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# Standard Practice for Rubber IRM 902 and IRM 903 Replacement Oils for ASTM No. 2 and ASTM No. 3 Oils<sup>1</sup>

This standard is issued under the fixed designation D 5964; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

#### INTRODUCTION

Test Method D 471 was revised in February 1995, establishing IRM 902 and IRM 903 as replacements for ASTM No. 2 and No. 3 immersion oils, respectively. Unlike ASTM No. 2 and No. 3 oils, the two IRM oils are severely hydrotreated, have a demonstrated negative Ames test and do not require cancer warning labels under the OSHA Hazard Communication Standard published in November 1983. Although it was attempted to match the effect of the ASTM oils on rubber properties in immersion testing as closely as possible, in general, neither of the IRM oils produces test results exactly identical to the ASTM oils it replaced.

The selections for replacement oils were made on the basis of an objective comprehensive test program as described in this practice and decisions on the data generated in this program were made in open meetings of Subcommittee D11.15. The SAE Committee on Automotive Rubber Specifications (CARS) made a recommendation on the replacement oils that was identical to the decisions made by D11.15.

This practice addresses the need for establishing a correlation between test results obtained with IRM versus ASTM oils, based on results of the described test program. Although the test program was quite comprehensive, it cannot begin to address the numerous variations in compound recipes used in the rubber industry. Correlations established by this practice may therefore not always provide satisfactory results. In this case it is suggested that other approaches be used, such as a direct comparison of each specific rubber compound in the respective ASTM and IRM oils. All new specifications, including oil immersion testing, shall be established using IRM 902 and IRM 903 in place of ASTM No. 2 and No. 3 oils, respectively.

# 1. Scope

- 1.1 This practice covers two new immersion oils to be used as replacements for ASTM No. 2 and No. 3 immersion oils as called for in Test Method D 471. The new immersion oils will be designated as IRM 902 as a replacement for ASTM No. 2 oil and IRM 903 as a replacement for ASTM No. 3 oil. The new reference oils have been developed under a new Committee D11 policy on reference materials (see Practice D 4678 for background on the new policy and procedures).
- 1.2 The new oils, IRM 902 and IRM 903, are similar but not fully equivalent to ASTM No. 2 and ASTM No. 3 oil, respectively.
- 1.3 This practice gives the necessary background and details on the changeover from the previous oils to the new oils. See Annex A1 for additional information on the commercial oils selected to replace the two ASTM oils and the test program

1.4 The EVS values are calculated on the basis of "correction equations" derived from one of two sources.

conducted for this selection process. The changeover from

Equivalent Volume Swell (EVS) for each of the two replace-

ment oils. EVS(902) is the ASTM No. 2 percent volume swell

value calculated from the measured percent volume swell value

using IRM 902 as the immersion liquid. A similar calculation

can be used to calculate the analogous EVS(903) value. Either

EVS value is obtained as a correction of the measured IRM 902

or 903 percent volume swell value. The EVS values may be

used to determine if volume swell specifications are met when

the specifications are expressed in terms of ASTM No. 2 or No.

1.3.2 Step 2—A longer term policy change or conversion of

1.3.1 Step 1-A transition phase that makes use of the

ASTM to IRM oils is proposed in two steps:

1.4.1 Correction equations derived from the results of the comprehensive evaluation program conducted to select each of

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3 limits, and

specifications from ASTM No. 2 and No. 3 values to IRM 902 and 903 values.

<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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the two replacement oils from a group of three candidate oils for each ASTM oil. This program is described in Annex A1.

