- Reacción típica de síntesis de un poliuretano [2]

La fabricación de los poliuretanos generalmente involucra a tres componentes: Isocianatos, polioles y extensores de cadena. El método más importante y usado es a través de la reacción entre un poliol y un disocianato. De acuerdo con la clasificación de los métodos, según la secuencia de participación de los componentes, tenemos el método de un paso y el de dos pasos, método del prepolímero (Esquema 2)



Esquema 2. Método del prepolímero para la formación un poliuretano [3]

Tanto para el método de un paso como de dos pasos, la estructura química de repetición típica de un poliuretano (Figura 1) se establece alternando los segmentos duros y blandos (Figura 2). Los segmentos duros forman domínios duros por medio de enlaces de hidrógeno (Figura 3). Esta morfología interna con fases duras y blandas de los poliuretanos le confiere una ventaja sobre el resto de los polímeros. Por ejemplo, en la resistencia al desgaste.

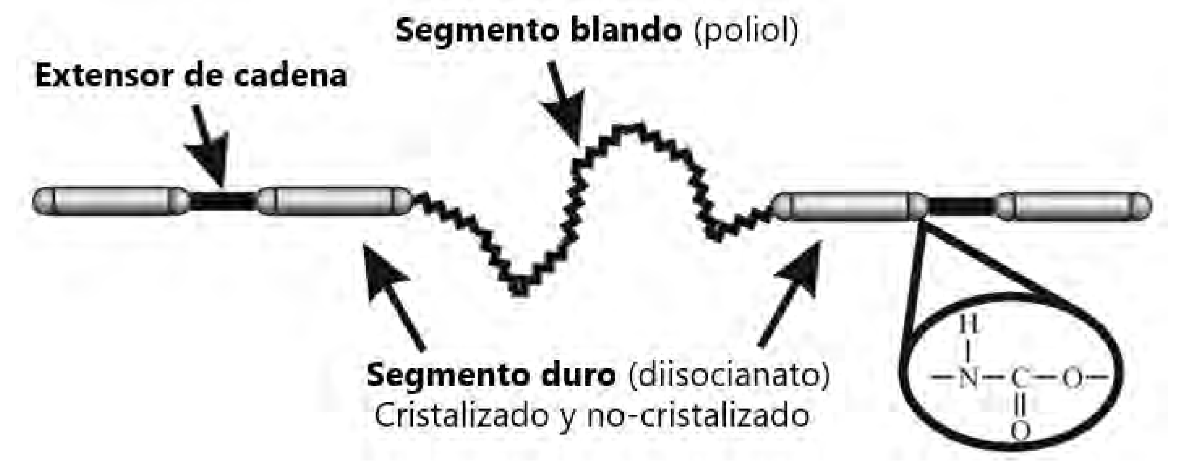


Figura 1.- Estructura de repetición típica en la cadena de un poliuretano[4]

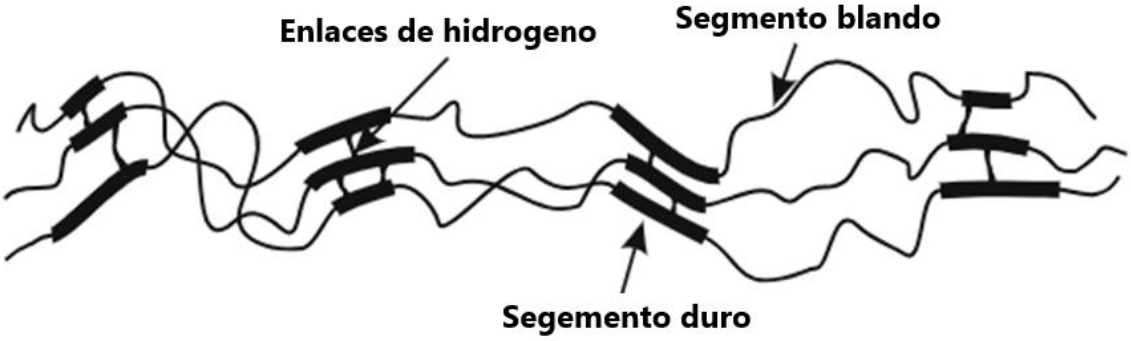


Figura 2.- Alternación de los segmentos duros y blandos en una estructura de poliuretano[4]

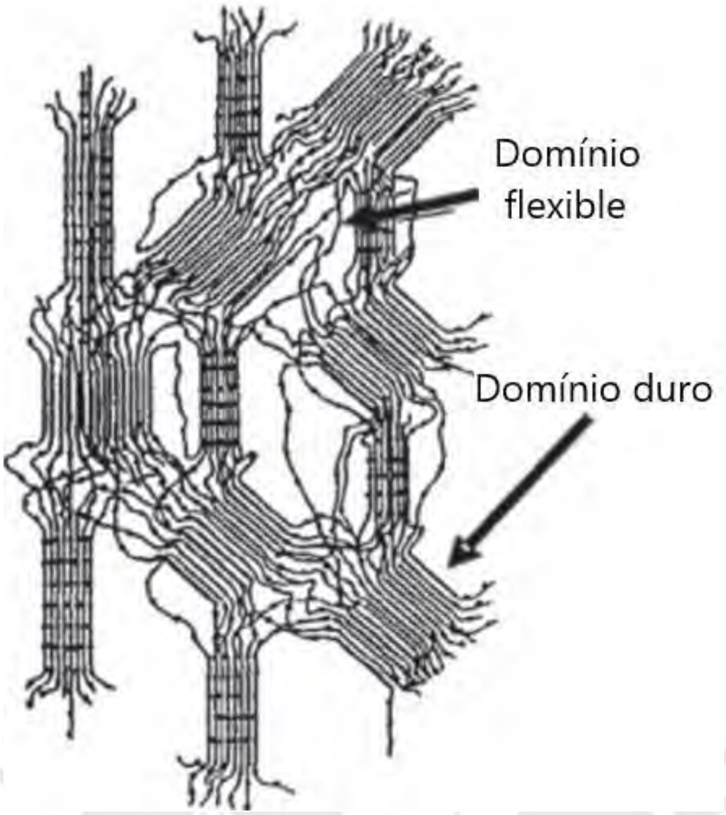


Figura 3.- Dominios duros y blandos en la estructura de un poliuretano [3]

Durante la reacción de síntesis de los poliuretanos se pueden producir subproductos tales como, por ejemplo, los alofanatos, biuretanos, ureas, ácido úrico y otros (Figura 4), relacionados directamente a los insumos utilizados y los parámetros de la reacción.

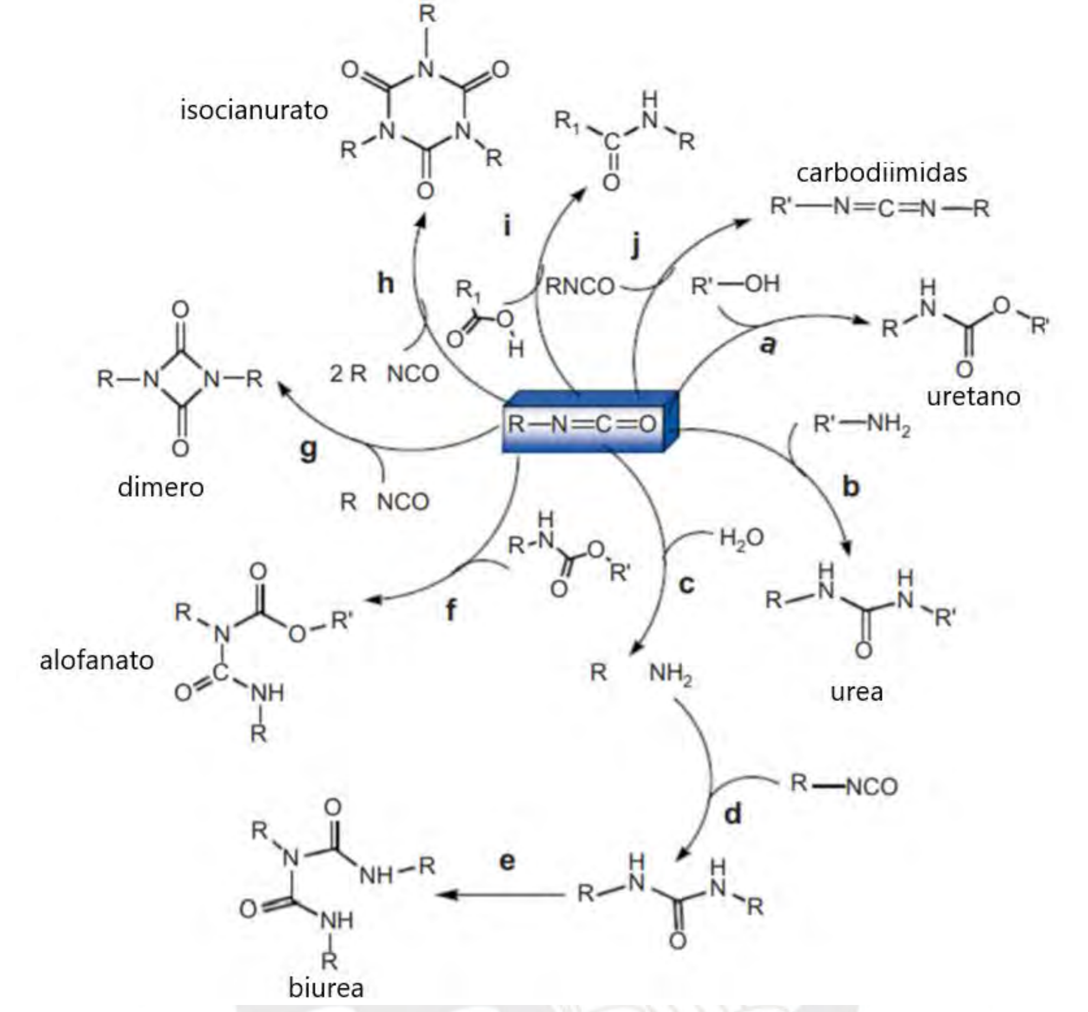


Figura 4.- Representación esquemática de los subproductos producidos durante la reacción entre dioles e isocianatos [5]

1.1.2 Tipos de poliuretanos

Dependiendo del tipo de reactantes y el método usado, los poliuretanos pueden ser producidos con diferentes propiedades. Actualmente existen diferentes tipos de isocianatos, polioles y extensores de cadena (ver Anexos I, II y II) que pueden ser usados en el diseño y síntesis de un poliuretano. Los grupos de poliuretanos más importantes son:

- Fibras
- Películas
- Materiales colables
- Termoplásticos
- Espumas
- Materiales maleables

1.1.3 Poliuretanos elastoméricos

Los elastómeros de poliuretano son materiales poliméricos que poseen una elasticidad similar al caucho, la cual es una característica de identificación de los elastómeros.

Para los poliuretanos en general es difícil mencionar una propiedad que los diferencie de los demás materiales poliméricos ya que el diseño de poliuretanos es un campo con innumerables opciones para una aplicación en particular. Los elastómeros de poliuretano, poseen una resistencia al desgaste superior a las del caucho y esto permitió desplazar a los cauchos en muchas aplicaciones, principalmente en la industria minera.

1.1.4 Aplicaciones de los poliuretanos

Hoy en día la cantidad de aplicaciones de los poliuretanos es enorme, por ejemplo, como: pinturas, lacas, espumas, implantes médicos, y aplicaciones industriales como: rodillos, aislantes eléctricos, componentes de ingeniería, suela de zapatos, sellos, etc. (Figura 5)

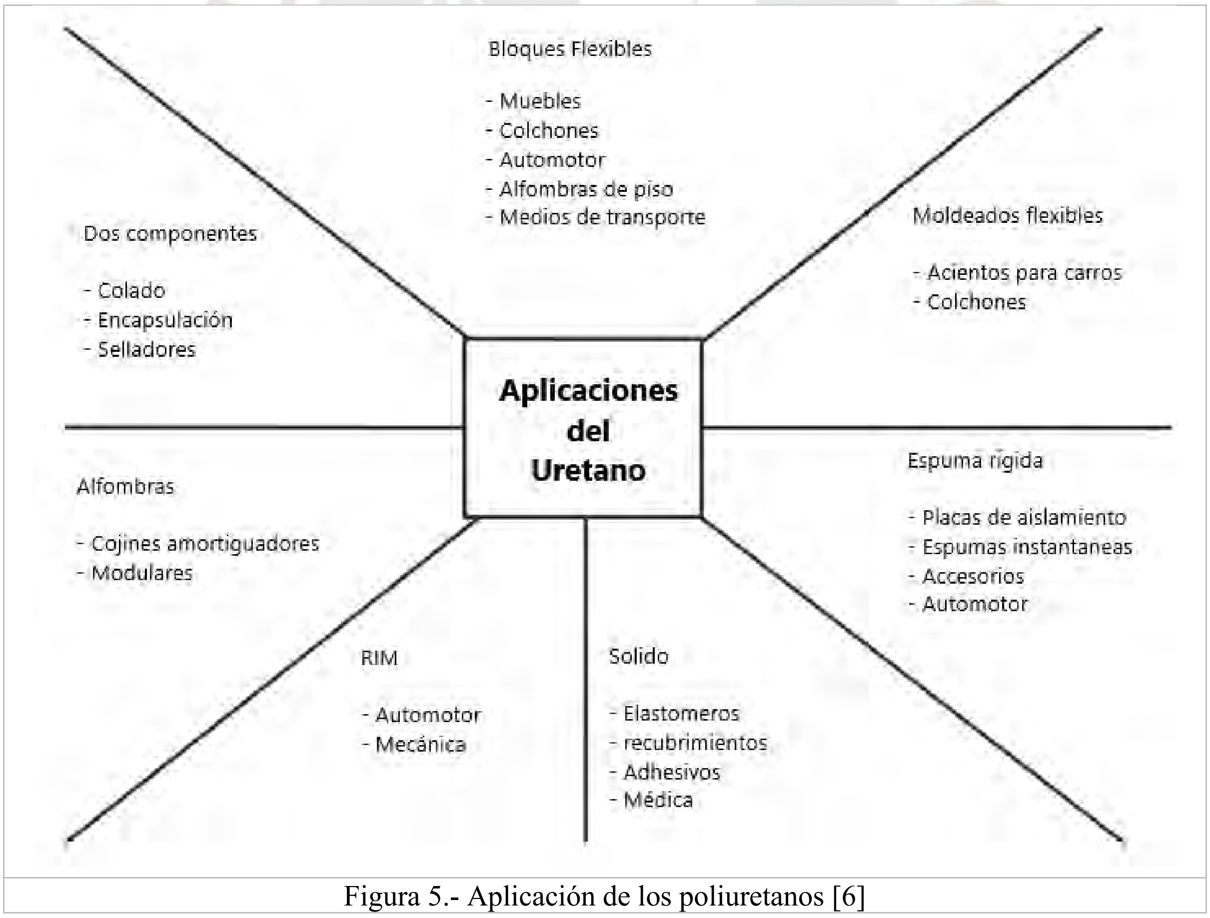


Figura 5.- Aplicación de los poliuretanos [6]

1.2 Introducción al trabajo de investigación

Los elastómeros de poliuretano (PUE) se consideran materiales de ingeniería cuyos componentes químicos provienen principalmente del petróleo. Los PUE se utilizan generalmente para contrarrestar el desgaste de los equipos en diferentes tipos de industrias. Estos materiales se utilizan por ejemplo en la industria minera [1, 7, 8]. En las últimas décadas, el elevado consumo de poliuretanos ha producido una importante cantidad de residuos. Entre los diferentes tipos de poliuretanos consumidos, los PUE tienen una participación suficientemente significativa como para generar problemas de contaminación [9, 10]. En este contexto, los PUEs de base biología pueden ser una alternativa amigable con el medio ambiente porque serían biodegradables por lo menos parcialmente.

La cadena de estructura segmentada del PUE está constituida por isocianatos, polioles y extensores de cadena. Entre estos componentes, los polioles han sido indicados como esenciales para determinar las propiedades finales de los elastómeros [11]. Por ello, muchos trabajos se han enfocado en la síntesis de PUE utilizando varios polioles tipo poliéster de base biológica (BBPE), por ejemplo, los dioles dímeros de ácidos grasos (FADD), debido a que estos polioles ofrecen ventajas sobre los polioles tipo poliéster convencionales, tales como mayor biodegradabilidad, resistencia térmica, mejor miscibilidad con poliéteres convencionales y una producción sostenible de bajo costo [12–14]. Sin embargo, el PUE sintetizado con BBPE tiene una aplicación reducida en la industria minera principalmente porque no puede cumplir con los requerimientos de la resistencia al desgaste en esta industria, lo que sí es posible lograr con PUEs de base poliéster y poliéter convencionales [15–17]

Generalmente, existen dos mecanismos de desgaste asociados con las superficies del PUE utilizadas en la industria minera, la abrasión y la erosión [1]. La resistencia a la abrasión y erosión del PUE está estrechamente relacionada con la estructura química y el grado de separación de fases en la microestructura polimérica que ocurre debido a la incompatibilidad entre los segmentos duros (isocianatos y extensores de cadena) y blandos (polioles) de las cadenas de poliuretano, donde los segmentos duros pueden interactuar entre sí a través de un enlace de hidrógeno, dando lugar a una fase dura que es miscible con la fase blanda [15, 17–19]. El grado de separación de fases (DPS) en la microestructura del PUE se puede determinar midiendo, a través de la Espectroscopia Infrarroja por Transformada de Fourier (FTIR), los cambios en la intensidad del enlace puente de hidrógeno entre el hidrógeno activo en el nitrógeno del grupo uretano y el átomo de oxígeno en el grupo carbonilo del uretano o en los grupos éster o éter de los polioles [20–22]. El grado de participación de los grupos carbonilo en el enlace de hidrógeno, denominado índice de enlace de hidrógeno (R), permite obtener el DPS [15, 21]

Por otro lado, los cambios en la intensidad de los enlaces de hidrógeno detectado por FTIR también permite analizar la miscibilidad entre polioles [22].

Se ha encontrado utilizando el FTIR que las cadenas colgantes en el BBPE impiden estéricamente la formación de enlaces de hidrógeno produciendo una disminución en la longitud entre los enlaces cruzados [23, 24]. El PUE obtenido mediante un BBPE puede contener cadenas colgantes cuyo tamaño y reactividad química dependerá del método utilizado para su síntesis. Las propiedades finales dadas al PUE por el efecto de cadena colgante son una función de su estructura química, cantidad y longitud de cadenas colgantes [12, 25–27].

Se ha estudiado, utilizando el método del prepolímero, el efecto de la composición química y la estructura física resultante de los polioles sobre la resistencia a la abrasión del elastómero final [15], donde la interacción entre los segmentos duros y blandos en un PUE de tipo poliéster es más fuerte que para un PUE de tipo poliéter, y a su vez, la resistencia a la abrasión del PUE poli (éster o éter) aumenta cuando el DPS disminuye [15, 17, 28]. Sin embargo, la relación entre la composición química de BBPE y la separación de fases en PUE aún no está clara. Actualmente, no hay suficiente información en la literatura sobre el uso de FADD en la síntesis de un PUE resistente al desgaste capaz de ser utilizado en componentes que sufren degaste severo por erosión y abrasión, como los de la industria minera.

Este trabajo de tesis tuvo como objetivo estudiar cual es la influencia de la formulación de un elastómero de poliuretano con un poliol de base biológica sobre las propiedades de desgaste del elastómero final. Por lo tanto, se evaluó el efecto de la adición de FADD sobre el grado de separación de fases y la resistencia al desgaste del elastómero de poliuretano obtenido. También se reportó la miscibilidad entre polioles, el FADD y el poliéter a base de petróleo.

1.3 Objetivo y justificación

Este trabajo tuvo como objetivo estudiar cual es la influencia de la formulación de un elastómero de poliuretano con un poliol de base biológica sobre las propiedades de desgaste del elastómero final, que podría ser aplicado en repuestos para maquinaria y equipos de la industria minera. De acuerdo con los mecanismos de desgaste encontrado (abrasión y erosión) en las superficies de los repuestos, estas se protegen en su mayoría con elastómeros de caucho y poliuretano [7, 29]. Hoy en día, entre los sistemas PUE utilizados en la producción de elastómeros de poliuretano, los MDI-Poli(éster o éter)-BDO son los preferidos por tener un alta resistencia al desgaste, un procesamiento amigable con la salud del operario y una dureza regulable [1, 30]. Se ha mostrado en la literatura una buena miscibilidad entre algunos polioles de tipo poliéster de base biológica (BBPE) y polioles de poliéteres convencionales [14, 31, 32]. Hubo algunos intentos de encontrar PUE de alto rendimiento utilizando BBPE [12,13]. Sin embargo, los resultados de resistencia a la abrasión [13] podrían no ser suficientes para las aplicaciones en la industria minera [15]. Por esa razón, evaluamos el efecto de la adición gradual de FADD en el comportamiento de desgaste de un PUE-poliéter elaborado mediante el método de polimerización de dos pasos (método de prepolímero). Se seleccionó un poliol a base de éter (PTMEG) con un peso molecular relativamente alto (PM = 2900 gr / mol) para aumentar la resistencia final a la abrasión del poliuretano elastomérico PUE. La cantidad de segmento blando en el PUE final se estableció en función de la dureza requerida en la producción estándar de paneles de clasificación de la industria minera [29].

CAPÍTULO II

PARTE EXPERIMENTAL

2.1 Materiales y síntesis

Se sintetizaron y caracterizaron elastómeros de poliuretano (PUE) a base de polioles, tipo poliéter, poliéster y sus combinaciones. Para el segmento blando del PUE se utilizó politetrametileno éter glicol con peso molecular 2900 (PTMEG 2900, Aldrich) y poliéster FADD el cual es derivado de ácido linoleico dimerizado e hidrogenado con peso molecular 2000 (PRIPLAST 1838 ™, Firma Croda), mientras que el segmento duro consistió en Di-isocianato de 4,4'-difenilmetano (MDI 98%, Aldrich) y 1,4-butanodiol (BDO 99%, Aldrich), este último se utilizó como extensor de cadena. Los polioles y BDO se secaron al vacío a 90-100°C durante 2 horas antes de su uso.

Los elastómeros se sintetizaron utilizando el método del prepolímero (método de dos pasos) conteniendo porcentajes similares de segmentos blandos.

Procedimiento típico: En la primera etapa, 76.1 gramos de MDI, previamente fundido a 50°C, se colocó en un reactor de vidrio de 1000 ml equipado con una termocupla y un agitador de paletas bajo una atmósfera de nitrógeno. Posteriormente, se añadió cloruro de benzoílo, hasta 33 microequivalentes por gramo de planeado prepolímero, para evitar reacciones secundarias y gelificación [1]. Se añadió al MDI lentamente 165.21 gramos de poliéster y luego 161.67 gramos de poliéter a 50-55ºC. Una vez añadidos los polioles, la temperatura de reacción se elevó a 80ºC y la mezcla se agitó continuamente durante 2 h hasta completar la reacción. El progreso de la reacción fue seguido por titulación con n-dibutilamina (Anexo V). Los prepolímeros obtenidos se secaron al vacío a 100°C durante 1 h antes de su uso. La denominación de las muestras se hizo de la siguiente manera: por ejemplo, para la síntesis descrita donde se utilizó 50% en peso del poliol de base bilógica con respecto al total de polioles fue denominada PP-50, análogamente se hizo la denominación con el resto de prepolímeros obtenidos.

En la segunda etapa, se pesó la cantidad requerida de prepolímero y 1,4-butanodiol en un vaso de precipitados, se agitó intensamente, se desgasificó al vacío y se vertió en un molde metálico calentado a 100ºC, tratado previamente con un desmoldeante de silicona. Transcurridos 30 min, las muestras se retiraron del molde y se post-curaron inmediatamente en un horno durante 18 h a 120ºC. Finalmente, todas las muestras se almacenaron durante 21 días a temperatura ambiente antes de realizar todos los ensayos de caracterización. La denominación de las muestras de poliuretanos elastoméricos obtenidos se hizo en función a la denominación del prepolímero usado, esto es, el elastómero E-50 fue obtenido a partir del prepolímero PP-50. Se procedió en forma análoga para el resto de los elastómeros obtenidos.

2.2 Métodos de caracterización

2.2.1 Determinación del porcentaje en peso de isocianato en las muestras de prepolímero de poliuretano

Se colocaron muestras de prepolímero con un peso aproximado de 1 g en un matraz Erlenmeyer de 250 ml. Luego se agregaron 50 ml de una solución de n-dibutilamina 0,05 N (99,5%, Aldrich) en tetrahidrofurano (THF), el cual fue previamente secado. Se añadió THF adicional con agitación constante hasta que el prepolímero se disolvió por completo. Se utilizó ácido clorhídrico 0,1 N, previamente estandarizado (Anexo IV), para la titulación del exceso de n-dibutilamina. El porcentaje en peso del contenido de isocianato (NCO) en la muestra se calculó mediante la Ecuación 1 (Anexo V).

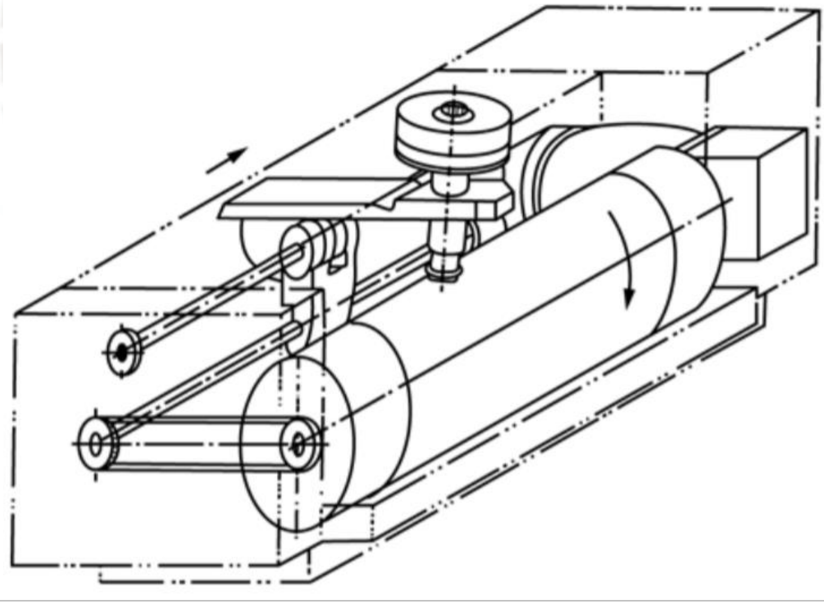
$$wt.\%\,NCO = \frac{(V_b - V_a) \times N \times 0.042}{m_p} \times 100 \qquad (1)$$

Donde: V_b es el volumen de HCl, en mililitros usado en la titulación de 50 ml de n-dibutilamina (sin contener la muestra, reactivo blanco), V_a es el volumen de HCl, en mililitros, usado en la evaluación de la muestra, y m_p es la masa del prepolímero, en gramos.

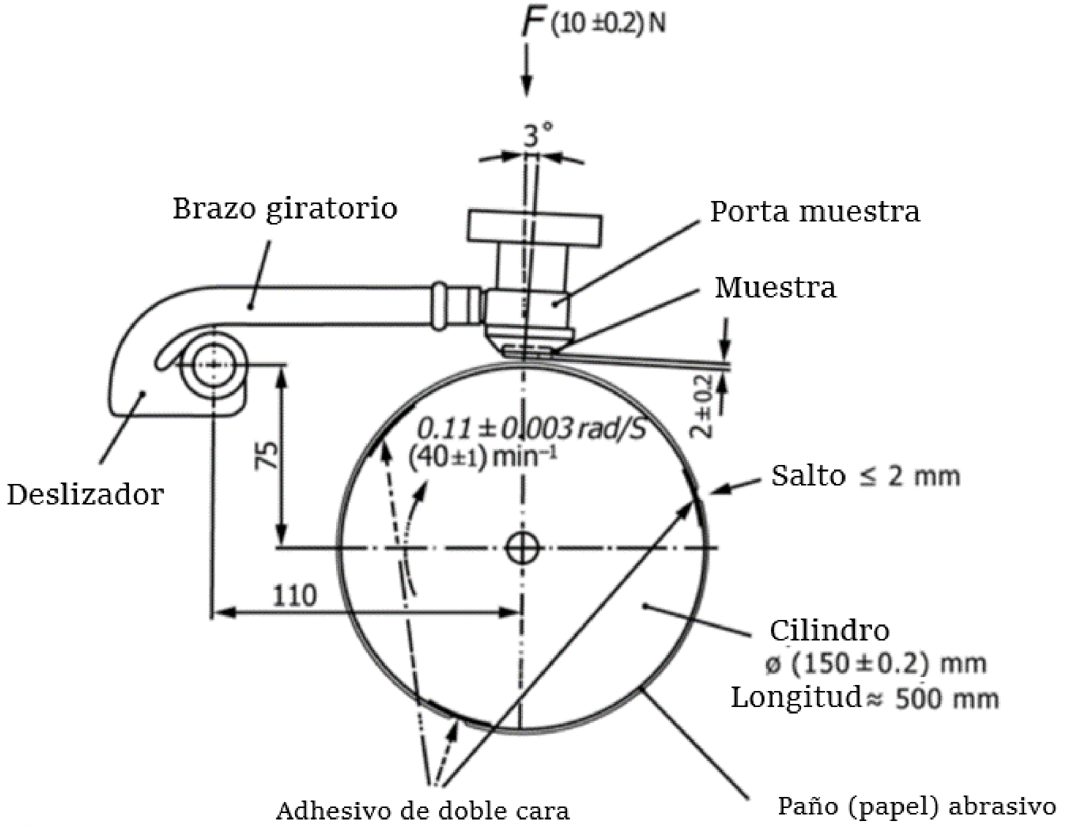
2.2.2. Resistencia a la abrasión

La resistencia a la abrasión se midió empleando un abrasímetro (Figura 6) de rodillo cilíndrico giratorio de acuerdo con las normas ASTM D 5963 (Anexo VI). Se usó una muestra de elastómero con una forma cilíndrica de 16 mm de diámetro y 2 mm de altura. El elastómero cilíndrico se fijó en la porta muestra para luego deslizarse sobre la hoja abrasiva adherida al rodillo metálico giratorio (Esquema 3 y 4). La distancia total de deslizamiento fue de 40 metros y la presión de contacto sobre la superficie rugosa y abrasiva fue de 10 N. La resistencia a la abrasión se determinó como una pérdida de volumen de la muestra, corregida con la abrasividad de la hoja abrasiva el cual es medido mediante un material de referencia. Se utilizó como material de referencia estándar una goma estándar proporcionada por el Instituto Federal de Investigación y Ensayo de Materiales (Berlín, Alemania).

Figura 6.- Abrasímetro de rodillo cilíndrico giratorio



Esquema 3.- Esquema del equipo de abrasión



Esquema 4.- Vista de perfil y dimensiones del equipo de abrasión

La hoja abrasiva se calibró para una pérdida de masa de material de referencia estándar, entre 180 y 200 mg para una distancia de abrasión de 40 m. Después de la prueba de abrasión, se determinó la pérdida de masa de las muestras y se calculó su volumen a partir de la densidad de los materiales. La pérdida de volumen de todas las muestras se comparó con los resultados de la referencia en las mismas condiciones. La resistencia a la abrasión se calculó utilizando la ecuación 2 [15] (Anexo VI).

$$A = \frac{\Delta m \ \times S_0}{d_1 \times S} \tag{2}$$

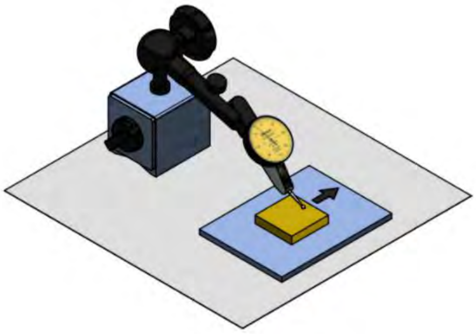
Donde, A es la resistencia a la abrasión definida por la pérdida de volumen de la muestra, en mm^3; Δm es la pérdida de masa de la muestra, en mg; d_1 es la densidad del elastómero analizado, en mg x mm^{-3}; S_0 es la pérdida de masa del material estándar (definida como 200 mg), S es la media aritmética de la pérdida de masa de tres muestras del material estándar, en mg.

