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Standard Reference Elastomers (SRE) for Characterizing the Effect of Liquids on Vulcanized Rubbers

***Foreword*—**The development of these standard reference elastomers (SRE) is based on static and dynamic engine oil sealing applications for passenger cars and light duty trucks.

***1. Scope*—**This SAE Standard specifies requirements for vulcanized rubbers in sheet form for use as standards in characterizing the effect of test liquids and service fluids. The annexes contain the standard reference elastomer formulas.

The property changes of the SRE in contact with the indicated fluid under specified test conditions are the responsibility of the user. See 7.3 and Table 1.

This standard is not designed to provide formulations of elastomeric product compositions for actual service.

1.1 Safety—This standard may involve hazardous materials, operations and equipment. It does not address the safety concerns which may be associated with its use. It is the responsibility of any user of this standard to consult and establish appropriate health and safety practices, and determine the applicability of regulatory limitations before use.

2. References

2.1 Applicable Publications—The following publications form a part of this standard to the extent specified herein. The latest issue of all standards shall apply.

2.1.1 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 412—ASTM for Vulcanized Rubber and Thermoplastic Elastomers—Tension
ASTM D 471—STM for Rubber Property—Effect of Fluids
ASTM D 1349—Standard Practice for Rubber—Standard Temperatures for Testing
ASTM D 1418—Standard Practice for Rubber and Rubber Latices—Nomenclature
ASTM D 3182—Standard Practice for Rubber Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
ASTM D 3187—Standard Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)
ASTM D 3767—Rubber—Measurement of Dimensions
ASTM D 4678—Standard Practice for Rubber—Preparation, Testing Acceptance, Documentation, and Use of Reference Materials

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2.1.2 ISO PUBLICATIONS—Available from ANSI, 25 West 43rd St., New York, NY 10036-8002.

ISO 1629—Rubber and latices—Nomenclature
ISO 2230—Vulcanized rubber property—Guide to storage

3. SRE Composition

3.1 The recommended mixing procedures and specified properties are from the identified materials in the annexes.

3.2 The compounding and preparation ensure the property profile;

a. agrees sufficiently with the material group and applications it represents;
b. exhibits reasonable "sensitivity" to designated fluid additive and base stock changes;
c. is consistent for reliable reproducibility.

3.3 SRE compound materials must be readily available worldwide.

4. Summary of Preparation

4.1 Each compound shall be a homogeneous mix of all materials shown in the annexes, weighed to the accuracy required in ASTM D 3182.

4.2 The mixing of compounds and vulcanization of test sheets follow ASTM D 3182, modified to details given in each annex condition and procedure.

4.3 Approved Mixing and Distribution Facilities—See Appendix.

4.3.1 An SRE by definition is mixed only by approved designated facilities authorized to mix, and distribute these compounds. See 4.3.2.

4.3.2 APPROVED FACILITY—Akron Rubber Development Lab, 2887 Gilchrist Road, Akron, OH 44305.

4.3.3 Labs can mix approved SRE formulations for internal use only.

5. Requirements and Testing

5.1 Sheet Dimensions—The vulcanized sheets meet ASTM D 3182 figure 1 mold cavity dimensions measured to ASTM D 3767.

5.1.1 All sheets are tested for thickness. One test sheet from each lot (all sheets from a single batch vulcanized under the same conditions) is tested for the specified properties for compliance with the tolerances given in the annexes.

5.2 Sheet Appearance—The molded sheet must be free of any surface defects or internal voids observed with normally corrected vision.

5.3 Identification—Using mold marks or visible, durable ink, mark each sheet along one edge with the ASTM D 1418 / ISO 1629 nomenclature letters with SRE formulation number, mix number, lot number, and grain direction (arrow). Separate each item with a slash (/) mark (see Figure 1).

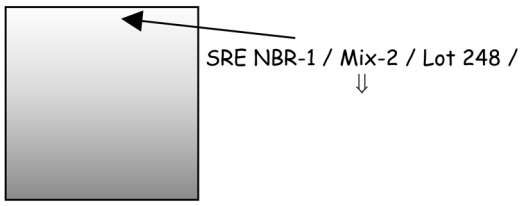

FIGURE 1—IDENTIFICATION PROTOCOL OF TEST SLABS EXAMPLE

5.4 Lot Testing—A sufficient number of molded test sheets from each lot (all sheets from a single batch vulcanized under the same conditions) is tested for compliance to specified properties using the appropriate test (see item 7.1).

5.4.1 Each shipped lot will include the approved lab(s) certification that the material complies with its annex requirements.