- 1.4.2 Correction equations derived from in-house customized or specific testing programs to make direct comparisons of the volume swell (and other important properties) of the two IRM and ASTM oils. These programs should be conducted in each laboratory of those organizations that engage in producer-user specification testing for rubber immersion performance.
- 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:
- D 92 Test Method for Flash and Fire Points by Cleveland Open Cup<sup>2</sup>
- D 97 Test Method for Pour Point of Petroleum Products<sup>2</sup>
- D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)<sup>3</sup>
- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension<sup>4</sup>
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)<sup>2</sup>
- D 471 Test Method for Rubber Property—Effect of Liquids<sup>4</sup>
- D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents<sup>2</sup>
- D 1414 Test Methods for Rubber O-Rings<sup>5</sup>
- D 1418 Practice for Rubber and Rubber Latices— Nomenclature<sup>4</sup>
- D 1500 Test Method for Color of ASTM Petroleum Products (ASTM Color Scale)<sup>2</sup>
- D 1747 Test Method for Refractive Index of Viscous Materials<sup>2</sup>
- D 2000 Classification System for Rubber Products in Automotive Applications<sup>5</sup>
- D 2008 Test Method for Ultraviolet Absorbance and Absorptivity of Petroleum Products<sup>2</sup>
- D 2140 Test Method for Carbon-Type Composition of Insulating Oils of Petroleum Origin<sup>6</sup>
- D 2240 Test Method for Rubber Property—Durometer Hardness<sup>4</sup>
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>4</sup>
- D 4678 Practice for Rubber—Preparation, Testing, Acceptance, Documentation, and Use of Industry Reference Materials<sup>4</sup>

## 3. Significance and Use

3.1 The two reference immersion oils described in this practice are required for the development of oil-resistant rubber

compounds for use in environments where contact with petroleum-based solvents and oils is encountered. Tests for tensile strength, percent elongation at break, hardness, and percent volume swell are performed after a specified immersion time period (at a specified temperature) in the evaluation of oil-resistant rubbers. The results of such testing by rubber product manufacturers and their customers are used to develop oil-resistant rubbers or compounds, or both.

3.2 Testing with ASTM Oils No. 2 and No. 3 is used to verify compliance with purchase specifications which reference the oil-resistant classes of rubbers and elastomers listed in Table 6 of Classification D 2000. These oils are also used in comparative performance evaluation testing of O-rings and O-ring compounds as cited in Test Methods D 1414. The use of these reference oils is required for the development and selection of oil-resistant rubber compounds having acceptable or optimum performance characteristics, or both.

# 4. Specifying IRM 902 and IRM 903 Reference Immersion Oils

- 4.1 The two oils selected to replace ASTM No. 2 and ASTM No. 3 immersion oils have commercial names. These new oils were selected on the basis of the closest match to ASTM No. 2 and ASTM No. 3 oils in the comprehensive evaluation program as outlined in Annex A1.
- 4.2 The (petroleum) specifications and typical properties of IRM 902 and IRM 903 are given in Table 1.

# 5. Converting "ASTM Oil-IRM Oil" Volume Swell Values

- 5.1 Basis of Conversion—One of the important issues for any user of the new IRM oils, especially producer-consumer operations, is the relationship and conversion of the customary ASTM oil volume swell values for proprietary and commercial compounds to volume swell values for the new IRM oils. As outlined in the scope, this can be done on the basis of two approaches.
- 5.1.1 Calculating EVS values for IRM 902 or IRM 903 for any commercial compound based on (*I*) selecting from Table A1.1 and Appendix X1, the compound nearest to the commercial compound in composition, and (*2*) using the percent difference (PC d) value for this compound in the conversion or correction calculation. This EVS value is an approximate value.
- 5.1.2 Organizing a special in-house testing program to obtain volume swell values under the appropriate conditions (time and temperature of immersion) for the selected ASTM and IRM oils for the proprietary or commercial compounds of interest. Once data for both oils are obtained, the relationship between the two oils is established.
- 5.1.3 If needed for future applications, calculations can be made to correct or convert the ASTM values to the IRM values or vice-versa for other compounds where the correction can be

 $<sup>^{2}\</sup> Annual\ Book\ of\ ASTM\ Standards,\ Vol\ 05.01.$ 

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 05.04.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 09.01.

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 09.02.

<sup>&</sup>lt;sup>6</sup> Annual Book of ASTM Standards, Vol 10.03.

<sup>&</sup>lt;sup>7</sup> Cal-2, designated as IRM 902, and Cal-3, designated as IRM 903, are manufactured by Calumet Lubricants Co., HC 62 Box 460, Princeton, LA 71067. The new reference immersion oils, IRM 902 and IRM 903, are distributed for the manufacturer by R. E. Carroll, Inc., P.O. Box 5806, Trenton, NJ 08638-0806, and Penreco Co., 4426 E. Washington Blvd., Los Angeles, CA 90023-4476.