2.2.3. Resistencia a la erosión

Las pruebas de erosión se llevaron a cabo en una máquina de erosión de fabricación propia basada en ASTM D-968 (Anexo VII). Las principales variaciones, con relación al estándar, del dispositivo ASTM fueron las siguientes: aumento de la longitud del tubo guía hasta 2 m, grano de acero abrasivo en lugar de arena y flujo constante que cae por medio del sistema elevador de cangilones (Figura 7). Estas variaciones permitieron generar huellas de desgaste con profundidad que era medible (mayor a 3 μm) en los materiales elastoméricos, y no era ese el caso con la configuración original de la norma usada. El borde afilado del abrasivo se gastó antes de su uso. Se cortó una muestra cuadrada con un tamaño de 40 mm x 40 mm para las pruebas de erosión. Las condiciones de la prueba de erosión se enumeran en la Tabla 1. Antes y después de las pruebas de erosión, se limpió la muestra con alcohol isopropílico (10% v / v), se secó y se escaneó tres veces para cada muestra con un palpador horizontal (Esquema 5) para medir la profundidad de desgaste. Antes y después de cada prueba, el rendimiento de la máquina de prueba de erosión se calibró utilizando el material de referencia estándar mencionado anteriormente en el apartado de resistencia a la abrasión. El grado de erosión se obtuvo mediante la medida de la profundidad de desgaste promedio (en μm).

Figura 7.- Esquema de la máquina de prueba erosiva de fabricación propia basada en ASTM D-968

Tabla 1.- Condiciones del ensayo de erosión	
Parámetros de ensayo	
Tipo de erosivo	Grano de acero angular - AMASTEEL® G14
Tamaño del erosivo	1.18-1.40 mm
Temperatura de ensayo	Temperatura ambiente (20ºC)[1]
Rapidez de alimentación del erosivo	70 kg/min
Tiempo de ensayo	6 horas
[1] hubo un pequeño aumento en la temperatura de la muestra debido a la colisión con las partículas abrasivas. Este aumento no fue superior a 2°C.	



Esquema 5.- Medición de la profundidad del desgaste con un palpador horizontal.

2.2.4. Densidad

La determinación de la densidad de los elastómeros se realizó según el método ASTM D1817(Anexo VIII).

2.2.5. Propiedades mecánicas de los elastómeros

Los resultados de dureza fueron obtenidos por medio de cinco medidas al azar en cada espécimen con durómetro modelo Rex® 1600 tipo A basado en ASTM D-2240 (Anexo IX). Mientras que, la resistencia a la tracción y el módulo a 300% se midieron en un medidor de fuerza Lutron FG-5020 acondicionado, donde la velocidad de ascenso fue de 50 mm / min según ASTM D-412 (Anexo X) utilizando al menos cinco muestras en forma de mancuerna de 1 mm de espesor para cada tipo de elastómero. Todas las pruebas se realizaron a temperatura ambiente (20°C) y 70% de humedad relativa.

2.2.6. Resiliencia

El ensayo de resiliencia se realizó mediante un equipo de rebote vertical basado en la norma ASTM D-2632 (Anexo XI). La resiliencia se determinó como la relación entre la altura de caída y la altura de rebote de un embolo de forma y masa conocidas. Se realizaron 5 medidas de la resiliencia a temperatura ambiente, y el resultado final se expresó como el valor medio de las mediciones.

2.2.7. Miscibilidad de polioles

La miscibilidad de los polioles fue estudiada mediante los espectros FTIR de las mezclas formadas por los polioles. Se usó un equipo de vórtice hecho a medida para mezclar polioles. Este dispositivo realiza un proceso de mezcla de doble eje sin paleta de mezcla (Figura 8). Los polioles se fundieron antes de mezclarse y de realizar la prueba. Todas las condiciones de mezcla se muestran en la Tabla 2. Todas las mezclas de polioles se caracterizaron a temperatura ambiente y 60°C.

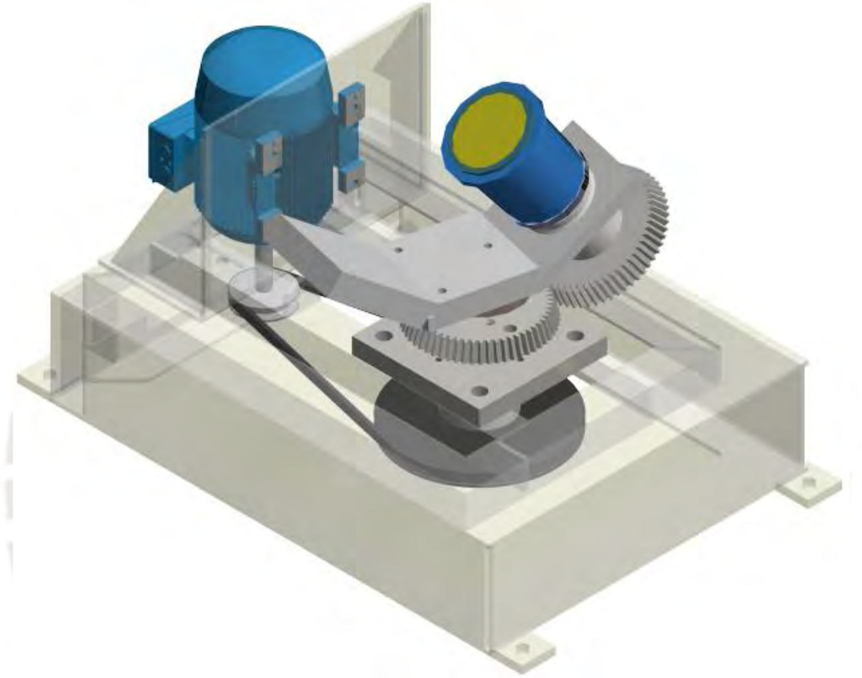


Figura 8.- Maquina mezcladora de vórtice

Tabla 2.- Parámetros para la elaboración de blendas	
Parámetros de ensayo	
Componentes de la blenda	PRIPLAST 1838 (FADD) y PTMEG 2900
Proporción de blenda	0, 25, 50, 75 y 100 % en peso de PRIPLAST 1838 (FADD)
Velocidad de la mezcladora	400 RPM
Temperatura de mezcla	40°C
Tiempo de mezcla	2 min

2.2.8. Espectroscopía infrarroja por transformada de Fourier

Se caracterizaron las mezclas de polioles y los prepolímeros usando un espectrofotómetro Perkin Elmer Spectrum 400 FTIR-ATR, para la medición se puso una gota de mezcla en la porta muestra del equipo. Mientras que los elastómeros PUE se caracterizaron utilizando un espectrofotómetro Alpha Bruker FTIR-ATR, se puso pequeños filamentos de 1 mm de espesor sobre la porta muestra del equipo. En ambos casos, las muestras usadas no recibieron ningún tratamiento adicional. Todos los resultados FTIR-ATR se muestran sin tratamiento adicional con excepción de las bandas que corresponden al grupo carbonilo C=O de los elastómeros, que se detallan más adelante. Los espectros se registraron a temperatura ambiente y 60°C en un rango de números de onda entre 400 y 4000 cm $^{-1}$ con una resolución de 4 cm $^{-1}$,

El grado de mezcla de fases (DPM) y de separación de fases (DPS) de los poliuretanos elastoméricos se calcularon utilizando las ecuaciones 3-5 [21].

$$R = \frac{A_b}{A_{nb}} \tag{3}$$

$$DPS = \frac{R}{R+1} \tag{4}$$

$$DPM = 1 - DPS \tag{5}$$

Donde, R es el índice de enlaces de hidrógeno originados por el grupo carbonilo y se determinó mediante la comparación de la intensidad de las vibraciones del grupo carbonilo enlazado (A_b) y del grupo carbonilo no enlazado (A_{nb}), cuyas bandas estaban ubicadas en 1700 cm $^{-1}$ y 1730 cm $^{-1}$, respectivamente [20]. El Índice R se calculó utilizando las áreas de bandas de absorción de carbonilo enlazados y no enlazados, proporcionados por la espectroscopia FTIR-ATR , donde se utilizó el Software Origin Pro para la corrección de la línea base y encontrar la superposición de picos (grupos de carbonilos libres y enlazados) por el método de deconvolución [15, 20, 21].

2.2.9. Calorimetría diferencial de barrido (DSC) y Análisis termogravimétrico (TGA)

Los análisis de DSC y TGA se realizaron en un equipo TA Instruments modelo DSC 2500 bajo atmósfera de nitrógeno, las muestras de poliuretanos elastoméricos con masa entre 6 y 10 mg se colocaron dentro de una capsula de aluminio. La DSC se realizó en un rango de -100 a 150°C con una velocidad de calentamiento de 5°C min $^{-1}$, y TGA de 25 a 700°C con una velocidad de calentamiento de 10°C min $^{-1}$. En el análisis DSC la temperatura de transición vítrea se tomó como el punto de inflexión de la curva de capacidad calorífica versus temperatura.

CAPÍTULO III

DISCUSIÓN DE RESULTADOS

3.1 Miscibilidad de polioles

El PRIPLAST 1838^{TM} es un poliol amorfo y compatible con componentes de baja polaridad, como lo indica el fabricante (Firma Croda). Por otro lado, el PTMEG es un poliol de tipo poliéter de polaridad relativamente baja [11]. Por tanto, cabría esperar que haya miscibilidad entre ambos polioles. Según la literatura, el grado de miscibilidad de la mezcla de polioles se puede determinar mediante el estudio de las interacciones entre sus grupos funcionales, como, por ejemplo, el enlace tipo puente de hidrógeno, y se puede estudiar directamente mediante la espectroscopía de infrarrojo por transformada de Fourier (FTIR) [22, 33, 34].

Los espectros FTIR de las mezclas de polioles se usaron para seguir la interacción química entre los grupos funcionales de los polioles causada por el reemplazo gradual del PTMEG por el FADD desde 0% en peso para B-0 hasta 100% en peso para B-100, a dos temperaturas, 25 y 60ºC, respectivamente. En la literatura, se puede encontrar una descripción detallada de las señales IR de poli (éter o éster) [33]. Las señales importantes están ubicadas entre 3300 y 3700 cm^{-1} (región O-H) y entre 1650 y 1800 cm^{-1} (región C = O). Un pico a 3485 cm^{-1} es atribuido a las vibraciones de estiramiento de los grupos hidroxilo unidos por enlaces de hidrógeno del PTMEG puro (B-0) [33,34]. Se encontró que este pico cambia progresivamente a números de onda más bajos al aumentar la cantidad de FADD, desde 3485 para B-0 hasta 3450 cm^{-1} para B-100 (FADD puro) a 25°C (Figura 9) y desde 3485 para B-0 hasta 3444 cm^{-1} para B-100 a 60°C (Figura 10). Este cambio podría significar la interacción mediante enlace de hidrógeno entre el grupo éster del FADD y el grupo hidroxilo del PTMEG, y una señal más difusa a 60°C podría indicar una interacción más fuerte entre estos grupos, mejorando la compatibilidad entre ellos lo cual fue corroborado cualitativamente por microscopía óptica. Además, la formación de hombros se puede ver aproximadamente a 3525 cm^{-1} (Figura 9 y 10), lo cual se puede atribuir a la vibración de estiramiento de un grupo hidroxilo, sin enlaces de hidrogeno, en la mezcla de polímeros debido al efecto estérico de la cadena colgante de la estructura química del FADD (Figura 11 a, b) [22, 23, 25, 27].

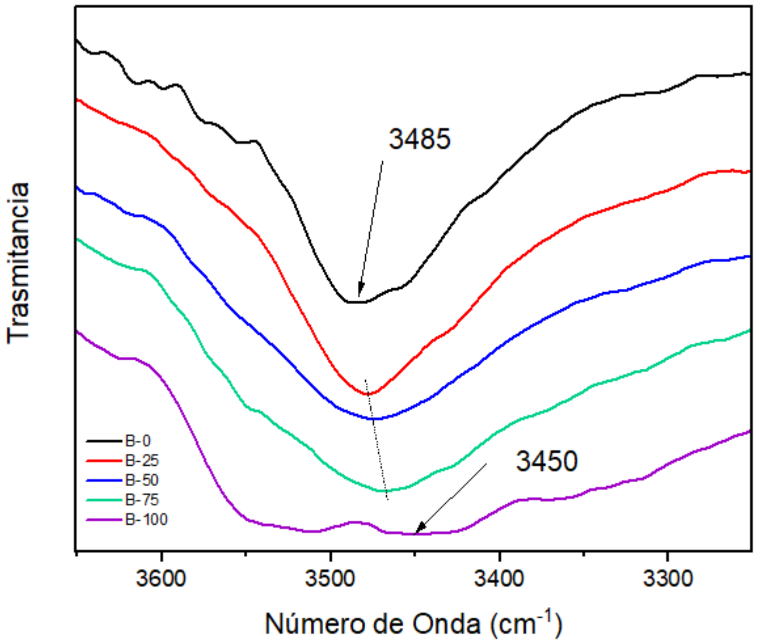


Figura 9.- Espectros ATR-FTIR de la mezcla de poliol PTMEG y FADD con contenido de FADD que varía de 0 (B-0) a 100% (B-100) en peso, espectros detallados de la región hidroxilo OH a 25°C

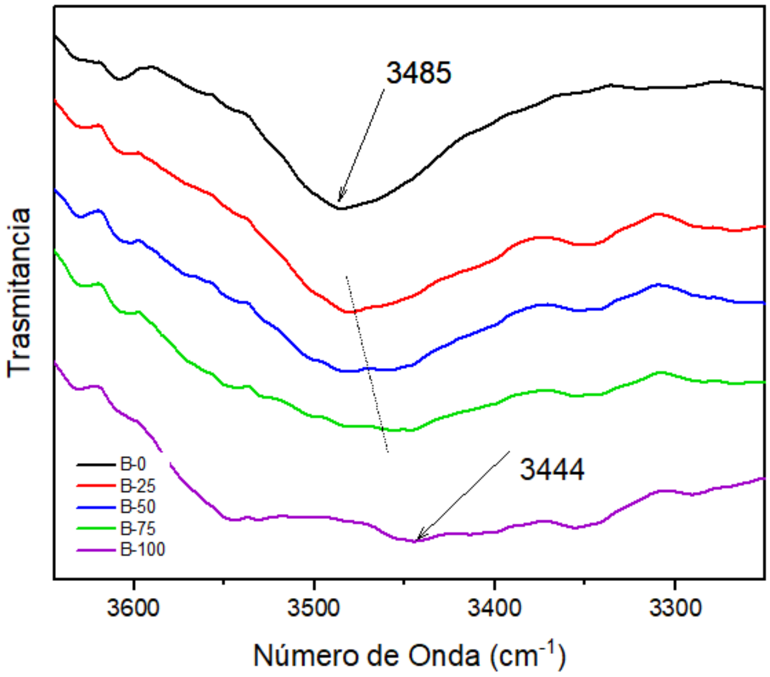


Figura 10.- Espectros ATR-FTIR de la mezcla de poliol PTMEG y FADD con contenido de FADD que varía de 0 (B-0) a 100% (B-100) en peso, espectros detallados de la región hidroxilo OH a 60°C

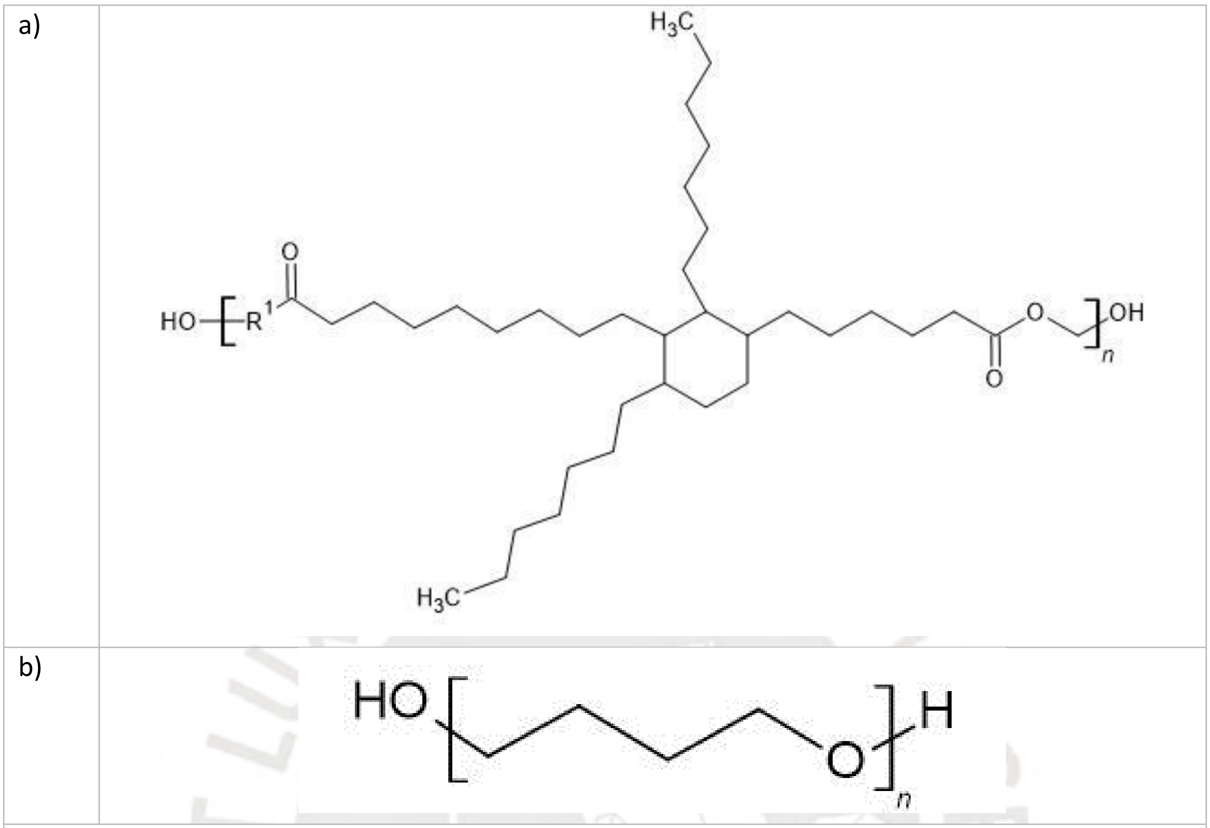


Figura 11.- Estructuras moleculares de los polioles en la mezcla a) diol dímero de ácido graso FADD (Priplast 1838), b) diol poliéter (PTMEG 2900)

En la región del carbonilo (C = O) entre 1710 y 1760 cm^{-1}, no hay señal de PTMEG puro, porque no contiene grupos carbonilo, y la intensidad de las vibraciones del grupo carbonilo del FADD crece con el aumento del contenido de FADD en la mezcla (Figura 12 y 13). La señal, aproximadamente a 1719 cm^{-1}, a 60°C, que se atribuye al grupo carbonilo que forma puentes de hidrogeno (Figura 13) aparece muy levemente. Esto debería indicar una posible interacción de enlaces puentes de hidrógeno de los grupos C = O del FADD con grupos O-H de la mezcla de polioles. Sin embargo, esta señal no indica claramente la mejora de la compatibilidad entre polioles con incremento de la temperatura desde 25 hasta 60°C. La señal de carbonilo es muy fuerte en el FTIR y no está significativamente influenciada por posibles enlaces de hidrógeno.

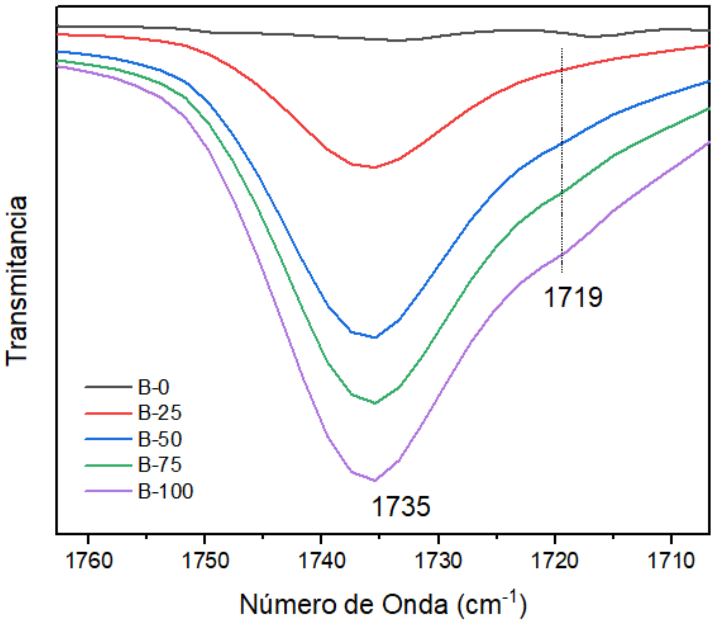


Figura 12.- Espectros ATR-FTIR de la mezcla de poliol PTMEG y FADD con contenido de FADD que varía de 0 (B-0) a 100% (B-100) en peso, espectros detallados de la región carbonilo C=O a 25°C

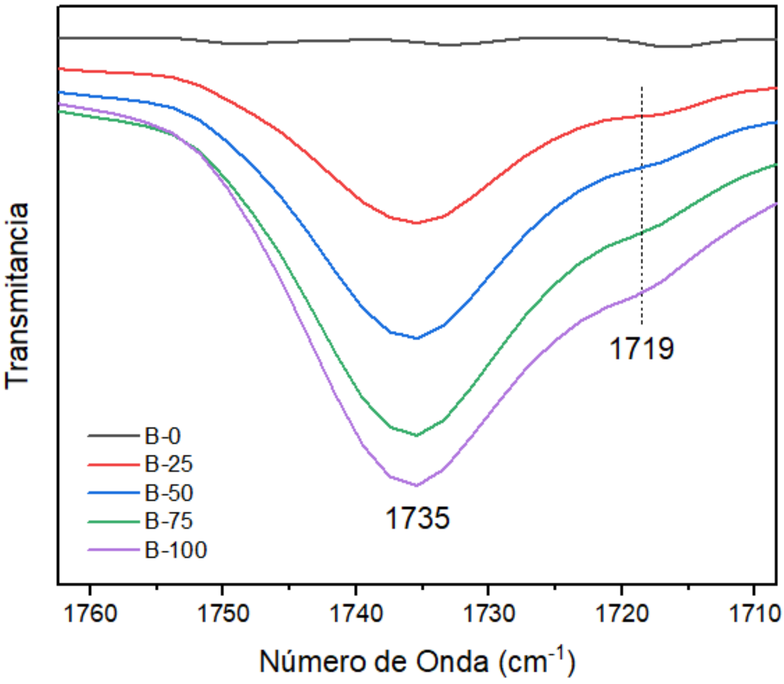


Figura 13.- Espectros ATR-FTIR de la mezcla de poliol PTMEG y FADD con contenido de FADD que varía de 0 (B-0) a 100% (B-100) en peso, espectros detallados de la región carbonilo C=O a 60°C

Por otro lado, a través de la inspección visual a temperatura ambiente de la mezcla de polioles, se verificó que estos formaban aparentemente una sola fase en cada mezcla con una variación de tonalidad dependiendo del contenido de FADD (Figura 14). Además, se pudo ver cualitativamente, a través de imágenes de la microscopía óptica, una mejora de la compatibilidad entre los polioles de base éster y éter (FADD y PTMEG) en las mezclas B-25, B-50 y B-75 al mezclar los polioles a 60°C (Figura 15d-f) con respecto a la mezcla a 25°C (Figura 15a-c).

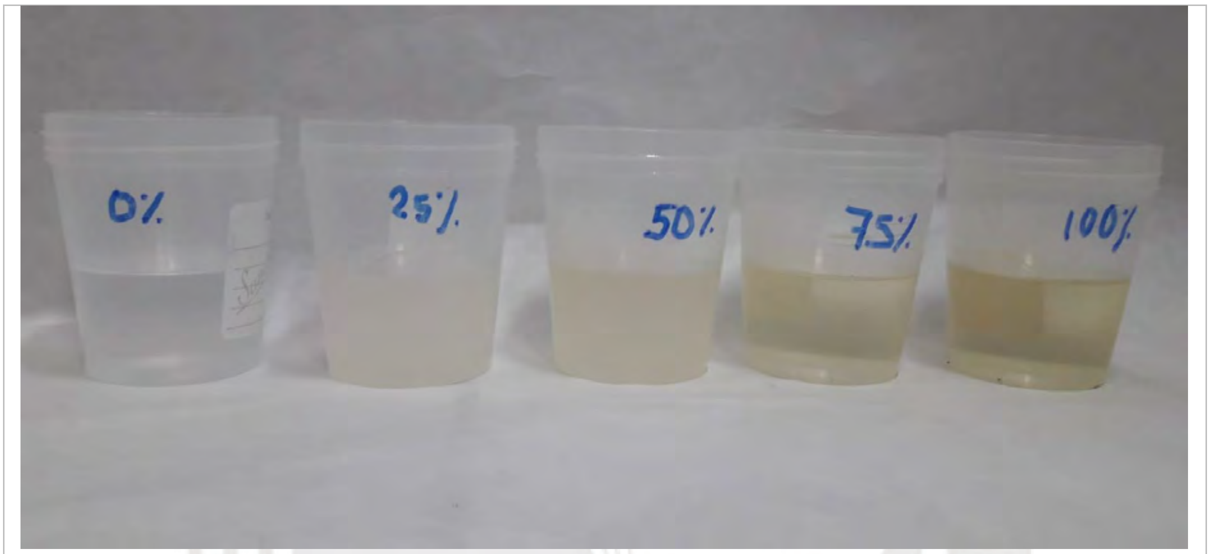


Figura 14.- Imagen de las blendas de polioles conteniendo desde 0% hasta 100% en peso de FADD.

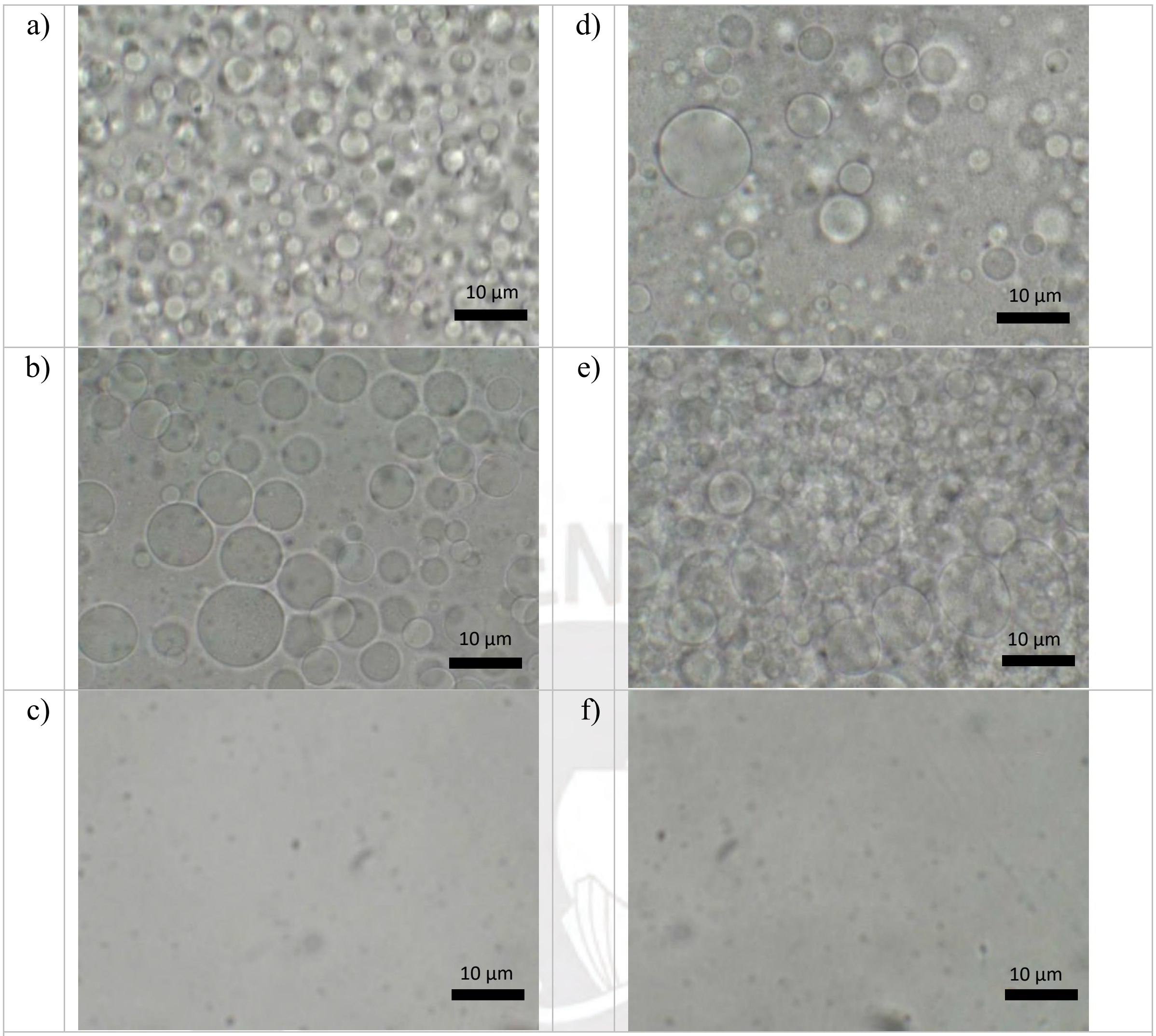


Figura 15.- Estudio de la compatibilidad de los polioles PTMEG y FADD a diferentes temperaturas mediante microscopía óptica convencional (con 40 aumentos) con contenido de FADD: a) 25%, b) 50%, y c) 75% en peso a 25 ° C y d) 25%, e) 50% y f) 75% en peso a 60ºC.

3.2. Síntesis de prepolímeros y elastómeros

Se realizó la reacción de los polioles e isocianato para obtener los prepolímeros, donde en todos los casos, después de dos horas de reacción se observó un contenido del NCO constante indicando la formación de los prepolímeros (Figura 16). Se realizó una reacción de extensión de cadena mediante la reacción de los prepolímeros, conteniendo grupos terminales isocianato, y el 1,4-butanodiol BDO y se obtuvieron poliuretanos elastoméricos de tipo poli(éter-éster). Las reacciones involucradas se pueden representar esquemáticamente (Esquema 6 a 11), donde las posibilidades de reacciones involucradas en la síntesis de los prepolímeros estarían relacionadas a la interacción de los compuestos utilizados, esto es, MDI+FADD, MDI+PTMEG y MDI+FADD+PTMEG (Esquemas 6, 7 y 8). De forma similar para las reacciones involucradas en la síntesis de los poliuretanos elastoméricos (Esquema 9, 10 y 11).

Para la síntesis de los diferentes tipos de elastómeros se utilizó el mismo porcentaje en peso del isocianato NCO del prepolímero resultante (Tabla 3 y 4) con el objetivo de obtener poliuretanos elastoméricos de similares segmentos blandos (Tabla 5 y 6). Esta reacción de extensión de cadena fue programada de tal manera que hubo tiempo suficiente para mezclar, desgasificar y colar en el molde a todas las muestras preparadas. El aumento en la cantidad de FADD en el prepolímero dio como resultado una mayor viscosidad del mismo. No obstante, se determinó cualitativamente que todas las mezclas de prepolímeros podían procesarse manualmente.

