



## Standard Specification for Physical and Chemical Properties of Industry Reference Materials (IRM)<sup>1</sup>

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### 1. Scope

1.1 This specification covers the chemical and physical quality specifications or requirements, or both, for Industry Reference Materials (IRMs) as cited in Practice D 4678 and other standards.

1.2 IRMs, as evaluated and referenced in Practice D 4678, are vitally important to conduct product, specification, and development testing in the rubber and carbon black industries.

1.3 Before a new lot of material can be accepted as an IRM, it must comply with the specifications prescribed in this specification. However, these specifications are only part of the requirements. Other requirements as given in Practice D 4678 shall be met before a candidate material can be formally accepted as an IRM.

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

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 88 Test Method for Saybolt Viscosity
- D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- D 97 Test Method for Pour Point of Petroleum Products
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
- D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
- D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
- D 1278 Test Methods for Rubber from Natural Sources—Chemical Analysis

- D 1416 Test Methods for Rubber From Synthetic Sources—Chemical Analysis<sup>2</sup>
- D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
- D 1519 Test Method for Rubber Chemicals—Melting Range
- D 1646 Test Method for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)
- D 1747 Test Method for Refractive Index of Viscous Materials
- D 1951 Test Method for Ash in Drying Oils and Fatty Acids
- D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids
- D 1960 Test Method for Loss on Heating of Drying Oils
- D 1965 Test Method for Unsaponifiable Matter in Drying Oils, Fatty Acids, and Polymerized Fatty Acids
- D 1980 Test Method for Acid Value of Fatty Acids and Polymerized Fatty Acids
- D 1982 Test Method for Titer of Fatty Acids
- D 1993 Test Method for Precipitated Silica—Surface Area by Multipoint BET Nitrogen Adsorption
- D 2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method
- D 2140 Test Method for Carbon-Type Composition of Insulating Oils of Petroleum Origin
- D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity
- D 2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils
- D 3037 Test Methods for Carbon Black—Surface Area by Nitrogen Adsorption<sup>2</sup>
- D 3157 Test Method for Rubber from Natural Sources—Color
- D 3194 Test Method for Rubber from Natural Sources—Plasticity Retention Index (PRI)
- D 3280 Test Methods for Analysis of White Zinc Pigments
- D 4004 Test Methods for Rubber—Determination of Metal

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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<sup>2</sup> Withdrawn.

Content by Flame Atomic Absorption (AAS) Analysis  
D 4075 Test Method for Compounding Materials—Flame Atomic Absorption Technique—Determination of Metals  
D 4315 Test Methods for Rubber Compounding Material—Zinc Oxide  
D 4569 Test Method for Determination of Acidity in Sulfur  
D 4570 Test Method for Rubber Chemicals—Determination of Particle Size of Sulfur by Sieving (Dry)  
D 4571 Test Methods for Rubber Chemicals—Determination of Volatile Material  
D 4572 Test Method for Rubber Chemicals—Wet Sieve Analysis of Sulfur  
D 4574 Test Methods for Rubber Chemicals—Determination of Ash Content  
D 4578 Test Methods for Rubber Chemicals—Determination of Percent Insoluble Sulfur by Solvent Extraction  
D 4678 Practice for Rubber—Preparation, Testing, Acceptance, Documentation, and Use of Reference Materials  
D 4934 Test Method for Rubber Compounding Materials: 2-Benzothiazyl Sulfenamide Accelerators—Insolubles  
D 4936 Test Method for Mercaptobenzothiazole Sulfenamide Assay by Reduction/Titration  
D 5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters  
D 5712 Test Method for Analysis of Aqueous Extractable Protein in Natural Rubber and its Products Using the Modified Lowry Method  
D 6499 Test Method for Immunological Measurement of Antigenic Protein in Natural Rubber and its Products

### 3. Significance and Use

3.1 IRMs are vitally important in product and specification testing, in research and development work, in technical service work, and in quality control operations in the rubber and carbon black industries. They are especially valuable for referee purposes. Many ASTM rubber standards for the evaluation of natural or synthetic rubber require the use of specific IRMs in their test recipes for better laboratory repeatability and reproducibility.