TABLE 1 Specifications and Typical Properties of IRM Reference Oils

Property Specifications	ASTM Test Method	IRM 902	IRM 903					
Aniline point, °C (°F) Kinematic viscosity (mm²/s (cSt))	D 611	93 ± 3 (199 ±	5)70± 1 (158 ± 2)					
38°C (100°F)	D 445		31.9-34.1					
99°C (210°F)	D 445	19.2-21.5						
Gravity, API, 16°C (60°F)	D 287	19.0-21.0	21.0-23.0					
Viscosity-Gravity constant	D 2140	0.860-0.870	0.875-0.885					
Flash point COC, °C (°F)	D 92	240 (464) min	163 (325) min					
Naphthenics, $C_N$ (%)	D 2140	35 min	40 min					
Paraffinics, $C_P$ (%)	D 2140	50 max	45 max					
Typical Properties								
Pour point, °C (°F)	D 97	-12 (10)	-31 (-24)					
ASTM Color	D 1500	L 2.5	L 0.5					
Refractive index	D 1747	1.5105	1.5026					
UV Absorbance, 260 nm	D 2008	4.0	2.2					
Aromatics, $C_A$ (%)	D 2140	12	14					

legitimately applied. This approach gives corrections that are specific to the compounds of interest; it is direct and substantially more accurate than the approximate approach and is the recommended conversion procedure for exact and critical specification applications if corrections of this sort are required.

5.2 Conversion Using the EVS Procedure—Two procedures are given: (1) for converting IRM values to equivalent ASTM values, and (2) for converting ASTM values to IRM values. The second operation may be of value in converting existing ASTM value specifications to IRM value specifications as the old ASTM oil specification values are phased out.

5.2.1 Eq 1 gives the EVS(902) value, the equivalent ASTM No. 2 oil percent volume swell value for IRM 902 oil, based on measured volume swell data in IRM 902 and data in Table A1.1 of Annex A1.

$$EVS(902) = \frac{MVS(902)}{\left(1 + \frac{PC d_1}{100}\right)}$$
(1)

where:

EVS (902) = EVS (approximate) for IRM 902 oil,

MVS (902) = measured percent volume swell in IRM 902

oil, and

 $PC d_1$  = difference between volume swells in IRM

902 oil and ASTM No. 2 oil expressed as a relative percentage (from Table A1.1, se-

lected in accordance with 5.1.1).

5.2.2 Eq 2 may be used for converting IRM values to ASTM values for compounds of commercial interest on the same basis as described above.

EVS(No. 2) = 
$$\frac{\text{MVS(No. 2)}}{\left(1 + \frac{\text{PC d}_2}{100}\right)}$$
 (2)

where:

EVS (No. 2) = EVS (approximate) for ASTM No. 2 oil, MVS (No. 2) = measured percent volume swell in ASTM

No. 2 oil, and

 $PC d_2$  = difference between volume swells in

ASTM No. 2 oil and IRM 902 oil expressed as a relative percentage (from Table A1.1, selected in accordance with

5.1.1 (Note 1)).

Note 1—By definition PC  $d_2 = -PC d_1$ .

5.2.3 Eq 1 and Eq 2 may be used for IRM 903 and ASTM No. 3 conversions or corrections by changing the parentheses values.

#### 6. Testing Precision

6.1 Although a precision statement is not a mandatory section in a practice, the precision of volume swell testing is an important issue for the conversion from the original ASTM oils to the new IRM oils. Annex A2 gives a review of the precision results obtained from the comprehensive program outlined in Annex A1. Refer to Annex A2 for precision information.