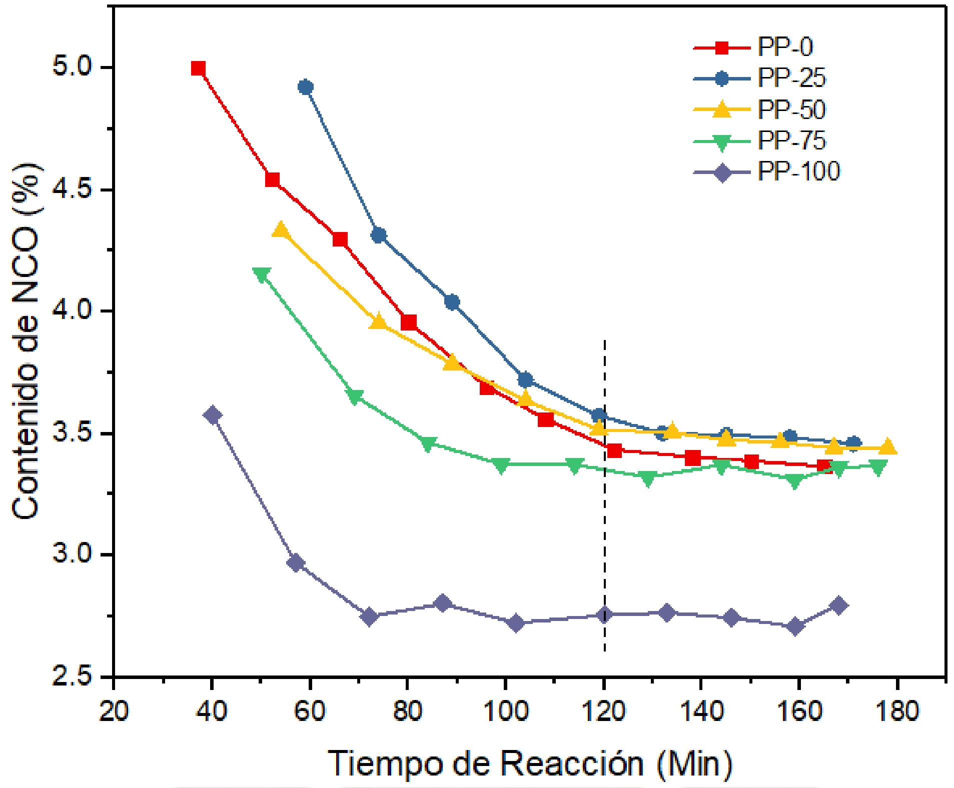
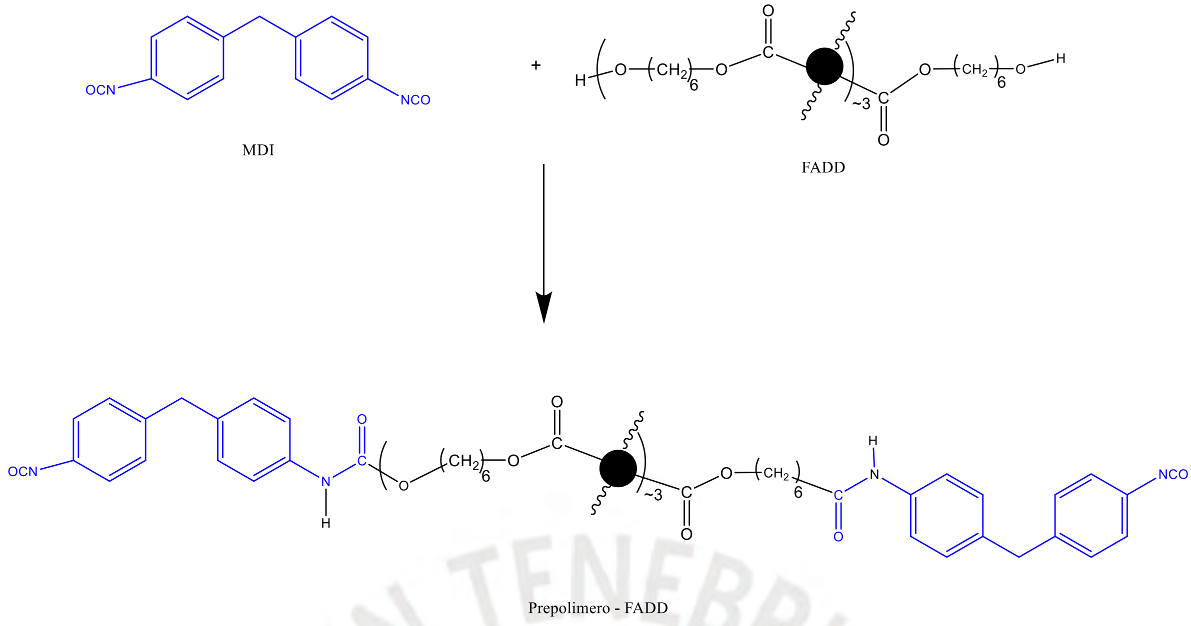
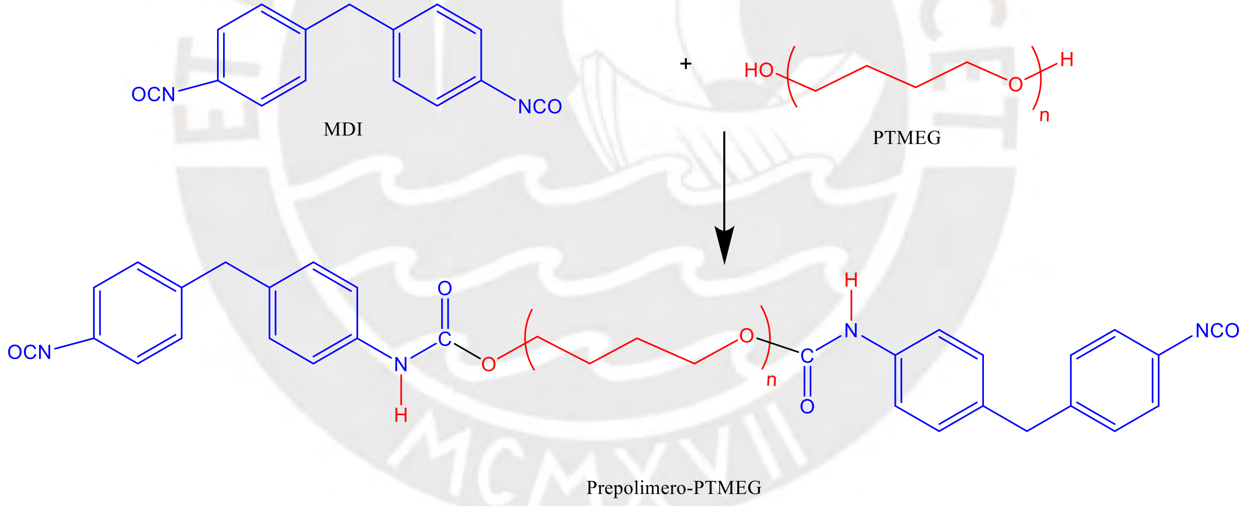


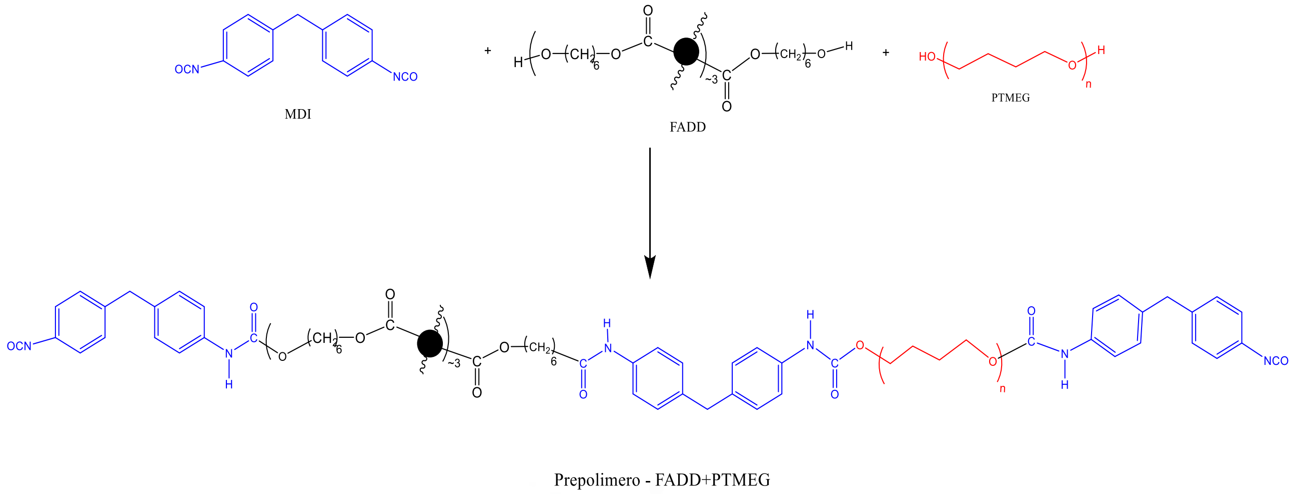
Figura 16.- Reacción de síntesis del prepolímero, Contenido de NCO vs tiempo de reacción



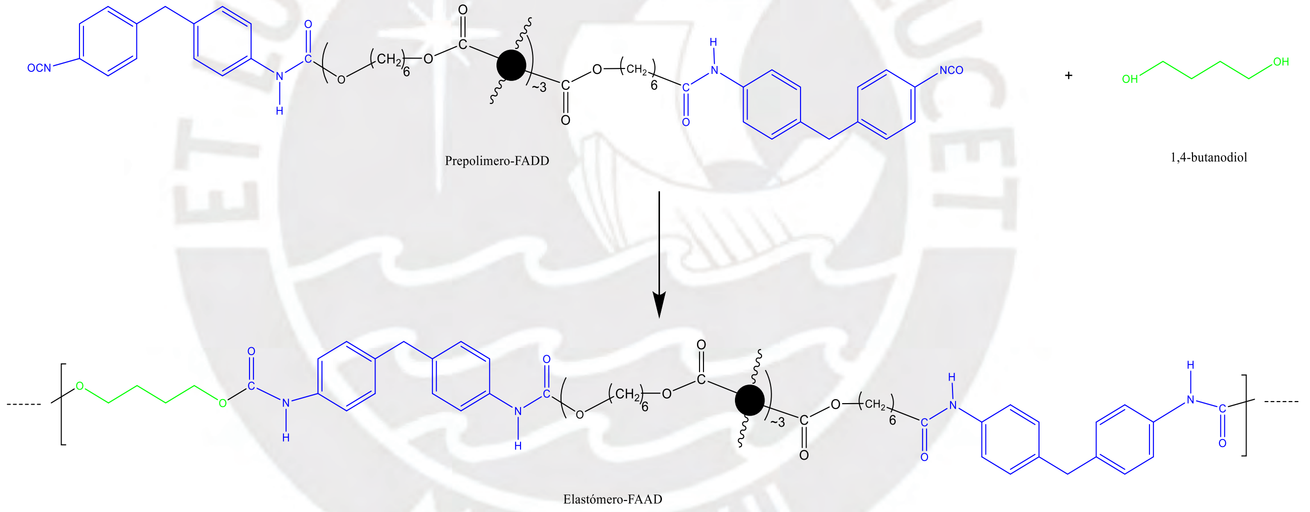
Esquema 6.- Obtención del prepolímero de poliuretano con grupos isocianatos (NCO) terminales a partir del isocianato MDI y poliol FADD



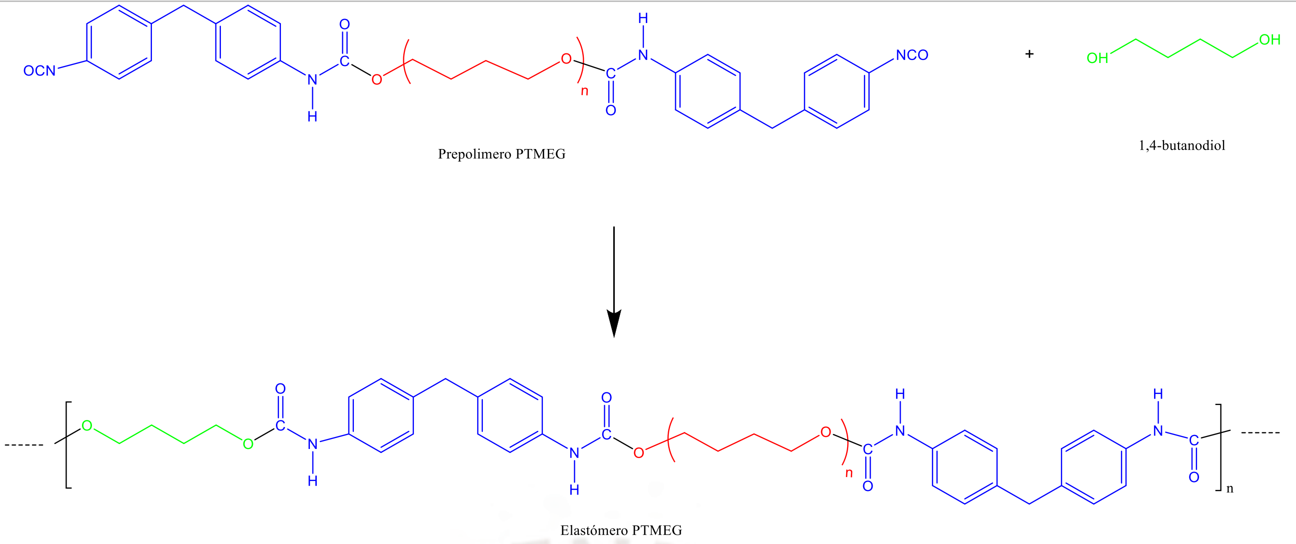
Esquema 7.- Obtención del prepolímero de poliuretano con grupos isocianatos (NCO) terminales a partir del isocianato MDI y poliol PTMEG.



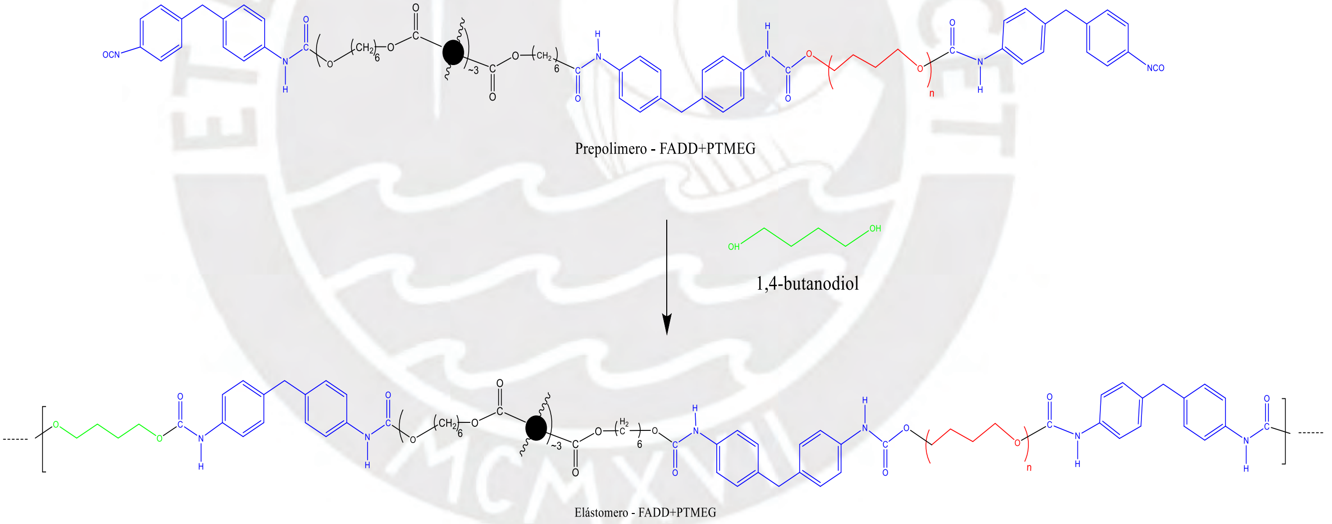
Esquema 8.- Obtención del prepolímero de poliuretano con grupos isocianatos (NCO) terminales a partir del isocianato MDI, poliol FADD y poliol PTMEG.



Esquema 9.- Obtención de poliuretanos elastoméricos a partir del prepolímero-FADD y el butanodiol, como extensor de cadena



Esquema 10.- Obtención de poliuretanos elastoméricos a partir del prepolímero-PTMEG y el butanodiol, como extensor de cadena



Esquema 11.- Obtención de poliuretanos elastoméricos a partir del prepolímero-FADD-PTMEG y el butanodiol, como extensor de cadena

Tabla 3.- Síntesis de prepolímeros

Muestra[a]	MDI[b] (%)	FADD[b] (%)	PTMEG-2900[b] (%)
PP-0	18,10	0,00	81,90
PP-25	18,52	18,78	62,70
PP-50	18,87	41,01	40,13
PP-75	19,25	62,51	18,24
PP-100	19,15	80,85	0,00

[a] Los prepolímeros fueron identificados por su contenido de FADD (% en peso) en la mezcla de polioles, p. ej., Muestra PP-25, significa que el prepolímero se sintetizó usando 25% en peso de FADD respecto de todos los polioles usados en su síntesis, [b] porcentaje en peso de todos los componentes que se utilizaron en la síntesis de los prepolímeros, diisocianato de 4,4'-difenilmetano (MDI), PRIPLAST 1838 (FADD) y Politetrametilen éter glicol (PTMEG-2900)

Tabla 4.- Caracterización del prepolímero

Muestra [a]	Relación NCO / OH [b]	Contenido de NCO (% en peso) [c]
PP-0	2,50	3,38
PP-25	2,32	3,46
PP-50	2,22	3,44
PP-75	2,13	3,37
PP-100	1,94	2,76

[a] Las muestras fueron identificados por su contenido de FADD (% en peso) en la mezcla de polioles, como en la Tabla 3, [b] NCO / OH es la relación de la cantidad molar inicial de isocianato presente en MDI y la cantidad molar de hidroxilo en mezcla de polioles presente en la síntesis del prepolímero, [c] el contenido de NCO representa el contenido final de grupos isocianato en el prepolímero después de la síntesis. Se determinó mediante análisis con dibutilamina y se calculó usando la Ec. 1, y este valor permaneció estable hasta antes de su uso.

Tabla 5.- Síntesis de los poliuretanos elastoméricos

Muestra[a]	MDI[b]	FADD[b]	PTMEG[b]	BDO[b]
E-0	1.00	0.00	0.39	0.54
E-25	1.00	0.13	0.29	0.54
E-50	1.00	0.27	0.18	0.53
E-75	1.00	0.41	0.08	0.51
E-100	1.00	0.53	0.00	0.41

[a] Los poliuretanos elastoméricos fueron identificados por su contenido de FADD (% en peso) en la mezcla de polioles, p. ej., Muestra E-25 significa que el elastómero se sintetizó usando un prepolímero, que contenía 25% en peso de FADD con respecto a todos los polioles utilizados en su síntesis. [b] La relación molar inicial de todos los componentes involucrados en la síntesis de elastómeros de poliuretano, diisocianato de 4,4'-difenilmetano (MDI), PRIPLAST 1838 (FADD), Politetrametilen éter glicol (PTMEG-2900) y el 1,4-butanodiol (BDO).

Tabla 6.- Caracterización de los poliuretanos elastoméricos

Muestra [a]	R [b]	DPS [c]	DPM [d]	SS (%) [d]
E-0	1,54	0,606	0,394	79,14
E-25	1,15	0,535	0,465	78,36
E-50	1,14	0,534	0,466	78,34
E-75	1,12	0,530	0,470	78,35
E-100	1,10	0,525	0,475	79,27

[a] Las muestras fueron identificado por su contenido de FADD (% en peso) en la mezcla de polioles, como en la Tabla 5, [b, c, d] Los valores de R, DPS y DPM se calcularon usando las Ec. 3, 4 y 5. [d] La composición (% en peso) del segmento blando (FADD y PTMEG) en la síntesis de elastómeros respecto a todos los componentes.

El espectro FTIR de los prepolímeros sintetizados (Figura 17) muestra, entre 2100-2400 cm^{-1}, las vibraciones correspondientes a los grupos isocianato NCO, los mismos que luego desaparecen tras la formación del elastómero (Figura 18), confirmando la reacción completa entre los grupos isocianato NCO de los prepolímeros y los grupos hidroxilo OH del extensor de cadena [33]. Además, la señal entre los números de onda 1675-1750 cm^{-1} se dividió en dos picos después de la síntesis del elastómero (Figuras 17 y 18), lo que indicaría un posible estiramiento del carbonilo con y sin enlace de hidrógeno [15, 20, 21]. El índice de enlaces de hidrogeno del carbonilo (R) se calculó como se indica en la Ec. (3), y el grado de separación de fases (DPS) y el grado de mezcla de fases (DPM) se obtuvieron mediante las ecuaciones (4) y (5) [21].

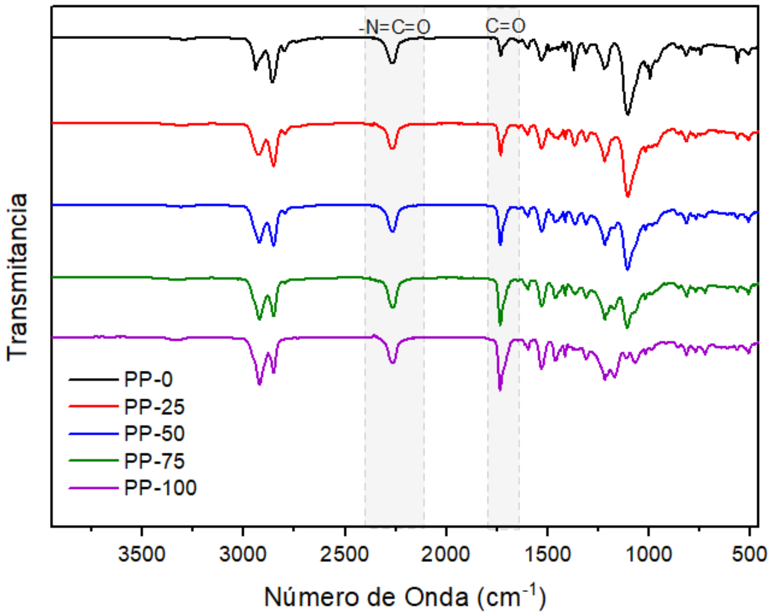


Figura 17.- Espectros ATR-FTIR de prepolímeros con contenido de FADD de 0 (PP-0) a 100% (PP-100) en peso de polioles

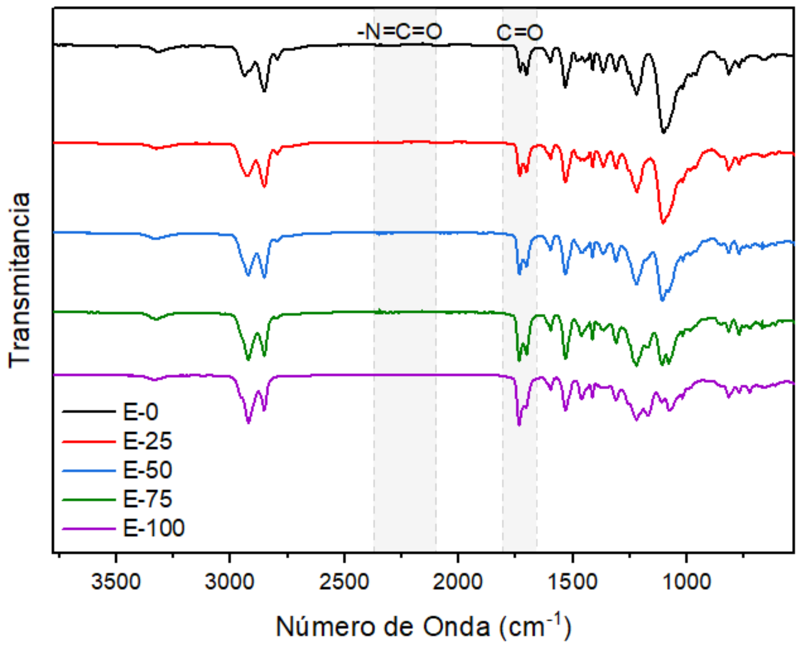


Figura 18.- Espectros ATR-FTIR de los poliuretanos elastoméricos con contenido de FADD de 0 (E-0) a 100%(E-100) en peso de polioles.

3.3. Configuración de las microfases y comportamiento frente al desgaste

El grado de separación de fases (DPS) en la microestructura del poliuretano elastomérico PUE se puede determinar midiendo los cambios en la intensidad del enlace de hidrógeno que existe entre el hidrógeno activo en el nitrógeno del grupo uretano y el átomo de oxígeno en el grupo carbonilo de otro grupo uretano o con los grupos éster o éter de los polioles [20–22]. Las bandas -NH (3200-3500 cm^{-1}) y -C = O (1700-1800 cm^{-1}) correspondientes a los segmentos duros en los elastómeros [21], se consideraron para evaluar el efecto del FADD en el grado de separación de fases de los elastómeros obtenidos (Figura 19 y 20). Las señales en ~ 3480 y ~ 3320 cm^{-1} podrían estar asociadas con el estiramiento del grupo N-H con y sin enlaces de hidrógeno [16] (Figura 19). Una sola señal fuerte a ~ 3320 cm^{-1} significaría que los grupos N-H estarían casi completamente unidos a los grupos que contienen oxigeno por enlaces de hidrógeno [16], dificultando la tarea de análisis del grado de dispersión de fases DPS en el elastómero final. Por lo tanto, se utilizaron las vibraciones de estiramiento del grupo carbonilo (Figura 20) para el análisis del DPS, donde hay claramente dos picos superpuestos ~ 1705 y ~ 1733 cm^{-1} correspondientes al estiramiento del carbonilo con y sin enlaces de hidrógeno [15, 21].

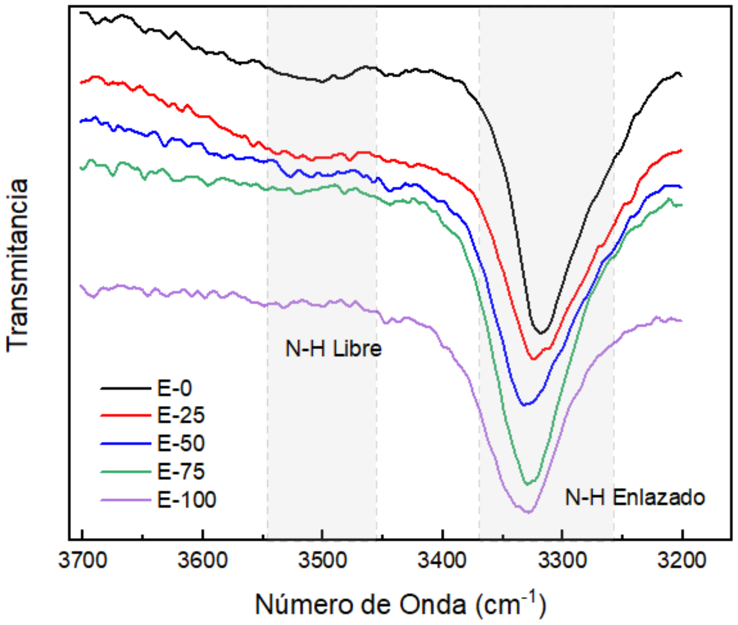


Figura 19.- Espectros ATR-FTIR de los poliuretanos elastoméricos obtenidos con contenido de FADD de 0 (E-0) a 100% (E-100 en peso de polioles, espectros relacionados con las vibraciones de estiramiento de los grupos amino N-H

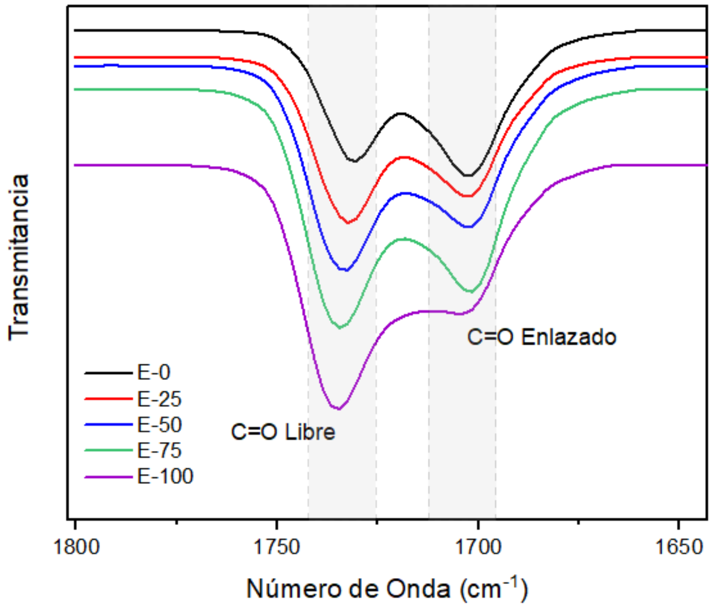


Figura 20.- Espectros ATR-FTIR de los poliuretanos elastoméricos obtenidos con contenido de FADD de 0 (E-0) a 100% (E-100) en peso de polioles, espectros relacionados con las vibraciones de estiramiento de los grupos carbonilo C=O

Los poliuretanos elastoméricos (PUE) se usan satisfactoriamente en aplicaciones donde se requiere la resistencia al desgaste. Se tiene dos tipos de desgaste asociado a los PUEs, desgaste por abrasión y desgaste por erosión [1]. La ASTM G40 "Terminología estándar relacionada con la erosión y el desgaste" define al desgaste por abrasión como "desgaste debido a partículas duras que se fuerzan y se mueven a lo largo de una superficie sólida", y al desgaste por la erosión como "pérdida progresiva de material original de una superficie sólida debido a la interacción mecánica entre esa superficie y un fluido, un fluido multicomponente, o líquido que incide o partícula sólida". El grado de desgaste por abrasión de los PUEs se expresó en mm^3 (volumen perdido). Mientras que el grado de desgaste por erosión se expresó en µm (profundidad de desgaste) debido a que las finas partículas del erosivo metálico podrían incrustarse dentro del material elastomérico y generarían un error en la diferencia de masa de la muestra después del ensayo, dato necesario para calcular el volumen perdido.

Los resultados del grado de dispersión de fases DPS y el comportamiento de desgaste de los elastómeros obtenidos se muestran en la Figura 21, donde se puede ver dos resultados de comportamiento opuestos. El primero ocurre desde E-0 hasta E-50, y el segundo ocurre desde E-50 hasta E-100. Hasta el E-50, tanto la resistencia a la erosión (45° y 90°) como la resistencia a la abrasión mejoran (por eso el desgaste disminuye), y simultáneamente el DPS disminuye, a pesar de la incorporación de cadenas colgantes y un poliol de menor peso molecular provenientes del FADD. Este comportamiento podría estar relacionado con la contribución del enlace de hidrógeno de los grupos éster provenientes del FADD, que produce un enlace de hidrógeno mucho más fuerte que el grupo éter y acorta el tamaño del dominio de la fase dura [15,17,28]. Después del E-50, la resistencia al desgaste (abrasión y erosión) disminuye y el DPS es aproximadamente constante y este comportamiento negativo podría estar asociado con el efecto predominante de la cadena colgante C9 sin grupos activos, que provocan un efecto de impedimento estérico que impide la formación de enlaces hidrogeno [23, 24]. La cadena colgante puede actuar como plastificante interno [25], y son imperfecciones en la red polimérica final. El E-100, elaborado al 100% con el poliol FADD, no tendría suficiente resistencia al desgaste, como un PUE convencional obtenido a partir de un poliol de tipo poliéster con el mismo peso molecular [15]. El E-0 muestra una mejor resistencia al desgaste por abrasión que el E-100, mientras que el E-100 muestra una mejor resistencia al desgaste por erosión (45° y 90°) que el E-0.

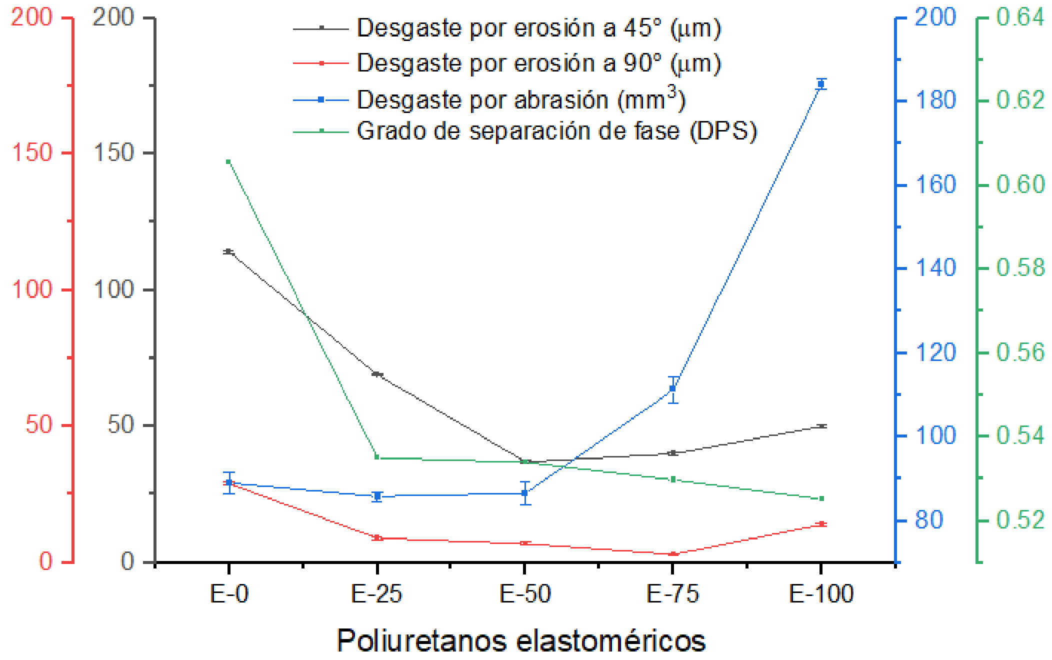


Figura 21.- Grado de separación de fases DPS y resistencia al desgaste de los elastómeros obtenidos con contenido de FADD de 0 (E-0) a 100% (E-100) en peso de polioles.

