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Mario Delgado Echeverry e Hijos Ltda.

Abogados Consultores – Marcas y Patentes

Bogotá D.C. – Colombia
Avda. Kra. 24 Park Way No. 37 – 31 Of.
202
Tels. (571) 2444096 – 3690624
Fax (571) 2442496 A.A. No. 6207
E-mail: mde@mdelaw.com
mdelawof@cablenet.co
mdeabogados@cablenet.co

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Bogotá D.C., 29 de Julio de 2013

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REFERENCIA: Expediente No. 11 –111.921
TÍTULO: “CAMISA INTERIOR DE FRIGORIFICO”
SOLICITANTE: TOTAL PETROCHEMICALS RESEARCH FELUY
TRÁMITE: Respuesta a Concepto Técnico notificado mediante Oficio No. 8.320 del 14 de mayo de 2013.

JAIME EDUARDO DELGADO VILLEGAS, en mi condición de apoderado de la sociedad **TOTAL PETROCHEMICALS RESEARCH FELUY**, dentro del término legal, presento respuesta a Concepto Técnico notificado mediante Oficio No. 8.320 del 14 de mayo de 2013.

1. DE LA SOLICITUD DE PATENTE.

Reivindicaciones

El Examinador Técnico de Patentes objeto el Capítulo Reivindicatorio porque:

“Las reivindicaciones 1, 5, 6, 7, 8, 14, 15, 16, 19 y 20 no son claras porque los términos “mayor que”, “de aproximadamente”, “sin básicamente ningún resalte”, “de baja a moderada”, “de al menos” “una cantidad moderada” y “de bajo Mw” son imprecisos. Por lo tanto, se debe sustituir estas expresiones por términos precisos o rangos concretos de valores”.

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Fax (571) 2442496 A.A. No. 6207
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“Las reivindicaciones 1, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 18, 19 y 20 no son claras por que presentan expresiones para describir la invención como “una distribución monomodal sin básicamente ningún resalte”, “una reticulación del caucho de baja a moderada expresada con un índice de hinchamiento (SI)”, “una fracción en volumen de fase de caucho (RPVF)”, “presenta una viscosidad en solución (SV) medida al 5,43% en peso de tolueno”,... que son efectos o resultados a alcanzar, lo cual no es permitido para definir las características técnicas esenciales de un procedimiento o producto, puesto que la reivindicación incluiría no sólo la solución propuesta por el solicitante, sino todas las alternativas presentes o futuras que lleguen a ese resultado. Se deben reemplazar por las características esenciales o estructurales, que contribuyen a la obtención de ese efecto”.

“Las reivindicaciones 1, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 18, 19 y 20 no son claras por que no deberían caracterizar la camisa interior del frigorífico solamente por los parámetros del polímero que lo compone, si es posible definirlo de otra manera. Se sugiere definirlo por sus características esenciales y estructurales.”.

“Las reivindicaciones 1 a 13 no son concisas, porque presentan una excesiva complejidad, lo cual dificulta determinar la extensión del objeto que se busca proteger”.

“Las reivindicaciones 14 y 15 no son concisas por que presentan la misma materia a proteger”.

Las reivindicación 14 es una reivindicación de proceso y presenta una falta presenta una falsa dependencia. Por lo anterior se tratara como una reivindicación independiente”.

La reivindicación 14 no es clara por que la expresión “para fabricar una camisa...” indica el uso que puede darse al producto reivindicado, el cual no es una característica técnica que defina a la invención. Se sugiere hacer la aclaración respectiva.”.

R/: Nuestro poderdante se permite manifestar que de acuerdo a las objeciones del Examinador Técnico de Patentes procedió a modificar el capítulo reivindicatorio, donde:

La **nueva reivindicación 1** está basada en el contenido de la reivindicación 1 original, en el que se han incluido las limitaciones definidas en las reivindicaciones 2 y 9. Además se han **incluido las características estructurales adicionales de la camisa** basadas en la primera frase citada en el apartado de antecedentes de la invención”, (*“La camisa del frigorífico se encuentra en contacto por un lado con el interior del frigorífico de la parte de los alimentos y por el otro lado con la espuma aislante”*), en concreto hoja 1, líneas 13-15.

Además, se ha **especificado el peso molecular y la identidad química del polímero monovinilaromático**, basándose en la descripción (página 14). También se ha detallado el porcentaje de partículas de caucho, así como el tipo de caucho empleado (polímeros alcadienos), basándose en las páginas 16 y 18 de la descripción. Consecuentemente, las referencias a una distribución monomodal, al índice de hinchamiento y la RPVF se han eliminado.

El **tercer componente (plastificante)** también se ha **definido** de forma más específica como **un aceite mineral o no mineral** (basándose en la página 22 de la descripción).

El **lenguaje superfluo o poco preciso** se ha eliminado.

Las reivindicaciones 2-13 se han eliminado.

La **nueva reivindicación 2** está basada en la reivindicación 14 original, donde se especifica de forma inequívoca que el procedimiento reivindicado es aquel que necesariamente resulta en la camisa de la invención, mediante un paso final de fabricación (de dicha camisa). Por eso, el último paso incluido (*“que además*

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comprende un paso para la fabricación de la camisa interior de frigorífico a partir de la composición obtenida”) se deriva de forma clara de la descripción y de la reivindicación 14 original.

Las reivindicaciones 15 y 16 se han eliminado.

La nueva reivindicación 3 está basada en la reivindicación 17 original.

Las reivindicaciones 18-20 se han eliminado.

Las modificaciones están soportadas por la solicitud originalmente presentada por lo que no suponen adición de materia.

Claridad

Como puede verse en el nuevo juego de reivindicaciones, la camisa interior del frigorífico ya no sólo se define por el material polimérico que la compone sino también por sus características estructurales, tal como requería el Examinador.

Además, la **naturaleza y cantidad específica de cada componente** (a, b y c) de la composición polimérica están ahora **definidas de forma explícita en la nueva reivindicación 1**. Al mismo tiempo, **cualquier término** que pudiera considerarse **ambiguo ha sido eliminado** por lo que la **nueva reivindicación 1 sólo hace referencia a las características técnicas esenciales de la composición polimérica**. Por tanto, la nueva reivindicación 1 es clara y concisa para cualquier experto en la materia.

Las otras **dos nuevas reivindicaciones (2 y 3)** están basadas en las reivindicaciones originales (14 y 17). Así, la **nueva reivindicación 2** se ha reescrito de forma que no queda duda de que **reivindica un proceso que lleva a**

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la obtención de una camisa bien definida, por lo que la referencia posterior queda completamente clara para un experto en la materia.