## 7. Keywords

7.1 ASTM oils; immersion tests; IRM oils; reference oils

#### **ANNEXES**

(Mandatory Information)

# A1. BRIEF SUMMARY OF EVALUATION PROGRAM TO SELECT IRM 902 AND IRM 903

# **A1.1 Program Organization**

A1.1.1 A comprehensive testing program was organized in early 1993 to evaluate three candidate immersion oils (commercially supplied by different manufacturers) for ASTM No. 2 oil and three candidate oils for ASTM No. 3 oil. The program consisted of evaluating each of the candidate oils along with the reference ASTM oil for their influence on compound physical properties in twelve typical oil-resistant rubber compounds, each prepared with a different rubber. Four properties were measured after 70 h immersion for each of the com-

pounds: tensile strength, in MPa and percent elongation in accordance with Test Methods D 412; hardness (Shore A) in accordance with Test Method D 2240; and percent volume swell in accordance with Test Method D 471.

A1.1.2 The program was conducted in nine laboratories to prevent an undue burden on any one laboratory to conduct all the immersion and physical tests. The nine laboratories were divided into three groups and in each group one set of four rubber compounds was tested. The program was conducted to give duplicate test results to obtain a typical "Day 1—Day 2"



estimate of test error for within-laboratory variation (repeatability) within each group. Between-laboratory variation was not assessed because the number of laboratories is too small to obtain a realistic reproducibility. A test result (obtained on each of the two days one week apart) is defined as the mean or median of the number of individual determinations as specified by each test method.

A1.1.3 The rubbers in each group and the immersion temperatures (70 h at each temperature) were as given below. Appendix X1 gives the formulations for the twelve compounds and identifies the rubbers according to the acronym (specified in Practice D 1418) used for each base rubber.

A1.1.3.1 Immersion at 100°C—CR, ECO, NBR, TPV,

A1.1.3.2 Immersion at 125°C—EPDM, ACM, AEM, EVM, and

A1.1.3.3 Immersion at 150°C—FKM, FVMQ, HNBR, VMO.

### **A1.2 Evaluation Program Results**

A1.2.1 From the results of this comprehensive evaluation program two commercial oils were selected as the closest match to ASTM No. 2 and ASTM No. 3 oil, respectively. These two selected oils will be referred to as IRM 902 and IRM 903 in the remainder of this practice. A formal report on the evaluation program has been prepared that gives the details and the analysis performed to select the two candidate oils.<sup>8</sup>

#### A1.3 Comparison of ASTM Oils and IRM Oils

A1.3.1 Table A1.1 gives the comparison of ASTM No. 2

versus IRM 902 and ASTM No. 3 versus IRM 903 oils. The results are given in terms of the percent difference between the ASTM oil and the IRM oil; this is designated as "PC d." The percent difference term is defined by the following equation:

$$PC d = \frac{(P_i - P_r)}{P_r} \cdot 100$$
 (A1.1)

where:

PC d = percent difference between IRM oil and ASTM oil (Note A1.1),

= any property value for (either) IRM oil, and

 $P_r$  = any property value for (either) ASTM reference

Note A1.1—This is a relative indication for any property and should not be confused with absolute percentages for elongation and volume swell.

A1.3.2 Table A1.1 is divided into four sections, one for each property. Under each property section there are two subsections, one for each IRM oil and the corresponding reference ASTM oil. The values on the line for IRM 902 are PC d values for each of the twelve rubbers. The tabulated PC d value for each rubber is an average of six values (three laboratories, duplicate test results in each laboratory) obtained from each group. The next line below gives the  $\pm 2S$  limits, where S = within-laboratory test result standard deviation for averages of six test results, pooled over all three laboratories and all four immersion oils (ASTM and three candidate oils). The next line, ASTM average, gives the average (six test results) test property value, in test measurement units, for the ASTM oil (ASTM No. 2 for IRM 902, ASTM No. 3 for IRM 903). The second

TABLE A1.1 Compilation of Immersion Test Results for Twelve Rubbers Percent Difference (PC d) from ASTM Oil