3.4 Propiedades físicas y mecánicas

Se sabe que en una síntesis de un poliuretano elastomérico PUE convencional los polioles de tipo poliésteres producen una densidad más alta que los polioles de tipo poliéteres [15, 17, 28]. El comportamiento físico y mecánico de los elastómeros obtenidos se muestran en la Tabla 7. Los resultados de densidad muestran primero un descenso leve y luego uno más pronunciado al pasar del E-0 al E-100, lo que se podría explicar debido a que posiblemente la cadena colgante del poliol FADD llenaría los espacios vacíos entre las cadenas poliméricas lineales obteniéndose un mejor empaquetamiento molecular. En la industria minera, particularmente en el proceso de clasificación de minerales, las mallas de clasificación hechas de poliuretano deben ser elásticas para evitar el taponamiento de los agujeros, y la dureza típica para lograr una buena elasticidad está entre 70 y 80 Shore A [1]. Todos los resultados de dureza de los poliuretanos elastoméricos PUE obtenidos están dentro del rango aplicable mencionado para la producción de paneles de clasificación. Sin embargo, una vez puesto el durómetro sobre la superficie del PUE se produce la pérdida de dureza durante un tiempo antes de su estabilización, esta pérdida de dureza muestra un perfil más plástico de los PUE al aumentar la cantidad de FADD, lo cual es concordante con los resultados de resiliencia y módulo donde muestran un comportamiento más elastomérico desde E-0 hasta E-50. La resistencia a la tracción concuerda con las disminuciones de DPS, donde después desde E-50, es más difícil soportar la tensión mientras que las imperfecciones en forma de cadenas colgantes provenientes del FADD aumentan [25–27].

Tabla 7.- Propiedades físicas y mecánicas de los elastómeros obtenidos

Muestra[a]	Densidad[b] (g/cm^3)	Resiliencia[b] (%)	Dureza[b,c] (Shore A)	Perdida de dureza[b] (Shore A)	Modulus[b] 300% (MPa)	Resistencia a la tracción [b] (MPa)
E-0	1,044	77,0	73.5 – 73.6	0,0	4,4	41,8
E-25	1,038	75,3	71.8 – 70.8	1,0	4,6	45,0
E-50	1,032	60,0	73.0 – 71.0	2,0	5,0	36,7
E-75	1,016	53,6	76.2 – 73.9	2,3	6,9	23,1
E-100	1,013	43,4	72.8 – 70.3	2,5	5,5	24,6

[a] Las muestras se han identificado por su contenido de FADD (% en peso) en el segmento blando en forma similar a la Tabla 2, [b] Propiedad calculada de acuerdo con las normas ASTM (Anexos VIII, IX, X y XI). [c] Se aplicó presión constante durante un minuto.

3.5 Comportamiento térmico

Se estudiaron los poliuretanos elastoméricos mediante la calorimetría diferencial de barrido (DSC) y el análisis termogravimétrico (TGA). El comportamiento térmico del elastómero E-0 (sistema MDI-PTMEG-BDO) siguió un patrón de DSC general [35], como se muestra en la Figura 22. Desde E-0 hasta E-100, los valores obtenidos de la temperatura de transición vítrea (Tg) mostraron un incremento desde -73 hasta -46°C lo que es consistente con la disminución del grado de separación de fases DPS, en ese mismo sentido [25, 27]. Además, los saltos de línea base similares entre 48 y 67°C estarían relacionados con la transición vítrea del mismo tipo y con la misma cantidad de segmento duro utilizado (MDI-BDO) en cada muestra. En los elastómeros que contienen FADD, una señal ancha de Tg se explicaría por el enrollamiento de las cadenas colgantes en la cadena principal que disminuiría la tasa de relajación [27]. Sin embargo, las cadenas colgantes no promovieron un cambio de la Tg a temperaturas más bajas [24]. Por tanto, todo esto se explicaría por un efecto predominante de los grupos éster sobre el efecto plastificante de la cadena colgante.

Por otro lado, una sola señal de Tg en los elastómeros con ambos polioles desde -69 (E-25) hasta -55°C (E-75) dentro de los extremos no combinados (-73 y -46°C) indicarían una miscibilidad entre ambos segmentos blandos (poliol de tipo éster y éter). El proceso de descomposición térmica de los elastómeros sigue una tendencia de la cantidad de FADD utilizada (Figura 23), esto se explicaría por la mejor estabilidad térmica del poliol tipo ester frente a un poliol tipo éter [6]. Prácticamente todas las muestras sufren descomposición a partir de las 250-300°C y hasta los 500°C.

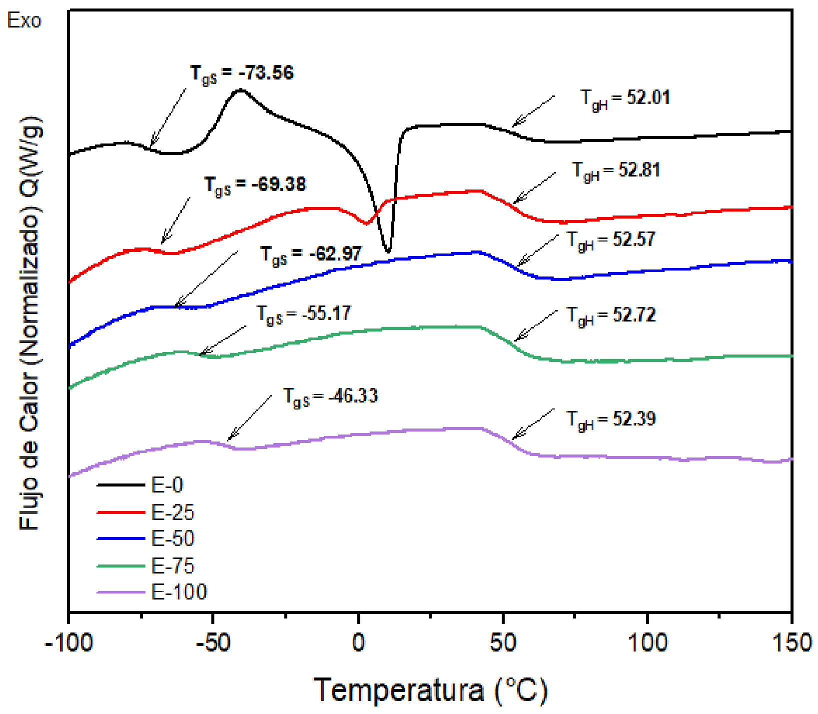


Figura 22.- Calorimetría diferencial de barrido DSC de las muestras de poliuretanos elastoméricos. TgS denota la transición vítrea del domínio blando y TgH la del domínio duro, respectivamente.

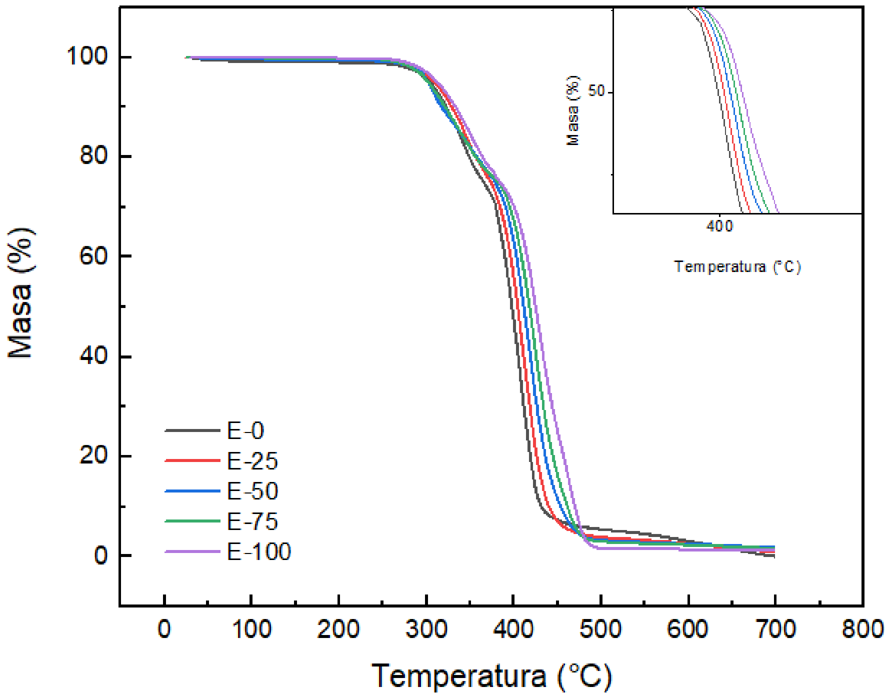


Figura 23.- Análisis termogravimétrico TGA de los poliuretanos elastoméricos

CAPÍTULO IV

CONCLUSIONES

Se sintetizaron elastómeros de poliuretano mediante el método de los prepolímeros (polimerización en dos pasos) utilizando como polioles una mezcla de poliéster de base biológica (diol dímero de ácidos grasos, FAAD) y poliéter de base petróleo (politetrametileno éter glicol PTMEG), los cuales reaccionaron con un exceso de diisocianato de 4,4'-difenilmetano (MDI) para obtener prepolímeros de poliuretano con terminales del grupo isocianato (NCO). Se utilizó el 1,4-butanodiol BDO como extensor de cadena para obtener los elastómeros finales.

Existe una miscibilidad parcial de los polioles de base biológica FADD y los polioles de base petróleo PTMEG debido a que los espectros FTIR de los polioles mezclados mostraron probables interacciones de enlace de hidrógeno entre el grupo éster provenientes del FADD y el grupo hidroxilo provenientes del PTMEG. También las imágenes resultantes de la microscopia óptica de las mezclas (25, 50 y 75% en peso de FADD) revelan una mejora en la compatibilidad de los polioles de base bióloga y de base petróleo, cuando hay un incremento de temperatura desde 25°C hasta 60°C.

El grado de separación de fases DPS de los poliuretanos elastoméricos obtenidos disminuye al aumentar el contenido de FADD.

Se estudio el comportamiento de desgaste de los poliuretanos elastoméricos PUE en función a la cantidad añadida de poliol de base biológica en remplazo del poliol de base petróleo. Se observó una mejora de la resistencia al desgaste que estaría relacionada a la disminución del grado de separación de fases DPS debido a la contribución del enlace de hidrógeno de los grupos éster provenientes del FADD, que lograría un enlace de hidrógeno mucho más fuerte que los grupos éter provenientes del PTMEG y entonces acortaría el tamaño del domínio de la fase dura. Sin embargo, cuando el PUE contiene más del 50% en peso del poliol de base biológica, las cadenas colgantes provenientes del FADD causarían un efecto de impedimento estérico que detendría dicha mejora.

Los resultados mostraron que tanto la resistencia a la erosión como a la abrasión de los poliuretanos elastoméricos sintetizados aumentan con el aumento del contenido de poliol de base biológica, al menos hasta llegar a un 50% en peso del poliol FADD.

La excelente flexibilidad de los poliuretanos elastoméricos obtenidos, con dureza entre 70-75 Shore A, hacen que esos elastómeros con hasta 50% en peso de poliéster de base biológica sean adecuados para su uso en la industria minera por que podrían producir una mejora sustancial en el rendimiento del desgaste, particularmente en la producción de mallas de clasificación, que deben ser elásticas para evitar taponamientos.

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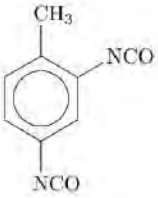
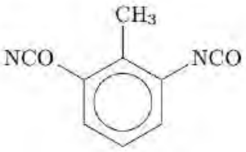
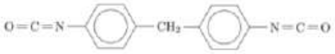
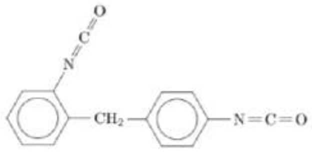
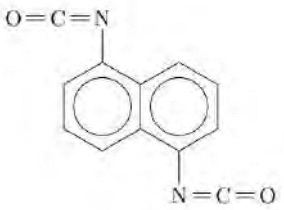
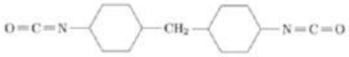
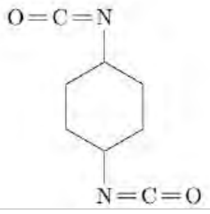
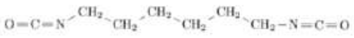
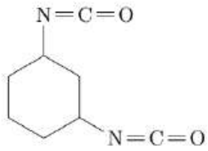
ANEXOS

ANEXO I

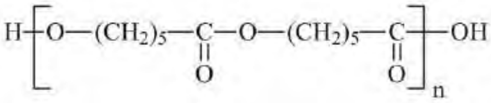
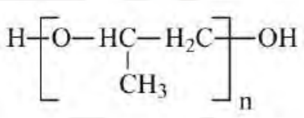
Tipos de diisocianatos para la síntesis de poliuretanos

Diisocianato	Nombre común	Nombre IUPAC	Estructura	Peso molecular
AROMATICOS	2,4 TDI	2,4-diisocianato-1-metil-benceno		174.2
	2,6 TDI	2,6-diisocianato-1-metil-benceno		174.2
	4,4’ MDI	1-isocianato-4-[(4-fenilisocianato)metil]benceno		250.3
	2,4’ MDI	1-isocianato-2-[(4-fenilisocianato)metil]benceno		250.3
	NDI	1,5-naftildiisocianato		210.2
ALIFATICOS	H_{12}MDI	1,1’-Metilenbis(4-ciclohexildiisocianato)		262.3
	CHDI	1,4-ciclohexildiisocianato		166.2
	HDI	1,6-hexanildiisocianato		168.2

	H_6XDI	1,3-Ciclohexildiisocianato		166.2

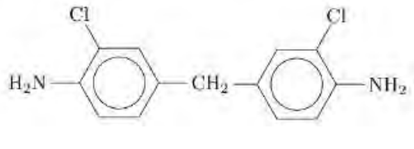
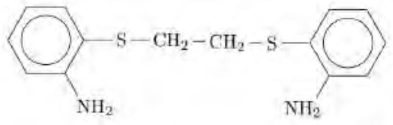
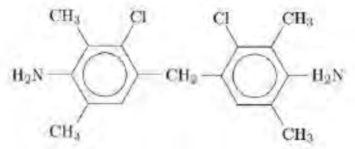
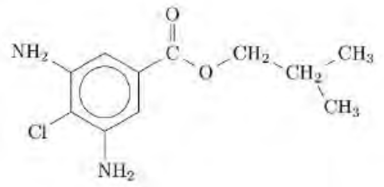
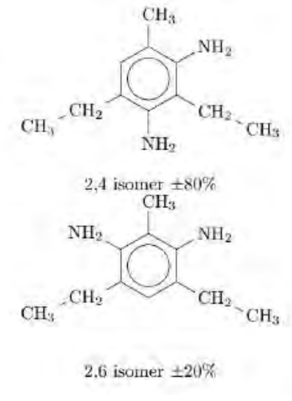
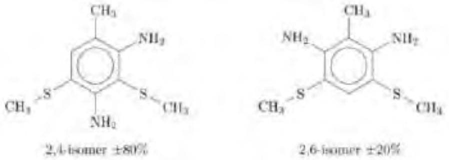
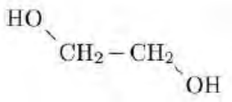
ANEXO II

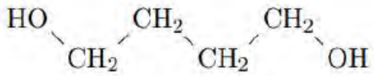
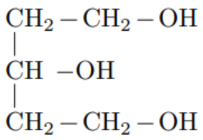
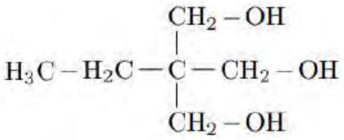
Tipos de polioles para la síntesis de poliuretanos

Polioles	Nombre común	Nombre IUPAC	Estructura
Poliésteres	PEA	Polietilenadipato	$H\left[O-(CH_2)_2-OCO-(CH_2)_4-CO\right]_n OH$
	PCL	Policaprolactona	 $H\left[O-(CH_2)_5-\underset{\underset{O}{\|}}{C}-O-(CH_2)_5-\underset{\underset{O}{\|}}{C}\right]_n OH$
	PBA	Polibutilenadipato	$H\left[O-(CH_2)_4-OCO-(CH_2)_4-CO\right]_n OH$
Poliéteres	PTMEG; PTHF o PTMO	Politetrahidrofurano	$H\left[O-(CH_2)_4\right]_n OH$
	PPO	Polipropilenoxido	 $H\left[O-\underset{\underset{CH_3}{\vert}}{HC}-H_2C\right]_n OH$
	PEG	Polietilenglicol	$H\left[O-CH_2-CH_2\right]_n OH$

ANEXO III

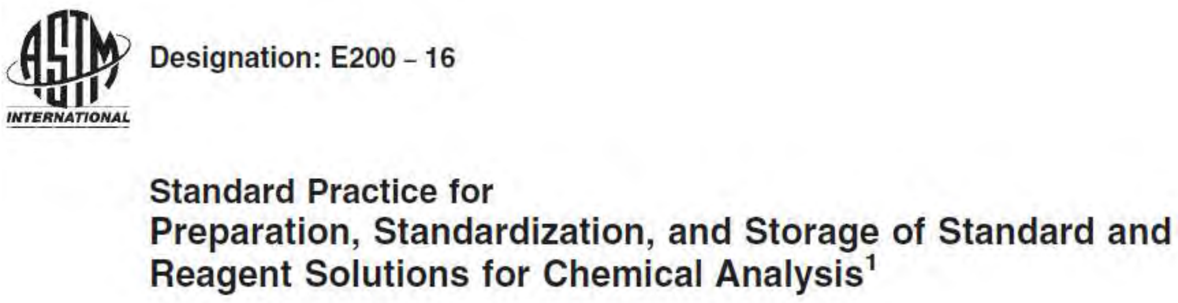
Tipos de extensores de cadena para la síntesis de poliuretanos

Extensor de cadena	Nombre común	Nombre IUPAC	Estructura
Diaminicos	Moca, Mboca	4,4-Metilenbis(2-cloroanilina)	
	Cyanocure	1,1’-(etano-1,2-diildisulfanodiil)bis(2-isocianatobenceno)	
	MCDEA	4,4-Metilen-bis-(3-cloro-2,6-dietilanilina)	
	BAYTEC 1604	Butil 3,5-diamino-clorobenzoato	
	ETHACURE 100	- 80% 2,4-dietil-6-metil-1,3-diamin-benceno - 20% 4,6-dietil-2-metil-1,3-diamin- benceno	
	ETHACURE 300	Dimetil-thio-diamin tolueno	
Dioles	Etilenglicol (EG)	1,2-etanodiol	

	Butanodiol (BDO)	1,4-butanodiol	
	Glicerina (Gly)	Glicerol	 $\begin{array}{l} CH_2-CH_2-OH \\ \vert \\ CH\ -OH \\ \vert \\ CH_2-CH_2-OH \end{array}$
	TMP	Trimetilolpropano	 $\begin{array}{c} CH_2-OH \\ \vert \\ H_3C-H_2C-C-CH_2-OH \\ \vert \\ CH_2-OH \end{array}$
	Hidroquinona (HQEE)	bis(2-hidroxietil)eter hidroquinona	

ANEXO IV

Norma de referencia usado para la estandarización del HCl



Designation: E200 – 16

Standard Practice for
Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[1]

HYDROCHLORIC ACID,
0.02 to 1.0 meq/mL (*N*)

20. Preparation

20.1 To prepare a 0.1 meq/mL (*N*) solution, measure 8.3 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) into a graduated cylinder and transfer it to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

20.2 For other normalities of HCl solution, use the requirements given in Table 4.

TABLE 4 Hydrochloric Acid Dilution Requirements

Desired meq/mL (*N*)	Volume of HCl to Be Diluted to 1 L, mL
0.02	1.66
0.04	3.32
0.1	8.3
0.2	16.6
0.5	41.5
1.0	83.0

21. Standardization with Sodium Carbonate [8]

21.1 Transfer 2 to 4 g of primary standard anhydrous sodium carbonate (Na_2CO_3) to a platinum dish or crucible, and dry at 250°C for 4 h. Cool in a desiccator.

21.2 To standardize a 0.1 meq/mL (*N*) solution, weigh accurately 0.22 ± 0.01 g of the dried Na_2CO_3, and transfer to a 500-mL conical flask. Add 50 mL of water, swirl to dissolve the carbonate, and add 2 drops of a 0.1 % solution of methyl red in alcohol. Titrate with the HCl solution to the first appearance of a red color, and boil the solution carefully, to avoid loss, until the color is discharged. Cool to room temperature, and continue the titration, alternating the addition of HCl solution and the boiling and cooling to the first appearance of a faint red color that is not discharged on further heating.

21.3 The weights of dried Na_2CO_3 suitable for other normalities of HCl solution are given in Table 5.

22. Calculation

22.1 Calculate the normality of the HCl solution, as follows:

$$A = \frac{B}{0.053 \times C} \qquad (3)$$

where:

A = meq/mL (*N*) normality of the HCl solution,
B = grams of Na_2CO_3 used, and
C = millilitres of HCl solution consumed.

TABLE 5 Weights of Dried Sodium Carbonate

meq/mL (*N*) of Solution	Weight of Dried Na_2CO_3 to Be Used, g
0.02	0.088 ± 0.001[A]
0.04	0.176 ± 0.001[A]
0.1	0.22 ± 0.01[B]
0.2	0.44 ± 0.01[B]
0.5	1.10 ± 0.01[B]
1.0	2.20 ± 0.01[B]

[A] A 100-mL buret should be used for this standardization.
[B] The listed weights are for use when a 50-mL buret is used. If a 100-mL buret is to be used, the weights should be doubled.

23. Stability

23.1 Restandardize monthly.

24. Precision and Bias (See Note 4)

24.1 The following criteria should be used for judging the acceptability of results:

24.1.1 *Hydrochloric Acid (1.0 meq/mL (N)) (See Note 5):*

24.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0004 meq/mL (*N*) normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0011 meq/mL (*N*) normality units.

24.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.0006 meq/mL (*N*) normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0017 meq/mL (*N*) normality units.

24.1.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.0015 meq/mL (*N*) normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0042 meq/mL (*N*) normality units.

24.1.2 *Hydrochloric Acid (0.1 meql/mL (N)) (See Note 6):*

24.1.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 meq/mL (*N*) normality units at 28 df. The 95 % limit for the difference between two such determinations is 0.0003 meq/mL (*N*) normality units.

24.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00007 meq/mL (*N*) normality units at 14 df. The 95 % limit for the difference between two such averages is 0.0002 meq/mL (*N*) normality units.

24.1.2.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be

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[8] A buret having a bent delivery tube is helpful in carrying out this standardization procedure.

0.00017 meq/mL (*N*) normality units at 13 df. The 95 % limit for the difference between two such averages is 0.0005 meq/mL (*N*) normality units.

25. Standardization with Tris(hydroxymethyl)-Aminomethane

25.1 Transfer 8 to 10 g of primary standard tris(hydroxymethyl)aminomethane[7] [$(HOCH_2)_3CNH_2$] to a suitable dish or crucible, and dry in a vacuum at 70°C for 24 h. Cool in a desiccator.

25.2 To standardize a 0.1 meq/mL (*N*) solution, weigh accurately 0.40 ± 0.02 g of the dried tris(hydroxymethyl) aminomethane, and transfer to a 250-mL beaker. Dissolve in 50 mL of ammonia- and carbon dioxide-free water, and titrate with the HCl solution to a pH of 4.70 using a suitable pH meter.

25.3 The weights of dried tris(hydroxymethyl)aminomethane suitable for other normalities of HCl solution are given in Table 6.

26. Calculation

26.1 Calculate the meq/mL (*N*) normality of the HCl solution, as follows:

$$A = \frac{B}{0.1211 \times C} \tag{4}$$

where:

A = meq/mL (*N*) normality of the HCl solution,
B = grams of tris(hydroxymethyl)aminomethane used, and
C = millilitres of HCl solution consumed.

27. Stability

27.1 Restandardize monthly.

28. Precision and Bias (See Notes 7 and 8)

28.1 The following criteria should be used for judging the acceptability of results:

28.1.1 *Hydrochloric Acid (1.0 meq/mL (N)):*

28.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0006 meq/mL (*N*) normality units at 16 df. The 95 % limit for the difference between two such determinations is 0.0017 meq/mL (*N*) normality units.

28.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.0007 meq/mL (*N*) normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0020 meq/mL (*N*) normality units.

TABLE 6 Weights of Dried Tris(Hydroxymethyl)Aminomethane

meq/mL (*N*) of Solution	Weight of Dried $(HOCH_2)_3CNH_2$ to be Used, g
0.02	0.16 ± 0.008[A]
0.04	0.32 ± 0.016[A]
0.1	0.40 ± 0.02[B]
0.2	0.80 ± 0.04[B]
0.5	2.0 ± 0.1[B]
1.0	4.0 ± 0.2[B]

[A] A 100-mL buret should be used for this standardization.

[B] The listed weights are for use when a 50-mL buret is used. If a 100-mL buret is to be used, the weights should be doubled.

28.1.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.0015 meq/mL (*N*) normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0042 meq/mL (*N*) normality units.

28.1.2 *Hydrochloric Acid (0.1 meq/mL (N):*

28.1.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 meq/mL (*N*) normality units at 16 df. The 95 % limit for the difference between two such determinations is 0.0003 meq/mL (*N*) normality units.

28.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00012 meq/mL (*N*) normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0003 meq/mL (*N*) normality units.

28.1.2.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00024 meq/mL (*N*) normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0007 meq/mL (*N*) normality units.

TABLE 7 Sulfuric Acid Dilution Requirements

Desired meq/mL (*N*)	Volume of H_2SO_4 to Be Diluted to 1 L, mL
0.02	0.60
0.1	3.0
0.2	6.0
0.5	15.0
1.0	30.0

NOTE 7—These precision estimates are based on an interlaboratory study conducted in 1973. One sample of each concentration was analyzed. One analyst in each of 9 laboratories performed duplicate determinations and repeated them on a second day, for a total of 36 determinations for each concentration herein. Practice E180 was used in developing these statements.

NOTE 8—Precision data have not been obtained for concentrations other than those listed in Section 28.

SULFURIC ACID, 0.02 TO 1.0 meq/mL (*N*)

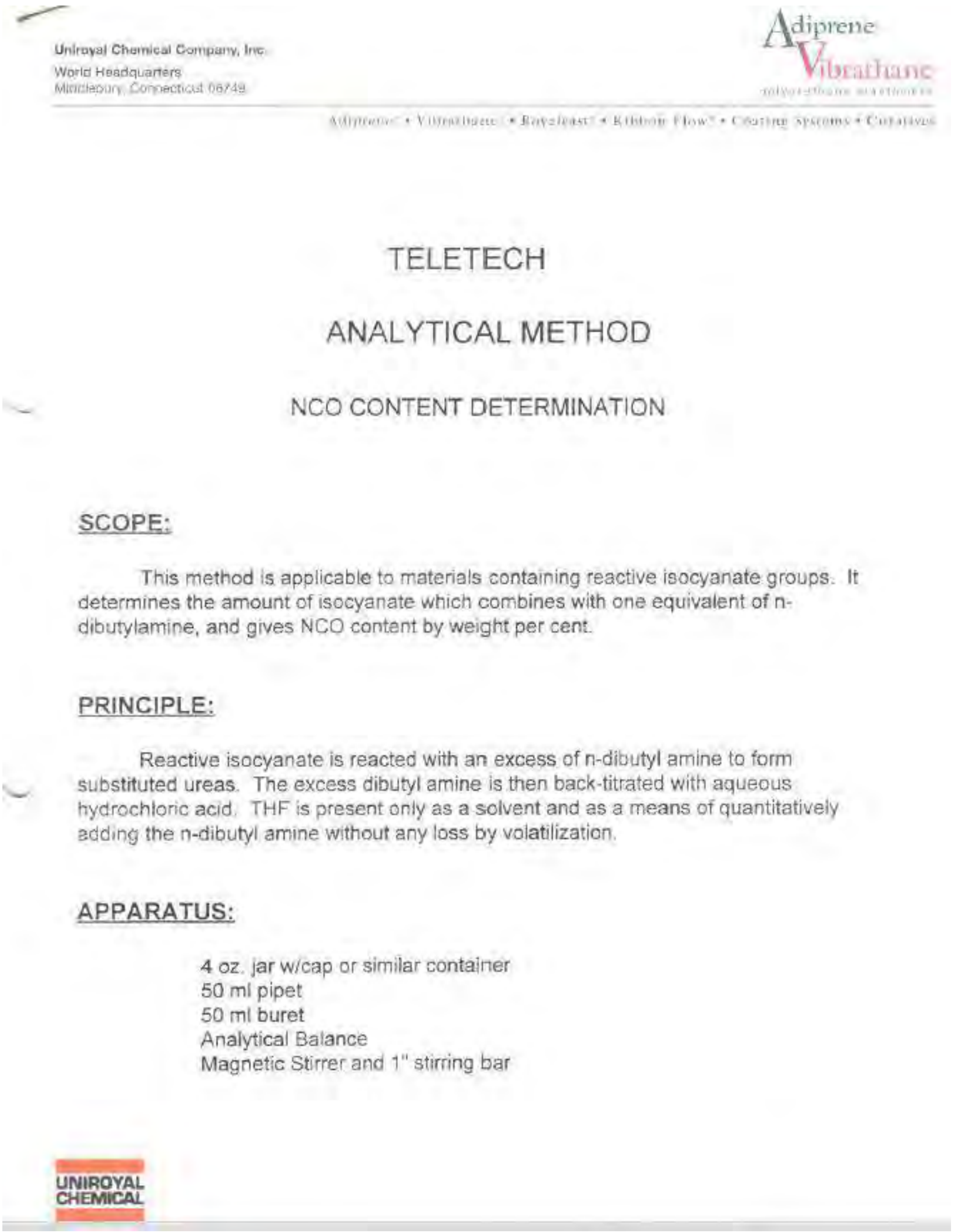
29. Preparation

29.1 To prepare a 0.1 meq/mL (*N*) solution, measure 3.0 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into a graduated cylinder and slowly add it to 400 mL of water in a 600-mL beaker. Rinse the cylinder into the beaker with water. Mix the acid-water mixture, allow it to cool, and transfer to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

29.2 For other meq/mL (*N*) normalities of the H_2SO_4

ANEXO V

Método de referencia usado para la determinación del % de NCO en los prepolímeros de poliuretano



Uniroyal Chemical Company, Inc.
World Headquarters
Middlebury, Connecticut 06749

Adiprene
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TELETECH

ANALYTICAL METHOD

NCO CONTENT DETERMINATION

SCOPE:

This method is applicable to materials containing reactive isocyanate groups. It determines the amount of isocyanate which combines with one equivalent of n-dibutylamine, and gives NCO content by weight per cent.

PRINCIPLE:

Reactive isocyanate is reacted with an excess of n-dibutyl amine to form substituted ureas. The excess dibutyl amine is then back-titrated with aqueous hydrochloric acid. THF is present only as a solvent and as a means of quantitatively adding the n-dibutyl amine without any loss by volatilization.

APPARATUS:

4 oz. jar w/cap or similar container
50 ml pipet
50 ml buret
Analytical Balance
Magnetic Stirrer and 1" stirring bar

UNIROYAL CHEMICAL

REAGENTS:

0.5 N Hydrochloric acid, aqueous (Standardized)

0.2 N n-dibutyl amine - prepared by adding 35 ml of A.R grade n-dibutyl amine to 1 liter of tetrahydrofuran (A.R. grade) dried over molecular sieves 4-A.

0.1% Bromphenol blue indicator - grind 0.1g of indicator with 14.9 ml of 0.01N NAOH. Dilute to 250 ml with distilled water.

PROCEDURE:

Accurately weigh, in duplicate, 2.0 - 5.0g ± .001g of sample into a 4 oz. jar. Pipet exactly 50 ml of 0.2 N n-dibutyl amine into jar, being careful not to splash any out of the jar. Add a 1" teflon coated stirring bar, cap jar tightly and place on a magnetic stirrer. Agitate sample gently until it is completely dissolved.

Add 6 drops of bromphenol blue indicator and titrate to a yellow-green endpoint with aqueous 0.5 N HCl.

Run a blank at the same time as the sample, following the same procedure, except omitting the sample.

CALCULATIONS:

$$\% \text{ NCO} = \frac{4202\ (\text{N HCl})(\text{B-A})}{(\text{Sample Wt.})\ (1000)}$$

B = Blank titer
A = Sample titer
N = Normality of the Hydrochloric Acid

Win:Data:3/23/95

ANEXO VI

Norma de referencia usado para los ensayos de abrasión de los poliuretanos elastoméricos

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D5963 – 04 (Reapproved 2019)

Standard Test Method for
Rubber Property—Abrasion Resistance (Rotary Drum Abrader)[1]

This standard is issued under the fixed designation D5963; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of abrasion resistance of rubbers (vulcanized thermoset rubbers and thermoplastic elastomers) that are subject to abrasive/frictional wear in actual service. The abrasion resistance is measured by moving a test piece across the surface of an abrasive sheet mounted to a revolving drum, and is expressed as volume loss in cubic millimetres or abrasion resistance index in percent. For volume loss, a smaller number indicates better abrasion resistance, while for the abrasion resistance index, a smaller number denotes poorer abrasion resistance.