El resto de reivindicaciones que habían sido objetadas por falta de claridad se han eliminado.

Al definir cada uno de los componentes de la camisa por medio de su naturaleza química y de su porcentaje dentro de la composición, y al haber eliminado o sustituido todos aquellos términos considerados poco claros por el Examinador, consideramos que ahora la materia reivindicada reúne todos los requerimientos de claridad y define de forma precisa la materia objeto de la presente invención.

Con el nuevo juego de reivindicaciones, consideramos que quedan suficientemente claros los límites y el alcance del contenido de las reivindicaciones.

Por lo anterior, nuestro poderdante pone a consideración del Despacho un **NUEVO CAPITULO REIVINDICATORIO** el cual incluye **TRES (3) Nuevas Reivindicaciones** en dos folios, que cumplen con los requisitos de claridad exigidos por el art. 30 de la Decisión 486.

2. DEL ANÁLISIS DE PATENTABILIDAD.

Nivel Inventivo (art.18 D 486)

El Examinador objeta el nivel Inventivo de la invención para las reivindicaciones 1, 2, 3, 4, 5, 6, 7, 9, 10, 11, 12, 13 con el documento **D1 (EP1201694)**; para las

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reivindicaciones 14, 15, 17, 18, 19, 20 con el documento **D2(EP1201693)**; para las reivindicaciones 1, 9, 10, 11, 12, 13 con el documento **D3 (EP0337569)** y para las reivindicaciones 14, 15, 16 con el documento **D4 (WO2005033176)** porque:

“Los documentos D1 y D3 se consideran el estado de la técnica cercano a la invención definida en la reivindicación 1, porque divulgan el documento D1, un componente de un refrigerador, por ejemplo el revestimiento de los mismos, que comprende el polímero de estireno modificado con caucho de cualquiera (Pág. 6, reivindicación 12, líneas 25 a 30) y el documento D2, una composición de resina que permite la producción de productos moldeados por inyección (Pág. 2, líneas 5 a 10) de poliestireno modificado con caucho de alto brillo y alta resistencia al impacto (Pág. 1, resumen)”.

La diferencia entre la invención, definida en la reivindicación 1 y el componente de un refrigerador de D1 consiste en que en este último (D1) no se menciona el uso de un plastificante”.

“La diferencia entre la invención, definida en la reivindicación 1 y los productos moldeados por inyección de D3 consiste que en este último (D3) no menciona el grado de hinchamiento que deben tener las partículas de caucho o su fracción de fase de caucho (contenido de gel).

“(…)”

“En consecuencia, la persona del oficio normalmente versada en la materia, incluiría Un plastificante del documento D3 en la composición del polímero del componente para refrigerador en el documento D1 para llegar así al objeto de la reivindicación 1 de la solicitud. Por lo cual se considera obvia”.

“(…)”

“En consecuencia, la persona del oficio normalmente versada en la materia, incluiría un agente de transferencia de cadena del documento D4 en el procedimiento del polímero del documento D2 para llegar así al objeto de la reivindicaciones 14 y 15 de la solicitud. Por lo cual se consideran obvias las reivindicaciones 14 y 15.

“(…)”

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R/: Con relación a las objeciones del Examinador de Patentes con respecto a la falta de **Nivel Inventivo** de la invención en estudio, el solicitante desea resaltar lo siguiente:

Con las modificaciones realizadas, queda claro que la **nueva reivindicación 2** es una reivindicación dependiente, por tanto el análisis sobre la patentabilidad se debe enfocar únicamente en la **reivindicación 1**.

En la acción oficial, los documentos **D1 y D3** se consideran relevantes para la patentabilidad **de la reivindicación 1**. Siguiendo el formato de tabla realizada por el Examinador para comparar las características de la presente invención con las del estado de la técnica, hemos realizado el siguiente esquema:

Invención reivindicación 1	D1 (EP 1201694 A2)	D3 (EP 0337569)
Camisa interior de frigorífico	Componente de un refrigerador pero no esencialmente una camisa.	No mencionado (el moldeado o inyección no se refiere a un frigorífico o camisa)
la cual se encuentra en contacto por un lado con el interior del frigorífico de la parte de los alimentos y por el otro lado con la espuma aislante	No se menciona	No se menciona
estando dicha camisa fabricada a partir de una composición de polímero monovinilaromático modificado de caucho que comprende:	Poliestireno	Poliestireno

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una matriz de polímero monovinilaromático que tiene un peso molecular ponderado medio Mw que varía de 150.000 a 250.000 g/mol	140.000 a 280.000	No se menciona
i. del 60 al 100 % en peso de uno o más monómeros monovinilaromáticos ii. C8-12 y del 0 al 40 % en peso de uno o más monómeros seleccionados de entre el grupo que consiste en ésteres alquilo C1-4 de ácido acrílico o metacrílico y acrilonitrilo y metacrilonitrilo;	No se mencionan rangos como tales, sólo valores discretos	No se mencionan rangos como tales, sólo valores discretos
del 2 al 20 % en peso, basado en el peso total del polímero monovinilaromático modificado de caucho, de partículas de caucho dispersas en la matriz,	No se menciona (el contenido en gel no es el contenido de partículas de caucho: ver el documento adjunto “Summary Standard Test Methods, 3.1.1.)	No se menciona
donde dichas partículas están hechas de polímeros de alcadieno	Aportan polibutadieno como ejemplo	En los ejemplos se Mencionan materiales particulares
y presentan un tamaño medio de partícula en volumen de 7 a 10 µm; y	13 µm o menos, preferiblemente de 8 a 11 µm	Co-presencia de partículas con dos tamaños diferentes 0.1-0.6 µm y 0.7-1.9 µm
Plastificantes (aceite mineral o no mineral)	No se menciona	Se menciona pero sólo dentro del contexto del polimetilsiloxano (página 4, líneas 13-14).
con una proporción en peso de c/(a+b+c) que varía del 0 al 5 %	No se menciona	0.25-4%

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El objeto de la presente invención es la obtención de una composición de una camisa interior de frigorífico con propiedades mejoradas de resistencia (a la tracción, al impacto y/o al ataque químico).

La composición de la invención presenta una combinación de características muy específica en cuanto a componentes, cantidad y naturaleza química de esos componentes. Esa combinación de características, y no otra, da lugar a una serie de ventajas inesperadas, demostradas debidamente en los ejemplos de la presente invención, cumpliendo así con el objetivo perseguido.