														Rang	ge	
	CR	ECO	NBR	TPV	EPDM	ACM	AEM	EVM	FKM	FVMQ	HNBR		Algebraic Average PC d	Low PC d	High PC d	Absolute Average PC d
Tensile Strength, MPa:																
IRM 902	5.1	2.0	0.4	-4.2	3.1	-2.6	-1.4	-2.5	-0.9	0.4	0.3	-2.7	-0.2	-4.2	5.	1 2.1
±2 <i>S</i>	6.1	1.6	3.4	3.2	6.6	5.1	4.1	4.7	3.2	1.7	3.1	3.2				
ASTM No. 2 Oil Average	18.2	12.8	20.9	5.1	5.2	12.7	14.8	17.2	14.3	9.1	26.7	9.2				
IRM 903	-9.9	-0.6	-0.8	8.0	-5.7	-8.9	-2.8	-0.6	-1.8	4.6	-0.9	4.9	-1.8	-9.9	4.6	3.5
±2 <i>S</i>	4.4	3.0	2.8	5.1	13.4	3.5	3.6	6.9	2.4	4.8	3.3	4.7				
ASTM No. 3 Oil Average	12.0	12.4	19.7	4.2	2.8	12.2	10.9	12.3	12.8	7.8	24.9	6.2				
Percent Elongation:																
IRM 902	2.0	-3.7	-0.2	-1.6	3.3	-0.6	0.8	-1.8	2.0	0.2	-0.9	-3.5	-0.3	-3.7	3.3	3 1.7
±2 <i>S</i>	5.1	7.4	3.5	6.0	4.1	4.5	7.0	5.1	2.7	1.8	3.6	2.7				
ASTM No. 2 Oil Average	209.0	373.0	577.0	285.0	322.0	130.0	306.0	177.0	257.0	286.0	322.0	315.0				
IRM 903	-5.0	-5.3	-3.6	-0.6	-7.1	-6.4	8.5	-0.2	-7.5	1.7	-3.6	-1.4	-2.5	-7.1	8.8	5 4.2
±2 <i>S</i>	2.7	8.1	2.8	5.9	4.3	2.8	8.2	4.6	6.1	3.7	3.7	10.9				
ASTM No. 3 Oil Average	161.0	366.0	569.0	230.0	250.0	137.0	227.0	131.0	273.0	264.0	298.0	229.0				
Hardness (Shore A):																
IRM 902	-2.4	4.2	1.1	-1.9	8.3	-0.0	-0.3	0.6	0.0	-0.5	-0.3	-0.6	0.7	-2.4	8.3	3 1.7
±2 <i>S</i>	2.0	1.6	3.3	1.2	8.4	1.1	1.7	1.6	1.0	0.8	1.0	1.4				
ASTM No. 2 Oil Average	57.0	62.7	59.0	54.2	14.7	78.8	59.7	59.3	73.5	63.0	66.0	55.5				
IRM 903	0.7	2.0	4.3	-0.8	4.4	3.1	1.8	2.9	-0.2	0.3	1.6	1.7	1.8	-0.8	4.4	4 2.0
±2 <i>S</i>	1.5	1.2	4.0	1.9	9.2	1.2	3.1	4.8	0.6	1.0	8.0	1.8				
ASTM No. 3 Oil Average	49.0	60.3	52.0	44.8	9.3	68.7	47.2	49.0	73.2	61.7	61.8	41.8				
Volume Swell, %:																
IRM 902	0.5	-13.6	-8.3	4.1	-1.9	-50.0	4.4	0.4	-15.8	-3.6	-6.7	4.2	$-3.4^{A}$	-15.8 <sup>A</sup>	4.4	<sup>4</sup> 5.8 <sup>A</sup>
±2 <i>S</i>	4.7	10.6	3.6	4.1	1.5	145.0	2.8	1.6	22.2	77.6	2.8	12.8				
ASTM No. 2 Oil Average	30.1	3.6	7.1	46.8	118.0	0.7	24.0	30.6	0.7	0.5	8.7	9.4				
IRM 903	-3.6	-29.0	-22.5	4.8	-18.3	-20.4	0.2	-3.4	-33.0	3.8	-17.0	8.5	-10.8	-29.0	8.8	5 13.7
±2 <i>S</i>	1.3	9.3	3.0	2.1	2.4	9.9	1.1	1.3	12.9	16.2	1.9	4.6				
ASTM No. 3 Oil Average	75.0	9.3	18.8	79.4	177.0	10.7	54.9	63.7	2.0	2.2	19.3	41.2				

<sup>&</sup>lt;sup>A</sup>Does not include ACM data.

