Designation: D5900 – 16

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

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D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)[3]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D1951 Test Method for Ash in Drying Oils and Fatty Acids (Withdrawn 2003)[3]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

[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.

[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
D6738 Test Method for Precipitated Silica—Volatile Content
D6739 Test Method for Silica—pH Value
D6845 Test Method for Silica, Precipitated, Hydrated—CTAB (Cetyltrimethylammonium Bromide) Surface Area
D6854 Test Method for Silica—Oil Absorption Number (OAN)

2.2 *ISO Standards:*[4]

ISO 787 General methods of test for pigments and extenders
ISO 3262 Extenders for paints -- Specifications and methods of test

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.

TABLE 1 Reference Material Number Assignment by Material Classification[A]

Material Classification	RM Number
Accelerators	1–10
Antioxidants	11–20
Fatty or stearic acids	21–30
Sulfur	31–40
Process oils	41–50
Plasticizers, physical	51–60
Waxes	61–70
Processing aids	71–80
ASTM and other reference liquids	81–90
Zinc-oxides	91–99
Silicas	100–110
Unassigned	111–120
Non-black fillers	121–140
NR	201–210
SBR	211–220
NBR	221–230
CR	231–240
IIR	241–250
BR	251–260
Other rubbers	261–300
Tackifying resins	301–320
Non-sulfur crosslinking agents	321–340
Inhibitors/retarders	341–360
Plasticizers, chemical (peptizers)	361–380
Reference liquids	901–930
Miscellaneous materials	931–999

[A] The assigned numbers apply to both IRM and CRM.

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.

4. Assignment and Tabulation of Reference Material (RM) Numbers

4.1 *Assigning RM Numbers*—Table 1 lists the numbering system that shall be used for assigning numbers for reference materials. These numbers shall be assigned to either IRM or CRM candidate materials, with the acronym preceding the number when referring to the reference material.

5. Specifications

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

5.2 *Specification for IRM 1*[5]*—Tetramethyl Thiuram Disulfide (TMTD):*

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[5] Lot IRM 1a is no longer available; IRM 1b has not been approved.

TABLE 2 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

5.2.1 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.

5.2.2 Specifications are given in Table 2.

5.3 *Specification for IRM 2*[6]*—Benzothiazyl Disulfide (MBTS)*

5.3.1 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.

5.3.2 Specifications are given in Table 3.

5.4 *Specification for IRM 3*[7]*—N-tert-butyl-benzothiazole Sulfenamide (TBBS)*

5.4.1 Material description: Appearance is light tan or beige pellets approximately ⅛ in. long. Specific gravity is 1.28.

5.4.2 Specifications are given in Table 4.

5.5 *Specification for IRM 21*[7]*—Stearic Acid*

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

5.5.2 Specifications are given in Table 5.

5.6 *Specification for IRM 31*[7]*—Sulfur:*

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

5.6.2 Specifications are given in Table 6.

5.7 *Specification for IRM 91*[8]*—Zinc Oxide (ZnO):*

5.7.1 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.

5.7.2 Specifications are given in Table 7.

5.8 *Specification for IRM 100*[9]*—Precipitated Silica:*

5.8.1 *Material Description*—Appearance is a white powder. Specific gravity is 2.0. Its CAS registry number is 112926-00-8. This silica is commonly used for the quality control monitoring of typical silica properties.

5.8.2 Specifications are given in Table 8.

TABLE 3 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 4 Specification for IRM 3—N-tert-butyl-benzothiazole Sulfenamide (TBBS)

Property	ASTM Designation	Limit/Target
Assay, %	D4936	96 min
Melting Point, Initial, °C	D1519	104 min
Melting Point, Final, °C	D1519	112 max
Volatile Matter, %	D1416	0.4 max
Ash, %	D4574	0.5 max
Insolubles in Methanol, %	D4934	0.5 max

TABLE 5 Specification for IRM 21—Stearic Acid

Property	ASTM Designation	Limit/Target
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 6 Specification for IRM 31—Sulfur

Property	ASTM Designation	Limit/Target
Purity, %	D4571 D4574 D4578 D4569	99.5 min
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 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

5.9 *Specification for IRM 241*[7]*—Butyl Rubber*

5.9.1 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 material is commonly used for the quality control monitoring of Mooney viscometers in accordance with Test Methods D1646.

[6] A lot IRM 2a has been depleted. Lot IRM 2b has not been approved.

[7] 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.