Ninguna de las composiciones de los documentos D1 o D3 reúne o sugiere todas las características esenciales de la presente invención. Ni siquiera la combinación de las características enumeradas en cada documento daría lugar a la composición de la invención. Por tanto, **tampoco sería obvio obtener los efectos técnicos sorprendentes conseguidos con la composición de la reivindicación 1.**

En los ejemplos 3 a 5 se demuestran dichos efectos o ventajas técnicas conseguidas. En dichos ejemplos se muestran composiciones que contienen monómeros de estireno, es decir, un compuesto monovinilaromático que da lugar a una matriz polimérica monovinilaromática (componente a); Intene®50, es decir, un caucho de butadieno que forma las partículas de caucho dispersadas (componente b) y aceite mineral con una viscosidad de 70 centi-Stokes como plastificante (componente c). Como puede verse en la tabla 3, las partículas de caucho de los ejemplos 4 y 5 tienen un volumen medio de partículas en la mezcla final de 8.0-8.3 μm , es decir dentro del

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rango 7-10 µm. Estos ejemplos muestran una mejorada resistencia frente al agrietamiento por tracción, es decir una mejorada ESCR.

Por tanto, el **contenido de la presente solicitud** demuestra que la **camisa interior de frigorífico de la invención presenta mejoras respecto al estado de la técnica (ej. mayor resistencia ESCR)**. El estado de la técnica no sugiere una combinación de características similar a la de la reivindicación 1, que permita resolver el problema técnico objetivo de la presente solicitud. Consecuentemente, la materia objeto de la reivindicación 1 implica actividad inventiva.

Es más, tras estudiar el contenido de los documentos **D1 y D3** se observan incompatibilidades en al menos las siguientes características:

Característica	D1	D3
Tamaño de la partícula caucho	13 µm o menos, preferiblemente 8-11	Otros tamaños completamente diferentes: 0.1-0-6 µm/ 0.7-1,9µm
Plastificantes (aceite mineral o no mineral)	No se menciona	Requiere la presencia de polidimetilsiloxano, no mencionado en D1
Contenido en plastificante	No se menciona	0,25-4% pero asociado al polidimetilsiloxano.

Es decir, existen características en el documento **D1** que son excluyentes de las del documento **D3**, y viceversa. Por tanto **un experto en la materia no se vería motivado a combinarlos**. No obstante, aunque si se

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combinaran estos documentos arbitrariamente y sin motivo, nunca se llegaría a la combinación de características de la presente invención, y además llevaría a la combinación de enseñanzas técnicas incompatibles. El experto en la materia simplemente no está capacitado para combinar sin más documentos **D1 y D3** y mucho menos para alcanzar el material objeto de la invención. Por tanto, el **contenido de la nueva reivindicación 1, y sus dependientes 2 y 3, tienen NIVEL INVENTIVO.**

Finalmente, el solicitante pone a consideración del Examinador Técnico de Patentes: Un **NUEVO CAPÍTULO REIVINDICATORIO** el cual incluye **tres (3) Nuevas Reivindicaciones** en dos folios; el cual cumple con los requisitos de **NOVEDAD, NIVEL INVENTIVO Y APLICACIÓN INDUSTRIAL** exigidos por la Decisión 486 del Acuerdo de la Comisión de la Comunidad Andina; además, estamos dando respuesta al Concepto Técnico notificado mediante Oficio No. 8.320 del 14 de mayo de 2013; por lo tanto, solicito respetuosamente se proceda a **CONCEDER** la Patente de Invención titulada: **“CAMISA INTERIOR DE FRIGORIFICO”** a nombre de la sociedad **TOTAL PETROCHEMICALS RESEARCH FELUY**, para las nuevas Reivindicaciones 1 a 3 inclusive.

Atentamente,



JAIME EDUARDO DELGADO VILLEGAS

C.C. No. 19.270.955 Bogotá

T.P. No. 43.308 C.S.J.

Anexos:
Nuevas (3) Reivindicaciones en dos folios.
“Summary of Standard Test Methods, 3.1.1.”

NUEVAS REIVINDICACIONES

1. Una camisa interior de frigorífico, la cual se encuentra en contacto por un lado con el interior del frigorífico de la parte de los alimentos y por el otro lado con la espuma aislante, estando dicha camisa fabricada a partir de una composición de polímero monovinilaromático modificado de caucho que comprende:
 - a) una matriz de polímero monovinilaromático que tiene un peso molecular ponderado medio M_w que varía de 150.000 a 250.000 g/mol, donde el polímero monovinilaromático comprende:
 - i. del 60 al 100 % en peso de uno o más monómeros monovinilaromáticos C_{8-12} y
 - ii. del 0 al 40 % en peso de uno o más monómeros seleccionados de entre el grupo que consiste en ésteres de alquilo C_{1-4} de ácido acrílico o metacrílico y acrilonitrilo y metacrilonitrilo;
 - b) del 2 al 20 % en peso, basado en el peso total del polímero monovinilaromático modificado de caucho, de partículas de caucho dispersas en la matriz, donde dichas partículas están hechas de polímeros de alcadieno y presentan un tamaño medio de partícula en volumen de 7 a 10 μm ; y
 - c) plastificantes, los cuales son un aceite mineral o un aceite no mineral, con una proporción en peso de $c/(a+b+c)$ que varía del 0 al 5 %.
2. Procedimiento para fabricar una camisa interior de frigorífico de acuerdo a la reivindicación 1, que comprende:
 - a) formar una mezcla polimerizable que comprende al menos un monómero monovinilaromático y, de manera opcional, uno o más co-monómeros,
 - b) disolver al menos un caucho viscoso de estructura esencialmente

lineal en dicha mezcla polimerizable para formar un caucho que contiene solución polimerizable,

- c) poner en contacto un iniciador de radicales libres y un agente de transferencia de cadena con la mezcla polimerizable en condiciones en las que tenga lugar posteriormente una inversión, donde el agente de transferencia de cadena es capaz de producir un aumento de la proporción de viscosidad de caucho con respecto a viscosidad fase de PS dentro del reactor de inversión,
- d) continuar la polimerización de la solución obtenida en la etapa c), de manera opcional en presencia de un iniciador de radicales libres, hasta que se obtenga una composición formada por una matriz de polímero monovinilaromático que tenga partículas de caucho dispersas en la misma,

que además comprende un paso para la fabricación de la camisa interior de frigorífico a partir de la composición obtenida.