1.2 Test results obtained by this test method shall not be assumed to represent the wear behavior of rubber products experienced in actual service.

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D297 Test Methods for Rubber Products—Chemical Analysis

D1765 Classification System for Carbon Blacks Used in Rubber Products

D2240 Test Method for Rubber Property—Durometer Hardness

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

2.2 *Other Standards:*

DIN 53516 Determination of Abrasion Resistance[3]

ISO 868 Plastics and Ebonite—Determination of Indentation Hardness by Means of a Durometer (Shore Hardness)[4]

ISO 2393 Rubber Test Mixes—Preparation, Mixing and Vulcanization—Equipment and Procedures[4]

ISO 2781 Rubber, Vulcanized—Determination of Density[4]

ISO 4649 Rubber—Determination of Abrasion Resistance Using a Rotating Cylindrical Drum Device[4]

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests[4]

ISO 7619 Rubber—Determination of Indentation Hardness by Means of Pocket Hardness Meters[4]

ISO 9298 Rubber Compounding Ingredients—Zone Oxide Test Methods[4]

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

Current edition approved Nov. 1, 2019. Published December 2019. Originally approved in 1997. Last previous edition approved in 2015 as D5963 - 04 (2015). DOI: 10.1520/D5963-04R19.

This test method is an adaptation of ISO Standard 4649 (1985 revision), Rubber—Determination of Abrasion Resistance Using a Rotating Cylindrical Drum Device; and is being coordinated with a current draft revision of that standard (see Appendix X1).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Beuth Verlag GmbH (DIN– DIN Deutsches Institut fur Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

3. Terminology

3.1 *abrasion loss, A,* (mm^3), *n*—the volume loss of a defined rubber test piece determined by sliding the test piece under specified conditions over the surface of an abrasive sheet of "nominal abrasiveness" (S_0) mounted to a rotating drum of specified dimensions.

NOTE 1—This corresponds to the test method of DIN 53516 and to Method A (Relative volume loss) of ISO 4649:1985. The same calculation could be made for the other test methods if the proper value for the "nominal abrasiveness" (S_0) for the test method were known or assumed. (150 mg has been indicated as a possible value for Method D but its accuracy has not been established to the degree of the Method A value.)

3.1.1 *Discussion*—The designation A_A is used to indicate that the test was run in accordance with Method A.

3.2 *abrasion resistance index ARI (%), n*—the ratio of the volume loss of a Standard Rubber to that of a test rubber expressed in percent, with the volume loss determined by sliding a defined rubber test piece under specified conditions over the surface of an abrasive sheet of known abrasiveness *S* (180 to 220 mg) mounted to a rotating drum of specified dimensions.

3.2.1 *Discussion*—The designations ARI_A, ARI_B, ARI_C, and ARI_D are used to indicate that the test was run in accordance with Method A, B, C, or D, respectively.

3.3 *"nominal abrasiveness," (S_0) of the abrasive sheet,* (mg), *n*—a specified (theoretical) mass loss of 200 mg of a defined test piece of a Standard Rubber, obtained by sliding the test piece under specified conditions over the surface of the abrasive sheet mounted to a rotating drum of specified dimensions.

3.4 *abrasiveness, S of the abrasive sheet,* (mg), *n*—the actual mass loss, within a specified range of 180 to 220 mg, of a defined test piece of a Standard Rubber, obtained by sliding the test piece under specified conditions over the surface of the abrasive sheet mounted to a rotating drum of specified dimensions.

3.5 *Standard Rubber*—a natural rubber compound of specified composition, mixed and vulcanized under defined conditions.

3.5.1 *Discussion*—Standard Rubber # 1 is used to determine the abrasiveness (*S*) of the abrasive sheet and to ensure that *S* is within the specified range of 180 to 220 mg. The ratio of the "nominal abrasiveness" (S_0) and the actual abrasiveness (*S*) is used to correct the abrasion loss of a test rubber for any deviation of the abrasiveness of the abrasive sheet from the specified "nominal abrasiveness" (200 mg).

4. Summary of Test Method

4.1 This test method provides procedures for preparing cylindrical test pieces of specified dimensions from vulcanized thermoset rubbers or thermoplastic elastomers and for evaluating their abrasion resistance by sliding a test piece across the surface of an abrasive sheet[5] attached to a rotating drum. It also describes the preparation and compliance testing of a Standard Rubber (see Annex A1).

4.2 The test is performed under specified conditions of contact pressure, sliding distance, and travel speed of the test piece, rotational speed of the drum, and degree of abrasiveness of the abrasive sheet.

4.3 The abrasiveness, *S*, of the abrasive sheet is defined by the mass loss in milligrams of a test piece prepared from Standard Rubber #1 when tested under the same specified conditions.

4.4 Four different methods may be used to test the abrasion resistance. Method A, uses a *non-rotating* test piece and Method B a *rotating* test piece, both utilizing Standard Rubber #1 as the reference. Methods C and D use Standard Rubber #2 as reference with a *non-rotating* and *rotating* test piece, respectively. The rotating test pieces provide, in many cases, a more uniform abrasion wear pattern.

4.5 The abrasion resistance, A_A, obtained in accordance with Method A is reported as abrasion (volume) loss in cubic millimetres, calculated from the mass loss of the test piece, density of the test rubber, and the abrasiveness of the abrasive sheet in relation to the "nominal abrasiveness" (S_0) defined by a mass loss of 200 mg. A smaller number indicates a higher resistance to abrasion.

4.6 The abrasion resistance, $ARI_{A–D}$, obtained in accordance with Methods A, B, C, and D is reported as the abrasion resistance index in percent, calculated from the mass losses and densities of the Standard Rubbers and test rubbers. A smaller number indicates a lower resistance to abrasion.

4.7 The mass loss obtained by the different methods can differ and the same method shall therefore be used if mass (and volume) losses are to be compared directly.

5. Significance and Use

5.1 Abrasion resistance is a performance factor of paramount importance for many rubber products, such as tires, conveyor belts, power transmission belts, hoses, footwear, and floor covering. A test capable of measuring resistance to abrasion of rubber, including uniformity of wear behavior under abrasive/frictional service conditions is therefore highly desirable.

5.2 This test method may be used to estimate the relative abrasion resistance of different rubbers. Since conditions of abrasive wear in service are complex and vary widely, no direct correlation between this accelerated test and actual performance can be assumed.

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[5] The sole source of supply of the abrasive sheet known to the committee at this time is CCSI, 221 Beaver St., Akron, OH 44304, http//:www.ccsi-inc.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

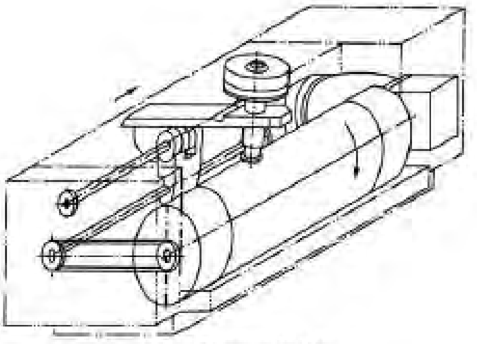

FIG. 1 Abrasion Tester

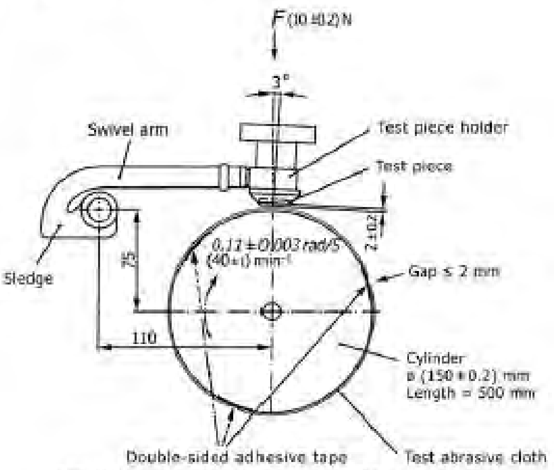

FIG. 2 Side View and Dimensions of Abrasion Tester

5.3 This test method is suitable for comparative testing, quality control, specification compliance testing, referee purposes, and research and development work.

6. Limitations

6.1 Test pieces containing voids or porosity, or both, will yield unreliable test results.

6.2 Test pieces that bounce (chatter) over the surface of the abrasive sheet rather than running smoothly will produce inaccurate test results.

6.3 Test pieces that tend to extensively smear the surface of the abrasive sheet will provide meaningless test results.

6.4 Test results obtained under any of the above conditions shall not be used to reach conclusions regarding the relative abrasion resistance of rubbers.

7. Apparatus and Materials

7.1 *Abrasion Tester:*

7.1.1 The abrasion tester consists of a machine frame holding a laterally movable test piece holder, a rotary cylindrical drum to which an abrasive sheet can be fastened, and a drive system as shown in Fig. 1. Dimensions are given in Fig. 2.

7.1.2 The diameter of the cylindrical drum shall be 150.0 ± 0.2 mm (5.906 ± 0.008 in.) and its length approximately 500 mm (20 in.), operating at a rotational frequency of 0.11 ± 0.003 rad/s (40 ± 1 rpm).

7.1.3 The abrasive sheet shall be bonded tightly to the drum using three evenly spaced double-faced pressure-sensitive adhesive tapes, extending the full length of the drum. The tapes shall be about 50 mm (2 in.) wide and not more than 0.2 mm (0.008 in.) thick. The gap where the ends of the abrasive sheets meet on the adhesive tapes shall not exceed 2 mm (0.08 in.).

7.1.4 It is mandatory that the abrasive sheet is bonded to the drum in the direction of rotation marked on the surface or back of the sheet.

7.1.5 The test piece holder shall be mounted on a pivoted swivel arm, which can be swung into vertical position to insert and remove the test piece (see Note 2).

NOTE 2—It is recommended to install a device preventing the test piece holder and abrasive sheet from coming into contact and damaging each other.

7.1.6 Suitable attachments may be provided to rotate the test piece around its own axis during the test run by rotation of the test piece holder (Methods B and D). The test piece shall rotate at the rate of 1 revolution for each 50 revolutions of the cylindrical drum with the abrasive sheet fastened to the drum.

7.1.7 The center axis of the test piece holder shall have an angle of 3° to the perpendicular in the direction of rotation and the center of the test piece shall be within ±1 mm (±0.04 in.) directly above the longitudinal axis of the drum.

7.1.8 The design of the swivel arm and holder shall be such that the test piece is pressed against the abrasive sheet with a force of 10.0 ± 0.2 N (2.25 ± 0.04 lbf). Both swivel arm and holder shall be free from vibration during operation.

7.1.9 The test piece holder shall consist of a cylindrical opening with an adjustable diameter of at least 15.5 mm (0.610 in.) to 16.3 mm (0.642 in.) and a device for adjusting the length of the test piece protruding from the opening to 2.0 ± 0.2 mm (0.08 ± 0.008 in.).

7.1.10 The swivel arm with the test piece holder is connected to a worm gear that moves the holder on a guide rod laterally across the surface of the abrasive sheet attached to the cylindrical drum. The lateral displacement shall be approximately 4.2 mm (0.165 in.) per revolution of the drum, so that the test piece passes only four times across the same area of the sheet during one test cycle.

7.1.11 Placement of the holder with the test piece on the drum at the beginning of the test and removal at the end shall be automatic. The normal length of the abrasion path shall be 40.0 ± 0.2 m (131.2 ± 0.7 ft). This is equivalent to about 84 revolutions when allowing for the thickness of 1 mm (0.04 in.) for the abrasive sheet.

7.1.12 For special cases of very high volume loss, half the length of the abrasion path, that is, 20 ± 0.1 m (65.6 ± 0.3 ft), equivalent to roughly 42 revolutions, may be used.

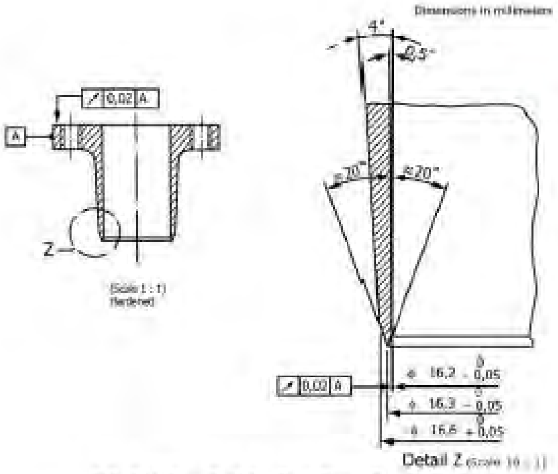

FIG. 3 Hollow Drill for Test Piece Preparation

7.1.13 The test machine may be equipped with a vacuum hose and a brush that contacts the drum and aids in the removal of debris.

7.2 *Abrasive Sheet:*

7.2.1 Corundum (aluminum oxide) of grit 60 bonded to a carrier sheet of at least 400 mm (15.8 in.) width, 474 ± 1 mm (18.66 ± 0.04 in.) length and an average thickness of 1 mm (0.04 in.) shall be used as the abrasive medium.

7.2.2 The sheets shall be of an abrasiveness as to cause a mass loss between 180 and 220 mg when tested over an abrasion path of 40 m (131.2 ft) using a non-rotating test piece (Method A) of Standard Rubber #1 specified in Section 8.

7.2.3 Since the abrasiveness of virgin abrasive sheets is usually higher than desired, it is necessary to blunt the sheets with one or two test runs using a steel test piece in place of the Standard Rubber to bring it into the desired range. The direction of rotation used for blunting shall be marked on the sheets.

7.2.4 After blunting, the abrasive sheets shall be thoroughly cleaned by brushing, blowing, or suction and two test runs with the Standard Rubber shall be made.

7.2.5 Test results obtained with abrasive sheets, thus calibrated, are more consistent, and it is normally possible to run several hundred single tests using the same sheet.

7.3 *Rotating Test Specimen Cutting Die:*

7.3.1 The rotating cutting die for specimen preparation is of a 'hollow drill' configuration, manufactured from A2 or P2 grade steel having a hardness of HRC 50, or above, of the dimensions shown in Fig. 3.

7.3.2 The rotational frequency of the circular cutting die should be approximately 2.65 rad/s (1000 rpm) and higher for rubbers (refer to 1.1) with a hardness of less than Type A50/1 (see Test Method D2240, ISO 868, or ISO 7619).

7.3.3 When obtaining test specimens using the rotating cutting die, a lubricant that does not negatively affect the integrity or character of the material shall be used to prevent deformation during the cutting process.

8. Standard Rubber

8.1 Sheets of Standard Rubber may be obtained commercially or prepared in-house, following the directions given in Annex A1.

8.2 If prepared in-house, it is mandatory to test a commercially obtained reference sheet for comparison.

8.3 Standard Rubber sheets shall be stored under cool, dry, and dark conditions, and wrapped in a suitable protective covering such as polyethylene film, which protects the sheets from ozone attack (see Note 3).

NOTE 3—Sheets of Standard Rubber stored under the above conditions remain usable for at least three years.

8.4 Standard Rubber sheets of satisfactory quality shall meet the requirements of A1.3.1 in Annex A1.

8.5 Standard Rubber test pieces may be used for up to three test runs per test piece (see 11.3).

9. Preparation of Test Specimens

9.1 Test specimens are cylindrical in shape with a diameter of 16 ± 0.2 mm (0.630 ± 0.008 in.) and a minimum thickness of 6 mm (0.24 in.).

9.2 Test specimens are obtained from sheets, vulcanized or formed in an appropriate mold, of no less than 6 mm (0.24 in.) in thickness, using a rotating cutting die in accordance with 7.3.

9.3 Preparation of test specimens by the use of a 'punch type' cutting die is not permitted.

9.4 If test specimens of the required thickness are not available, the necessary thickness may be obtained by bonding specimen of at least 2 mm (0.08 in.) to a base piece having a durometer hardness of no less than Type A80/1 (see Test Method D2240, ISO 868, or ISO 7619).

9.4.1 Care shall be taken that test specimens prepared in this manner are not abraded to the bond line during the course of the testing procedure.

9.4.2 Exposing the bond during the test procedure invalidates the test determination.

9.5 When testing finished products, such as fabric-reinforced conveyor belts, test specimens shall be obtained directly, when possible, from the entire finished product, including the fabric or any integral layers. They may be obtained from test pieces, when necessary.

9.5.1 In this case, it is also important that test specimens (having been obtained from the finished product or sample) are not abraded to an adhesive layer, layer of fabric, or other integral, but heterogeneous material, and that only the rubber layer being tested for abrasion is subjected.

9.5.2 Exposing bonded or heterogeneous layers during the test procedure invalidates the test determination.

10. Procedure

10.1 The test shall be carried out at 23 ± 5°C (73 ± 9°F) and no sooner than 16 h after vulcanization or forming of the test compounds.

10.2 The density of the rubbers to be tested shall be determined using a hydrostatic method (see Test Methods D297 or ISO 2781).

10.3 *Method A—Test Run:*

10.3.1 Method A is run with a non-rotating test piece, using Standard Rubber #1 as reference.

10.3.2 Prior to each test, any debris left on the abrasive sheet from a previous abrasion test shall be removed by vigorous brushing, blowing, or suction. If necessary, the sheet may be cleaned by running a blank test with the Standard Rubber in case the sheet has been smeared by a test piece from the previous test.

10.3.3 First, at least three test runs shall be made with the Standard Rubber, followed by a maximum of ten runs (see Note 4) with one or more rubbers to be tested (test series). This shall be followed by at least another three runs with the Standard Rubber.

NOTE 4—Do not split tests of one test rubber. Run nine tests, and so forth, if that completes the tests for a test rubber.

10.3.4 When more than one rubber is to be tested, the test runs for each rubber shall be carried out consecutively. Only one test run per test rubber piece is permitted.

10.3.5 At least three test pieces and, for referee purposes, ten test pieces shall be run. The results shall be expressed as the mean value.

10.3.6 The test pieces shall be weighed to the nearest 1 mg and firmly fixed into the holder so that it protrudes 2.0 ± 0.2 mm (0.08 ± 0.008 in.) from the opening of the holder. Turn the vacuum on if it is provided. The swivel arm is moved into starting position and the automatic test run is started (see Note 5).

NOTE 5—A preparatory run to hollow-grind the test piece is not necessary.

10.3.7 There shall be no vibration in the test piece holder during the run. After completing an abrasion path of 40 m (131.2 ft), the test piece shall automatically disengage from the abrasive sheet.

10.3.8 The test piece is reweighed to the nearest 1 mg. Test pieces that have heated up during testing shall be conditioned to room temperature prior to weighing. Any loose material shall be removed from the test piece prior to weighing.

10.3.9 If there is a considerable loss in mass (400 mg or more per 40 m (131.2 ft) abrasion path), the test may be run by stopping the abrasion drum approximately halfway through the test, readjusting the test piece to protrude 2.0 ± 0.2 mm (0.08 ± 0.008 in.) from the opening of the holder and then complete the run. Care shall be taken that the test piece does not abrade to a thickness of less than 5 mm (0.2 in.).

10.3.10 If very high mass losses are encountered (600 mg or more per 40 m (131.2 ft) abrasion path), it may be necessary to terminate the test after an abrasion path of 20 m (65.6 ft). This shall be noted in the test report and the volume loss reported for an abrasion distance of 40 m (131.2 ft) by multiplying the value by two.

10.4 *Method B—Test Run:*

10.4.1 Method B is run with a rotating test piece. This applies to the test rubber as well as the Standard Rubber. Standard Rubber #1 is used as reference.

10.4.2 All other test conditions are the same as described in 10.3.

10.5 *Method C—Test Run:*

10.5.1 Method C is run with a non-rotating test piece. This applies to the test rubber as well as the Standard Rubber. Standard Rubber #2 is used as reference.

10.5.2 All other test conditions are the same as described in 10.3.

10.6 *Method D—Test Run:*

10.6.1 Method D is run with a rotating test piece. This applies to the test rubber as well as the Standard Rubber. Standard Rubber #2 is used as reference.

10.6.2 All other test conditions are the same as described in 10.3.

11. Determination of Abrasiveness of the Abrasive Sheet

11.1 The abrasiveness, S, of the sheet used for the test (expressed as mass loss in mg) shall be determined with the aid of the Standard Rubber #1 in at least three, or, for referee purposes, at least five test runs, both before and after each test series. The test procedure described in 10.3 shall be followed (Method A, non-rotating test piece).

11.2 The abrasiveness is reported as the mean value of the individual mass losses before and after each test series.

11.3 Up to three runs may be carried out with the same test piece of Standard Rubber, making certain that the test piece is placed into the holder the same way each time.

11.4 For test rubbers that have a tendency to smear, it may be necessary to determine the abrasiveness after each test run.

12. Calculation of Test Results

12.1 *Calculation of Abrasion Loss—Method A:*

12.1.1 Calculate the abrasion loss, A_A, the loss in mass (mean value of 3 to 10 single tests) shall be converted into volume loss using the density (volume loss equals mass loss divided by density) of the test rubber (see Note 6). The volume loss is then corrected by using the ratio $\frac{S_O}{S}$ where S_O is the "nominal abrasiveness" of the abrasive sheet and S that of the abrasive sheet used for the test. See Eq 1.

$$A_A = \frac{\Delta m_t \cdot S_0}{d_t \cdot S} \tag{1}$$

where:

A_A = abrasion loss in mm^3 (non-rotating test piece),
Δm_t = mass loss of the test piece in mg,
d_t = density of the test rubber in mg/m^3,
S_0 = "normal abrasiveness" = 200 mg, and
S = abrasiveness in mg.

NOTE 6—The volume loss of non-porous test pieces may also be determined by forced immersion in a buoyant medium, such as water, before and after the test run, instead of weighing the loss in mass and converting this from the density. The volume for the test piece is then obtained by weighing the displaced mass of the buoyant medium.

12.2 *Calculation of Abrasion Resistance Index—Method A:*

TABLE 1 Type 1 Precision (Method A: Non-Rotating Test Piece)

Test Rubber Compound	Property Mean Abrasion Loss A (mm^3)	Within Laboratory			Between Laboratory		
		S_r	r	(r)	S_R	R	(R)
A (NR/BR)	68	2.3	6.5	9.6	6.6	18.8	27.6
B (NR/SBR)	106	3.8	10.8	10.2	7.6	21.4	20.2
C (NR)	160	8.2	23.2	14.5	10.7	30.4	19.0
D (IIR)	257	10.7	30.2	11.8	20.3	57.5	22.4
E (EPDM)	345	14.1	39.8	11.5	29.3	83.0	24.1

NOTE 1—S_r = repeatability standard deviation in measurement units; r = repeatability = 2.83 × S_r (in measurement units); (r) = repeatability in percent of the mean; S_R = reproducibility standard deviation in measurement units; R = reproducibility = 2.83 × S_r (in measurement units); and (R) = reproducibility in percent of the mean.

12.2.1 To calculate the abrasion resistance index, ARI_A, the loss in mass (mean value of 3 to 10 single tests) shall be converted into volume loss using the density of the test rubber (see Note 6). The ratio of the volume loss of Standard Rubber #1 to that of the test rubber is then expressed in percent, as shown in Eq 2.

$$ARI_A = \frac{\Delta m_1 \cdot d_t}{\Delta m_t \cdot d_1} \cdot 100 \tag{2}$$

where:

ARI_A = abrasion resistance index in percent (non-rotating test piece),
Δm_1 = mass loss of the Standard Rubber #1 test piece in mg,
d_1 = density of Standard Rubber #1 in mg/m^3,
Δm_t = mass loss of the test rubber piece in mg, and
d_t = density of the test rubber in mg/m^3.

12.3 *Calculation of Abrasion Resistance Index—Method B:*

12.3.1 The abrasion resistance index, ARI_B, is calculated as described in 12.2.1 using Eq 3:

$$ARI_B = \frac{\Delta m_1 \cdot d_t}{\Delta m_t \cdot d_1} \cdot 100 \tag{3}$$

where:

ARI_B = abrasion resistance index in percent (rotating test piece),
Δm_1 = mass loss of the Standard Rubber #1 test piece in mg,
d_1 = density of Standard Rubber #1 in mg/m^3,
Δm_t = mass loss of the test rubber piece in mg, and
d_t = density of the test rubber in mg/m^3.

12.4 *Calculation of Abrasion Resistance Index—Method C:*

12.4.1 The abrasion resistance index, ARI_C, is calculated as described in 12.2.1 using Eq 4, except that Standard Rubber #2 is used instead of Standard Rubber #1.

$$ARI_C = \frac{\Delta m_2 \cdot d_t}{\Delta m_t \cdot d_2} \cdot 100 \tag{4}$$

where:

ARI_C = abrasion resistance index in percent (non-rotating test piece),
Δm_2 = mass loss of the Standard Rubber #2 test piece in mg, and
d_2 = density of Standard Rubber #2 in mg/m^3.

12.5 *Calculation of Abrasion Resistance Index—Method D:*

12.5.1 The abrasion resistance index, ARI_D, is calculated as described in 12.2.1 using Eq 5, except that Standard Rubber #2 is used instead of Standard Rubber #1.

$$ARI_D = \frac{\Delta m_2 \cdot d_t}{\Delta m_t \cdot d_2} \cdot 100 \tag{5}$$

where:

ARI_D = abrasion resistance index in percent (rotating test piece),
Δm_2 = mass loss of the Standard Rubber #2 test piece in mg, and
d_2 = density of Standard Rubber #2 in mg/m^3.

13. Test Report

13.1 Report the following information:

13.1.1 Type and identification of the product under test,

13.1.2 Date and temperature of testing,

13.1.3 Rubber compound details, if available (rubber type, vulcanization temperature, and time),

13.1.4 Density of the test rubbers,

13.1.5 Method of test piece preparation (cut or molded),

13.1.6 Test method used: Method A, B, C, or D,

13.1.7 The mean value of either the abrasion loss in mm^3 or the abrasion resistance index in percent,

13.1.8 The standard deviation of the test result (optional), and

13.1.9 Any deviations from standard test methods, especially if the length of the abrasion path was reduced from the specified 40 m (131.2 ft) to 20 m (65.6 ft).

14. Precision and Bias[6]

14.1 The precision and bias section deals with test results of Methods A, C, and D obtained in an international interlaboratory program organized in accordance with ISO 5725ISO 5725. This section has been prepared in accordance with Practice D4483, which is equivalent to ISO 5725. Refer to this practice for terminology and other statistical calculation details.

14.2 The precision results in this section give an estimate of the precision of Methods A, C, and D using the materials of the particular interlaboratory test program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the group of

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1079.

TABLE 2 Type 1 Precision (Method C: Non-Rotating Test Piece)

Test Rubber Compound	Property Mean Abrasion Loss A (mm^3)	Within Laboratory			Between Laboratory		
		S_r	r	(r)	S_R	R	(R)
A (NR/BR)	157	10.9	30.8	19.6	21.1	59.6	38.0
B (NR/SBR)	102	5.3	15.0	14.7	6.7	19.1	18.7
C (NR)	67	3.2	9.1	13.6	4.8	13.6	20.3
D (IIR)	43	2.8	7.9	18.4	3.6	10.2	23.7
E (EPDM)	32	1.3	3.8	11.9	2.5	7.2	22.5

NOTE 1—S_r = repeatability standard deviation in measurement units; r = repeatability = 2.83 × S_r (in measurement units); (r) = repeatability in percent of the mean; S_R = reproducibility standard deviation in measurement units; R = reproducibility = 2.83 × S_r (in measurement units); and (R) = reproducibility in percent of the mean.

materials and the specific testing protocols of the test method. Abrasion tests were conducted for each rubber in triplicate on each of two days, separated by three to four days. A test result was reported as the mean of three individual test runs (abrasion loss in mm^3).

14.3 A Type 1 interlaboratory test program was conducted in 1986 with 16 participating laboratories, using five different vulcanized rubbers at different abrasion levels. The vulcanized rubbers were distributed in sheet form by one laboratory, and test pieces were cut from these sheets by each of the participating laboratories.

14.4 The precision is given in terms of *Sr, r,* (*r*), *SR, R*, and (*R*) for the measured abrasion loss values.

14.5 The precision of the test method may be expressed in the format of the following statements, which use an "appropriate value" of *r, R*, (*r*), or (*R*), that is, that value to be used in decisions about test results obtained with this test method. The appropriate value is that value of *r* or *R* associated with the mean level in Tables 1-3 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

14.6 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Tables 1-3. 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.

14.7 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Tables 1-3. 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 value) must be considered to have come from different or nonidentical sample populations.

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

14.9 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 values of abrasion loss are exclusively defined by this test method. Bias, therefore, cannot be determined.

15. Keywords

15.1 abrader; abrasion; abrasion resistance; abrasion resistance index; abrasion test; abrasion tester; ARI; frictional wear; mass loss; rotary drum abrader; rotating drum abrader; volume loss; wear behavior

ANEXO VII

Norma de referencia usado para los ensayos de erosión de los poliuretanos elastoméricos

Designation: D968 – 17

Standard Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive[1]

This standard is issued under the fixed designation D968; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the determination of the resistance of organic coatings to abrasion produced by abrasive falling onto coatings applied to a plane rigid surface, such as a metal or glass panel.

1.2 Two test methods based on different abrasives are covered as follows:

	Sections
Method A—Falling Sand Abrasion Test	8 – 13
Method B—Falling Silicon Carbide Abrasion Test	14 – 21

1.3 These methods should be restricted to testing in only one laboratory when numerical values are used because of the poor reproducibility of the methods (see 13.1.2 and 21.1.2). Interlaboratory agreement is improved significantly when ranking is used in place of numerical values.

1.4 The values stated in SI units are to be regarded as the standard with the exception of mils when determining coating thickness. The values given in parentheses are for information only.

1.5 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels

D1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *Other Standards:*

ANSI B74.12 Specifications for the Size of Abrasive Grain — Grinding Wheels, Polishing and General Industrial Uses[3]

FEPA Standard 42-2:2006 Grains of Fused Aluminum Oxide, Silicon Carbide and other Abrasive Materials for Bonded Abrasives and for General Applications — Microgrits F230 to F2000[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *abrasion resistance, n*—the amount of abrasive required to wear through a unit film thickness of the coating.

4. Summary of Test Method

4.1 Abrasive is allowed to fall from a specified height through a guide tube onto a coated panel until the substrate becomes visible. The amount of abrasive per unit film thickness is reported as the abrasion resistance of the coating on the panel. Silica sand or silicon carbide may be used, as specified.

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[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved June 1, 2017. Published June 2017. Originally approved in 1948. Last previous edition approved in 2015 as D968 – 15. DOI: 10.1520/D0968-17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from Federation of European Producers of Abrasives (FEPA), 20 av., Reille, Paris, F-75014, www.fepa-abrasives.com.

*A Summary of Changes section appears at the end of this standard

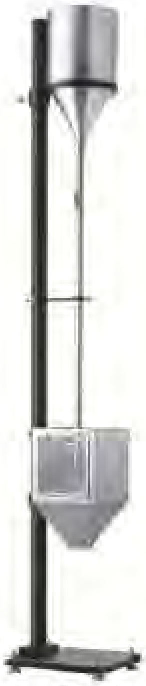
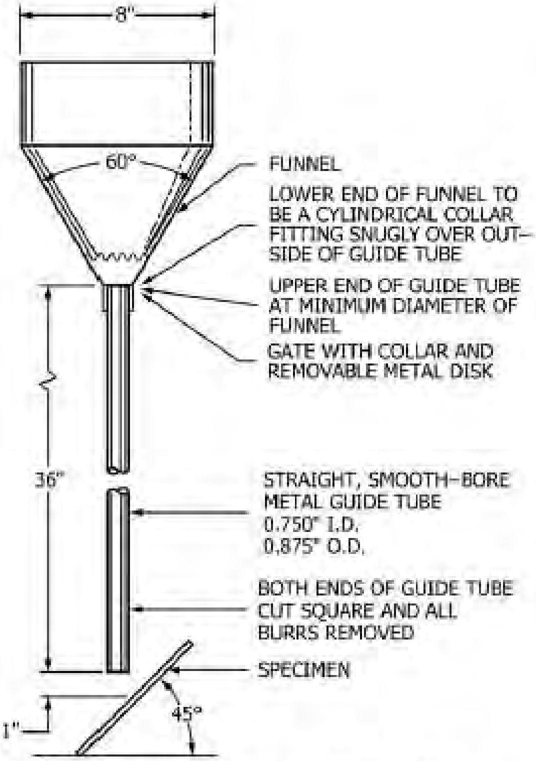


FIG. 1 Apparatus for Falling Sand Abrasion Test



NOTE 1—All dimensions are given in inches. 1 in. = 25.4 mm.

FIG. 2 Design Details of Abrasion Test Apparatus

5. Significance and Use

5.1 Silica sand produces a slower rate of abrasion for organic coatings than that provided by silicon carbide. For some types of coatings, it may also provide greater differentiation.