[8] An approved lot has been reserved and is available from G.H. Chemicals Ltd., PO Box 456, Saint Hyacinthe, Québec, Canada, J2S 7B8.

[9] An approved lot has been reserved and is available from Balentine Enterprises, Inc. dba Laboratory Standards and Technologies, 227 Somerset Street, Borger, Texas 79007, http://www.irmsilicastandard.com.

TABLE 8 Specification for IRM 100—Precipitated Silica

Property	ASTM Designation	Limits/Targets
Surface area (BET), m^2/g	D1993	175 ± 15
Surface area (CTAB), m^2/g	D6845	167 ± 15
Volatile matter, % (2 h at 105°C)	D6738	3.5 ± 0.7
pH value, (5 %)	D6739	5.5 ± 0.5
Conductivity, µS/cm (4 %)	ISO 787	≤350
Loss on ignition, % (orig. mat.)	ISO 3262	6.5 ± 1.0
OAN Silica (DOA), mL/100 g (orig. mat.)	D6854	260 ± 15

TABLE 9 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 10 Specification for IRM 901—Petroleum Oil

Property	ASTM Designation	Limit/Targets
Aniline point, °C	D611	124 ± 1
Kinematic Viscosity (mm^2/s [cSt]) 99°C	D445	18.12–20.34
Gravity, API @ 16°C	D287	28.8 ± 1.0
Viscosity-gravity Constant	D2140	0.790 – 0.805
Flash pt. COC, °C	D92	243 min
Naphthenics Cn%	D2140	27 (avg)
Paraffinics Cp%	D2140	65 min
Pour Point, °C (°F)	D97	–12
ASTM Color	D1500	*L* 3.5
Refractive Index	D1747	1.4848
UV Absorbance, 260 nm	D1747	0.8
Aromatics, C_A (%)	D2140	3

TABLE 11 Specification for IRM 902—Petroleum Oil

Property	ASTM Designation	Limits/Targets
Aniline point, °C	D611	93.0 ± 3.0
Kinematic Viscosity (mm^2/s [cSt]) 99°C	D445	19.2–21.5
Gravity, API @ 16°C	D287	19.0–21.0
Viscosity-gravity constant	D2140	0.860–0.870
Flash pt. COC, °C	D92	232 min
Naphthenics Cn%	D2140	35 min
Pour Point, °C (°F)	D97	–15
ASTM Color	D1500	*L* 2.0
Refractive Index	D1747	1.5083
UV Absorbance, 260 nm	D1747	1.43
Aromatics, C_A (%)	D2140	10

5.9.2 Specifications are given in Table 9.

5.10 *Specification for IRM 901*[8]—Petroleum Oil:

5.10.1 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 –12ºC; 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.

5.10.2 Specifications are given in Table 10.

TABLE 12 Specification for IRM 903—Petroleum Oil

Property	ASTM Designation	Limits/Targets
Aniline point, °C	D611	70.0 ± 1.0
Kinematic Viscosity (mm^2/s [cSt]) 38°C	D445	31.9–34.1
Gravity, API @ 16°C	D287	21.0–23.0
Viscosity-gravity constant	D2140	0.875–0.885
Flash pt. COC, °C	D92	163 min
Naphthenics Cn%	D2140	40 min
Pour Point, °C (°F)	D97	–42.8
ASTM Color	D1500	*L* 0.5
Refractive Index	D1747	1.5004
UV Absorbance, 260 nm	D1747	1.43
Aromatics, C_A (%)	D2140	12

TABLE 13 Specification for IRM 905—Petroleum Oil

Property	ASTM Designation	Limit/Targets
Aniline point, °C	D611	115 ± 1.0
Kinematic Viscosity (mm^2/s [cSt]) 99°C	D445	10.8–11.9
Flash pt. COC, °C	D92	243 min
Pour Point, °C	D97	–15
ASTM Color	D1500	*L* 1.0
Refractive Index	D1747	1.4808
Aromatics, C_A (%)	D2140	4

5.11 *Specification for IRM 902*[8]*—Petroleum Oil:*

5.11.1 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.

5.11.2 Specifications are given in Table 11.

5.12 *Specification for IRM 903*[8]—Petroleum Oil:

5.12.1 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.

5.12.2 Specifications are given in Table 12.

5.13 *Specification for IRM 905*[8]—Petroleum Oil:

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

5.13.2 Specifications are given in Table 13.

6. Keywords

6.1 industry reference materials; IRM; reference materials