5.4.2 Test specimens are cut 15mm in from the sheet edge, with the longitudinal axis parallel to the sheet grain direction.

5.4.3 Test 5 specimens for each physical property, and 5 specimens for volume change.

6. Storage

6.1 Test sheets are stored in accordance to ISO 2230. After one year they are retested or discarded.

6.2 When the composition of any SRE is revised or changed, authorized lab(s) shall not discard stored test sheets for that SRE until new changes are approved by SAE Committee on Automotive Rubber Specifications (CARS).

6.3 In the event SRE changes are made, do not distribute the previous SRE formulation, except for comparative purposes. Authorized lab(s) will phase in the revised SRE when changes are approved.

7. Application

7.1 Development of SRE—Candidate selection (sensitivity) is based on percent change per ASTM D 412, die C, tensile strength, elongation at break, and tensile stress at 50%. ASTM D 471 percent volume change rounds out the requirements. Hardness measurements may be made but evidence from the Inter Lab Test program indicates that hardness is much less sensitive to differences in "rubber stiffness" compared to 50% modulus.

7.1.1 Test conditions reflect OEM engine oil system requirements and the material classification limits. Test temperature conditions follow ASTM D 1349 practice. To achieve equilibrium, immerse test specimens 168 h or longer (as designated).

7.1.2 For each material classification, the tester should be aware of the effect of aeration on a candidates physical properties during immersion testing. Use the fluid treatment (static or aerated) most representative of the intended use. Appendix tables are based on static immersion.

7.2 Test Fluid—Use ASTM Service Fluid 105 to evaluate SRE candidate selection and establish a property loss baseline for engine oil applications using the properties in 7.1. Service Fluid 105 is available from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206.

7.3 Appendices A-J—Appendices A-J gives all formulations for the selected SRE's of this standard. Also included in the appendices is a Summary Table for SRE properties. Part 1 contains delta or percent change in property values for each SRE after immersion for 168 hr in ASTM Service Fluid 105 at temperatures as specified in Table 1. Part 2 contains the original properties for each SRE prior to immersion. The summary table results were obtained in an interlaboratory test program with all of the SREs as listed in the annex and table, using data from eight typical industry laboratories. Prior to the final analysis as indicated in the Summary Table, outlier values were deleted.

7.3.1 The interlaboratory test program did not contain a sufficiently large database to obtain interlaboratory standard deviations based on 20 or more degrees of freedom, DF. Thus the value equivalent to the classical or standard (±) 3 sigma limit (at the DF applicable to each SRE), is defined in the table as t*SDev, where t is the tabulated 95% confidence or p=0.05 level t value at the DF applicable to the standard deviation, SDev, as evaluated in the test program.

TABLE 1—SRE TEST TEMPERATURE

SRE	Temperature (°C)
NBR-1, NBR-2, ECO-1	100
All others	150

7.4 Appendix K—Appendix K provides protocol for creation, maintenance and control of formulations for the selected SRE's of this standard.

PREPARED BY THE SAE ENGINE OIL COMPATABILITY WITH RUBBER GASKETS AND SEALS SUBCOMMITTEE OF THE SAE COMMITTEE ON AUTOMOTIVE RUBBER STANDARDS

APPENDIX A

A.1 Standard Reference Formulas and Procedures

A.1.1 Nitrile Rubber

A.1.1.1 Standard formulas for the following nitrile rubber compounds are given in Tables A2 and A3:

TABLE A1—NBR CURE TYPES

Number	Type
NBR-1	sulfur-cured
NBR-2	peroxide-cured

A.1.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

A.1.1.3 RECOMMENDED STANDARD TENSILE SHEET CURES—Cure NBR-1 specimen 10 minutes at 168 °C [335 °F]. Cure NBR-2 specimen 10 minutes at 182 °C [360 °F].

A.1.1.4 COMPOSITION

TABLE A2—COMPOSITION OF SRE NBR-1 (28/S)

Ingredient	Parts by mass
NBR with 28 ± 0.5% by mass of acrylonitrile(1)	100.0
Zinc oxide(2)	5.0
Stearic acid, triple press(3)	2.0
STANGARD 500(4)	2.0
Carbon black N774	70.0
PLASTHALL 7050(5)	5.0
SPIDER Sulfur(6)	0.5
N-Cyclohexyl-2-benzothiazolesulphenamide (CBTS)	1.0
Tetramethyl thiuram disulfide (TMTD)	1.0
Tetraethyl thiuram disulfide (TETD)	1.0
Total	187.5

1. NIPOL DN2850, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 920C, activator, Zinc Corp. of America. No equivalent.
3. C.P. Hall.
4. Blend from Harwick standard.
5. Monomeric plasticizer, C. P. Hall Co. No equivalent.
6. SPIDER S, C.P. Hall. No equivalent.