5.2 The abrasion resistance scales produced by the two methods differ, but the methods provide approximately the same rankings of coatings for abrasion resistance.

5.3 Each of the methods has been found useful for rating the abrasion resistance of specific types of coatings. For example Method A (falling sand) has been used for rating floor coatings while Method B (falling silicon carbide) has been used for rating coatings for ship decks.

METHOD A—FALLING SAND ABRASION TEST [5]

6. Apparatus and Materials

6.1 *Abrasion Tester,* as illustrated in Fig. 1 and Fig. 2. A gate for starting the flow of abrasive is located near the top of the guide tube. It consists of a metal disk inserted into a slit in the side of the guide tube with a collar covering the slit. The guide tube shall be firmly supported in a vertical position over a suitable receptacle, which shall contain a support for holding the coated panel at an angle of 45° to the vertical. The opening of the tube is directly above the area to be abraded and the distance from the tube to the coated surface face at the nearest point is 25 mm (1 in.) when measured in the vertical direction. The base of the apparatus shall be fitted with adjusting screws for properly aligning the equipment.

6.2 *Standard Abrasive*—Natural silica sand from the St. Peters or Jordan sandstone deposits (located in the central United States) shall be considered standard when graded as follows after 5 min of continuous sieving. Use the sieves described in Specification E11.

0 % retained on a No. 16 (1.18 mm) sieve
Maximum 15 % retained on a No. 20 (850 µm) sieve
Minimum 80 % retained on a No. 30 (600 µm) sieve
Maximum 5 % passing a No. 30 (600 µm) sieve

The sand is characterized by its grain shape and has a silicon dioxide content greater than 99 %.

NOTE 1—The abrading qualities of sand obtained from different sources may differ slightly even though the sand meets the sieve requirements. Therefore, for maximum precision of test results, purchaser and seller should use sand from the same source.

7. Test Specimens

7.1 Apply uniform coatings of the material to be tested to a plane, rigid surface such as a metal or glass panel. Prepare a minimum of two coated panels for the material.

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[5] Hipkins, C. C., and Phain, R. J., "The Falling Sand Abrasion Tester," *ASTM Bulletin*, No. 143, December 1946, pp. 18–22.

7.2 Cure the coated panels under the conditions of humidity and temperature agreed upon between the purchaser and seller.

NOTE 2—The coatings should be applied in accordance with Practices D823, or as agreed upon between the purchaser and the seller.

NOTE 3—The thickness of the dry coatings should be measured in accordance with Test Methods D1005 or D7091.

NOTE 4—While the minimum of two coated panels is acceptable, evaluating three or more panels per material will provide greater confidence in your test results.

8. Standardization

8.1 Pour a quantity of standard sand into the funnel and examine the sand stream falling from the lower end of the guide tube. Align the apparatus by means of the adjusting screws in the base until the inner concentrated core of the sand stream falls in the center of the flow when viewed at two positions at 90° to each other. Introduce a measured volume of sand (2000 ± 10 mL is a convenient amount) and determine the time of efflux. The rate of flow shall be 2 L of sand in 21 to 23.5 s.

8.2 Secure a trial panel in the testing position, as described in 6.1, and introduce the sand in increments until a spot 4 mm (5⁄32 in.) in diameter is worn through to the base material. The overall abraded area shall be elliptical in shape, about 25 mm (1 in.) in width and 30 mm (1¼ in.) in length. The center of the area of maximum abrasion shall be on the centerline through the longer axis of the abraded pattern and within 14 to 17 mm (9⁄16 to 11⁄16 in.) of the top edge. Slight final adjustment of the instrument may be required to center the abrasion spot in the pattern.

8.3 A final check on alignment is made by determining the amount of sand that passes through a 4-mm (5⁄32-in.) hole in a metal panel placed directly under the tube. Place a container under the hole in the panel and allow a weighed amount of sand to pass through the tube onto the panel. Weigh the amount of sand that passed through the hole into the container. The apparatus can be considered to be in calibration if the amount of sand that passed through the hole is 90 to 93 % of the amount of sand that impinged on the panel.

9. Conditioning

9.1 Unless otherwise agreed upon between purchaser and seller, condition the coated test panels for at least 24 h at 23 ± 2°C and 50 ± 5 % relative humidity. Conduct the test in the same environment or immediately on removal therefrom.

10. Procedure

10.1 On each coated panel mark three circular areas, each approximately 25 mm (1 in.) in diameter, and so arranged that each can be properly positioned in the panel support of the abrasion tester. Measure the thickness of the coating by Test Methods D1005 or D7091 in at least three locations in each area. Record the mean of each set of measurements as the thickness of the coating over the respective area.

10.2 After conditioning, secure the coated panel in the tester as described in 6.1. Adjust the panel so that one of the marked areas will be centered under the guide tube.

10.3 Pour standard sand, measured volumetrically, into the funnel. Withdraw the gate and allow the sand to flow through the guide tube and impinge on the coated panel. Collect the sand in a container located at the bottom of the tester. Repeat this operation until a 4-mm (5⁄32-in.) diameter area of the coating has worn through to the substrate. A convenient increment of sand to employ during the test is 2000 ± 10 mL. As the end-point is approached, increments of 200 ± 2 mL may be introduced into the funnel.

NOTE 5—When the gate is withdrawn from the guide tube, make certain that a collar covers the slit opening in the tube.

10.4 Abrade each of the remaining marked-off areas of the coated panel as outlined in 10.2.

NOTE 6—Check the alignment of the guide tube at frequent intervals to ensure that the concentrated inner core of the sand stream is falling in the center of the flow.

NOTE 7—After 25 passes through the apparatus, resieve the sand with a No. 30 sieve to remove fines. Replace the sand after 50 passes.

NOTE 8—Sand should be stored in a humidity controlled environment. Excessive moisture level of the sand may influence the results and can be corrected by drying the sand for 1 h in an oven at approximately 82°C (180°F).

10.5 Repeat 10.1 – 10.4 on at least one additional panel coated with the material under test.

11. Calculation

11.1 For each area of the coated panel tested, calculate the abrasion resistance, A, in litres per mil from the following equation:

$$A_{volume} = V/T \tag{1}$$

where:

V = volume of abrasive used, L (to one decimal place) and
T = thickness of coating, mils (to one decimal place).

11.2 Calculate the mean of the abrasion resistance values obtained for different areas of the coated panel and the mean value of the replicate panels.

12. Report

12.1 Report the following information for each coated panel tested:

12.1.1 Temperature and humidity during curing and at the time of testing,

12.1.2 Type and source of abrasive,

12.1.3 Litres of abrasive used for each area tested,

12.1.4 Wear pattern measurements, if different than 8.2,

12.1.5 Coating thickness in mils for each area tested,

12.1.6 Abrasion resistance values for each area tested,

12.1.7 Mean abrasion resistance for each coated panel tested, and

12.1.8 Mean abrasion resistance and range of the replicate coated panels.

13. Precision [6]

13.1 On the basis of an interlaboratory test of this test method in which three laboratories tested four types of coatings

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1037. Contact ASTM Customer Service at service@astm.org.

differing in their abrasion resistance, the within-laboratory coefficient of variation was found to be 9 % with 22 df and the between-laboratories coefficient of variation 35 % with 7 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

13.1.1 *Repeatability*—Two results, each the mean of three runs, obtained by the same operator should be considered suspect if they differ by more than 25 % of their mean value.

13.1.2 *Reproducibility*—Two results, each the mean of three runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 118 % of their mean value.

NOTE 9—The reproducibility of this test is improved substantially when rankings of the coatings by magnitude of abrasion resistance are used. In the interlaboratory test for evaluating precision, all laboratories ranked the coatings in the same order.

13.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining bias for this test method, therefore no statement on bias is being made.

METHOD B—FALLING SILICON CARBIDE TEST

14. Apparatus and Materials

14.1 *Abrasion Tester,* as described in 6.1, with two exceptions:

14.1.1 A metal washer with an opening of 8.5 ± 0.1 mm is centered in the bottom opening of the funnel to restrict the flow of the abrasive.

14.1.2 The disk gate installed in a slit at the top of the guide tube may be replaced by a gate in the bottom of the funnel. This gate consists of a solid metal disk attached to a long vertical screw and mounted above the washer.

14.2 *Standard Abrasive*—Silicon carbide grain shall be considered standard when graded as follows after 5 min of continuous sieving:

0 % retained on a No. 10 (2.00 mm) sieve
Maximum of 20 % retained on a No. 14 (1.40 mm) sieve
Minimum of 45 % retained on a No. 16 (1.18 mm) sieve
Minimum of 70 % retained on a No. 16 (1.18 mm) and No. 18 (1.00 mm) sieve
3 % Maximum passes a No. 20 (850 µm) sieve

These grading requirements correspond to No. 16 grit in accordance with ANSI B74.12. These grading requirements also correspond to F16 grit in accordance with FEPA Standard 42-2:2006.

14.2.1 Use the sieves described in Specification E11.

15. Test Specimens

15.1 Prepare the coated panels as outlined in 7.1 and 7.2.

16. Standardization

16.1 Standardize the abrasion tester by the procedures given in 8.1 – 8.3, with the following exceptions:

16.1.1 Use silicon carbide grain where sand is specified.

16.1.2 Weigh the volume of silicon carbide grain to be introduced into the tester. Determine the efflux time for this volume. The rate of flow shall be 10 ±1 g/s.

17. Conditioning

17.1 Unless otherwise agreed upon between purchaser and seller, condition the coated panels for at least 24 h at 23 ± 2°C and 50 ± 5 % relative humidity. Conduct the test in the same environment or immediately on removal therefrom.

18. Procedure

18.1 Measure the thickness of the coating by the procedures given in 10.1.

18.2 Abrade the coated panel by the procedures given in 10.1 – 10.4 using silicon carbide as the abrasive. Determine the volume or weight, or both, of abrasive used to reach the end point.

18.3 Repeat 18.1 and 18.2 on at least one additional coated panel of the material under test.

19. Calculation

19.1 For each area of the coated panel tested, calculate the abrasion resistance in litres per mil from the equation given in 11.1 or, calculate the abrasion resistance, A, in kilograms per mil from the equation:

$$A_{weight} = W/T \qquad (2)$$

where:

W = weight of abrasive used, kg (to one decimal place) and
T = thickness of coating, mils (to one decimal place).

19.2 Calculate the mean of the abrasion resistance values obtained at different areas of the coated panel and the mean values of the replicate panels.

20. Report

20.1 Report the information specified in Section 12 for each coated panel tested. Abrasion resistance may be reported as litres per mil or as kilograms per mil.

21. Precision [6]

21.1 An interlaboratory test was conducted in which operators in three laboratories tested four coatings having a broad range of abrasion resistance. The within-laboratory coefficient of variation was 19 % with 16 df. The between-laboratories coefficient of variation was 45 % with 8 df. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

21.1.1 *Repeatability*—Two results, each the mean of three runs, obtained by the same operator should be considered suspect if they differ by more than 56 % of their mean value.

21.1.2 *Reproducibility*—Two results, each the mean of three runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 147 % of their mean value. See Note 7.

21.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining bias for this test method, therefore no statement on bias is being made.

22. Keywords

22.1 abrasion; abrasion (of paints/related coatings); falling abrasive tester; falling sand abrasion test; falling silicon carbide abrasion test; resistance

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D968–16) that may impact the use of this standard. (Approved June 1, 2017.)

(*1*) Added Note 8, suggesting sand be stored in a humidity controlled environment.

Committee D01 has identified the location of selected changes to this standard since the last issue (D968-15) that may impact the use of this standard. (Approved December 1, 2016.)

(*1*) Replaced image in Fig. 2.
(*2*) Added requirement to report wear pattern measurement in section 12.1.4.

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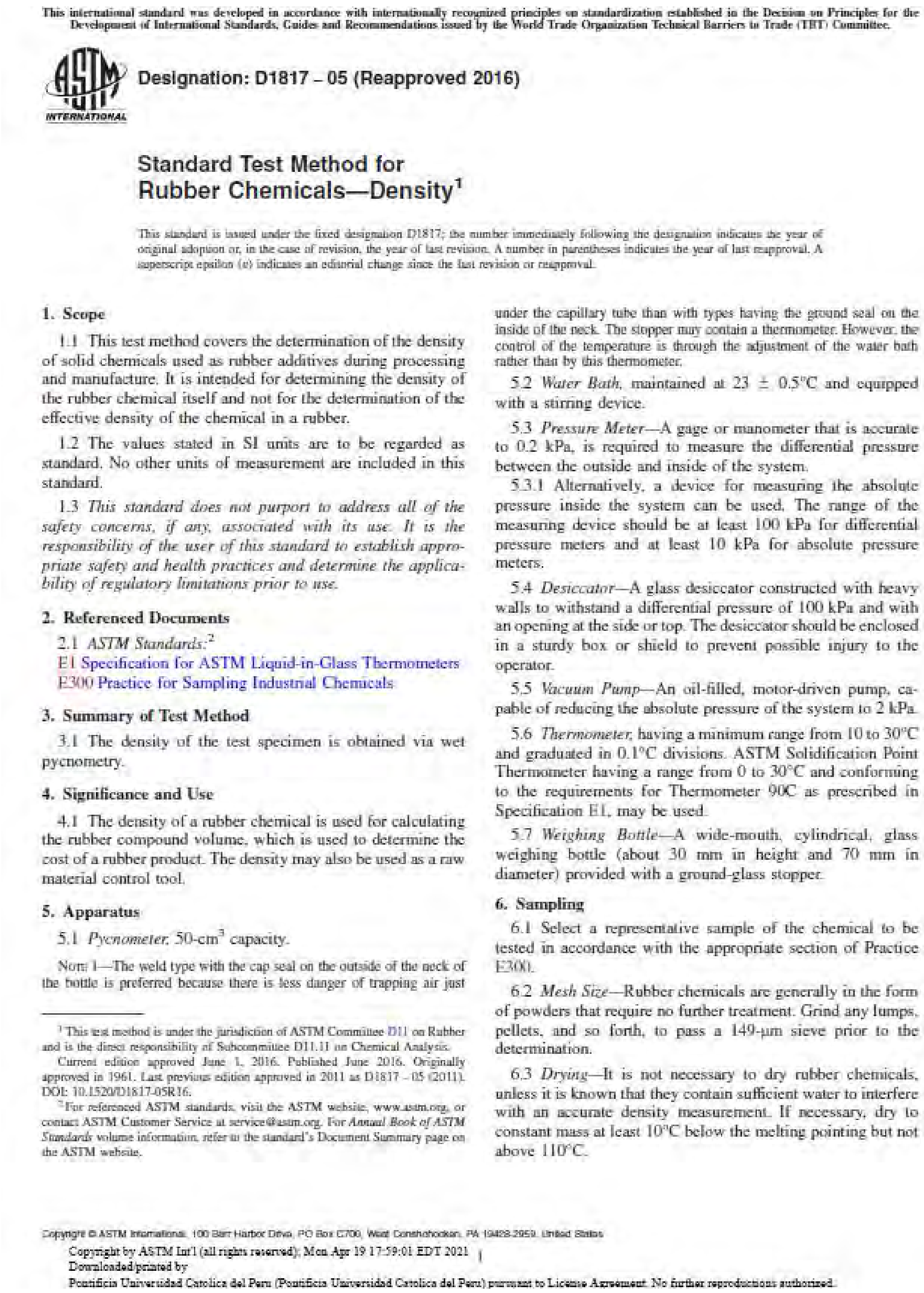
ANEXO VIII

Norma de referencia usado para la determinación de la densidad de los poliuretanos elastoméricos



Designation: D1817 – 05 (Reapproved 2016)

Standard Test Method for Rubber Chemicals—Density[1]

This standard is issued under the fixed designation D1817; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the density of solid chemicals used as rubber additives during processing and manufacture. It is intended for determining the density of the rubber chemical itself and not for the determination of the effective density of the chemical in a rubber.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1 Specification for ASTM Liquid-in-Glass Thermometers
E300 Practice for Sampling Industrial Chemicals

3. Summary of Test Method

3.1 The density of the test specimen is obtained via wet pycnometry.

4. Significance and Use

4.1 The density of a rubber chemical is used for calculating the rubber compound volume, which is used to determine the cost of a rubber product. The density may also be used as a raw material control tool.

5. Apparatus

5.1 *Pycnometer,* 50-cm^3 capacity.

NOTE 1—The weld type with the cap seal on the outside of the neck of the bottle is preferred because there is less danger of trapping air just under the capillary tube than with types having the ground seal on the inside of the neck. The stopper may contain a thermometer. However, the control of the temperature is through the adjustment of the water bath rather than by this thermometer.

5.2 *Water Bath,* maintained at 23 ± 0.5°C and equipped with a stirring device.

5.3 *Pressure Meter*—A gage or manometer that is accurate to 0.2 kPa, is required to measure the differential pressure between the outside and inside of the system.

5.3.1 Alternatively, a device for measuring the absolute pressure inside the system can be used. The range of the measuring device should be at least 100 kPa for differential pressure meters and at least 10 kPa for absolute pressure meters.

5.4 *Desiccator*—A glass desiccator constructed with heavy walls to withstand a differential pressure of 100 kPa and with an opening at the side or top. The desiccator should be enclosed in a sturdy box or shield to prevent possible injury to the operator.

5.5 *Vacuum Pump*—An oil-filled, motor-driven pump, capable of reducing the absolute pressure of the system to 2 kPa.

5.6 *Thermometer,* having a minimum range from 10 to 30°C and graduated in 0.1°C divisions. ASTM Solidification Point Thermometer having a range from 0 to 30°C and conforming to the requirements for Thermometer 90C as prescribed in Specification E1, may be used.

5.7 *Weighing Bottle*—A wide-mouth, cylindrical, glass weighing bottle (about 30 mm in height and 70 mm in diameter) provided with a ground-glass stopper.

6. Sampling

6.1 Select a representative sample of the chemical to be tested in accordance with the appropriate section of Practice E300.

6.2 *Mesh Size*—Rubber chemicals are generally in the form of powders that require no further treatment. Grind any lumps, pellets, and so forth, to pass a 149-μm sieve prior to the determination.

6.3 *Drying*—It is not necessary to dry rubber chemicals, unless it is known that they contain sufficient water to interfere with an accurate density measurement. If necessary, dry to constant mass at least 10°C below the melting pointing but not above 110°C.

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

Current edition approved June 1, 2016. Published June 2016. Originally approved in 1961. Last previous edition approved in 2011 as D1817 – 05 (2011). DOI: 10.1520/D1817-05R16.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

7. Immersion Liquid

7.1 An immersion liquid should be chosen in which no portion of the rubber chemical (major component, impurity, or added component) is soluble and which will not react with any portion of the rubber chemical. Refined white kerosene of narrow boiling range and low evaporation rate may sometimes be used. Other immersion liquids such as ethylene glycol, tetrahydronaphthalene, and so forth, may be suitable. If an immersion liquid cannot be found that meets these solubility requirements, an immersion liquid saturated with the soluble components of the sample shall be used.

8. Standardization of Pycnometer

8.1 Fill the pycnometer with freshly boiled distilled water at 20 to 22°C; gradually bring to 23 ± 0.5°C in the water bath, and then remove, dry, and weigh it as described in 9.1. Empty the pycnometer, then clean, dry, and reweigh it. Next, fill the pycnometer with the immersion liquid at 22°C or less, and bring to 23 ± 0.5°C in the water bath. Remove from the bath, dry, and weigh as before. Calculate the density of the immersion liquid as follows:

$$\text{Density at } 23^{\circ}C, Mg/m^3 = (A/B) \times 0.997 \qquad (1)$$

where:

A = mass of immersion liquid, and
B = mass of water,
0.9976 = density of water at 23°C, Mg/m^3.

9. Procedure

9.1 *Weighing*—Transfer a sufficient amount of the test specimen to a clean, dry, weighed pycnometer to form a layer approximately 19 mm (¾ in.) deep and reweigh. Weigh rubber chemicals of a hygroscopic nature from a weighing bottle.

9.2 *Addition of Immersion Liquid*—Add sufficient immersion liquid to the pycnometer to form a thin layer above the rubber chemical. When necessary, swirl the contents of the pycnometer by hand, to wet the sample.

9.3 *Removal of Occluded Air*—Place the pycnometer in the desiccator, close, and attach to the pump for the removal of air. Take care not to decrease the pressure too quickly, otherwise some of the sample may be lost due to the sudden removal of the entrapped air. A pinch-cock may be used to control the rate of evacuation. The pressure meter is used to indicate whether the oil pump is giving the proper vacuum, which is an absolute pressure of 0.2 kPa. Bubbles of air rise from the sample rapidly at first, then decrease and finally stop. The time required for complete removal of air may vary from 30 min to 24 h. When no more bubbles can be seen, it may be assumed that occluded air has been removed and the rubber chemical is thoroughly wet with immersion liquid. Slowly readmit air to the desiccator.

NOTE 2—If an immersion liquid is used whose vapor pressure is greater than 0.2 kPa at room temperature, the liquid will evaporate, often with "bumping." Therefore it is necessary to adjust the vacuum to a higher pressure with some loss in effectiveness of air removal. The use of such immersion liquids should be limited to those cases in which no low vapor pressure liquid can be used. In no case should a mixed liquid be used in which any component has an appreciable vapor pressure.

9.4 *Final Adjustment*—Remove the pycnometer from the desiccator, fill with immersion liquid at 22°C or less, taking care to add a sufficient quantity to prevent air bubbles remaining in the pycnometer when closed. Insert the stopper, being careful not to trap any air bubbles. Place the pycnometer in the water bath and permit it to come to constant temperature at 23 ± 0.5°C. Remove from the water bath, wipe the end of the capillary with lint-free toweling or lens paper, making sure not to suck any liquid from the capillary. Cap the capillary. Dip the pycnometer (up to the side arm) in a beaker of alcohol to remove any residual immersion liquid. Thoroughly dry the outside of the pycnometer and weigh.

9.5 *Number of Specimens*—Make duplicate tests on all specimens.

10. Calculation

10.1 Calculate the density of the rubber chemical as follows:

$$\text{Density at } 23^{\circ}C, Mg/m^3 = PS/[(P+K)-F] \qquad (2)$$

where:

P = mass of rubber chemical used,
S = density of the immersion liquid,
K = mass of the pycnometer filled with immersion liquid, and
F = final mass of the pycnometer with rubber chemical and immersion liquid.

11. Report

11.1 Report the following information:

11.1.1 Sample identification

11.1.2 Density at 23°C, Mg/m^3, and

11.1.3 Immersion liquid used.

12. Precision and Bias

12.1 *Precision*—This test method has not been tested for reproducibility or repeatability, but duplicate determinations on the same sample should not differ by more than 0.02 Mg/m^3 at 23°C.

12.2 *Bias*—No statement about bias is being made at this time.

13. Keywords

13.1 density; rubber chemicals

ANEXO IX

Norma de referencia usado para los ensayos de la dureza de los poliuretanos elastoméricos

Designation: D2240 – $15^{\varepsilon 1}$

Standard Test Method for Rubber Property—Durometer Hardness[1]

This standard is issued under the fixed designation D2240; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

ε^1 NOTE—Editorially corrected 8.3 in November 2017.

1. Scope

1.1 This test method covers twelve types of rubber hardness measurement devices known as durometers: Types A, B, C, D, DO, E, M, O, OO, OOO, OOO-S, and R. The procedure for determining indentation hardness of substances classified as thermoplastic elastomers, vulcanized (thermoset) rubber, elastomeric materials, cellular materials, gel-like 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 D1415.

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

1.4 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 organizations parallel in nature.

1.5 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.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D374 Test Methods for Thickness of Solid Electrical Insulation (Metric) D0374_D0374M

D618 Practice for Conditioning Plastics for Testing

D785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

D1349 Practice for Rubber—Standard Conditions for Testing

D1415 Test Method for Rubber Property—International Hardness

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

F1957 Test Method for Composite Foam Hardness-Durometer Hardness

2.2 *ISO Standard:*[3]

ISO/IEC 17025: 1999 General Requirements for the Competence of Testing and Calibration Laboratories

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

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Aug. 1, 2015. Published January 2016. Originally approved in 1964. Last previous edition approved in 2010 as D2240 – 05 (2010). DOI: 10.1520/D2240-15E01.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland.

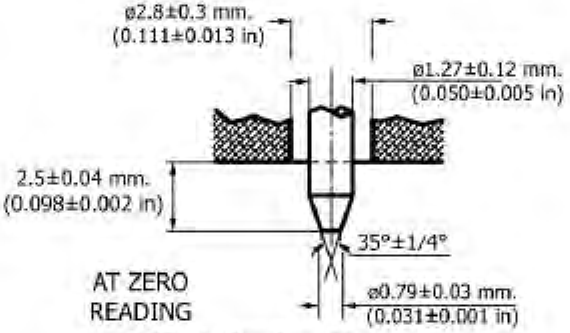

FIG. 1 (a) Type A and C Indentor

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 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 indentor 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 indentor 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 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 D785 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 (see 5.1.2) consisting of the following components:

5.1.1 *Durometer:*

5.1.1.1 *Presser Foot*, the configuration and the total area of a durometer presser foot may produce varying results when there are significant differences between them. It is recommended that when comparing durometer hardness determinations of the same type (see 4.1), that the comparisons be between durometers of similar presser foot configurations and total area, and that the presser foot configuration and size be noted in the Hardness Measurement Report (see 10.2.4 and 5.1.1.3).

5.1.1.2 *Presser Foot*, Types A, B, C, D, DO, E, O, OO, OOO, and OOO-S, with an orifice (to allow for the protrusion of the indentor) having a diameter as specified in Fig. 1 (a, b, c, d, e, f, and g), with the center a minimum of 6.0 mm (0.24 in.) from any edge of the foot. When the presser foot is not of a flat circular design, the area shall not be less than 500 mm^2 (19.7 in.^{2}).

NOTE 1—The Type OOO and the Type OOO-S, designated herein, differ in their indentor configuration, spring force, and the results obtained. See Table 1 and Fig. 1 (e and g).

5.1.1.3 *Presser Foot*—flat circular designs designated as Type *xR*, where *x* is the standard durometer designation and *R* indicates the flat circular press foot described herein, for example, Type *aR*, *dR*, and the like. The presser foot, having a centrally located orifice (to allow for the protrusion of the indentor) of a diameter as specified in Fig. 1 (a through g). The flat circular presser foot shall be 18 ± 0.5 mm (0.71 ± 0.02 in.) in diameter. These durometer types shall be used in an operating stand (see 5.1.2).

(a) Durometers having a presser foot configuration other than that indicated in 5.1.1.3 shall not use the Type *xR* designation, and it is recommended that their presser foot configuration and size be stated in the Hardness Measurement Report (see 10.2.4).

5.1.1.4 *Presser Foot, Type M*, with a centrally located orifice (to allow for the protrusion of the indentor), having a diameter as specified in Fig. 1 (d), with the center a minimum of 1.60 mm (0.063 in.) from any edge of the flat circular presser foot. The Type M durometer shall be used in a Type 3 operating stand (see 5.1.2.4).

5.1.1.5 *Indentor*, formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1 (a, b, c, d, e, or g), polished over the contact area so that no flaws are visible under 20× magnification, with an indentor extension of 2.50 ± 0.04 mm (0.098 ± 0.002 in.).

5.1.1.6 *Indentor, Type OOO-S*, formed from steel rod and hardened to 500 HV10, shaped in accordance with Fig. 1 (f), polished over the contact area so that no flaws are visible under 20× magnification, with an indentor extension of 5.00 ± 0.04 mm (0.198 ± 0.002 in.).

5.1.1.7 *Indentor, Type M*, formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1 (d), polished over the contact area so that no flaws are visible under 50× magnification, with an indentor extension of 1.25 ± 0.02 mm (0.049 ± 0.001 in.).

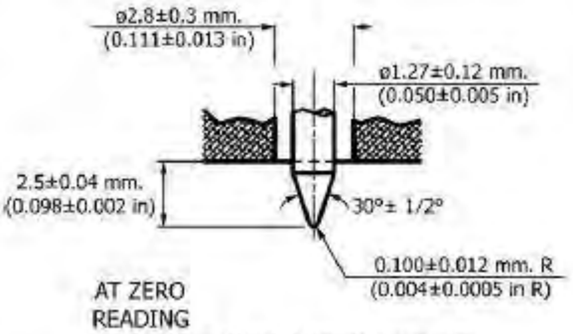

FIG. 1 (b) Type B and D Indentor *(continued)*

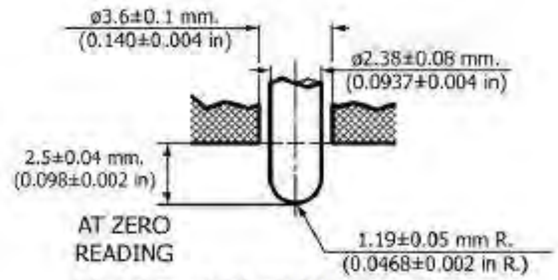

FIG. 1 (c) Type O, DO, and OO Indentor *(continued)*

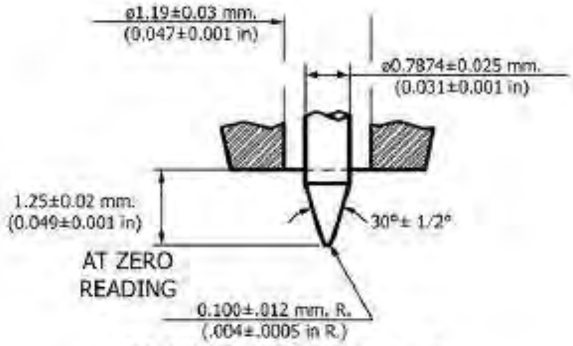

FIG. 1 (d) Type M Indentor *(continued)*

5.1.1.8 *Indentor Extension Indicator*, analog or digital electronic, having a display that is an inverse function of the indentor extension so that:

(1) The display shall indicate from 0 to 100 with no less than 100 equal divisions throughout the range at a rate of one hardness point for each 0.025 mm (0.001 in.) of indentor movement,

(2) The display for Type OOO-S durometers shall indicate from 0 to 100 with no less than 100 equal divisions throughout the range at a rate of one hardness point for each 0.050 mm (0.002 in.) of indentor movement,

(3) The display for Type M durometers 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 indentor movement, and

(4) In the case of analog dial indicators having a display of 360°, the points indicating 0 and 100 may be at the same point on the dial and indicate 0, 100, or both.

5.1.1.9 *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

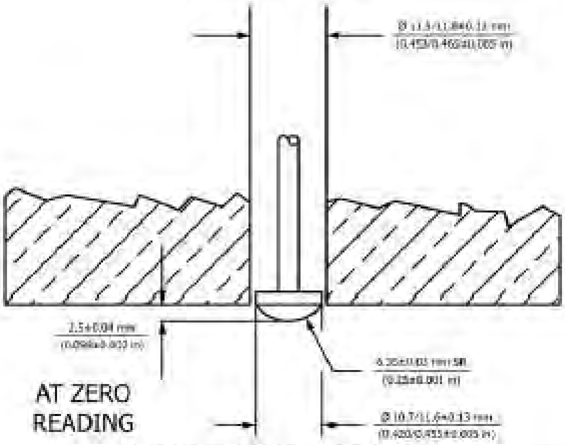

FIG. 1 (e) Type OOO Indentor *(continued)*

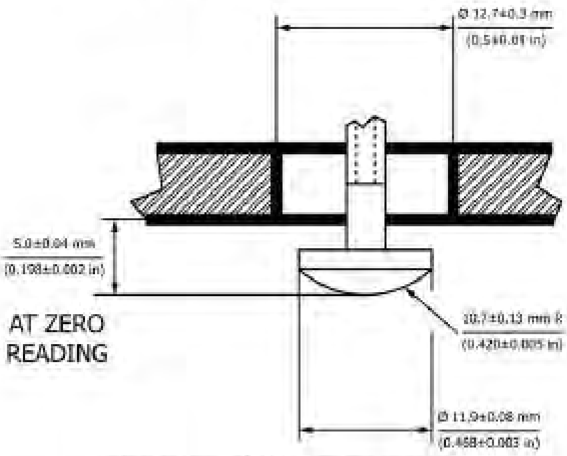

FIG. 1 (f) Type OOO-S Indentor *(continued)*

presser foot is in contact with the specimen being tested, for example, the initial indentor 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 of the calibration tolerance stated in Table 1.

5.1.1.10 *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 valued achieved until reset by the operator.

5.1.1.11 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 (see 10.1.5), and when reporting hardness determinations (see 10.2.4). Analog Type M, OO, OOO, and Type OOO-S durometers shall not be equipped with maximum indicating pointers.

5.1.1.12 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.

5.1.1.13 *Calibrated Spring*, for applying force to the indentor, in accordance with Fig. 1 (a through g) and capable of applying the forces as specified in Table 1.