<sup>&</sup>lt;sup>8</sup> A research report is on file at ASTM International Headquarters. Request RR:D11-1069.



subsection is a repeat of the first subsection, for the IRM 903 oil

A1.3.3 The last four columns to the right in Table A1.1 give (1) the algebraic average of the PC d values, (2) and (3) the low and high of the range of values across the twelve rubbers and (4) the absolute average PC d, (with sign ignored). The average and low-high values for IRM 902 for percent volume swell do not contain the individual values for the ACM rubber. The absolute volume swell is very close to zero for ACM and this inflates the PC d values because of the division-by-near-zero problem. These atypical results were excluded from the four column values.

A1.3.4 A review of the results of Table A1.1 illustrates the comparative performance of IRM 902 versus ASTM No. 2 and IRM 903 versus ASTM No. 3 oils. A PC d value of zero indicates identical performance for the IRM oil versus the ASTM oil. A positive PC d value indicates that for any property and any compound, immersion in the IRM oil gives a higher property value than immersion in the respective ASTM oil. A negative value on the same basis indicates a lower IRM value compared to the ASTM value.

A1.3.5 The use of the  $\pm 2S$  limits permits a decision about the significance of the PC d value for any rubber, that is, does the tabulated PC d value differ significantly from zero? If PC d is less than the tabulated 2S value, there is no demonstrated significant difference at this level of testing (averages of two

test results across three laboratories; 12 degrees of freedom (DF) for *S*). Subsequent testing of a more comprehensive nature (more laboratories, greater test precision) may demonstrate that small non-significant differences as shown in Table A1.1 are in fact significant and represent real differences in oil immersion performance.

A1.3.6 Although a substantial number of the tabulated PC d values for the four test properties are not significantly different from zero, the tabulated PC d values in Table A1.1 represent the best estimate of the difference between the IRM oil and the respective ASTM oil as of this date in the transition from ASTM to IRM oils.

A1.3.7 A review of Table A1.1 will show that volume swell is the property that has the greatest PC d values and the greatest number of significant PC d values. This is especially noted for IRM 903 versus ASTM No. 3 oil. Volume swell, therefore, is the most sensitive and most important property for immersion performance. The tabulated volume swell PC d values will be used in the correction equations as developed in 5.2 to calculate the ESV values for the initial transition phase corrections of IRM values to (equivalent) ASTM values. No corrections will be given for the other three properties. If desired, the user of this practice may make corrections for these other three properties based on the procedures as outlined for volume swell.

#### A2. PRECISION RESULTS FOR IRM 902 AND IRM 903 TESTING

A2.1 *Introduction*—The comprehensive evaluation program as described in Annex A1 may be used to derive estimates of within-laboratory variation or repeatability. Reproducibility or between-laboratory variation will not be evaluated because of the inadequate number of laboratories (three). Reproducibility estimates with this number of laboratories can be misleading. Some terminology used in Annex A2 is contained in Practice D 4483. Refer to Practice D 4483 for background details.

A2.2 Evaluating Precision—In the evaluation program of Annex A1, the twelve compounds were tested in groups of four; each group of four was tested by three laboratories. For any one type of oil (three candidate plus ASTM No. 2; three candidate plus ASTM No. 3) there are only three duplicates (one duplicate set of tests in each laboratory) for an estimate of within-laboratory variation for any combination of compound and oil. This is an inadequate number of DF for such an estimate. However, a reasonable assumption may be made that for any given compound, the true test variation with each of the four oils is equivalent. On this basis for any candidate set of four oils, the three DF estimates of test standard deviation for each of the four oils may be pooled to obtain a twelve DF estimate of test standard deviation. The within-laboratory precision of this annex is based on such pooled values. The estimates of the standard deviation S, used to calculate  $\pm 2S$ limits in Annex A1, were obtained on the same basis.

A2.3 Precision Results—The precision results are con-

tained in Table A2.1 for percent volume swell. This is the only property that was evaluated for precision since Annex A1 shows that it is the most important and is the most sensitive to variations in oil physical/chemical properties.

A2.3.1 Table A1.1 in Annex A1 contains precision results ( $\pm 2S$  limits) for the PC d values and thus should also be consulted for specific comparisons of oils on the basis of PC d values as defined in Annex A1.