TABLE A3—COMPOSITION SRE NBR-2 (28/P)

Ingredient	Parts by mass
NBR with 28 ± 0.5% by mass of acrylonitrile[1]	100.0
Zinc oxide[2]	5.0
Stearic acid, triple press[3]	2.0
STANGARD 500[4]	2.0
Carbon black N774	70.0
PLASTHALL 7050[5]	
Dicumyl peroxide, 40% active MA carrier[6]	
	187.0

1. NIPOL DN2850, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 920C, activator, Zinc Corp. of America. No equivalent.
3. C.P. Hall.
4. Blend from Harwick standard.
5. Monomeric plasticizer, C. P. Hall Co. No equivalent.
6. DICUP 40 KE Hercules. Burgess clay carrier. No equivalent.

APPENDIX B

B.1 Polyacrylate Rubber

B.1.1 Standard formulas for the following polyacrylate rubber compounds are given in Table B2:

TABLE B1—ACM CURE TYPE

Number	Type
ACM-1	sodium stearate-cured

B.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

B.1.3 Recommended Standard Tensile Sheet Cures—Cure ACM-1 specimen 10 minutes at 168 °C [335 °F].

B.1.4 Composition

TABLE B2—COMPOSITION OF SRE ACM-1

Ingredient	Parts by mass
Acrylic rubber(1)	100.0
Stearic acid	1.0
NAUGARD 445(2)	2.0
Carbon black N550	80.0
Sodium stearate	4.0
conc. fatty acid ester(3)	2.0
NPC-25(4)	4.0
Total	191.0

1. HYTEMP 4052, Zeon Chemicals, Inc. No equivalent known.
2. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
3. Process aid, Strukol WB222. No equivalent.
4. Vulcanizing agent, Zeon Chemicals, Inc. No equivalent.

APPENDIX C

C.1 Epichlorohydrin Rubber

C.1.1 Standard formulas for the following epichlorohydrin rubber compounds are given in Table C2:

TABLE C1—ECO CURE TYPE

Number	Type
ECO-1	ETU-cured

C.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

C.1.3 Recommended Standard Tensile Sheet Cures—Cure ECO-1 specimen 25 minutes at 191 °C [375 °F]. Post cured 4 h at 177 °C [350 °F].

C.1.4 Composition

TABLE C2—COMPOSITION SRE ECO-1

Ingredient	Parts by mass
Hydrin C2000L[1]	100.0
Carbon black N550	70.0
TP95[2]	10.0
Stearic acid	1.0
4- and 5-methylmercaptobenzimidazole[3]	0.5
NAUGARD 445[4]	1.0
MAGLITE D	3.0
GND-75 (ETU) [5]	1.0
Total	186.5

1. HYDRIN C2000L, Zeon Chemicals, Inc. No equivalent known.
2. Plasticizer, Rohm & Haas. No equivalent.
3. VULKANOX MB-2/MG antioxidant, Bayer AG. No equivalent.
4. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
5. Ethylene thiourea, accelerator, 75% active.

APPENDIX D

D.1 Ethylene Acrylic Rubber

D.1.1 Standard formulas for the following ethylene acrylic rubber compounds are given in Table D2:

TABLE D1—AEM CURE TYPE

Number	Type
AEM-1	DOTG- diamine cured

D.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

D.1.3 Recommended Standard Tensile Sheet Cures—Cure AEM-1 specimen 10 minutes at 177 °C [350 °F] Post cure: 4h at 177 °C [350 °F].

D.1.4 Composition

TABLE D2—COMPOSITION SRE AEM-1

Ingredient	Parts by mass
VAMAC® G(1)	100.0
NAUGARD 445(2)	2.0
Stearic acid	1.5
ARMEEN 18D	0.5
VANFRE VAM(3)	1.0
FEF Carbon black N550	60.0
DIAK #1(4)	1.5
di-o-tolylguanidine (DOTG)	4.0
Total	170.5

1. VAMAC® a registered tradename for ethylene/acrylic rubber from DuPont. No equivalent known.
2. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
3. Organic phosphate ester free acid, processing aid, R. T. Vanderbilt Co., Inc. No equivalent.
4. Accelerator, DuPont. No equivalent.