3.2 New material lots that have been selected as candidates for IRM approval shall conform to the appropriate specifications given in this standard and meet requirements given in Practice D 4678 before the lots may be accepted as IRMs.

3.3 The chemical and physical IRM specifications shown will ensure some consistency in IRM properties from one lot to the next. However, the specifications cannot ensure exact inter-lot consistency.

### 4. Specifications

4.1 The following are specifications for Industry Reference Materials (IRMs).

4.2 *Specification for IRM 1—Tetramethyl Thiuram Disulfide (TMTD)*.<sup>3</sup>

**TABLE 1 Specification for IRM 1—Tetramethyl Thiuram Disulfide (TMTD)**

Property	ASTM Designation	Limits
Melting point, °C	D 1519	142 min
Ash, %	D 4574	0.10 max
Loss on heating at 105°C, % loss	D 4571	0.5 max
Wet sieve analysis, % retaining on 100 mesh screen	D 4572	0.05 max

4.2.1 Material description: Appearance is light buff to white powder. Specific gravity is 1.4. This material is commonly used in certain ASTM rubber test recipes as an accelerator in sulfur vulcanization.

4.2.2 Specifications are given in Table 1Table 6.

4.3 *Specification for IRM 2—Benzothiazyl Disulfide (MBTS)*.<sup>4</sup>

4.3.1 Material description: Appearance is cream-colored powder. Specific gravity is 1.5. This material shall contain  $2 \pm 0.2$  % mineral oil. This material is commonly used as an accelerator in certain ASTM rubber test recipes in sulfur vulcanization.

4.3.2 Specifications are given in Table 2.

4.4 *Specification for IRM 3—N-tert-butyl-benzothiazole Sulfenamide (TBBS)*.<sup>5</sup>

4.4.1 Material description: Appearance is light tan or beige pellets approximately  $\frac{1}{8}$  in. long. Specific gravity is 1.28.

4.4.2 Specifications are given in Table 3.

4.5 *Specification for IRM 21—Stearic Acid*.<sup>6</sup>

4.5.1 Material description: Appearance is a fine powder or flakes.

4.5.2 Specifications are given in Table 4.

4.6 *Specification for IRM 31—Sulfur*.<sup>7</sup>

4.6.1 Material description: Appearance is light to pale yellow powder.

4.6.2 Specifications are given in Table 5.

4.7 *Specification for IRM 43—Naphthenic Process Oil*.<sup>8</sup>

4.7.1 Material description: Appearance is translucent dark liquid. Specific gravity is 0.9. This material is commonly used in certain ASTM rubber test recipes as a process oil. This oil has been hydrotreated.

4.7.2 Specifications are given in Table 6.

4.8 *Specification for IRM 91—Zinc Oxide (ZnO)*.<sup>9</sup>

4.8.1 Material description: Appearance is a white powder. Specific gravity is 5.6. This material is commonly used in many ASTM rubber test recipes as an activator and in ASTM test recipes for testing halogenated elastomers as a vulcanizing agent.

<sup>4</sup> A lot IRM 2a has been depleted. Lot IRM 2b has not been approved.

<sup>5</sup> An approved lot, IRM 3, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: [www.ardl.com](http://www.ardl.com).

<sup>6</sup> An approved lot, IRM 21, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: [www.ardl.com](http://www.ardl.com).

<sup>7</sup> An approved lot, IRM 31, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: [www.ardl.com](http://www.ardl.com).

<sup>8</sup> No approved lot of IRM 43 has been established.

<sup>9</sup> An approved lot, IRM 91a, has been reserved and is available from R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806.

<sup>3</sup> Lot IRM 1a is no longer available; IRM 1b has not been approved.