3. Procedimiento de acuerdo con la reivindicación 2 en el que existe una etapa adicional para desgasificar el producto de la etapa d) con el fin de separar los monómeros no polimerizados opcionales y los co-monómeros, diluyentes opcionales y recuperar el polímero monovinilaromático de alto impacto.

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Designation: D 2765 – 01

Standard Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics¹

This standard is issued under the fixed designation D 2765; 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 The gel content (insoluble fraction) produced in ethylene plastics by crosslinking can be determined by extracting with solvents such as decahydronaphthalene or xylenes. Such extraction test methods are described herein. They are applicable to crosslinked ethylene plastics of all densities, including those containing fillers, and all provide corrections for the inert fillers present in some of those compounds.

1.2 Test Method A, which permits most complete extraction in least time, is to be used for referee tests, but two alternative nonreferee Test Methods B and C are also described. The first of these differs from the referee test method only in sample preparation; that is, it requires use of shavings taken at selected points in cable insulation, for example, rather than the ground sample required by the referee test method. Because the shaved particles are larger, less total surface per sample is exposed to the extractant, so this test method ordinarily yields extraction values about 1 to 2 % lower than the referee method. The second of the alternative test methods requires that a specimen in one piece be extracted in xylenes at a constant temperature of 110°C. At this temperature and with a one-piece specimen, even less extraction occurs (from 3 to 9 % less than the referee test method) but swell ratio (a measure of the degree of crosslinking in the gel phase) can be determined.

1.3 Extraction tests can be made on articles of any shape. They have been particularly useful for electrical insulations since specimens may be selected from those portions of the insulation most susceptible to insufficient crosslinking.

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

NOTE 1—ISO 10147 is similar to this test, but is not equivalent.

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

bility of regulatory limitations prior to use. Specific precautionary statements are given in Sections 6, 10, and 25.

2. Referenced Documents

2.1 ASTM Standards:

- D 297 Test Methods for Rubber Products—Chemical Analysis²
- D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing³
- D 883 Terminology Relating to Plastics³
- D 1603 Test Method for Carbon Black in Olefin Plastics³
- D 1998 Specification for Polyethylene Upright Storage Tank³
- D 3351 Test Method for Gel Count of Plastic Film⁴
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁵

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *gel content*—the percentage by mass of polymer insoluble in a specified solvent after extraction under the specified conditions.

3.1.2 *soluble*—capable of being loosened or dissolved, susceptible of being dissolved in or as if in a fluid. (See Webster's Ninth New Collegiate Dictionary, 1988.)

3.1.3 *swell ratio*—the ratio of the gel volume in the swollen state to its volume in the unswollen state.

3.2 Terms as shown in Terminology D 883 are applicable to this test method.

4. Summary of Test Methods

4.1 Specimens of the crosslinked ethylene plastic are weighed and then immersed in the extracting solvent at the temperature specified by the procedure selected and for the time designated by that procedure. After extraction, the specimens are removed, dried, and reweighed as directed. The amount of material extracted is calculated and, if desired, swell ratio also may be determined by the means described in alternative Test Method C.

¹ These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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² Annual Book of ASTM Standards, Vol 09.01.

³ Annual Book of ASTM Standards, Vol 08.01.

⁴ Annual Book of ASTM Standards, Vol 08.02.

⁵ Annual Book of ASTM Standards, Vol 14.02.

5. Significance and Use

5.1 Many important properties of crosslinked ethylene plastics vary with the gel content. Hence, determination of the gel content provides a means of both controlling the process and rating the quality of finished products.

5.2 Extraction tests permit verification of the proper gel content of any given crosslinked ethylene plastic and they also permit comparison between different crosslinked ethylene plastics, including those containing fillers, provided that, for the latter, the following conditions are met:

5.2.1 The filler is not soluble in either decahydronaphthalene or xylenes at the extraction temperature.

5.2.2 The amount of filler present in the compound either is known or can be determined.

5.2.3 Sufficient crosslinking has been achieved to prevent migration of filler during the extraction. Usually it has been found that, at extraction levels up to 50 %, the extractant remains clear and free of filler.

5.3 Since some oxidative degradation of the material can occur at the reflux temperature of the extractants, a suitable antioxidant is added to the extractant to inhibit such degradation.

5.4 Before proceeding with this test method, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or a combination thereof, covered in the materials specification shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply.

6. Precautions

6.1 This test method measures a much larger three-dimensional polymer network than that measured by Test Method D 3351 and should not be confused with it.

6.2 It has been reported that crosslinked ultra-high molecular weight polyethylene fails to completely dissolve in this procedure at times.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases of disagreement, the tolerance shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$).

7.2 *Test Conditions*—Prepare samples in the standard laboratory atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$), unless otherwise specified in the test methods. In cases of disagreement, the tolerance shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$).

TEST METHOD A (REFEREE TEST METHOD)

8. Apparatus

8.1 The extraction apparatus shall be of the following general type, as illustrated in Fig. 1:

8.1.1 *Round-Bottom Flask*, with large-mouth ground-glass or cork joint. For one or two determinations at one time, a 500-mL flask is appropriate. For several determinations at one time, but not exceeding six, a 2000-mL flask is suitable.

8.1.2 *Heating Mantle* to fit the flask and with sufficient heating capacity to boil decahydronaphthalene (boiling point 190 to 193°C) or xylenes (boiling point 138 to 141°C).

8.1.3 *Reflux Condenser* with ground-glass or cork joint to fit into flask.

8.1.4 *Ring Stand and Appropriate Clamps*.

8.2 *Grinding Equipment*, suitable for reducing the sample to a fineness between 30 and 60 mesh. A bench-top laboratory mill⁶ is satisfactory, although any procedure which will produce a sample of the required fineness without generating excessive heat may be used.

8.2.1 *U.S. No. 30 and U.S. No. 60 Sieves*.

8.2.2 *U.S. No. 120 Stainless Steel Wire Cloth*.

8.3 *Vacuum Oven*, with vacuum source capable of creating a vacuum of at least 710 mm (28 in.) Hg and equipped with a thermometer capable of measuring 150°C .

8.4 *Analytical Balance*, capable of weighing to 0.001 g.

