



Standard Specification for Physical and Chemical Properties of Industry Reference Materials (IRM)¹

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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 D4678 and other standards.

1.2 IRMs, as evaluated and referenced in Practice D4678, 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 D4678 shall be met before a candidate material can be formally accepted as an IRM.

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

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

2. Referenced Documents

2.1 ASTM Standards:²

D88 Test Method for Saybolt Viscosity

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D97 Test Method for Pour Point of Petroleum Products

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments

¹ 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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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.

D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents

D1278 Test Methods for Rubber from Natural Sources—Chemical Analysis

D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)³

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D1519 Test Methods for Rubber Chemicals—Determination of Melting Range

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D1747 Test Method for Refractive Index of Viscous Materials

D1951 Test Method for Ash in Drying Oils and Fatty Acids (Withdrawn 2003)³

D1959 Test Method for Iodine Value of Drying Oils and Fatty Acids (Withdrawn 2006)³

D1960 Test Method for Loss on Heating of Drying Oils (Withdrawn 2003)³

D1965 Test Method for Unsaponifiable Matter in Drying Oils, Fatty Acids, and Polymerized Fatty Acids (Withdrawn 2007)³

D1980 Test Method for Acid Value of Fatty Acids and Polymerized Fatty Acids (Withdrawn 2007)³

D1982 Test Method for Titer of Fatty Acids

D1993 Test Method for Precipitated Silica—Surface Area by Multipoint BET Nitrogen Adsorption

D2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method

D2140 Practice for Calculating Carbon-Type Composition

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

- of Insulating Oils of Petroleum Origin
- D2161** Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity
- D2501** Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils
- D3037** Test Method for Carbon Black—Surface Area by Nitrogen Adsorption (Withdrawn 1999)³
- D3157** Test Method for Rubber from Natural Sources—Color
- D3194** Test Method for Rubber From Natural Sources—Plasticity Retention Index (PRI)
- D3280** Test Methods for Analysis of White Zinc Pigments
- D4004** Test Methods for Rubber—Determination of Metal Content by Flame Atomic Absorption (AAS) Analysis
- D4075** Test Methods for Rubber Compounding Materials—Flame Atomic Absorption Analysis—Determination of Metals
- D4315** Test Methods for Rubber Compounding Material—Zinc Oxide
- D4569** Test Method for Rubber Compounding Materials—Determination of Acidity in Sulfur
- D4570** Test Method for Rubber Chemicals—Determination of Particle Size of Sulfur by Sieving (Dry)
- D4571** Test Methods for Rubber Compounding Materials—Determination of Volatile Material
- D4572** Test Method for Rubber Chemicals—Wet Sieve Analysis of Sulfur
- D4574** Test Methods for Rubber Compounding Materials—Determination of Ash Content
- D4578** Test Methods for Rubber Chemicals—Determination of Percent Sulfur by Extraction
- D4678** Practice for Rubber—Preparation, Testing, Acceptance, Documentation, and Use of Reference Materials
- D4934** Test Method for Rubber Compounding Materials: 2-Benzothiazyl Sulfenamide Accelerators—Insolubles
- D4936** Test Method for Mercaptobenzothiazole Sulfenamide Assay by Reduction/Titration
- D5289** Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

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 **D4678** 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.

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

Property	ASTM Designation	Limits
Melting point, °C	D1519	142 min
Ash, %	D4574	0.10 max
Loss on heating at 105°C, % loss	D4571	0.5 max
Wet sieve analysis, % retaining on 100 mesh screen	D4572	0.05 max

4. Specifications

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

4.2 *Specification for IRM 1⁴—Tetramethyl Thiuram Disulfide (TMTD):*

4.2.1 Material description: Appearance is light buff to white powder. Specific gravity is **1.5**. 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 1**.

4.3 *Specification for IRM 2⁵—Benzothiazyl Disulfide (MBTS)*

4.3.1 Material description: Appearance is cream-colored powder. Specific gravity is 1.5. This material shall contain 2 ± 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 3⁶—N-tert-butyl-benzothiazole Sulfenamide (TBBS)*

4.4.1 Material description: Appearance is light tan or beige pellets approximately 1/8 in. long. Specific gravity is 1.28.