**TABLE 2 Specification for IRM 2—Benzothiazyl Disulfide (MBTS)**

Property	ASTM Designation	Limits
Melting point, °C	D 1519	165 min
Ash, %	D 4574	0.7 max
Loss on heating at 105°C, % loss	D 4571	0.5 max
Wet sieve analysis, % retained on 100 mesh screen	D 4572	0.1 max

**TABLE 3 Specification for IRM 3—N-tert-butyl-benzothiazole Sulfenamide (TBBS)**

Property	ASTM Designation	Limit/Target
Assay, %	D 4936	96 min
Melting Point, Initial, °C	D 1519	108 min
Melting Point, Final, °C	D 1519	109 min
Volatile Matter, %	D 1416	0.1 max
Ash, %	D 4574	0.2 max
Insolubles in Methanol, %	D 4934	0.3 max

**TABLE 4 Specification for IRM 21—Stearic Acid**

Property	ASTM Designation	Limit/Target
Mineral Acid		None
Acid Value	D 1980	193 to 199 min
Titer, °C	D 1982	66 min
Iodine Value, %	D 1959	1.0 max
Loss on Heating, %	D 1960	0.2 max
Ash, %	D 1951	0.05 max
Fat, Unsaponifiables and Insoluble, %	D 1965	0.5 max

**TABLE 5 Specification for IRM 31—Sulfur**

Property	ASTM Designation	Limit/Target
Organic Material, %		0.05 max
Volatile Matter, °C	D 4571	0.5 max
Ash, %	D 4574	0.3 max
Insoluble in CS <sub>2</sub> , %	D 4578	0.3 max
Acidity, %	D 4569	0.005 max
Dry analysis, % retained on 100 mesh screen	D 4570	0.0 max
Dry analysis, % retained on 200 mesh screen	D 4570	5 to 10

**TABLE 6 Specification for IRM 43—Naphthenic Process Oil**

Property	ASTM Designation	Limits/Targets
Clay-gel absorption chromatographic analysis	D 2007	
% Asphaltenes		0.3 max
% Polar compounds		6.0 max
% Saturated hydrocarbon		35.1 to 65.0
Viscosity @ 100°C, mm <sup>2</sup> /s	D 88 and D2161	16.8 ± 1.2
Viscosity-gravity constant	D 2501	0.889 ± 0.002

4.8.2 Specifications are given in Table 7.

#### 4.9 Specification for IRM 241—Butyl Rubber<sup>10</sup>

4.9.1 Material description: Appearance is pale white solid rubber. This is a copolymer of isobutylene and isoprene (IIR). The rubber shall be stabilized with a non-staining antioxidant suitable for long-term storage. Specific gravity is 0.92. This

**TABLE 7 Specification for IRM 91—Zinc Oxide (ZnO)**

Property	ASTM Designation	Limits/Targets
Surface area, m <sup>2</sup> /g	D 4315 and D 3037	4.3 ± 0.3
% Zinc oxide	D 3280	99.5 min
% Lead	D 4075	0.08 max
% Cadmium	D 4075	0.08 max
Loss on heating @ 105°C % loss	D 280	0.50 max
Wet sieve analysis, %, retains on 45 µm	D 4315	0.10 max

**TABLE 8 Specification for IRM 241—Butyl Rubber**

Property	ASTM Designation	Limits/Targets
Mooney Viscosity mL 1 + 8 125°C (unmassed)	D 1646	50.0 ± 1.0
Mooney Viscosity mL 1 + 4 100°C (unmassed)	D 1646	73.5 ± 2.0
Volatile matter, %	D 1416	0.30 max
Ash, %	D 1416	0.50 max

**TABLE 9 Specification for IRM 902—Petroleum Oil**

Property	ASTM Designation	Limits/Targets
Viscosity: cst @ 99°C	D 445	20.35 ± 1.15
Gravity, API @ 16°C	D 287	20.0 ± 1.0
Flash pt. COC, ° C	D 92	240 min
Aniline point, °C	D 611	93.0 ± 3.0
Viscosity-gravity constant	D 2140	0.865 ± 0.005
Naphthenics Cn%	D 2140	35 min
Paraffinics Cp%	D 2140	50 max

**TABLE 10 Specification for IRM 903—Petroleum Oil**

Property	ASTM Designation	Limits/Targets
Viscosity: cst @ 38°C	D 445	33 ± 1.1
Gravity, API @ 16°C	D 287	22.0 ± 1.0
Flash pt. COC, ° C	D 92	163 min
Aniline point, °C	D 611	70.0 ± 1.0
Viscosity-gravity constant	D 2140	0.880 ± 0.005
Naphthenics Cn%	D 2140	40 min
Paraffinics Cp%	D 2140	45 max

material is commonly used for the quality control monitoring of Mooney viscometers in accordance with Test Method D 1646.

