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Standard Test Method: Test Method for Determining Automotive Engine Oil Compatibility with Typical Seal Rubbers and Elastomers"

Introduction

This test method standard calls for the use of reference immersion oils and reference test materials (rubber or elastomer compounds) that are used in the evaluation of candidate oils or candidate compounds. Most of the reference materials of all types used in various in Committee D11 standards are materials that have been evaluated and accepted by the use of D4678, " Preparation, Testing, Acceptance, Documentation and Use of Reference Materials". These D4678 materials are designated as Industry Reference Materials or IRMs.

For a number of reasons the reference materials of this engine oil compatibility standard were not able to be evaluated by the procedures of D4678 and therefore do not bear the name IRM. The reference materials of this engine oil standard are offered for use in the evaluation of candidate rubbers, elastomers and oils on an ad hoc basis.

1. Scope -

1.1 Effective sealing action requires that the rubber or elastomer compound used for any seal have a high level of resistance to the liquid or oil in which it is immersed. When a high level of resistance exists, the rubber is said to be compatible with the liquid or oil.

1.2 This standard provides test method procedures for a *preliminary* or first order evaluation of the compatibility of (1) oils classified as Automotive Engine Oils and (2) rubbers or elastomers used in the sealing materials in contact with these oils. Since seals may be static or dynamic and may operate over a range of conditions, a complete evaluation of the potential sealing performance of any rubber-oil combination in any service condition usually requires additional tests.

1.3 This test method may be used to determine the compatibility of rubbers and elastomers such as nitrile (NBR), polyacrylate (ACM), fluoroelastomer (FKM) and silicone (VMQ) with Automotive Engine oil. Other candidate rubbers and elastomers as proposed for use in conjunction with any candidate Automotive Engine oil may also be evaluated.

1.4 The testing procedures as described in D412, on stress strain evaluation, in D471, on the effect of rubber immersion in liquids, in D865 on the use of special test tubes for aging evaluation, in D2240, on the measurement of hardness and in D5662, gear oil compatibility with typical oil seal rubbers and elastomers, are all used in the execution of the operations of this standard. The user of this standard should be proficient in the use of these additional cited standards.

1.5 (Standard ASTM safety boilerplate text here)

2. Referenced Documents

2.1 ASTM Standards

D412 - Standard Test Method - Rubber Properties in Tension
D471 - Standard Test Method - Rubber Property - Effect of Liquids
D1349 - Standard Practice - Rubber: Standard Temperatures for Testing
D2240 - Standard Test Method - Rubber: Durometer Hardness
D5662 - Standard Test Method - Determining Automotive Gear Oil Compatibility with Typical Oil Seal Elastomers
D865 - Rubber - Deterioration by Heating in Air (Test Tube Enclosure)

3. Terminology

3.1 *Description of Terms Specific for this Standard* - These terms are defined in a sequential order; the simple or more basic definitions are defined first then the more complex terms. The basic terms are then used as a part of later definitions. This produces the most meaningful and succinct definitions.

3.1.1 *compatibility*, n. - a characteristic of a rubber or elastomer 'compound-liquid' combination that signifies a complete or high level of resistance of the compound, to deleterious effects imparted by contact with or immersion in, the liquid.

Discussion: The phrase 'high compatibility' indicates that after contact or immersion, the compound properties are maintained at or near their original level.

3.1.2 *immersion test*, n. - an operation to evaluate compatibility; it determines the effect of a liquid on rubber or elastomer compound test specimens maintained beneath the surface of the liquid for a specified time and temperature.

Discussion: The effect of the liquid is evaluated by the difference in (typical) compound physical test properties before and after immersion.

3.1.3 *reference compound*, n. - a compound prepared using a specified rubber or elastomer (formulation) that has well established immersion test properties with selected oils or liquids, obtained by the use of recognized and accepted testing and documentation procedures.

3.1.4 *reference oil*, n. - an immersion liquid that has well-established properties, obtained by the use of recognized and accepted testing and documentation procedures.

Discussion: The 'established properties' may be chemical or compositional properties or properties as developed from immersion tests using reference compounds.

3.1.5 *candidate compound*, n. - a non-reference or experimental rubber or elastomer compound to be evaluated for compatibility with reference or candidate oils or liquids.

3.1.6 *candidate oil*, n. - a non-reference or experimental oil or liquid to be evaluated for its effect on (compatibility with) reference compounds or experimental compounds.

3.1.7 *Tensile strength, n* – the maximum tensile stress applied in stretching a specimen to rupture.