4.4.2 Specifications are given in **Table 3**.

4.5 *Specification for IRM 21⁶—Stearic Acid*

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 31⁶—Sulfur*

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 43⁷—Naphthenic Process Oil:*

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 91⁸—Zinc Oxide (ZnO):*

⁴ Lot IRM 1a is no longer available; IRM 1b has not been approved.

⁵ A lot IRM 2a has been depleted. Lot IRM 2b has not been approved.

⁶ An approved lot has been reserved and is available from Akron Rubber Development Lab Inc., 300 Kenmore Blvd., Akron, OH 44301, website: www.ardl.com.

⁷ No approved lot of IRM 43 has been established.

⁸ An approved lot has been reserved and is available from R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806.

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

Property	ASTM Designation	Limits
Melting point, °C	D1519	165 min
Ash, %	D4574	0.7 max
Loss on heating at 105°C, % loss	D4571	0.5 max
Wet sieve analysis, % retained on 100 mesh screen	D4572	0.1 max

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

Property	ASTM Designation	Limit/Target
Assay, %	D4936	96 min
Melting Point, Initial, °C	D1519	108 min
Melting Point, Final, °C	D1519	109 min
Volatile Matter, %	D1416	0.1 max
Ash, %	D4574	0.2 max
Insolubles in Methanol, %	D4934	0.3 max

TABLE 4 Specification for IRM 21—Stearic Acid

Property	ASTM Designation	Limit/Target
Mineral Acid		None
Acid Value	D1980	193 to 199 min
Titer, °C	D1982	66 min
Iodine Value, %	D1959	1.0 max
Loss on Heating, %	D1960	0.2 max
Ash, %	D1951	0.05 max
Fat, Unsaponifiables and Insoluble, %	D1965	0.5 max

TABLE 5 Specification for IRM 31—Sulfur

Property	ASTM Designation	Limit/Target
Organic Material, %		0.05 max
Volatile Matter, °C	D4571	0.5 max
Ash, %	D4574	0.3 max
Insoluble in CS ₂ , %	D4578	0.3 max
Acidity, %	D4569	0.005 max
Dry analysis, % retained on 100 mesh screen	D4570	0.0 max
Dry analysis, % retained on 200 mesh screen	D4570	5 to 10

TABLE 6 Specification for IRM 43—Naphthenic Process Oil

Property	ASTM Designation	Limits/Targets
Clay-gel absorption chromatographic analysis	D2007	
% Asphaltenes		0.3 max
% Polar compounds		6.0 max
% Saturated hydrocarbon		35.1 to 65.0
Viscosity @ 100°C, mm ² /s	D88 and D2161	16.8 ± 1.2
Viscosity-gravity constant	D2501	0.889 ± 0.002

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.

4.8.2 Specifications are given in **Table 7**.

4.9 Specification for IRM 241⁶—Butyl Rubber

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

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

Property	ASTM Designation	Limits/Targets
Surface area, m ² /g	D4315 and D3037	4.3 ± 0.3
% Zinc oxide	D3280	99.5 min
% Lead	D4075	0.08 max
% Cadmium	D4075	0.08 max
Loss on heating @ 105°C % loss	D280	0.50 max
Wet sieve analysis, %, retains on 45 µm	D4315	0.10 max

TABLE 8 Specification for IRM 241—Butyl Rubber

Property	ASTM Designation	Limits/Targets
Mooney Viscosity mL 1 + 8 125°C (unmassed)	D1646	51 ± 1
Volatile matter, %	D1416	0.30 max
Ash, %	D1416	0.50 max

TABLE 9 Specification for IRM 901—Petroleum Oil

Property	ASTM Designation	Limit/Targets
Viscosity: cst @ 99°C	D445	19.23 ± 1.11
Gravity, API @ 16°C	D287	28.8 ± 1.0
Flash pt. COC, °C	D92	243 min
Aniline point, °C	D611	124 ± 1
Viscosity-gravity Constant	D2140	0.790 – 0.805
Naphthenics Cn%	D2140	27 (avg)
Paraffinics Cp%	D2140	65 min