APPENDIX E

E.1 Hydrogenated Nitrile Rubber

E.1.1 Standard formulas for the following hydrogenated nitrile rubber compounds are given in Table E2:

TABLE E1—HNBR CURE TYPE

Number	Type
HNBR-1	peroxide-cured

E.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

E.1.3 Recommended Standard Tensile Sheet Cures—Cure HNBR-1 specimen 10 minutes at 177 °C [350 °F]. Post-cure none.

E.1.4 Composition

TABLE E2—COMPOSITION OF SRE HNBR-1

Ingredient	Parts by mass
HNBR with 36 ± 0.5% by mass of nitrile groups and £5% of residual double bonds[1]	100.0
Zinc oxide[2]	5.0
Stearic acid	0.5
NAUGARD 445[3]	1.5
Zinc 2-mercapto-toluimidazole[4]	1.0
Carbon black N774	50.0
PLASTHALL TOTM[5]	5.0
40% a,a'-bis-(t-butyl peroxy) diisopropylbenzene on Burgess KE Clay[6]	8.0
Total	171.0

1. ZETPOL 2010, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 911C, activator, Zinc Corp. of America. No equivalent.
3. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
4. VANOX ZMTI, antioxidant, R. T. Vanderbilt Co. No equivalent.
5. PLASTHALL, monomeric plasticizer, C. P. Hall Co. No equivalent.
6. VULCUP 40KE from Hercules. No equivalent.

APPENDIX F

F.1 Silicone Rubber

F.1.1 Standard formulas for the following silicone rubber compounds are given in Table F2:

TABLE F1—VMQ CURE TYPE

Number	Type
VMQ-1	peroxide-cured

F.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

F.1.3 Recommended Standard Tensile Sheet Cures—Cure VMQ-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: none.

F.1.4 Composition

TABLE F2—COMPOSITION VMQ SRE-1

Ingredient	Parts by mass
vinyl methyl silicone base polymer(1)	40.0
vinyl methyl silicone base polymer(2)	60.0
HT-1(3)	1.0
Magnesium oxide	3.0
2,5-dimethyl-2,5-di (tertbutylperoxy) hexane(4)	1.0
Total	105

1. Dow Corning Q4-4758. Base. No equivalent known.
2. Dow Corning Q4-4768. Base. No equivalent known.
3. Thermal stabilizing additive, Dow Corning. No equivalent.
4. Vulcanizing agent, VAROX DBPH-50, R. T. Vanderbilt Co., Inc. or equivalent.

APPENDIX G

G.1 Fluorosilicone Rubber

G.1.1 Standard formulas for the following fluorosilicone rubber compounds are given in Table G2:

TABLE G1—FVMQ CURE TYPE

Number	Type
FVMQ-1	peroxide-cured

G.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

G.1.3 Recommended Standard Tensile Sheet Cures—Cure FVMQ-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 4h at 200 °C [392 °F].

G.1.4 Composition

TABLE G2—COMPOSITION FVMQ SRE-1

Ingredient	Parts by mass
Fluoro-vinyl methyl silicone base polymer(1)	100.0
HT-1(2)	1.0
2,5-dimethyl-2,5-di (tertbutylperoxy) hexane(3)	1.0
Total	102

1. Dow Corning Silicones LS-2860. No equivalent known.
2. Thermal stabilizing additive, Dow Corning. No equivalent.
3. Vulcanizing agent, VAROX DBPH-50, R. T. Vanderbilt Co., Inc. or equivalent.

APPENDIX H

H.1 Fluorocarbon Rubber

H.1.1 Standard formulas for the following fluorocarbon rubber compounds are given in Table H2:

TABLE H1—FKM CURE TYPE

Number	Type
FKM-1	bisphenol-cured

H.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

H.1.3 Recommended Standard Tensile Sheet Cures—Cure FKM-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 24h at 200 °C [392 °F].

H.1.4 Composition

TABLE H2—COMPOSITION OF SRE FKM-1

Ingredient	Parts by mass
Fluorocarbon base polymer(1)	97.5
Calcium hydroxide	3.0
Magnesium Oxide(2)	6.0
N990 MT Carbon Black(3)	25
VC 20(4)	0.5
VC 50(5)	2
VPA-1 (6)	1
Total	135.0

1. Viton B600 from DuPont Dow Elastomers. No equivalent.
2. Elastomag 170 from Akrochem. No equivalent.
3. Thermax Flowform from Cancarb, Ltd. No equivalent.
4. Curative from DuPont Dow Elastomers. No equivalent
5. Curative from DuPont Dow Elastomers. No equivalent.
6. Processing aid from DuPont Dow Elastomers. No equivalent.