NOTE 2—If a slightly higher degree of accuracy is desired (about 1 to 2 %) a modified Soxhlet Extractor; Kontes Catalog No. 586100-0023, Size C, Kontes Glass Co., Vineland, NJ or equivalent may be employed wherein the specimen is held in an extraction thimble. The extraction thimble, with extra-coarse fritted glass disk sealed in; Kontes 586500-0023, 45-mm body diameter, 130-mm height or equivalent (height must subsequently be cut to 75 mm). The fritted disk is extra-coarse with fused edge; Kontes 952000-4025, 40-mm diameter or equivalent. Glass wool 13 to 19 mm thick ($\frac{1}{2}$ to $\frac{3}{4}$ in.) placed on the bottom of the extraction thimble to support one gram 30–60 mesh ground sample which is covered with 13 to 19 mm thick layer of glass wool, a fritted glass disk and a small glass weight. The extraction thimble assembly rests upon the 55-mm portion of the thimble previously cut off, the latter inserted into the modified Soxhlet.

9. Reagents

9.1 *Decahydronaphthalene*, anhydrous, boiling point 189 to 191°C .

9.2 *Xylenes*, ACS reagent grade, boiling point 138 to 141°C .

9.3 *2,2'-methylene-bis (4-methyl-6-tertiary butyl phenol)*.⁷

10. Safety Precautions

10.1 Xylenes and decahydronaphthalene are toxic and flammable solvents and as such should be handled carefully. Use only in a ventilated hood. Check the effectiveness of the hood before starting the tests. Do not inhale the vapors. Excessive inhalation of the vapors may cause dizziness or headache, or both. In the event of excessive inhalation, seek fresh clean air.

11. Test Specimens

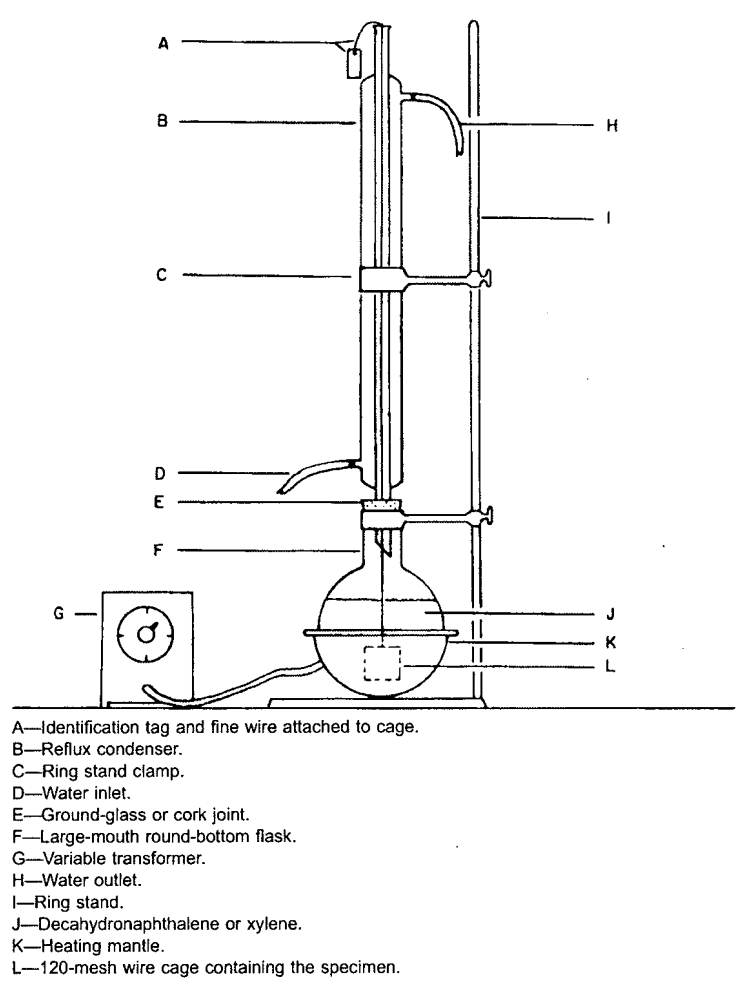
11.1 At least two specimens each containing 0.300 ± 0.015 g of ground polymer weighed to the nearest 0.001 g shall be tested.

11.2 The test samples shall be ground to a fineness that will pass through a 30-mesh sieve. Shake this sieved material over a 60-mesh sieve and reject any material that passes through.

⁶ A Wiley Cutting Mill, Intermediate Model, or equivalent, Catalog No. 08-338, available from Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219, has been found satisfactory for this purpose.

⁷ Antioxidant, available from Cytec under the trade name Cyanox 2246, has been found satisfactory for this purpose. Cytec Industries, Polymer Additive Department, 5 Garret Mountain Plaza, West Paterson, NJ 07424.

ASTM D 2765



A—Identification tag and fine wire attached to cage.
 B—Reflux condenser.
 C—Ring stand clamp.
 D—Water inlet.
 E—Ground-glass or cork joint.
 F—Large-mouth round-bottom flask.
 G—Variable transformer.
 H—Water outlet.
 I—Ring stand.
 J—Decahydronaphthalene or xylene.
 K—Heating mantle.
 L—120-mesh wire cage containing the specimen.

FIG. 1 Extraction Apparatus

11.3 Test specimens originating from insulation cut from wire should be homogeneous and consist of an equal mixture of samples representative of insulation nearest the conductor and samples representative of insulation nearest the outer periphery of the construction unless specific portions of the insulation are being examined for insufficient crosslinking.

12. Procedure

12.1 Prepare a specimen holder by cutting a piece of 120-mesh stainless steel cloth measuring approximately 80 by 40 mm (3 by 1½ in.). Fold this to form a square measuring approximately 40 mm (1½ in.). Fold two sides of this square closed by folding the cloth at the edges about 6 to 7 mm (¼ in.) and stapling those folds. In that manner, a pouch open at the top is obtained. Weigh this pouch (*W1*).

12.2 Place approximately 0.3 g of the ground and screened sample in the weighed pouch. Weigh the pouch and specimen (*W2*). Fold over the open side of the pouch and staple the edge to form a cage. Weigh the cage and sample (*W3*).

12.3 Fill a round-bottom flask as described in 8.1.1 with enough solvent to immerse the 120-mesh cage and sample completely. Three hundred fifty grams in a 500-mL flask or 1000 g in a 2000-mL flask is sufficient. Dissolve 1 % of

antioxidant in the decahydronaphthalene or xylenes to inhibit further crosslinking of the specimen.

12.4 Boil the decahydronaphthalene or xylenes vigorously enough to ensure good agitation of the solution. Twenty to 40 drops/min from the condenser, while using 1000 g of solvent, is a satisfactory boiling rate. Suspend the cage and specimen in the solvent so that the bottom edge of the cage is almost touching the bottom of the flask. A small wire, attached to the cage and extending through the reflux condenser can be used for suspending the cage. Extract the specimen for 6 h in decahydronaphthalene or for 12 h in xylenes.