A2.3.2 Table A2.1 lists the repeatability standard deviation, Sr; the repeatability, r, in units of percent volume swell; the relative repeatability, (r), which is a percent of a percent for volume swell measurements and for completeness the coefficient of variation, CV in percent. The mean volume swell values range from near zero to slightly over 160 %. The precision parameters also display a wide range. Regression and graphical analysis reveal that: (I) there is a direct (positive slope) log-log relationship for Sr versus mean volume swell, and (2) an inverse (negative slope) relationship between the relative repeatability (r) and mean volume swell. These relationships apply to both sets of data and to the combined data sets.

A2.4 Using the Precision Parameters—The results of Table A2.1 may be applied to within-laboratory data comparisons using either IRM 902 or IRM 903 for any commercial or proprietary compound by selecting the Table A2.1 rubber (compound) closest to the commercial compound and selecting the level of volume swell value in Table A2.1 closest to the

TABLE A2.1 Precision Results for Percent Volume Swell Within-Laboratory Variation

Note 1—Sr = Repeatability standard deviation,

 $r = \text{Repeatability} = Sr \times 2.83,$ 

(r) = Repeatability on relative basis (percent of percent), and

CV % = Coefficient of variation = (Sr/mean) 100.

Rubber	Candidate Set <sup>A</sup>	Mean <sup>B</sup>	Sr	r	(r)	CV,%
CR	ASTM No. 2	36.0	1.63	4.61	12.8	4.5
	ASTM No. 3	68.1	1.15	3.26	4.8	1.7
ECO	ASTM No. 2	3.9	0.58	1.65	42.7	15.1
	ASTM No. 3	7.1	1.22	3.48	48.5	17.1
NBR	ASTM No. 2	7.6	0.35	0.99	13.1	4.6
	ASTM No. 3	14.8	0.72	2.05	13.8	4.9
TPV	ASTM No. 2	48.4	1.98	5.62	11.6	4.1
	ASTM No. 3	79.5	1.92	5.43	6.8	2.4
EPDM	ASTM No. 2	120.2	2.17	6.14	5.1	1.8
	ASTM No. 3	161.5	5.19	14.70	9.1	3.2
ACM	ASTM No. 2	1.3	0.57	1.60	119.0	42.2
	ASTM No. 3	8.3	1.31	3.71	44.6	15.8
AEM	ASTM No. 2	27.8	0.82	2.33	8.4	3.0
	ASTM No. 3	50.3	0.71	2.01	4.0	1.4
EVM	ASTM No. 2	34.1	0.60	1.68	4.9	1.7
	ASTM No. 3	58.7	1.04	2.94	5.0	1.8
FKM	ASTM No. 2	0.7	0.20	0.57	76.5	27.0
	ASTM No. 3	1.6	0.34	0.95	59.1	20.9
FVMQ	ASTM No. 2	0.4	0.31	0.87	198.0	70.0
	ASTM No. 3	2.0	0.43	1.21	59.5	21.0
HNBR	ASTM No. 2	9.4	0.30	0.85	9.1	0.3
	ASTM No. 3	15.9	0.46	1.29	8.1	2.9
VMQ	ASTM No. 2	10.4	1.36	3.84	37.0	13.1
	ASTM No. 3	43.4	2.30	6.52	15.0	5.3

ASTM No. 2 = Results for set of candidate oils to replace ASTM No. 2. ASTM No. 3 = Results for set of candidate oils to replace ASTM No. 3.

measured volume swell of the commercial compound.

A2.4.1 Two test results of the commercial compound that differ by more than the tabulated r or (r) values as selected by the above process, must be considered to have come from different sample populations (that is, be significantly different).

A2.4.2 Alternatively normal testing operations should produce values for duplicate test results that are within the r and

(r) intervals as selected by the above process.

A2.5 *Bias*—As is usual with most physical property testing, reference values do not exist for this type of testing since the value of the test property is defined exclusively by the test method. Bias therefore cannot be determined.