4. Summary of the Test Method

4.1 Rubbers and elastomers immersed in selected oils are aged at a selected temperature specific for each oil - rubber or elastomer combination, for 14 days or 336 hrs. The performance of the rubber or elastomer is determined by its resistance to change in typical physical properties after immersion such as; stress-strain, elongation at break, tensile strength, hardness (Durometer Type A), and dimensional properties (volume change). The results of the immersion testing are usually expressed in terms of the percent change in the selected properties, (after immersion - before immersion). A negative percent change indicates a reduction in property level.

4.2 Both reference oils and candidate or experimental oils may be evaluated. When comparisons among any series of oils are to be made, the test conditions for all oils (reference and candidate) shall be well controlled especially the concurrent immersion test aging, in the same temperature controlled heating device.

5. Significance and Use

5.1 Engine oil formulations have been shown to lack compatibility (cause deleterious effects) with certain rubbers and elastomers. These deleterious effects are greatest under two conditions (1) with virgin or new engine oil, i.e.. oil that has not been exposed to an engine's operating environment and (2) when the exposure is at elevated temperatures.

5.2 This method evaluates the relative effects of new or candidate engine oil formulations using four reference compounds as cited in the Scope. See also Annex A2. The performance of the new engine oils is determined by a comparison of the reference compound immersion test data for the candidate oils with the reference compound immersion test data for the reference oils. This comparison permits decisions on the anticipated or predicted performance of the candidate oils in service. The standard also permits an evaluation of candidate compounds with either reference or candidate oils or liquids. This comparison permits decisions on the anticipated or predicted performance of the candidate compounds in service.

5.3 This test method is suitable for specification compliance testing, quality control, referee testing and research and development.

6. Apparatus

6.1 The testing equipment as specified in D412, D471, D865, D2240 and D5662 is required for the use of this standard.

6.1.1 *Hardness Durometer* - See D2240.

6.1.2 *Tension Testing Machine* - See the appropriate sections of D412. The rate of grip separation for the tension testing shall be 500 +/- 50 mm per min (8.5 +/- 0.8 inches per min).

6.1.3 *Glass tubes*, of borosilicate glass if possible, shall be used, having an outside diameter of 38 (+0, -1 mm) and an overall length of 300 mm. Each tube is fitted loosely with an aluminum-covered cork stopper or an equivalent inert sealing device that will not contaminate the immersion oil. An inert (stainless steel) wire rod is hung over the edge of the glass tube and held in place by the stopper. It is used to hold the test specimens submerged in the immersion liquid. See Figure 1 and 2 and see D865, sec 5.

6.1.4 *A Heated Immersion Test Bath or Block,* or equivalent shall be used. This heating device should be capable of maintaining the oil sample in the glass tube within +/- 1 deg C of the test temperature. The

immersion test bath or block shall contain a rack or holes which will accept the glass tubes specified in 6.1.3 and hold them in a vertical position.

6.1.5 D*umbbell cutting die* should use that referenced in ASTM D-412. Die C is required.

7. Reference Materials

7.1 *Reference Oils* - The reference oils are maintained and distributed by the ASTM Test Monitoring Center (TMC), see Annex A1. .

7.1.1 The TMC is responsible for managing a system that ensures the performance and formulation consistency of the reference oils. Reference oils shall be stored in locations where the ambient temperature does not exceed 32 deg C. Under these conditions the shelf life of the reference oils is five years unless otherwise specified by the TMC using documented analysis procedures for a longer projected shelf life.

7.2 *Reference Compounds* - Reference compounds are available from an organization known as the Parts Distributor (PD), see Annex A1. The four reference compounds are (1) a fluoroelastomer (FKM), (2) a polyacrylate material (ACM), (3) a silicone rubber (VMQ) and (4) a nitrile rubber (NBR). See Annex A2 for formulations. A numbering system has been established by the PD of the format: [type] X; where 'type' is either (FKM), (ACM), (VMQ) or (NBR). and X = batch number for the particular formulation. The physical properties as reported in Annex A2 are typical values.

7.2.1 The PD is responsible for (1) maintaining the numbering and tracking system for the reference compounds and (2) for managing the procurement of rubbers and elastomers that meet the specifications of this standard. The reference compounds shall be stored in a location shielded from light, where the relative humidity is in the range 40 to 55% and the temperature in the range of 10 to 25 deg C. Under these conditions the shelf life of the reference compounds is three years from the date of cure. Any immersion test using a reference compound older than three years shall be treated with caution.