NOTE 3—Since most of the extraction occurs within less than the specified time, the period of extraction may be shortened for control tests on the basis of experience. If this is done, the time of extraction must be reported. For referee tests, extraction time is to be as specified by this test method.

12.5 After extraction, place the cage and specimen immediately in a vacuum oven preheated to 150°C. Dry the specimen to constant weight under at least 710 mm (28 in.) Hg vacuum. Cool and weigh (*W4*). If the compound absorbs moisture, cool the specimen in a desiccator before weighing.

NOTE 4—It has been reported that drying time sometimes can be

materially shortened if the cage and sample are cooled for 15 min and then are placed either on a suspended screen or on lint-free absorbent material to remove excess solvent before being put into the vacuum drying oven.

12.6 If extraction tests are regularly made, the apparatus may be left assembled and the extracting solvent (containing inhibitor as directed) may be reused until it darkens. However, if there is any doubt about the values obtained with reused extractant, the test should be repeated with fresh extractant.

NOTE 5—If the modified Soxhlet apparatus is used, the complete thimble assembly containing 1 g of sample is weighed to the nearest ± 0.0001 g before extraction and after vacuum drying.

13. Calculation

13.1 Calculate the solvent extraction in percent as follows:

$$\begin{aligned} \text{Extract, \%} &= (\text{weight lost during extraction}) / \\ &(\text{weight of original specimen} - \text{weight of filler}) \quad (1) \\ &= [(W_3 - W_4) / ((W_2 - W_1) - F(W_2 - W_1))] \times 100 \\ &= [(W_3 - W_4) / ((1 - F)(W_2 - W_1))] \times 100 \end{aligned}$$

where:

- W_1 = weight of the pouch (sealed on three sides, one side open),
- W_2 = weight of the specimen and the pouch (sealed on three sides, one side open),
- W_3 = weight of the specimen and the cage, after being stapled shut,
- W_4 = weight of the specimen and the cage after extraction and drying,
- F = fraction of filler (which must be insoluble in decahydronaphthalene or xylenes) in the polyethylene compound, and
- gel content = 100 - percent extract.

NOTE 6—If the fraction of filler in the material is not known, the test methods described in Test Method D 1603 or in Test Methods D 297, may be used to determine filler content.

14. Report

14.1 Report the following information:

- 14.1.1 Complete identification of the compound,
- 14.1.2 Density of the polyethylene in the compound (usually as identified by its supplier),
- 14.1.3 Weight percent of polyethylene in the compound (usually as identified by its supplier, although it may be determined by the test methods identified in Note 6),
- 14.1.4 Percent extract; the value for each specimen and the average for the sample,
- 14.1.5 Solvent used, time of extraction, and any pertinent sample preparation, and
- 14.1.6 Any unusual or abnormal behavior observed during the test.

TEST METHOD B (NONREFEREE TEST METHOD)

15. Scope

15.1 This modification of the referee test method was developed particularly for wire and cable insulations and differs from the referee test method only in specimen preparation.

15.2 In using this alternative test method, it should be recognized that the values obtained may be lower than those obtained by referee Test Method A for the reason given in 1.2.

16. Test Specimens

16.1 Using a wood plane, shave a strip approximately 0.4 mm (0.015 in.) thick parallel to the axis of the insulated conductor. The test strip used for extraction should be taken next to the conductor only when strand shielding tapes or compounds are not employed. In high-voltage cable employing strand shielding tapes or compounds, the strip should be shaved as closely as possible along the shielded surface, being careful not to include any of the semiconducting compound (see Fig. 2).

16.2 A section approximately 6 mm (1/4 in.) wide, depending on cable size, is cut longitudinally through the center portion of the strip obtained as described in 15.1. The outer sections of this strip are discarded, thus leaving only a sample of the material closest to the conductor or shielded surface. This sample then is cut or diced into pieces measuring approximately 6 by 6 mm (1/4 by 1/4 in.) for extraction.

16.3 The dimensions given in 16.2 are for cable sizes 1/0 AWG and larger. For Size 2 AWG and smaller, the dimensions should be proportionately less.

17. Procedure

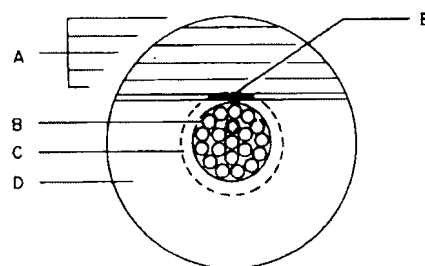
17.1 Place approximately 0.3 g of the diced sample, prepared as described in Section 16, in a previously weighed pouch (W_1) and then reweigh (W_2). Close the pouch by folding over the open side and stapling it to form a cage. Weigh this cage and the sample again (W_3) (see Section 12 of Test Method A).

17.2 Conduct the extraction as described in Section 12, using decahydronaphthalene as the extractant.

17.3 After extraction, dry the specimen and reweigh also as described in Section 12.

18. Calculation

18.1 Calculate solvent extraction in percent as described in Section 13 of the referee test method.



- A—Shaved strips to be discarded.
- B—Semiconducting strand shield.
- C—Area most susceptible to insufficient crosslinking.
- D—Crosslinked polyethylene insulation.
- E—Specimen for solvent extraction (shaded area).

FIG. 2 Enlarged Cross-Sectional View of High-Voltage Cable Showing Sample Location

19. Report

19.1 See Section 14 but include identification of the alternative test method used (Test Method B).

TEST METHOD C (NONREFEREE TEST METHOD)

20. Scope

20.1 Degree of crosslinking is determined by measuring the swell and extraction occurring in a solvent that attaches that portion of the polymer which is not crosslinked. Both measurements are obtained in one test. The degree of crosslinking is not expressed as a percent of total crosslinkability or similar expression, but is judged from swell ratio and percent extract based on experience with the particular polymer-solvent system under consideration.

20.2 This test method is applicable to crosslinked ethylene plastics of any density and including those containing fillers, provided that the fillers are insoluble in the extractant.

20.3 In using this alternative test method, it should be recognized that the extraction values obtained will be lower than those obtained by either Test Method A or Test Method B for the reasons given in 1.2. However, this test method permits swell ratio to be determined.

21. Summary of Test Method

21.1 Specimens of the crosslinked polymer are weighed, immersed in hot xylene for 24 h, removed, weighed in the swollen state, and dried and reweighed. The swell ratio and percent extract are calculated from these weight measurements.