#### **APPENDIX**

(Nonmandatory Information)

#### X1. RUBBER ACRONYM IDENTIFICATION AND COMPOUND FORMULATIONS

X1.1 Part 1—Acronyms for Rubbers:

ACM copolymer of ethyl (or other) acrylate and cure site monomers

AEM ethyl (or other) acrylate-ethylene copolymer

CR chloroprene (rubber)

ECO ethylene oxide—chloromethyloxirane (epichlorohydrin copolymer)

EPDM ethylene propylene diene terpolymer

EVM vinyl acetate—ethylene copolymer

FKM fluoro polymethylene (rubber)

FVMQ fluoro silicone (rubber) with vinyl and methyl groups HNBR hydrogenated acrylonitrile butadiene (rubber)

NBR acrylonitrile butadiene (rubber)

VMQ silicone (rubber) with vinyl and methyl groups

X1.1.1 Special Commercial Acronym Not in D1418:

TPV special thermoplastic elastomer

X1.2 Part 2—Compound Formulations:

CR—(Cure: 38 min at 16	60°C)	EVM—(Cure: 9 min at 1	EVM—(Cure: 9 min at 177°C)			
Baypren 111	100.0	Levapren KA-8385	100.0			
Maglite D	4.0	Carnauba wax	2.0			
Stearic acid	0.5	Maglite D	1.0			
N-774 black	50.0	N-550 black	50.0			
Vulkanox OCD	2.0	Vulkanox DDA	1.0			
Kadox 911	5.0	Diak No. 7	1.5			
Vulkacit CRV	0.5	Vulcup 40 KE	6.0			
Total	162.0	Total	162.0			
EPDM—(Cure: 12 min a	t 166°C)	NBR—(Cure: 15 min at	170°C)			
Polysar EPDM 5465X	200.0	Nipol 1052	100.0			
Zinc oxide	5.0	Zinc oxide	5.0			

<sup>&</sup>lt;sup>B</sup>Mean % volume swell for all four oils.



Stearic acid	1.0	Stearic acid	1.0
N-330 black	80.0	Agerite Stalite S	1.5
Vulkacit Merkapto	0.5	N-550 black	40.0
Vulkacit Thiuram	1.0	Sulfasan R	0.3
Sulfur	1.0	TMTD	3.0
Total	288.5	Total	150.8
HNBR—(Cure: 30 min	at 170°C)	ECO—(Cure: 30 min at	170°C)
Zetpol 2010	100.0	Hydrin 2000	100.0
N-774 black	50.0	N-550 black	40.0
Kadox 911C	5.0	NBC	1.0
Stearic acid	0.5	Maglite D	3.0
Naugard 445	1.5	Span 80	2.0
Vanox ZMT1	1.0	Calcium carbonate	5.0
Vulcup 40 KE	8.0	Zisnet	1.0
Total	166.0	Total	152.0
		FKM—(Cure: 10 min at	177°C;
ACM—(Cure: 4 min at	190°C)	Post-Cure: 16 h at 232°	C)
Hytemp 4051 EP	100.0	Fluorel FC-2181	100.0
Stearic acid	2.0	N-990 black	30.0
Agerite Stalite S	2.0	Maglite D	3.0
TE-80	2.0	Calcium hydroxide	6.0
N-550 black	70.0	,	

Sodium stearate Hytemp NPC-50	4.0 2.0	Total	139.0		
Total	182.0	_			
AEM—(Cure: 10 min at 4 h at 175°C)	t 177°C; Post-Cure:	VMQ—(Cure: 15 min at 17	77°C)		
Vamac	100.0	GE-Silicone SE 6160	100.0		
Naugard 445	2.0	GE-Silicone SE 910MO	1.0		
Stearic acid	2.0	Varox DBPH50	0.8		
Armeen 18 D	0.5				
Vanfre VAM	1.0	Total	101.8		
N-550 black	60.0				
TP-759	5.0	FVMQ—(Cure: 10 min at 177°C; Post-Cure: 4 h at 200°C)			
DOTG	4.0				
Diak No. 1	1.5	Silastic LS-2880	100.0		
		 HT-1	1.0		
Total	176.0	Varox DBPH-50	1.0		
		Total	102.0		
TPV—Santoprene® Ru	bber 101-64				

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