5.1.2 *Operating Stand* (Fig. 2):

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. 3) throughout the travel of each. The durometer presser foot to specimen support table parallelism shall be verified each time the test specimen support table

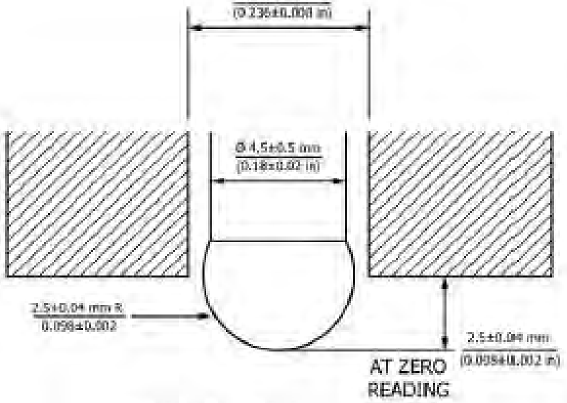

FIG. 1 (g) Type E Indentor *(continued)*

TABLE 1 Durometer Spring Force Calibration[A]
All Values are in N

Indicated Value	Type A, B, E, O	Type C, D, DO	Type M	Type OO, OOO	Type OOO-S
0	0.55	0	0.324	0.203	0.167
10	1.3	4.445	0.368	0.294	0.343
20	2.05	8.89	0.412	0.385	0.520
30	2.8	13.335	0.456	0.476	0.696
40	3.55	17.78	0.5	0.566	0.873
50	4.3	22.225	0.544	0.657	1.049
60	5.05	26.67	0.589	0.748	1.226
70	5.8	31.115	0.633	0.839	1.402
80	6.55	35.56	0.677	0.93	1.579
90	7.3	40.005	0.721	1.02	1.755
100	8.05	44.45	0.765	1.111	1.932
N/durometer unit	0.075	0.4445	0.0044	0.00908	0.01765
Spring Calibration Tolerance	± 0.075 N	± 0.4445 N	± 0.0176 N	± 0.0182 N	± 0.0353 N

[A] Refer to 5.1.1.3 for the Type *xR* designation.

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 indentor type), shall be capable of applying the specimen to the indentor in a manner that minimizes shock.

5.1.2.3 *Operating Stand, Type 2* (indentor to specimen type), shall be capable of applying the indentor to the specimen in a manner that minimizes shock.

5.1.2.4 *Operating Stand, Type 3* (indentor 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 indentor 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. 3) 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 indentor to the center of the insert, or the point at which the indentor is to contact the specimen. Care should be exercised to assure that the indentor does not abruptly contact the specimen support table as damage to the indentor 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

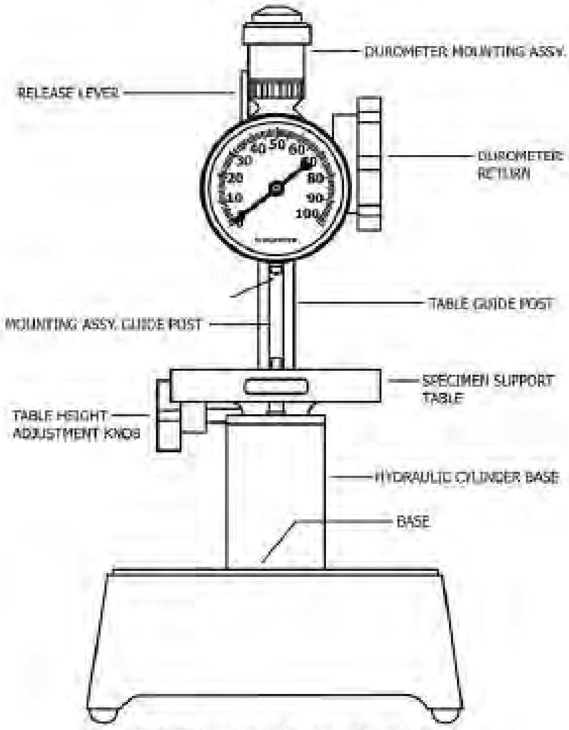

FIG. 2 Durometer Operating Stand

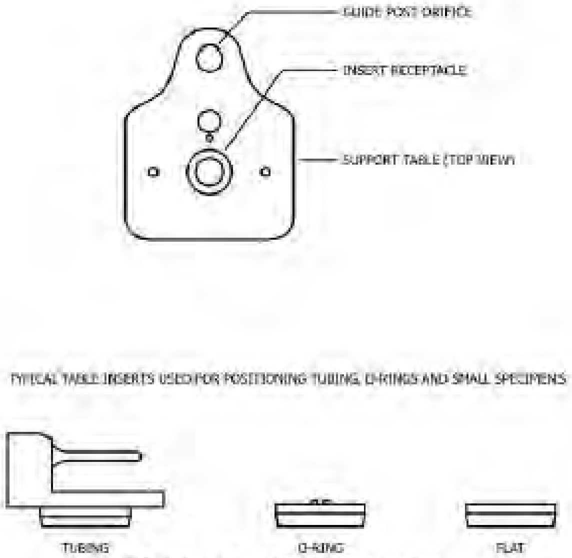

FIG. 3 Small Specimen Support Table

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 indentor 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 indentor.*

6.2 Type OOO, OOO-S, and 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 indentor.*

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 indentor 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 *Indentor 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 indentor. Arrange the blocks so that the durometer presser foot contacts the larger block(s) and the indentor tip just contacts the smaller block (Fig. 4). It is necessary to observe the arrangement of the blocks and the presser foot/indentor under a minimum of 20× magnification to assure proper alignment.

7.1.2 Indentor extension and shape shall be in accordance with 5.1.1.5, 5.1.1.6, or 5.1.1.7, respective to durometer type. See Fig. 1 (a through g). Examination of the indentor under 20× magnification, 50× for Type M indentors, is required to examine the indentor 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 + 0.00/–0.0254 mm (0.100 + 0.00/–0.001 in.) between them. For Type OOO-S durometers, the gage block dimensions are 5.08 + 0.00/–0.0508 mm (0.200 + 0.00/–0.002 in.). For Type M durometers, the gage block dimensions are 1.27 + 0.0/–0.0127 mm (0.050 + 0.00/–0.0005 in.) between them (Fig. 4).

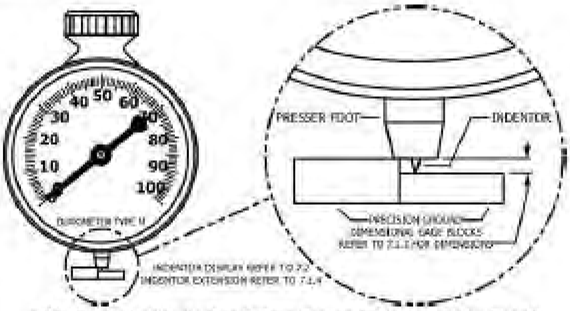

FIG. 4 Detail of Indentor Extension and Display Adjustment

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

7.1.5 Adjust the indentor extension to 2.50 ± 0.04 mm (0.098 ± 0.002 in.). For Type OOO-S durometers, adjust the indentor extension to 5.0 ± 0.04 mm (0.198 ± 0.002 in.). For Type M durometers, adjust the indentor 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 indentor tip. Fig. 4 depicts a suitable arrangement for gaging indentor extension.

7.1.6 Parallelism of the durometer presser foot to the support surface, and hence the dimensional gage blocks, at the time of instrument calibration, may be in accordance with Test Methods D374, Machinist's Micrometers, or otherwise accomplished in accordance with the procedures specified by the manufacturer.

7.2 *Indentor Display Adjustment:*

7.2.1 After adjusting the indentor extension as indicated in 7.1, use a similar arrangement of dimensional gage blocks to verify the linear relationship between indentor travel and indicated display at two points: 0 and 100. Following the manufacturer's recommendations, make adjustments so that:

7.2.2 The indicator displays a value equal to the indentor travel measured to within:

–0.0 +1.0 durometer units measured at 0;

±0.50 durometer units measured at 100;

±1 durometer units at all other points delineated in 7.4.

7.2.3 Each durometer point indicated is equal to 0.025 mm (0.001 in.) of indentor travel, except for:

7.2.3.1 Type M Durometers, each indicated point is equal to 0.0125 mm (0.0005 in.) of indentor travel;

7.2.3.2 Type OOO-S Durometers, each indicated point is equal to 0.050 mm (0.002 in.) of indentor travel.

7.2.4 The indicator shall not display a value greater than 100 or less than 0 at the time of calibration.

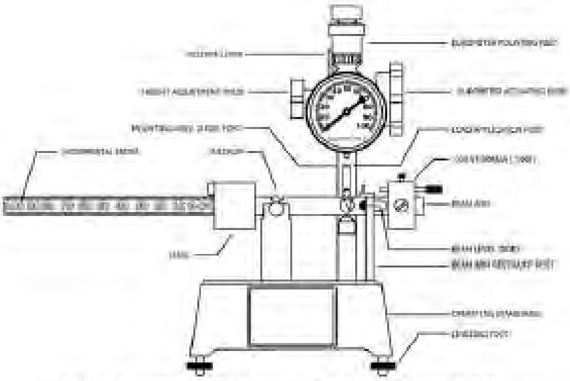

FIG. 5 Example of Durometer Calibration Apparatus

7.2.5 Other means of determining indentor extension or indentor travel, such as optical or laser measurement methods, are acceptable. The instrumentation used shall have traceability as described in 1.4.

7.2.6 The durometer shall be supported in a suitable fashion when performing the procedures described in 7.1 and 7.2.

7.3 *Calibration Device:*

7.3.1 The durometer spring shall be calibrated by supporting the durometer in a calibrating device, see Fig. 5, in a vertical position and applying a measurable force to the indentor tip. The force may be measured by means of a balance as depicted in Fig. 5, or an electronic force cell. The calibrating device shall be capable of measuring applied force to within 0.5 % of the maximum spring force necessary to achieve 100 durometer units.

7.3.2 Care should be taken to ensure that the force is applied vertically to the indentor tip, as lateral force will cause errors in calibration. See 7.1.5.1 and 7.1.6.

7.4 *Spring Calibration*—The durometer spring shall be calibrated at displayed readings of 10, 20, 30, 40, 50, 60, 70, 80, and 90. The measured force (9.8× mass in kilograms) shall be within the spring calibration tolerance specified in Table 1. Table 1 identifies the measured force applied to the indentor for the entire range of the instrument, although it is necessary only to verify the spring calibration at points listed herein.

7.5 *Spring Calibration Procedure:*

7.5.1 Ensure that the indentor extension has been adjusted in accordance with 7.1, and the linear relationship between indentor travel and display is as specified in 7.2.

7.5.2 Place the durometer in the calibration device as depicted in Fig. 5. Apply the forces indicated in Table 1 so that forces applied are aligned with the centerline of the indentor in a fashion that eliminates shock or vibration and adjust the durometer according to manufacturers' recommendations so that:

7.5.3 At the points enumerated in 7.4, the display shall indicate a value equal to 0.025 mm (0.001 in.) of indentor travel. For Type OOO-S durometers, the display shall indicate a value equal to 0.05 mm (0.002 in.) of indentor travel. For Type M durometers, the display shall indicate a value equal to 0.0125 mm (0.0005 in.) of indentor travel within the spring calibration tolerances specified in 7.6.

7.6 Spring calibration tolerances are ±1.0 durometer units for Types A, B, C, D, E, O, and DO, ±2.0 durometer units for Types OO, OOO, and OOO-S, and ±4.0 durometer units for Type M, while not indicating below 0 or above 100 at the time of calibration (see Table 1).

7.7 *Spring Force Combinations:*

7.7.1 For Type A, B, E, and O durometers:
Force, N = 0.55 + 0.075 HA
Where HA = hardness reading on Type A, B, E, and O durometers.

7.7.2 For Type C, D, and DO durometers:
Force, N = 0.4445 HD
Where HD = hardness reading on Type C, D, and DO durometers.

7.7.3 For Type M durometers:
Force, N = 0.324 + 0.0044 HM
Where HM = hardness reading on Type M durometers.

7.7.4 For Type OO and OOO durometers:
Force, N = 0.203 + 0.00908 HOO
Where HOO = hardness reading on Type OO durometers.

7.7.5 For Type OOO-S durometers:
Force, N = 0.167 + 0.01765 HOOO-S
Where HOOO-S = hardness reading on Type OOO-S durometers.

7.8 The rubber reference block(s) provided for verifying durometer operation and state of calibration are not to be relied upon as calibration standards. The calibration procedures outlined in Section 7 are the only valid calibration procedures.

7.8.1 The use of metal reference blocks is no longer recommended (see Note 2).

7.9 Verifying the state of durometer calibration, *during routine use*, may be accomplished by:

7.9.1 Verifying that the zero reading is no more than 1 indicated point above zero, and not below zero (on durometers so equipped), when the durometer is positioned so that no external force is placed upon the indentor.

7.9.2 Verifying that the 100 reading is no more than 100 and no less than 99 when the durometer is positioned on a flat surface of a non-metallic material so that the presser foot is in complete contact, causing the indentor to be fully retracted.

7.9.2.1 It is important that when performing the verification of 100, as described in 7.9.2, that extreme care be taken so as to not cause damage to the indentor. Verification of the 100 value is not recommended for durometers having a spring force greater than 10 N (Types C, D, and DO).

7.9.2.2 When performing the verification of 100, as described in 7.9.2, the non-metallic material shall be of a hardness value greater than 100 of the type (scale) of the durometer being employed. Tempered glass of a thickness greater than 6.35 mm (0.25 in.) has been found satisfactory for this application.

7.9.3 Verifying the displayed reading at any other point using commercially available rubber reference blocks which are certified to a stated value of the type (scale) of the durometer being employed. The displayed value of the durometer should be within ±2 durometer points of the reference block's stated value.

7.9.4 Verification of the zero and 100 readings of a durometer provide reasonable assurance that the linear relationship between the indicated display and the durometer mechanism remain valid.

7.9.5 Verification of points between zero and 100 provide reasonable assurance that the curvilinear relationship between the indicated display and the durometer mechanism remain valid.

7.9.6 *This is not a calibration procedure, it is a means by which a user may routinely verify that the durometer may be functioning correctly.* (See Note 2.)

8. Laboratory Atmosphere and Test Specimen Conditioning

8.1 Tests shall be conducted in the standard laboratory atmosphere, as defined in Practice D618, Section 4.2.

8.2 The instrument shall be maintained in the standard laboratory atmosphere, as defined in Practice D618, Section 4.1, for 12 h prior to performing a test.

8.3 The specimen shall be conditioned in accordance with condition 40/23 exclusive of humidity control, as described in Practice D618, Section 9.1, Procedure A and tested under the same conditions, exclusive of humidity control.

8.4 These procedures may be modified if agreed upon between laboratories or between supplier and user and are in accordance with alternative procedures identified in Practice D618.

8.5 No conclusive evaluation has been made on durometers at temperatures other than 23.0 ± 2.0°C (73.4 ± 3.6°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 D1349).

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 ± 2.5 mm (1.00 ± 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 indentor is in accordance with Section 6, unless it is known that identical results are obtained when measurements are made with the indentor at a 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 indentor" type operating stands, operate the lever or other mechanism to apply the specimen to the indentor 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 stand for the Type M durometer is not an acceptable practice, see 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, when the initial indentor travel has ceased, the maximum indicated reading shall be recorded. The time interval of 1 s, between initial indentor travel cessation and the recording of the indicated reading, shall be considered standard. Other time intervals, when agreed upon among laboratories or between supplier and user, may be used and reported accordingly. The indicated hardness reading may change with time.

9.1.7.1 If the durometer is equipped with an electronic maximum indicator or timing device (refer to 5.1.1.9) the indicated reading shall be recorded within 1 ± 0.3 s of the cessation of indentor travel and reported (refer to 10.2.9 for reporting protocols), unless otherwise noted.

9.1.7.2 If the durometer is equipped with an analog type maximum indicator (refer to 5.1.1.10), the maximum indicated reading may be recorded and shall be reported (refer to 10.2.9), unless otherwise noted.

9.1.7.3 If the durometer is not equipped with the devices described in 5.1.1.9 or 5.1.1.10, the indicated reading shall be recorded within 1 s as is possible and reported (refer to 10.2.9), unless otherwise noted.

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 indentor 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 indentor 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 ± 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 ± 0.1 s of the cessation of initial indentor 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 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 (handheld) 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 indentor. Recommended masses are 1 kg for Type A, B, E, and O durometers, 5 kg for Type C, D, and DO durometers, and 400 g for Type OO, OOO, and OOO-S durometers. The introduction of an additional mass on Type M durometers is not permitted. Further improvement may be achieved by the use of a durometer operating stand that 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 Calibration due date (see Note 2).

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

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

10.1.6 Ambient temperature.

10.1.7 Relative humidity.

10.1.8 Technician identification.

10.1.9 Applicable standards to which the instrument is calibrated.

10.1.10 Calibrating instrument information to include type, serial number, manufacturer, date of last calibration, calibration due date (see Note 2), and a statement of traceability of standards used to NIST or other acceptable organization. See 1.4.

10.2 *Hardness Measurement Report:*

10.2.1 Date of test.

TABLE 2 Type 1 Precision—Type M Durometer Method

Material		Within Laboratories			Between Laboratories		
	MEAN	Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	31.8	1.26	3.58	11.24	3.76	10.63	33.41
2	40.8	1.14	3.23	7.90	2.47	7.00	17.13
3	54.0	0.975	2.76	5.11	2.38	6.73	12.46
4	62.8	0.782	2.21	3.52	2.24	6.34	10.10
5	70.9	0.709	2.01	2.83	0.974	2.76	3.89
6	80.6	1.686	4.77	5.92	1.61	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	8.26	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	3.56	2.29	6.49	12.17
11	63.2	0.485	1.37	2.17	2.19	6.20	9.80
12	69.8	0.737	2.09	3.00	0.99	2.80	4.02
13	78.3	0.784	2.22	2.84	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	0.635	1.80	4.25	1.20	3.39	8.01
17	54.6	0.56	1.59	2.90	2.15	6.09	11.15
18	62.9	1.12	3.17	5.04	1.47	4.16	6.61
19	70.3	0.689	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	5.86	6.67
AVERAGE	61.4						
POOLED VALUES		0.924	2.62	4.26	2.148	6.07	9.89

[A] Sr = repeatability standard deviation, measurement units.
[B] r = repeatability = 2.83 × Sr, measurement units.
[C] (r) = repeatability, relative, (that is, in percent).
[D] SR = reproducibility standard deviation, measurement units.
[E] R = reproducibility = 2.83 × SR, measurement units.
[F] (R) = reproducibility, relative, (that is, in percent).

TABLE 3 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	51.4	0.646	1.83	3.56	1.56	4.41	8.59
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	61.6	0.677	1.92	3.11	2.018	5.72	9.28

[A] Sr = repeatability standard deviation, measurement units.
[B] r = repeatability = 2.83 × Sr, measurement units.
[C] (r) = repeatability, relative, (that is, in percent).
[D] SR = reproducibility standard deviation, measurement units.
[E] R = reproducibility = 2.83 × SR, measurement units.
[F] (R) = reproducibility, relative, (that is, in percent).

TABLE 4 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	42.6	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	82.3	1.01	2.86	3.47	3.54	10.0	12.2
Pooled	59.8	0.762	2.16	3.61	3.32	9.40	15.7

[A] Sr = repeatability standard deviation, measurement units.
[B] r = repeatability = 2.83 × Sr, measurement units.
[C] (r) = repeatability, relative, (that is, in percent).
[D] SR = reproducibility standard deviation, measurement units.
[E] R = reproducibility = 2.83 × SR, measurement units.
[F] (R) = reproducibility, relative, (that is, in percent).

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, date of last calibration, and calibration due date (see Note 2).

NOTE 2—The calibration interval (calibration due date) for a durometer is to be determined by the user, based upon frequency of use, severity of conditions, environmental factors, and other variables.

Periodic checking of the operation and state of durometer calibration using commercially available rubber test blocks (refer to 7.8), specifically designed for this purpose, is recommended.

An instrument that has been exposed to severe shock, is visibly damaged, produces test determinations more than 2 points different from calibrated rubber test blocks or other reference standard, or is otherwise suspected of unreliability, should be removed from service and returned to a qualified calibration facility.

A calibration interval of one year is recommended for durometer test blocks and durometer instruments that are infrequently used, more often for others.

The calibration interval for instruments and peripheral devices employed in the calibration of durometers is to be determined by the calibration service provider. It is recommended that the protocols outlined in ISO/IEC 17025, as required by the manufacturer, and those to which the service is provided, be followed.

10.2.5 Means of testing, whether manual (hand held), Type 1 operating stand (specimen to indentor), Type 2 operating stand (indentor 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/60/1 where M is the type of durometer, 60 the reading, and 1 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 D4483. 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,[4] Table 3 shows the precision results for Type A method,[5] and Table 4 gives the precision results for Type D method.[5]

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.

NOTE 3—A Type 1 precision statement for Types E, OOO, OOO-S, and R have not yet been made available.

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 tabulated 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)*, and 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

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1091.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1029.

ANEXO X

Norma de referencia usado para los ensayos de tracción de los poliuretanos elastoméricos

Designation: D412 – 16$^{\varepsilon 1}$

Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension[1]

This standard is issued under the fixed designation D412; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

ε[1] NOTE—Corrected 2.2 editorially in March 2021.

1. Scope

1.1 These test methods cover procedures used to evaluate the tensile (tension) properties of vulcanized thermoset rubbers and thermoplastic elastomers. These 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

NOTE 1—These two different methods do not produce identical results.

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 concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1349 Practice for Rubber—Standard Conditions for Testing
D1566 Terminology Relating to Rubber
D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products
D3767 Practice for Rubber—Measurement of Dimensions
D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries
E4 Practices for Force Verification of Testing Machines

2.2 *ASTM Adjunct:*

Cut Ring Specimens, Method B (D412)[3]

2.3 *ISO Standards:*

ISO 37 Rubber, Vulcanized and Thermoplastic Determination of Tensile Stress-Strain Properties[4]

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. (D1566)

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. (D1566)

3.1.4 *tensile stress*—a stress applied to stretch a test piece (specimen). (D1566)

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. (D1566)

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[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Nov. 1, 2016. Published December 2016. Originally approved in 1935. Last previous edition approved in 2015 as D412 – 15a. DOI: 10.1520/D0412-16E01.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Detailed drawings are available from ASTM Headquarters, 100 Barr Harbor Drive, Conshohocken, PA 19428. Order Adjunct No. ADJD0412-E-PDF.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

3.1.6 *thermoplastic elastomers*—a diverse family of rubber-like 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. (D1566)

3.1.9 *yield strain*—the level of strain at the yield point. (D1566)

3.1.10 *yield stress*—the level of stress at the yield point. (D1566)

4. Summary of Test Method

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 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 ± 50 mm/min (20 ± 2 in./min) for a distance of at least 750 mm (30 in.) (see Note 2). The testing machine shall have both a suitable dynamometer and an indicating or recording system for measuring the applied force within ±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 ±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 2—A rate of elongation of 1000 ± 100 mm/min (40 ± 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 at 500 ± 50 mm/min (20 ± 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°C (3.6°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 *Micrometer*—A digital or analogue dial micrometer conforming to the requirements of Practice D3767 (Procedure A). Ring specimens are addressed in 14.10.

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

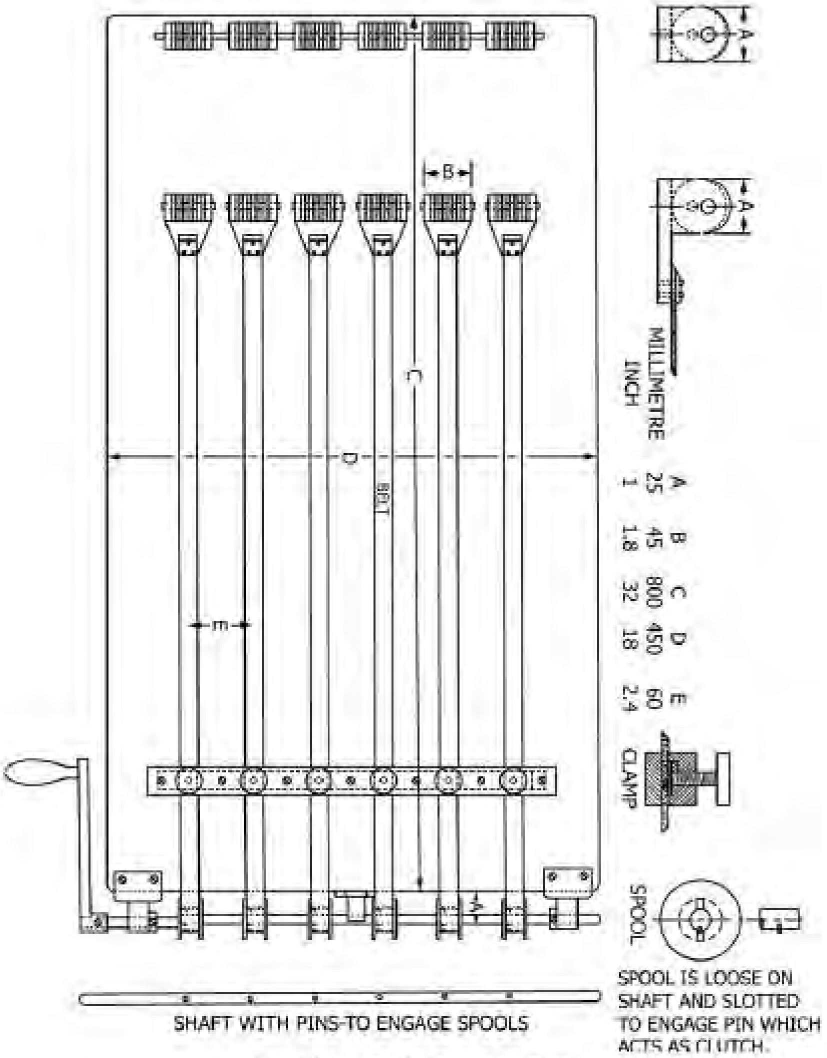


FIG. 1 Apparatus for Tensile Set Test

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. Selection of Test Specimens

7.1 Consider the following information in making selections:

7.1.1 Since anisotropy or grain directionality due to flow introduced during processing and preparation may have an influence on tensile properties, dumbbell or straight specimens should be cut so the lengthwise direction of the specimen is parallel to the grain direction when this direction is known. Ring specimens normally give an average of with and across the grain properties.

7.1.2 Unless otherwise noted, thermoplastic rubber or thermoplastic elastomer specimens, or both, are to be cut from injection molded sheets or plaques with a thickness of 3.0 ± 0.3 mm. Specimens of other thickness will not necessarily give comparable results. Specimens are to be tested in directions both parallel and perpendicular to the direction of flow in the mold. Sheet or plaque dimensions must be sufficient to do this.

7.1.3 Ring specimens enable elongations to be measured by grip separation, but the elongation across the radial width of the ring specimens is not uniform. To minimize this effect the width of the ring specimens must be small compared to the diameter.

7.1.4 Straight specimens tend to break in the grips if normal extension-to-break testing is conducted and should be used only when it is not feasible to prepare another type of specimen. For obtaining non-rupture stress-strain or material modulus properties, straight specimens are quite useful.

7.1.5 The size of specimen type used will be determined 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.

8. Calibration of the Testing Machine

8.1 Calibrate the testing machine in accordance with Procedure A of Practices E4. If the dynamometer is of the strain-gauge type, calibrate the tester at one or more forces in addition to the requirements in Sections 7 and 18 of Practices E4. Testers having pendulum dynamometers may be calibrated as follows:

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

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

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

8.1.4 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).

8.1.5 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.

8.1.6 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.

8.1.7 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 rachets 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.

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

9. Test Temperature

9.1 Unless otherwise specified, the standard temperature for testing shall be 23 ± 2°C (73.4 ± 3.6°F). Specimens shall be conditioned for at least 3 h when the test temperature is 23°C (73.4°F). If the material is affected by moisture, maintain the relative humidity at 50 ± 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 D1349.

9.2 For testing at temperatures above 23°C (73.4°F) preheat specimens for 10 ± 2 min for Method A and for 6 ± 2 min for Method B. 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.

9.3 For testing at temperatures below 23°C (73.4°F) condition the specimens at least 10 min prior to testing.

TEST METHOD A—DUMBBELL AND STRAIGHT SPECIMENS

10. Apparatus

10.1 *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 9.2).

NOTE 4—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.

10.2 *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 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 a contact extensometer is used to measure elongation, bench marks are not necessary.

10.3 *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.

10.4 *Grips*—The testing machine shall have two grips, one of which shall be connected to the dynamometer.

10.4.1 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

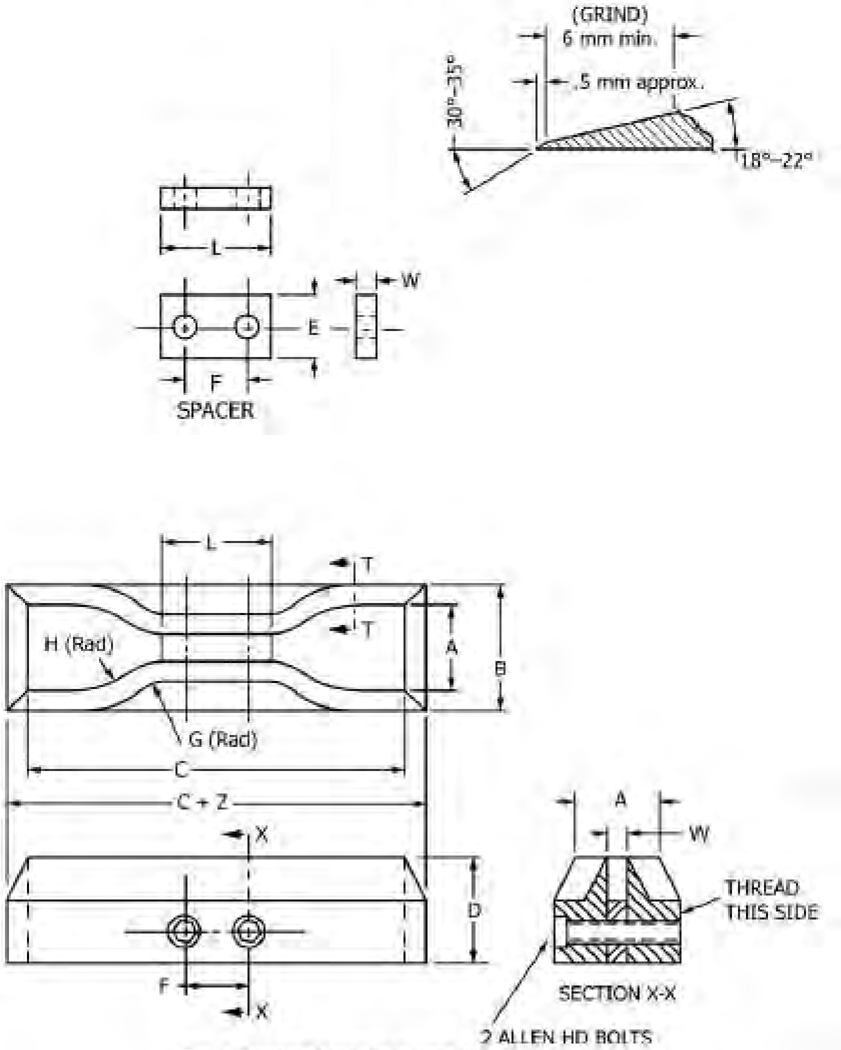


FIG. 2 Standard Dies for Cutting Dumbbell Specimens

Dimensions of Standard Dumbbell Dies[A] (Metric Units)

Dimension	Units	Tolerance	Die A	Die B	Die C	Die D	Die E	Die F
A	mm	±1	25	25	25	16	16	16
B	mm	max	40	40	40	30	30	30
C	mm	min	140	140	115	100	125	125
D	mm	±6[B]	32	32	32	32	32	32
D-E	mm	±1	13	13	13	13	13	13
F	mm	±2	38	38	19	19	38	38
G	mm	±1	14	14	14	14	14	14
H	mm	±2	25	25	25	16	16	16
L	mm	±2	59	59	33	33	59	59
W	mm	+0.05, −0.00	12	6	6	3	3	6
Z	mm	±1	13	13	13	13	13	13

[A] Dies whose dimensions are expressed in metric units are not exactly the same as dies whose dimensions are expressed in U.S. customary units. Dies dimensioned in metric units are intended for use with apparatus calibrated in metric units.

[B] For dies used in clicking machines it is preferable that this tolerance be ±0.5 mm.