22. Significance and Use

22.1 This test method provides useful measurements when the temperature of the solvent in which the polymer is immersed is above the crystalline melting point of the polymer and below the temperature at which chemical degradation begins to occur. Xylene, which thermodynamically is a good solvent for polyethylene, depresses the melting point of polyethylene (in accordance with Flory's theory of diluent forces) about 30°C. In the presence of xylene, polyethylene in the density range from 0.910 to 0.925 g/cm³ (low density) has a melting point less than 80°C, and polyethylene in the density range from 0.941 to 0.965 g/cm³ (high density) has a melting point less than 110°C. Thus, tests on both types of polyethylene can be made at 110°C.

22.2 The percent extract is a measure of the amount of polymer that is soluble or is not entrapped in the main gel phase, or both, at the end of the immersion period. Provided that no chemical degradation occurs, the greater the degree of crosslinking the lower the amount of extract (lower percent extract).

22.3 Swell ratio is a relative measure of crosslinking in the main gel phase. Provided that no chemical degradation occurs, the lower the swell ratio the greater the crosslinking and the lower the molecular weight between crosslinks.

23. Apparatus

23.1 *Oil Bath* at 110°C, agitated to ensure temperature uniformity.

23.2 *Wide-Mouth Glass Jars*, 8-oz, with screw caps.

23.3 *Analytical Balance*, accurate to 0.001 g.

23.4 *Forceps*, long, 250-mm (10-in.)

23.5 *Cutting Devices* to cut specimens from sample.

23.6 *Vacuum Oven* at 100°C, with pump and cold trap.

23.7 *Flasks*, 100-mL, 24/40 joint.

23.8 *Desiccator*, containing drying agent.

23.9 *Weighing Bottles*, 30-mL and 60-mL.

23.10 *Pipet*, 100 mL, or *Bottle Top Dispenser* capable of dispensing 100 mL of liquid.

24. Reagents and Materials

24.1 *Xylenes*, ACS reagent grade, boiling point 138 to 141°C.

24.2 *Oil*, stable at 110°C.

24.3 *Acetone*, technical grade.

24.4 *Dry Ice*.

25. Safety Precautions

25.1 Xylenes are toxic and flammable and as such should be handled carefully. Use only in a ventilated hood. Check the effectiveness of the hood before starting the tests. Do not inhale the vapors. Excessive inhalation of the vapors may cause dizziness or headache, or both. In case of excessive inhalation, seek fresh clean air.

26. Test Specimens

26.1 At least two specimens each containing 0.500 ± 0.020 g of polymer shall be weighed to the nearest 0.001 g.

26.2 The test specimens shall be cut with sharp tools from the sample and all edges shall be smooth and clean. The surface-to-volume ratio shall be kept as small as reasonably possible.

26.3 Test specimens consisting of insulation cut from wire shall be slit longitudinally. If necessary, they may be cut into two or three pieces crosswise so that they can be completely submerged in the solvent.

27. Procedure

27.1 Place the weighed test specimen in an 8-oz jar; add 100 ± 0.1 mL of xylenes with the automatic pipet; make sure the test specimen is completely immersed in the solvent (Note 7); screw the cap on the jar finger tight.

NOTE 7—The solvent must cover the *swollen* test specimen at the conclusion of the immersion period to obtain reliable results. Tests shall be discarded and repeated with a fresh test specimen if specimens are not covered with solvent at the conclusion of the extraction step.

27.2 Place the jar in the oil bath so that the level of the oil is at or slightly above the level of the solvent in the jar. The temperature of the oil bath must be 110°C and the bath must be at that temperature within $\pm 0.5^\circ\text{C}$ no less than $\frac{1}{2}$ h after the jar is placed therein. If necessary, place a weight on the jar or clamp it to hold it in place.

27.3 Maintain the oil bath at the proper temperature within $\pm 0.5^\circ\text{C}$ for 24 h, with agitation.

NOTE 8—The immersion period specified seems to be adequate to reach an approximate equilibrium state for swell measurements. Increasing this period to 72 h had no significant effects on the results. However, a longer immersion period does slightly increase the values for percent extract.

27.4 Remove the jar after 24 h in the hot oil bath and transfer the swollen test specimen with the forceps without delay to a clean, dry tared weighing bottle (Note 9). Handle the swollen test specimen gently and carefully. Blow the surfaces (Note 10). Stopper the weighing bottle.

NOTE 9—The size of the swollen test specimen dictates the size of the weighing bottle to be used.

NOTE 10—Extreme care must be used in handling the swollen test specimen to obtain reliable results. The test specimen should not be squeezed with the forceps. The transfer of the test specimen should be made quickly. The surfaces should not be subjected to excess blowing (too long or more than once on each side); otherwise more than the surface xylene will be removed.

Test specimens that break up during the immersion period (usually very low degrees of crosslinking) can be caught on a 60-mesh or finer stainless steel screen, transferred to a weighing bottle, and treated in the specified manner. Results obtained from such specimens are of limited value. If the system is unfilled, the swell ratio will be in error because it is impossible to eliminate the surface xylene, but the percent extract will be correct if all the gel particles were collected. If the system is filled, both the swell ratio and the percent extract will be erroneous because of loss of filler.

27.5 Place the weighing bottle in the desiccator, allow to cool to room temperature, and weigh to the nearest 0.001 g.

27.6 Place the opened weighing bottle with the swollen test specimen in the vacuum oven at 100°C. Apply vacuum slowly. Cool cold trap with dry ice and acetone to catch and condense xylene vapor. Heat in vacuum until xylene is all removed, as determined by repeated weighing. Formulations containing low-density polyethylene usually require 24 h and those containing high-density polyethylene require 16 h to reach weight equilibrium.