4.9.2 Specifications are given in Table 8.

#### 4.10 Specification for IRM 902—Petroleum Oil<sup>11</sup>

4.10.1 Material description: Appearance is a light yellow, translucent liquid. Specific gravity is 0.8. This oil is a severely hydrotreated naphthenic distillate. Its CAS registry number is 64742-52-5. This oil is commonly used in ASTM oil immersion tests. Typical properties are as follows: Aromatics, Ca in accordance with Test Method D 2140 is 12 %; pour point in accordance with Test Method D 97 is – 12°C; color in accordance with Test Method D 1500 is L2.5; refractive index in accordance with Test Method D 1747 is 1.5105; and UV absorbance @ 260 nm is 4.0.

4.10.2 Specifications are given in Table 9.

#### 4.11 Specification for IRM 903—Petroleum Oil<sup>12</sup>:

<sup>11</sup> An approved lot, IRM 902a, has been reserved and is available from R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806.

<sup>12</sup> An approved lot, IRM 903a, has been reserved and is available from R.E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806.

<sup>10</sup> An approved lot, IRM 241b, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301.

**TABLE 11 Specification for IRM 913—Ammoniated Latex Antigenic Protein**

Property	ASTM Designation	Limit/Target
Source	D 6499	Ammoniated NR Latex (high and low)
Protein Amount	D 5712	Stock solution should be adjusted to 1 mg protein per ml
AA Analysis	D 5712	The correlation coefficient of AA composition between batches should be at least 0.90.
SDS-PAGE	D 6499	Full distribution of NR proteins (between 5 kDa and 200 kDa)
Western blot profile	D 6499	Full distribution of NR antigens (between 5 kDa and 200 kDa)
Inhibition ELISA	D 6499	Adjust antigen content based on D 6499 determination.

4.11.1 Material description: Appearance is a light yellow, translucent liquid. Specific gravity is 0.8. This oil is a severely hydrotreated naphthenic distillate. Its CAS registry number is 64742-52-5. This IRM is commonly used in ASTM oil immersion tests. Typical properties are as follows: Aromatics, Ca in accordance with Test Method D 2140 is 14 %; pour point in accordance with Test Method D 97 is – 31°C; color in accordance with Test Method D 1500 is L0.5; refractive index in accordance with Test Method D 1747 is 1.5026; and UV absorbance @ 260 nm is 2.2.

4.11.2 Specifications are given in Table 10.

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**TABLE 12 Specification for IRM 914—Rabbit Anti-AL Antisera**

Property	ASTM Designation	Limit/Target
Animal	D 6499	Rabbits
Antigen	D 6499	Ammoniated NR reference protein
Adjuvant Titer	D 6499	Freund's complete and incomplete Minimum 1 / 8 300
Western blot profile <sup>A</sup>	D 6499	Reactivity with entire spectrum of SDS-PAGE <sup>B</sup> visible AL protein
Inhibition ELISA reactivity <sup>C</sup>	D 6499	Comparative analysis of extracts from a minimum of 5 NR products

<sup>A</sup> Western blot represents the transfer of protein from SDS-Page to a membrane and subsequent antigen analysis using NR latex anti-sera.

<sup>B</sup> SDS-PAGE represents sodium dodecylsulfate polyacrylamide gel electrophoresis.

<sup>C</sup> ELISA represents the enzyme linked immuno-sorbant assay.

#### 4.12 Specification for IRM 913—Ammoniated Latex Antigenic Protein<sup>13</sup>

4.12.1 Material description: Sealed vial for hypodermic extraction — 500 test quantity.

4.12.2 Specifications are given in Table 11.

#### 4.13 Specification for IRM 914—Rabbit Anti AL Antisera<sup>14</sup>

4.13.1 Material description: Sealed vial for hypodermic extraction — 7500 test quantity.

4.13.2 Specifications are given in Table 12.

### 5. Keywords

5.1 industry reference materials; IRM; reference materials

<sup>13</sup> An approved lot, IRM 913, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: www.ardl.com.

<sup>14</sup> An approved lot, IRM 914, has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: www.ardl.com.