FIG. 2 *(continued)*

Dimensions of Standard Dumbbell Dies[A] (U.S. Customary Units)

Dimension	Units	Tolerance	Die A	Die B	Die C	Die D	Die E	Die F
A	in.	±0.04	1	1	1	0.62	0.62	0.62
B	in.	max	1.6	1.6	1.6	1.2	1.2	1.2
C	in.	min	5.5	5.5	4.5	4	5	5
D	in.	±0.25[B]	1.25	1.25	1.25	1.25	1.25	1.25
D-E	in.	±0.04	0.5	0.5	0.5	0.5	0.5	0.5
F	in.	±0.08	1.5	1.5	0.75	0.75	1.5	1.5
G	in.	±0.04	0.56	0.56	0.56	0.56	0.56	0.56
H	in.	±0.08	1	1	1	0.63	0.63	0.63
L	in.	±0.08	2.32	2.32	1.31	1.31	2.32	2.32
W	in.	+0.002, −0.000	0.500	0.250	0.250	0.125	0.125	0.250
Z	in.	±0.04	0.5	0.5	0.5	0.5	0.5	0.5

[A] Dies whose dimensions are expressed in metric units are not exactly the same as dies whose dimensions are expressed in U.S. customary units.

[B] For dies used in clicking machines it is preferable that this tolerance by ±0.02 in.

FIG. 2 *(continued)*

recommended for inserting specimens to the same depth in the grip and for alignment with the direction of pull.

10.4.2 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.

11. Specimens

11.1 *Dumbbell Specimens*—Prepare five specimens for testing.

11.1.1 The test specimens may 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 (refer to Fig. 2 for the standard methods). Refer to 7.1 regarding thermoplastic rubber or thermoplastic elastomer specimens.

11.1.1.1 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 D3183.

11.1.2 The preferred method of preparing specimens is by compression molding. The mold shall have cavities in depth and of a configuration described in subsection 8.2.2 of Practice D3182.

11.1.2.1 The specimens may be die-cut from the molded piece using Die C (refer to Fig. 2) unless otherwise specified. Cut the specimens from the sheet with a single impact stroke (hand or machine) to ensure smooth cut surfaces.

11.1.3 All specimens shall be cut so that the lengthwise portion of the specimens is parallel to the grain unless otherwise specified. Refer to 7.1.1 regarding anisotropy or grain directionality.

11.1.4 An alternative method of preparing dumbbell specimens is to mold them directly in the form of the dies described in Fig. 2 to the depth and of a configuration described in subsection 8.2.2 of Practice D3182.

11.1.5 *Marking Dumbbell Specimens*—Dumbbell specimens shall be marked with the bench marker described in 10.2, 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.).

11.1.6 *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.

11.2 *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 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 11.1.5. 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:

$$A = M/DL \tag{1}$$

where:

A = cross-sectional area, cm^2,
M = mass, g,
D = density, g/cm^3, and
L = length, cm.

NOTE 6—A in square inches = A (cm^2) × 0.155.

12. Procedure

12.1 *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 ± 50 mm/min (20 ± 2 in./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 13 for calculations.

NOTE 7—For materials having a yield point (yield strain) under 20 % elongation when tested at 500 ± 50 mm/min (20 ± 2 in./min), the rate of elongation shall be reduced to 50 ± 5 mm/min (2.0 ± 0.2 in./min). If the material still has a yield point (strain) under 20 % elongation, the rate shall be reduced to 5 ± 0.5 mm/min (0.2 ± 0.002 in./min). The actual rate of separation shall be reported.

12.2 *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 13 for calculations.

12.3 *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 13 for calculations.

13. Calculation

13.1 Calculate the tensile stress at any specified elongation as follows:

$$T_{(xxx)} = F_{(xxx)}/A \tag{2}$$

where:

$T_{(xxx)}$ = tensile stress at (xxx) % elongation, MPa (lbf/in.^{2}),
$F_{(xxx)}$ = force at specified elongation, MN or (lbf), and
A = cross-sectional area of unstrained specimen, m^2 (in.^{2}).

13.2 Calculate the yield stress as follows:

$$Y_{(stress)} = F_{(y)}/A \tag{3}$$

where:

$Y_{(stress)}$ = yield stress, that stress level where the yield point occurs, MPa (lbf/in.^{2}),
$F_{(y)}$ = magnitude of force at the yield point, MN (lbf), and
A = cross-sectional area of unstrained specimen, m^2 (in.^{2}).

13.3 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.

13.4 Calculate the tensile strength as follows:

$$TS = F_{(BE)}/A \tag{4}$$

where:

TS = tensile strength, the stress at rupture, MPa (lbf/in.^{2}),
$F_{(BE)}$ = the force magnitude at rupture, MN (lbf), and
A = cross-sectional area of unstrained specimen, m^2 (in.^{2}).

13.5 Calculate the elongation (at any degree of extension) as follows:

$$E = 100\left[L - L_{(0)}\right]/L_{(0)} \tag{5}$$

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 $L_{(0)}$).

13.6 The breaking or ultimate elongation is evaluated when L is equal to the distance between bench marks at the point of specimen rupture.

13.7 Calculate the tensile set, by using Eq 5, where L is equal to the distance between bench marks after the 10 min retraction period.

13.8 *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.

13.8.1 *Exception 1*—If one or two of the three measured values do not meet specified requirement values when testing for compliance with specifications.

13.8.2 *Exception 2*—If referee tests are being conducted.

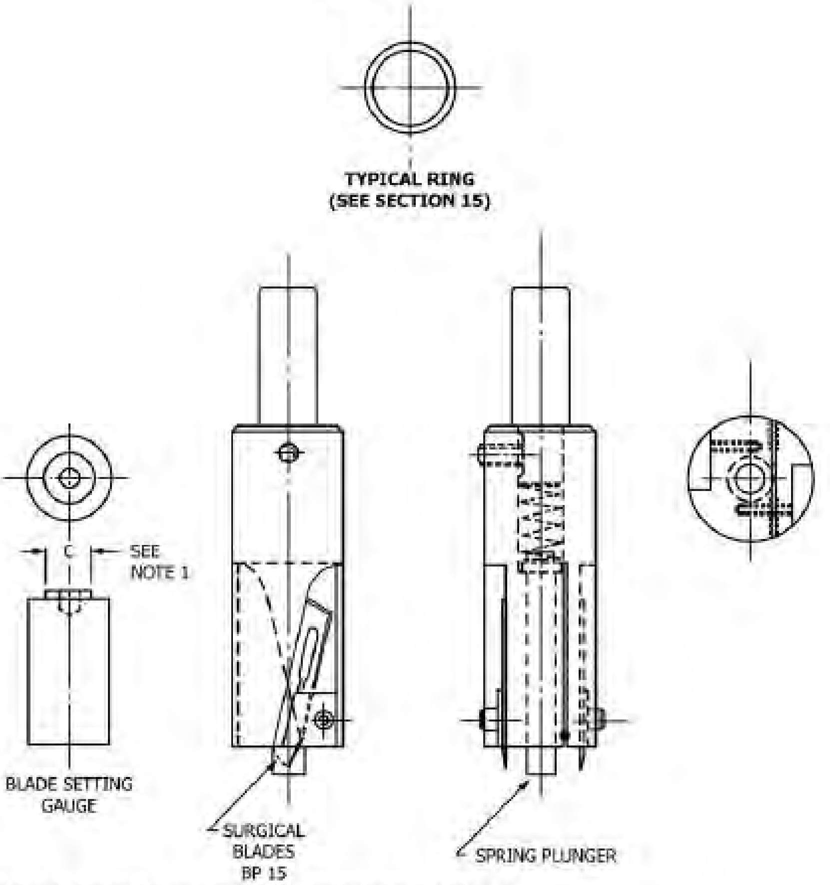
TEST METHOD B—CUT RING SPECIMENS

14. Apparatus

14.1 *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.

14.1.1 *Blade Depth Gauge*—This gauge 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.

14.2 *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. Fig. 4 illustrates the design of an apparatus for holding standard sheets (approximately 150 × 150 × 2 mm) during cutting.



NOTE 1—Dimension *C* to be 2 mm (0.08 in.) less than the inside diameter of the ring.

FIG. 3 Typical Ring Cutter Assembly

14.3 *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.

14.4 *Soap Solution*—A mild soap solution shall be used on the specimen sheet to lubricate the cutting blades.

14.5 *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.).

14.6 *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.

14.7 *Tensile Testing Machine*—A machine as specified in 6.1 shall be provided.

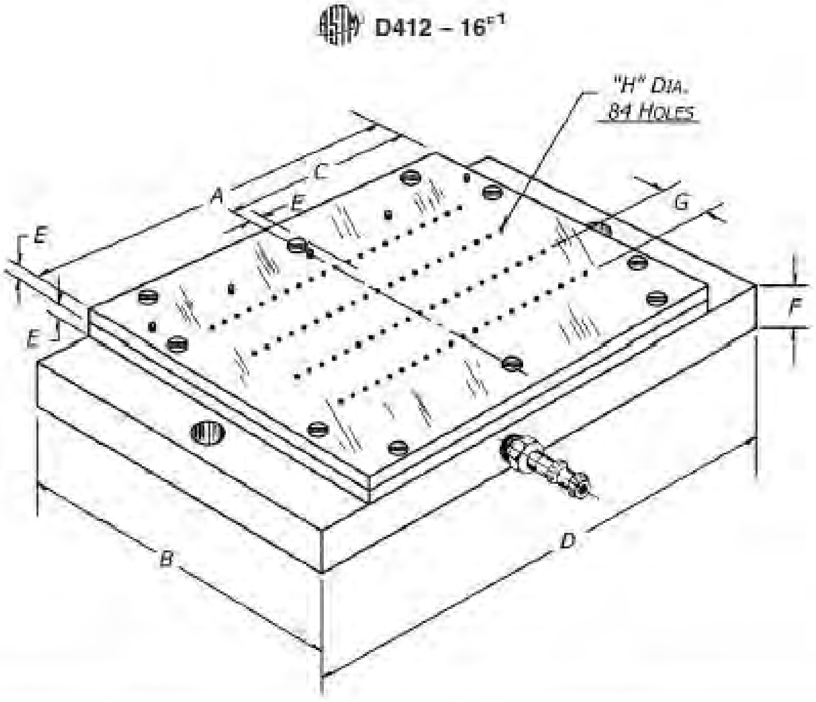
14.8 *Test Fixture*—A test fixture as shown in Fig. 5 shall be provided for testing the ring specimens. The testing machine shall be calibrated as outlined in Section 8.

14.9 *Test Chamber*—A chamber for testing at high and low temperatures shall be provided as specified in 6.2.

14.9.1 The fixtures specified in 14.8 are satisfactory for testing at other than room temperature. However at extreme temperatures, a suitable lubricant shall be used to lubricate the spindle bearings.

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

14.10 *Micrometer*—A digital or analogue dial micrometer conforming to the requirements of Practice D3767. Procedure A2 is the preferred method for measuring the dimensions of the



Dimension	mm	in.	Dimension	mm	in.
A	178	7.0	F	19	0.75
B	152	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

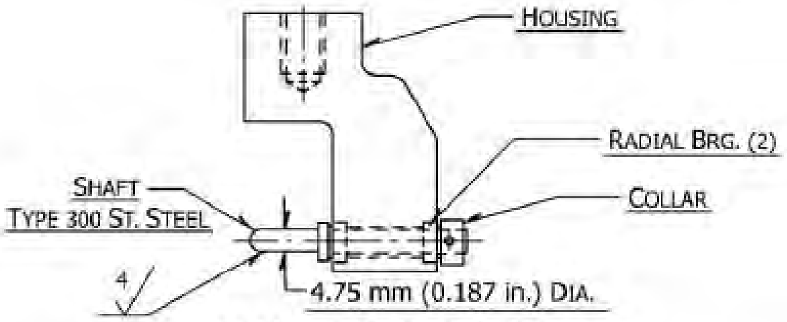


FIG. 5 Assembly, Ring Tensile Test Fixture

cut-rings. Either the dome-dome contact arrangement described in 9.2.2.1 or the dome-flat contact arrangement described in 9.2.2.3 of Practice D3767 are acceptable.

14.11 The radial width of the cut-ring is measured using a micrometer with a base that 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 ± 0.5 mm (0.61 ± 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 the dial micrometer shaft to fit the curvature of the ring(s), may be used.

15. Ring Specimen

15.1 *ASTM Cut Rings*—Two types of cut ring specimens may be used. Unless otherwise specified, the Type 1 ring specimen shall be used.

15.1.1 *Ring Dimensions:*

	mm	in.
Type 1		
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
Type 2		
Circumference mean	100.0 ± 0.2	4.0 ± 0.0004
Diameter (inside)	29.8 ± 0.06	1.19 ± 0.0001
Radial width	2.0 ± 0.02	0.08 ± 0.0008
Thickness, minimum	1.0	0.04
maximum	3.3	0.13

15.2 *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

15.3 *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.

15.4 *Preparation of Cut Ring Specimens*—Place the blades in the slots of the cutter and adjust the blade depth using the blade depth gauge. 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.

15.5 *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.

15.6 *Ring Dimension Measurements:*

15.6.1 *Circumference*—The inside circumference can be determined by a stepped cone or by "go-no go" gauges. 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 π (3.14).

15.6.2 *Radial Width*—The radial width is measured at three locations distributed around the circumference using the micrometer described in 14.11.

15.6.3 *Thickness*—For cut rings, the thickness of the disk cut from the inside of the ring is measured with a micrometer described in Practice D3767, Procedure A2, refer to 14.10.

15.6.4 *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.

16. Procedure

16.1 *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 a 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:

$$IS = \left[C_{(TS)} - C_{(SP)}\right]/2 \qquad (6)$$

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 Note 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 17 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).

16.2 *Tests at Temperatures Other than Standard*—Use the test chamber described in 6.2. 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 D1349. Place each specimen in the test chamber at intervals such that the recommendations of 9.2 are followed.

17. Calculation

17.1 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.

17.2 The following options are used to calculate stress at a specified elongation (strain) and breaking or ultimate elongation.

17.2.1 *Stress at a Specified Elongation*—The mean circumference of the ring is used for determining the elongation. The rationale for this choice is that the mean circumference best represents the average strain in each leg of the ring.

17.2.2 *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.

17.3 Calculate the tensile stress at any specified elongation by using Eq 2 in 13.1.

17.3.1 The elongation to be used to evaluate the force as specified in Eq 2 (13.1), is calculated as follows:

$$E = 200\left[L/MC_{(TS)}\right] \tag{7}$$

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.).

17.3.2 The grip separation for any specified elongation can be found by rearranging Eq 7, as given below:

$$L = E \times MC_{(TS)}/200 \tag{8}$$

17.4 Calculate the yield stress by using Eq 3 in 13.2.

17.5 Evaluate the yield strain as given in 13.3. Since yield strain may be considered to be an average bulk property of any material, use the mean circumference for this evaluation.

17.6 Calculate the tensile strength by using Eq 4 in 13.4.

17.7 Calculate the breaking or ultimate elongation as follows (see Notes 8 and 9):

$$E = 200 \times \left[L/IC_{(TS)}\right] \tag{9}$$

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.).

17.8 The inside circumference is used for both types of rings, see 15.1.1 for dimensions. Use the inside diameter to calculate the inside circumference for Type 2 rings.

NOTE 9—Eq 7, Eq 8, and Eq 9 are applicable only if the initial setting of the spindle centers is adjusted in accordance with Eq 6.

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 17.1 and 17.2), it is possible that a stress at a specified elongation, slightly less (4 to 5 %) than the ultimate elongation cannot be measured (calculated).

18. Report

18.1 Report the following information:

18.1.1 Results calculated in accordance with Section 13 or 17, whichever is applicable,

18.1.2 Type or description of test specimen and with Section 13 which type of die, either U.S. Customary Units or Metric Units, was used.

18.1.3 Date of test,

18.1.4 Rate of extension if not as specified,

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

18.1.6 Temperature of test if at other than 23 ± 2°C (73.4 ± 3.6°F) and

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

19. Precision and Bias[5]

19.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

19.2 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.

19.3 *Test Method A (Dumbbells):*

19.3.1 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.

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

19.3.3 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, gauged, 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°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."

19.3.4 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.

19.3.5 The precision of this test method may be expressed in the format of the following statements that use what is called

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1099.

ANEXO XI

Norma de referencia usado para los ensayos de resiliencia de los poliuretanos elastoméricos

Designation: D2632 – 15 (Reapproved 2019)

Standard Test Method for Rubber Property—Resilience by Vertical Rebound[1]

This standard is issued under the fixed designation D2632; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of impact resilience of solid rubber from measurement of the vertical rebound of a dropped mass.

1.2 This test method is not applicable to the testing of cellular rubbers or coated fabrics.

1.3 A standard test method for impact resilience and penetration of rubber by a rebound pendulum is described in Test Method D1054.

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

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D832 Practice for Rubber Conditioning For Low Temperature Testing
D1054 Test Method for Rubber Property—Resilience Using a Goodyear-Healey Rebound Pendulum (Withdrawn 2010)[3]
D1349 Practice for Rubber—Standard Conditions for Testing
D1566 Terminology Relating to Rubber
D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products
D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

2.2 *Other Documents:*[4]

ISO-10012-1 Quality Assurance Requirements for Measuring Equipment—Part 1: Metrological Confirmation System for Measuring Equipment[5]
ANSI/NCSL-Z540-1 American National Standard for Calibration—Calibration Laboratories and Measuring and Test Equipment—General Requirements[6]

3. Summary of Test Method

3.1 Resilience is determined as the ratio of rebound height to drop height of a metal plunger of prescribed mass and shape which is allowed to fall on the rubber specimen.

4. Significance and Use

4.1 Resilience is a function of both dynamic modulus and internal friction of a rubber. It is very sensitive to temperature changes and to depth of penetration of the plunger. Consequently, resilience values from one type of rebound instrument may not, in general, be predicted from results on another type of rebound instrument.

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Nov. 1, 2019. Published December 2019. Originally approved in 1967. Last previous edition approved in 2015 as D2632 – 15. DOI: 10.1520/D2632-15R19.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] This test method previously referenced MIL-STD-4662a *Military Standard: Calibration System Requirements*, which was subsequently canceled by the Department of Defense in February 1995. These are the DoD recommended replacement documents.

[5] Available from the International Organization for Standardization, 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland.

[6] Available from the American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

4.2 This test method is used for development and comparison of materials. It may not directly relate to end-use performance.

5. Apparatus

5.1 A diagram of the essential features and dimensions of the apparatus appears in Fig. 1. It includes means for suspending a plunger at a given height above the specimen, its release, and measuring the subsequent rebound height.

5.1.1 Each resilience instrument shall have a unique identification number assigned and permanently and visibly imprinted or affixed upon it.

5.2 The plunger dimensions are also given in Fig. 1. Its mass shall be 28 ± 0.5 g.

5.3 The height of the drop point and of the resilience scale above the base of the instrument shall be adjustable so that the drop height is always 400 ± 1 mm (16 ± 0.04 in.) above the specimen surface. The resilience scale shall be marked in 100 equally spaced divisions.

5.3.1 The top of the plunger should be in line with 100 on the scale when the plunger is locked in the elevated position. Some models of the apparatus do not meet this requirement, but may be modified to do so.

5.4 The descent of the plunger and its ensuing ascent (rebound) is guided by a vertical rod (plunger guide). In order to minimize friction between the plunger and the vertical rod, a means shall be provided for leveling the base of the instrument and adjusting the perpendicularity of the vertical rod to the instrument base.

5.4.1 The bottom of the vertical rod shall have a 4 mm diameter sharp point formed by a 60° angle, to secure the location of the bottommost end of the vertical rod. This point should indent the test specimen, providing a secure location for the free end of the guide rod.

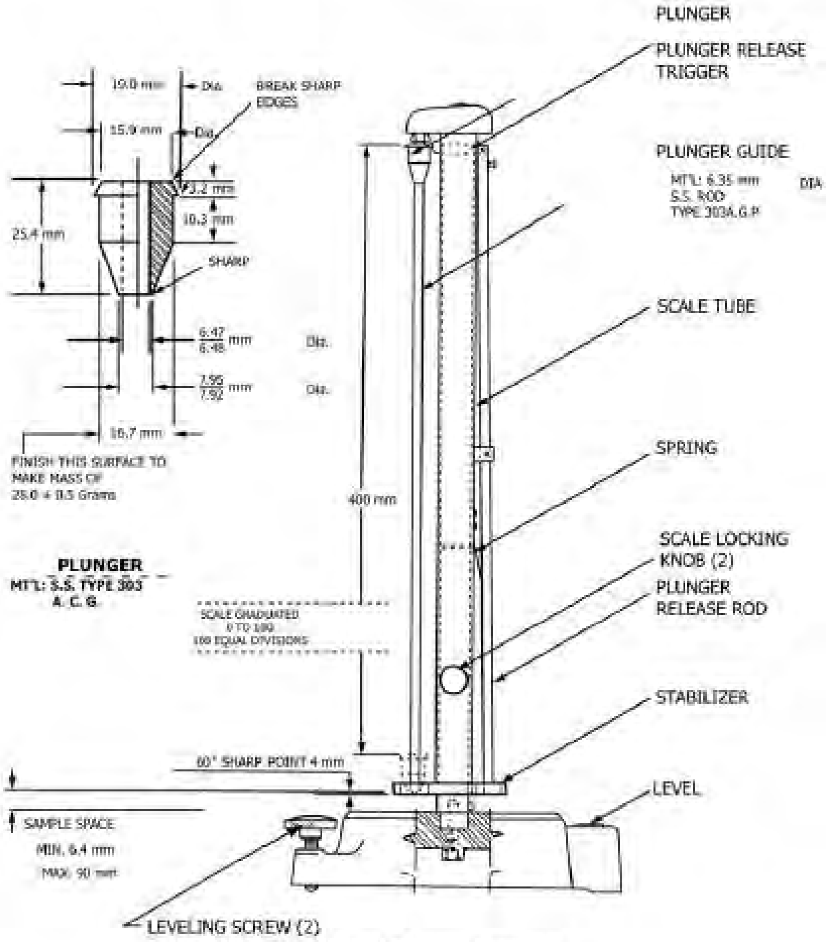

FIG. 1 Vertical Rebound Apparatus

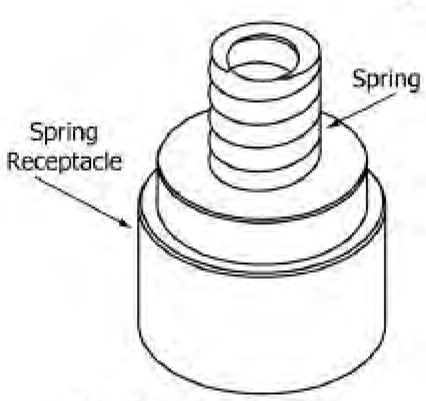

FIG. 2 Spring Calibration Device

5.4.2 The plunger shall be allowed to rest at the lowest point of travel and act as a guide to position the rod in the center of the stabilizer, as is visually practical under 10× magnification, as it is lowered onto the test specimen.

5.5 An opaque shield may be mounted between the operator and the plunger scale to be used for pass-fail test determinations. In use, the shield is adjusted so that its upper edge (or central-most graduation within a range) is even with the desired test determination. If the top of the rebounding plunger is visible above the shield (or within graduations demarcating a predetermined range of acceptability), the specimen passes.

6. Test Specimen

6.1 Mixing, sheeting, and curing shall be performed in accordance with Practices D3182 and D3183, unless otherwise specified.

6.2 The standard test specimen shall have a thickness of 12.5 ± 0.5 mm (0.50 ± 0.02 in.). The specimen shall be cut from a slab or specifically molded so that the point of plunger impact is a minimum distance of 14 mm (0.55 in.) from the edge of the specimen.

6.2.1 Any variation from the standard test specimen shall be reported (see 11.3).

6.3 Alternative specimens may be prepared by plying samples cut from a standard test slab. These samples shall be plied, without cementing, to the thickness required. Such plies shall be smooth, flat, and of uniform thickness. The results obtained with these specimens will not necessarily be identical with those obtained using a solid specimen of the same material and state of cure.

6.3.1 A thin specimen reaches a higher state of cure at a given time and temperature of cure than does a thicker specimen. Therefore, if plied specimens are used, their cure time should be appropriately lower than that of unplied specimens used for comparison.

6.4 Specimens may be prepared from finished products by cutting and buffing to the required dimensions, making sure that the opposing faces are parallel and that grain direction, where applicable, is uniform.

6.4.1 When buffing is required, it is recommended that only one side be buffed and the unbuffed side tested or, if both sides must be buffed, comparisons should not be made between buffed and unbuffed specimens.

7. Calibration

7.1 All materials, instruments, or equipment used for the determination of force, mass, or dimension in the calibration of this instrument or mechanical spring calibration device shall be traceable to the National Institute for Standards and Technology (NIST) or other internationally recognized organization parallel in nature and scope.

7.2 *Calibration Device:*

7.2.1 A mechanical spring calibration device (see Fig. 2) shall be used to calibrate this instrument.[7]

7.2.2 The force required to compress the spring 3.302 ± 0.0254 mm (0.130 ± 0.010 in.), while mounted in the receptacle, shall be 44.45 ± 0.4445 N (4532.6 ± 45.33 gf).

7.2.2.1 The spring shall be deflected 3.302 mm while mounted in the receptacle, and the force required shall be measured and reported.

7.2.2.2 A force of 44.45 N shall be applied to the spring while mounted in the receptacle, and the deflection shall be measured and reported.

7.2.2.3 The results of 7.2.2.1 and 7.2.2.2 shall be within the tolerances stated in 7.2.2 and, accordingly, shall determine the calibration of the spring.

7.2.2.4 The results achieved on a Resiliometer instrument, described in Section 5, that is in current calibration and properly used, will typically be in the range of 89 ± 2 Resiliometer points.

7.2.3 The mechanical spring calibration device shall have a unique identification number assigned and permanently and visibly imprinted or affixed upon it.

7.2.4 The resilience values assigned to an instrument using the mechanical spring calibration device shall be established at the time of calibration. These values are recorded as part of the calibration report.

7.3 *Calibration Procedure:*

7.3.1 The instrument shall be situated on a flat, level, vibration-free platform. The instrument shall be adjusted so that it is plumb and level, verified either by the integral level or by an external device designed for this purpose and in accordance with the manufacturer's instructions.

7.3.2 Perpendicularity of the vertical rod (plunger guide) to the support surface shall be verified by a device designed for this purpose and in accordance with the manufacturer's instructions.

7.3.3 The dimensions and mass of the plunger (see 5.2 and Fig. 1), the scale graduations, height of the drop point, and of the resilience scale above the base of the instrument (see 5.3 and Fig. 1) shall be verified by devices designed for this purpose.

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[7] The sole source of supply of the calibration device known to the committee at this time is CCSi, Inc., University Park, 221 Beaver Street, Akron, OH 44304. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

7.3.4 The calibration procedure shall be performed in the standard laboratory atmosphere as defined in Practice D618. The instrument, mechanical spring calibration device, and any instrument or equipment used in the calibration procedure shall equilibrate at the standard laboratory temperature for a minimum of 12 h prior to performing the calibration.

7.3.5 Situate the mechanical spring calibration device securely in the instrument as described in Section 5 and in accordance with the manufacturer's instructions.

7.3.6 Make three sets of five readings, averaging each set. Each set becomes a test determination. Average the three test determinations to the nearest whole number. This whole number becomes the resilience calibration value for the instrument.

7.3.7 The instrument shall be considered in calibration if the resilience calibration value is within ±2 points of the resilience calibration value established for the mechanical spring calibration device.

7.3.8 The resilience calibration value is assigned to the mechanical spring calibration device by the manufacturer or by the calibration service supplier using an identical resilience instrument, following the procedure outlined herein.

7.3.9 Calibration frequencies and calibration records should be kept in accordance with procedures outlined in the documents described in 2.2 or as required by the user's proprietary quality system.

7.4 *Reference Spring:*

7.4.1 If the instrument is provided with a mechanical spring device as a reference spring, this device shall have a resilience calibration value assigned, following the procedure outlined herein.

7.4.2 This device may be used to determine the state of calibration of the instrument during routine testing at a frequency determined by the user.

7.4.3 It shall not be used as a calibration device, however it should be routinely calibrated (refer to 7.2.2) to ensure the validity of its assigned values.

7.5 *Report*—The calibration report shall contain the following information:

7.5.1 Date of calibration.

7.5.2 Date of last calibration.

7.5.3 Manufacturer, type, model, and serial number of the instrument.

7.5.4 Manufacturer, type, model, and serial number of the mechanical spring calibration devices.

7.5.5 Values obtained (pre- and post-calibration results), following the procedure outlined in 7.3.

7.5.6 Ambient temperature.

7.5.7 Relative humidity.

7.5.8 Technician identification.

7.5.9 Applicable standards to which the instrument is calibrated.

7.5.10 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 7.1.

8. Test Temperature

8.1 Test procedures shall be performed in the standard laboratory atmosphere as defined in Practice D1349 unless otherwise agreed upon between customer and supplier or between laboratories.

8.2 The instrument and test specimens shall be conditioned in the standard laboratory atmosphere as described in Practice D618 unless otherwise agreed upon between customer and supplier or between laboratories.

8.3 When test procedures are conducted at temperatures or conditions other than those specified in 8.1 and 8.2, they shall be chosen from those enumerated in Practices D618, D832, or D1349 and the procedures described therein shall be followed unless otherwise agreed upon between customer and supplier or between laboratories.

9. Procedure

9.1 Level the instrument (see 7.3.1) and raise the plunger to the top of its guide rod.

9.2 Position the resilience scale (see 5.3) so that its full mass rests upon the specimen (see 5.3.1). Lock it in this position.

9.3 Release the plunger, ensuring that it slides freely on the vertical rod (plunger guide) (see 5.3.1).

9.3.1 Lateral force or impact on the guide rod may result in the hindrance of the descent of the plunger. Do not lubricate any part of the instrument. Always keep a standard test specimen under the stabilizer when not in use to avoid damage to the plunger.

9.4 Test three specimens from the same sample, making six test determinations on each specimen. Refer to Terminology D1566 for definitions of specimen and sample and to Practice D4483 for definitions of determinations and results.

9.4.1 Do not reposition the specimen once the initial test determination has been made.

9.4.2 Do not record the first three test determinations, as these condition and stabilize the specimen.

9.4.3 Record the last three test determinations.

10. Calculations

10.1 The instrument scale is divided into 100 equal parts, therefore a test determination is equal to the resilience value in percent.

10.2 Average the 4th, 5th, and 6th test determinations (see 9.4.3) from a specimen to calculate the test result from the specimen.

10.3 Average or alternatively, determine the median, the test results from the three specimens to the nearest whole number. This whole number is the resilience value of the sample. The median may also be used.

11. Report

11.1 Report the test results from the specimens (see 10.2).

11.2 Report the average or alternatively, the median, of the three test results (see 10.3).

11.3 Describe and report any variation from standard test specimen or standard conditions.

12. Precision and Bias

12.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical calculation details.

12.2 A Type 1 (interlaboratory) precision was evaluated in 1987. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. A test result is the average value, as specified by this test method, obtained on three determination(s) or measurement(s) of the property or parameter in question.

12.3 Three different materials were used in the interlaboratory program; these were tested in six laboratories on two different days.

12.4 The results of the precision calculations for repeatability and reproducibility are given in Table 1, in ascending order of material average or level, for each of the materials evaluated.

TABLE 1 Type 1 Precision

NOTE 1—
Sr = repeatability standard deviation.
r = repeatability.
(*r*) = repeatability (on relative basis, %).
S_R = reproducibility standard deviation.
R = reproducibility.
(*R*) = reproducibility (on relative basis, %).

Material	Average Test Level[A]	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	S_R	*R*	(*R*)
RA	37.9	0.48	1.36	3.58	2.65	7.50	19.8
RE	45.7	0.53	1.50	3.28	1.41	3.99	8.73
RF	48.5	0.59	1.66	3.42	1.37	3.89	8.02

[A] Resilience value, scale reading.

12.5 The precision of this test method may be expressed in the format of the following statements that use 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 the precision table closest to the mean level under consideration at any given time, for any given material in routine testing operations.

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

12.7 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the *appropriate value* tabulated in the precision table. 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.

12.8 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.

12.9 *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.

13. Keywords

13.1 impact; rebound; resilience; rubber

ANEXO XII

Registro fotográfico del trabajo de investigación

Obtención de los prepolímeros

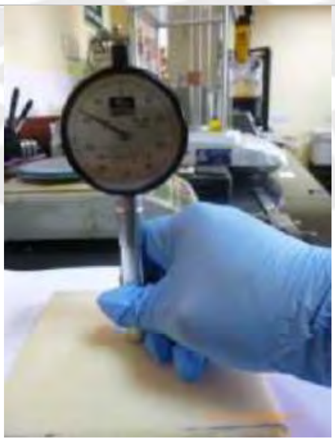
Obtención de los elastómeros

Obtención de la malla a escala piloto

Ensayos de tracción

Ensayos de erosión

Ensayos de resiliencia



Ensayos de dureza