28. Calculation (See Note 11 for Derivation)

28.1 Calculate the swell ratio as follows:

$$\text{Swell ratio} = [(W_g - W_d)/(W_o - W_e)] K + 1 \quad (2)$$

28.2 Calculate the percent extract as follows:

$$\text{Extract, \%} = [(W_s - W_d)/W_o] \times 100 \quad (3)$$

where:

- f = polymer factor (the ratio of the weight of the polymer in the formulation to the total weight of the formulation)
- W_o = original polymer weight (the amount of polymer in the specimen being tested)
- W_s = weight of specimen being tested
- W_e = fW_s
- W_e = weight of extract (amount of polymer extracted from the specimen in the test)
- W_g = weight of swollen gel after the immersion period
- W_d = weight of dried gel
- W_e = $W_s - W_d$, weight of xylene in swollen gel
- W_p = weight of insoluble polymer in swollen gel
- W_p = $W_o - W_e$
- D_p = density of polymer at the immersion temperature
- V_p = volume of polymer in gel
- V_p = $(W_o - W_e)/D_p = W_p/D_p$
- W_x = weight of solvent in gel
- W_x = $W_g - W_d$
- D_x = density of solvent at the immersion temperature
- V_x = volume of solvent in gel

$$V_x = (W_g - W_d)/D_x = W_x/D_x$$

K = ratio of density of polymer to that of the solvent at the immersion temperature. This ratio is approximately 1.07 for low-density polyethylene at 80°C and 1.17 for high-density polyethylene at 110°C

28.3 High swell ratios indicate a low degree of crosslinking, that is, a high molecular weight between crosslinks. Low swell ratios indicate a more tightly bound structure. Low values of percent extract indicate a high degree of crosslinking.

NOTE 11—Derivation of calculations:

$$\begin{aligned} \text{Swell ratio} &= (\text{volume of polymer in gel} + \text{volume of absorbed xylene})/(\text{volume of polymer in gel}) \\ &= (V_p + V_x)/V_p = [(W_p/D_p) + (W_x/D_x)]/(W_p/D_p) \\ &= 1 + (D_p/W_p)(W_x/D_x) \\ &= 1 + (D_p/D_x)(W_x/W_p) = 1 + K(W_x/W_p) \\ &= 1 + K[(W_g - W_d)/(W_o - W_e)] \\ &= 1 + K[(W_g - W_d)/(W_o - (1 - f)W_o)] \\ \text{Extract, \%} &= (\text{weight of extract/original polymer weight}) \times 100 \\ &= (W_e/W_o) \times 100 = [(W_s - W_d)/W_o] \times 100 \\ &= [(W_s - W_d)/fW_o] \times 100 \end{aligned}$$

29. Report

29.1 Report the following information:

29.1.1 Complete identification of the compound,

29.1.2 Density of the polyethylene polymer in the compound (usually as identified by its supplier),

29.1.3 Weight percent of polyethylene in the compound (usually as identified by its supplier, although it may be determined by the methods identified in Note 6),

29.1.4 Swell ratio; the value for each specimen and the average for the sample,

29.1.5 Percent extract; the value for each specimen and the average for the sample,

29.1.6 Identification of the method used, that is, Method C,

29.1.7 Any deviations from this test method and the reasons for such deviations, and

29.1.8 Any unusual or abnormal behavior observed during the test.

30. Precision and Bias ⁸

30.1 *Test Method A*—Table 1 is based on a round robin conducted in 1967 involving six materials tested by three laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the result of one individual determination. Each laboratory obtained three test results for each material.

30.2 *Test Method B*—Table 1 is based on a round robin conducted in 1967 involving six materials tested by one laboratory. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratory that tested them. Each test result was the result of one individual determination. Each laboratory obtained three test results for each material.

30.3 *Test Method C*—Table 1 is based on a round robin conducted in 1967 involving six materials tested by three laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at

⁸ Supporting data are available from ASTM Headquarters. Request RR:D20-45.

TABLE 1 Percent Extract and Swell Ratio

NOTE 1— S_r = within-laboratory standard deviation of the average.
 SR = between-laboratories standard deviation of the average.
 $I_r = 2.80 S_r$
 $IR = 2.80 SR$

Test Method A (Percent Extract)					
Material	Average	S_r	SR	I_r	IR
1	19.57	0.635	1.57	1.78	4.40
2	20.57	0.743	1.38	2.08	3.86
3	18.59	0.529	1.60	1.48	4.48
4	18.97	1.89	1.56	5.29	4.37
5	10.71	0.687	2.37	1.92	6.64
6	14.09	1.35	1.34	3.78	3.75

Test Method B (Percent Extract)					
Material	Average	S_r	SR	I_r	IR
1	18.00	0.200	0.00	0.560	0.00
2	17.97	0.057	0.00	0.160	0.00
3	17.30	0.100	0.00	0.280	0.00
4	15.73	0.305	0.00	0.854	0.00
5	10.53	0.379	0.00	1.06	0.00
6	12.97	0.251	0.00	0.703	0.00

Test Method C (Percent Extract)					
Material	Average	S_r	SR	I_r	IR
1	14.63	1.04	0.741	2.91	02.07
2	16.51	1.26	0.205	3.53	0.570
3	14.17	0.160	0.180	0.448	0.500
4	11.74	0.402	1.01	1.12	2.83
5	7.044	0.734	1.39	2.06	3.89
6	10.05	0.786	1.86	2.20	5.21

Test Method C (Swell Ratio)					
Material	Average	S_r	SR	I_r	IR
1	6.500	0.187	2.54	0.524	7.11
2	7.583	0.216	2.71	0.605	7.59
3	5.917	0.191	2.43	0.535	6.80
4	6.367	0.258	2.64	0.722	7.39
5	4.417	0.153	1.20	0.428	3.36
6	3.833	0.082	0.894	0.230	2.50

the laboratory that tested them. Each test result was the result

of one individual determination. Each laboratory obtained three test results for each material.

NOTE 12—**Caution:** The following explanations of r and R (30.4-30.4.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The test data in Table 1 should not be rigorously applied to acceptance or rejection of these materials, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 30.4-30.4.3 would then be valid for such data.

30.4 *Concept of r and R* —If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages (medians\other functions) from testing two specimens:

30.4.1 *Repeatability, r* —(Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day) the two test results should be judged not equivalent if they differ by more than the r value for that material.

30.4.2 *Reproducibility, R* —(Comparing two test results for the same material, obtained by different operators using different equipment on different days) the two test results should be judged not equivalent if they differ by more than the R value for that material.

30.4.3 Any judgment in accordance with 30.4.1 or 30.4.2 would have an approximate 95 % (0.95) probability of being correct.

30.5 There are no recognized standards by which to estimate bias of this test method.

31. Keywords

31.1 crosslinked ethylene plastics; gel content; insoluble fraction; swell ratio.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this test method since the last issue that may impact the use of this procedure.

D 2765 – 95:

- (1) ISO statement was changed.
- (2) Changed xylene to xylenes where appropriate.
- (3) Updated equipment catalog numbers in Note 2.
- (4) Removed humidity requirement as per D 4976.

(5) Corrected an error in the Precision and Bias statement as per Research Report.

(6) General editorial changes were made throughout the standard.

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