TABLE 10 Specification for IRM 902—Petroleum Oil

Property	ASTM Designation	Limits/Targets
Viscosity: cst @ 99°C	D445	20.35 ± 1.15
Gravity, API @ 16°C	D287	20.0 ± 1.0
Flash pt. COC, °C	D92	232 min
Aniline point, °C	D611	93.0 ± 3.0
Viscosity-gravity constant	D2140	0.865 ± 0.005
Naphthenics Cn%	D2140	35 min
Paraffinics Cp%	D2140	50 max

suitable for long-term storage. Specific gravity is 0.92. This material is commonly used for the quality control monitoring of Mooney viscometers in accordance with Test Methods **D1646**.

4.9.2 Specifications are given in **Table 8**.

4.10 Specification for IRM 901⁸—Petroleum Oil:

4.10.1 Material description: Appearance is a dark liquid. Specific gravity is 0.8. This oil is a severely solvent refined heavy paraffinic petroleum oil. Its CAS registry number is 64741-88-4. This oil is commonly used in ASTM oil immersion tests. Typical properties are as follows: Aromatics, Ca in accordance with Test Method **D2140** is 3 %; pour point in accordance with Test Method **D97** is –12°C; color in accordance with Test Method **D1500** is L3.5; refractive index in accordance with Test Method **D1747** is 1.4848; and UV absorption @ 260 nm is 0.8.

4.10.2 Specifications are given in **Table 9**.

4.11 Specification for IRM 902⁸—Petroleum Oil:

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 oil is commonly used in ASTM oil immersion tests. Typical properties are as follows: Aromatics, Ca in accordance with Test Method **D2140** is 12 %; pour point in accordance with Test Method **D97** is – 12°C; color in accordance with Test Method **D1500** is L2.5; refractive index in accordance with Test Method **D1747** is 1.5105; and UV absorbance @ 260 nm is 4.0.

4.11.2 Specifications are given in **Table 10**.

4.12 *Specification for IRM 903*⁸—Petroleum Oil:

4.12.1 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 **D2140** is 14 %; pour point in accordance with Test Method **D97** is – 31°C; color in accordance with Test Method **D1500** is L0.5; refractive index in accordance with Test Method **D1747** is 1.5026; and UV absorbance @ 260 nm is 2.2.

4.12.2 Specifications are given in **Table 11**.

4.13 *Specification for IRM 905*⁸—Petroleum Oil:

4.13.1 Material description: Appearance is an amber liquid. Specific gravity is 0.88. This oil is a severely solvent refined heavy paraffinic petroleum oil. Its CAS registry number is 64741-88-4. This oil is commonly used in ASTM oil immersion tests. Typical properties are as follows: Aromatics, Ca in accordance with Test Method **D2140** is 4 %; pour point in

TABLE 11 Specification for IRM 903—Petroleum Oil

Property	ASTM Designation	Limits/Targets
Viscosity: cst @ 38°C	D445	33 ± 1.1
Gravity, API @ 16°C	D287	22.0 ± 1.0
Flash pt. COC, °C	D92	163 min
Aniline point, °C	D611	70.0 ± 1.0
Viscosity-gravity constant	D2140	0.880 ± 0.005
Naphthenics Cn%	D2140	40 min
Paraffinics Cp%	D2140	45 max

TABLE 12 Specification for IRM 905—Petroleum Oil

Property	ASTM Designation	Limit/Targets
Viscosity: cst @ 99°C	D445	11.35 ± 0.55
Flash pt. COC, °C	D92	243 min
Aniline point, °C	D611	115 ± 1.0
Pour Point, °C (typical)	D97	–15
ASTM Color (typical)	D1500	L 1.0
Refractive Index (typical)	D1747	1.4808
Aromatics, C _A (%) (typical)	D2140	4

accordance with Test Method **D97** is –5°C; color in accordance with Test Method **D1500** is L1.0; and refractive index in accordance with Test Method **D1747** is 1.4808.

4.13.2 Specifications are given in **Table 12**.

5. Keywords

5.1 industry reference materials; IRM; reference materials

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