APPENDIX I

I.1 Copolymer of Tetrafluoroethylene and Propylene Rubber

I.1.1 Standard formulas for the following fluorocarbon rubber compounds are given in Table I2:

TABLE I1—FEPM CURE TYPE

Number	Type
FEPM-1	peroxide-cured

I.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

I.1.3 Recommended Standard Tensile Sheet Cures—Cure FEPM-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 24 hrs at 232 °C [450 °F].

I.1.4 Composition

TABLE I2—COMPOSITION SRF-FEPM-1

Ingredient	Parts by mass
Tetrafluoroethylene-propylene copolymer(1)	100.0
N990 MT Carbon Black(2)	15.0
Calcium metasilicate(3)	10.0
Sodium Stearate	1.0
Triallyl isocyanurate (TAIC)(4)	4.0
40% a,a'-bis-(t-butyl peroxy) diisopropylbenzene on Burgess KE Clay(5)	4.0
Total	134.0

1. AFLAS 100S Polymer from Asahi Glass, Japan, distributed by 3M/dyneon. No equivalent.
2. Thermax Flowform from Cancarb, Ltd. No equivalent.
3. Nyad 400 from Nyco Inc. No equivalent.
4. DIAK #7 from DuPont Dow Elastomers. No equivalent.
5. VULCUP 40KE from Hercules. No equivalent.

APPENDIX J

J.1 Summary Table

Delta Elongation, % for Lab Averages ; Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-6.9	-18.8	-50.6	-9.8	-54.1	-35.3	-14.9	-23.1	-25.5	-76.9
Min =	Avg - t*SDev		-13.9	-22.8	-59.2	-19.0	-59.8	-48.8	-21.8	-35.7	-32.3	-81.3
Max =	Avg + t*SDev		-0.04	-14.73	-41.97	-0.54	-48.35	-21.77	-8.01	-10.46	-18.80	-72.57
		Range	13.8	8.1	17.3	18.5	11.4	27.0	13.7	25.3	13.5	8.7

Delta 50% Modulus, % , for Lab Averages ; Outliers deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-8.8	-15.7	25.5	-19.6	7.5	6.4	-4.9	-25.3	32.5	
Min =	Avg - t*SDev		-39.7	-23.0	11.5	-37.7	-13.7	-7.1	-9.6	-40.1	18.7	
Max =	Avg + t*SDev		22.1	-8.4	39.4	-1.5	28.7	19.9	-0.2	-10.4	46.4	
		Range	61.8	14.6	28.0	36.1	42.4	27.0	9.3	29.7	27.7	

Delta Tensile Strength, % , for Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-2.78	-19.9	-56.4	-11.7	-33.1	-33.2	-4.00	-25.3	-20.6	-44.1
Min =	Avg - t*SDev		-12.5	-24.7	-63.2	-25.1	-59.0	-46.5	-15.9	-41.3	-33.4	-53.4
Max =	Avg + t*SDev		6.9	-15.1	-49.6	1.8	-7.1	-19.9	7.9	-9.2	-7.7	-34.8
		Range	19.4	9.6	13.5	26.9	52.0	26.7	23.9	32.1	25.7	18.6
		Avg	-2.8	-19.9	-56.4	-11.7	-33.1	-33.2	-4.0	-25.3	-20.6	-44.1

Delta Hardness; % for Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-7.8	-16.1	3.2	-7.0	-1.8	-1.8	-5.0	-31.6	11.0	10.3
Min =	Avg - t*SDev		-16	-21	1	-9	-12	-7	-7	-36	3	7
Max =	Avg + t*SDev		1	-11	6	-5	9	4	-3	-28	19	14
		Range	17	11	5	4	21	11	3	8	16	6

Percent Volume Swell ; Lab Averages - Outliers deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	2.00	24.5	0.60	8.73	4.42	5.51	4.07	30.7	-0.18	-0.0
Min =	Avg - t*SDev		0.4	22.0	-0.0	8.0	2.3	3.7	3.5	26.5	-1.3	-2.0
Max =	Avg + t*SDev		3.4	26.6	1.1	9.4	6.4	7.2	4.6	34.0	1.0	2.0
		Range	2.9	4.6	1.2	1.4	4.1	3.5	1.1	7.5	2.3	4.1

FIGURE J1A—SUMMARY TABLE PART 1: CHANGE (DELTA) IN PROPERTIES AFTER IMMERSION