7.3 Table 1 lists the reference compounds, the specified immersion test temperatures and the reference oils. As noted above these are available from the Parts Distributor and/or the Test Monitoring Center. See Annex A1.

8. Procedure for Immersion Testing

8.1 *General Background* - The immersion tests for any candidate oil (or oils) shall be conducted on the basis of a 'test series' operation. A test series is a complete evaluation program using the specified physical tests, for any selected number of candidate and reference oils and/or candidate (experimental) or reference compounds. The test results for the specified physical tests obtained for the reference compounds in the candidate oils shall be compared to the test results, for the specified physical tests, for the reference compounds in the reference oils. For this comparison insure that the same compound batch is used for both candidate and reference oils. Refer to Table 1 for the immersion test temperatures and reference oils for each reference compound.

8.2 *Number of Test Specimens* - There are two types of test specimens, (1) dumbbells for stress-strain testing and (2) 25 x 50 mm rectangular sheets for dimensional changes or volume swell and hardness testing. For the dumbbell specimens, twelve (12) test specimens shall be prepared; six (6) for original property testing and six (6) for aged (after immersion) testing. For the rectangular specimens, six (6) test specimens should be prepared; the same specimen is used for original property testing and for aged (immersion) testing. The total number of specimens of both types is determined by the scope of the

testing; the number of candidate compounds or candidate oils to be evaluated and the number of selected reference compounds or reference oils in the test series. The assessment of the total number of specimens is based on the use of the standard to evaluate both candidate compounds as well as candidate oils. It is important that all reference compounds in one test set be from the same batch.

8.3 *Test Specimen Preparation* - Using the total number of specimens as required for each candidate or reference compound for the entire test series as determined in 8.2. , select the number of sheets for each compound as required for the projected testing. This will depend on the number of oils to be used for immersion. Condition the sheets from which test specimens are to be cut for 3 hr at 23 +-2 deg C as specified in D412.

8.3.1 *Stress-Strain Testing* - For each candidate or reference compound, cut the required number of dumbbell specimens from the sheets. This cutting shall be with Die C as specified in D412, parallel to the grain, using sharp well prepared dies that are unaltered though out the entire cutting operation. Use a die press for the operation to cut only one sheet thickness for all cutting operations. Since two or more sheets will be required for the total number of dumbbells for any compound, it is necessary that each sample of six dumbbells (original and aged sample sets) contain as close as possible an equal number of dumbbells from each of the individual sheets as required for the testing.

8.3.2 *Dimensional Properties (Volume Swell)* - As specified in D471, cut the number of required number of 25 x 50 x 2.0 +- 0.1 mm (1.0 x 2.0 x 0.08 +- 0.005 inch) specimens from the sheets. Since two or more sheets will be required (to prepare at least 12 specimens) for the total number of specimens for any compound, it is necessary that each sample of six (original and aged sample sets) contain an equal number of specimens from each of the individual sheets as required for the testing.

8.3.3 *Hardness Testing* - The hardness testing is conducted on the specimens prepared for the volume swell testing.

8.4 *Physical Testing: Initial Measurements* - Conduct the initial or original physical testing measurements using six of the prepared test specimens. Measurements that must be recorded include: (1) Initial and final (after immersion) modulus, elongation at break and tensile strength, (2) initial and final (after immersion) mass of 25 x 50 specimens in air and water (or other liquid used for the weighing operation) and (3) initial and final (after immersion) hardness using the 25 x 50 specimens.

8.4.1 *Stress-Strain* - Using the procedure as specified in D412, test six dumbbells, recording for each (1) the elongation at break and (2) the maximum tensile stress (tensile strength) applied to the specimen in stretching the specimen to break. Calculate and record the original average and standard deviations of the six measurements for each of the two properties.

8.4.2 *Dimensional Properties: Original (Initial) Mass* - Use the water displacement method as described in D471 to conduct the initial mass (weight) measurements for the 25 x 50 mm specimens. Weigh each specimen in air to the nearest 1 mg. This original mass is recorded as M_1. Immerse the specimen into a 1% solution of Aerosol OT in water before weighing in distilled water, insuring that there are no air bubbles clinging to the specimen. Record this original 'in water' mass as M_2.

8.4.3 *Hardness* - Using a Type A Durometer as described in D2240, stack three of the 25 x 50 mm specimens on top of each other for a 6 mm thickness. Take three readings from the topside of the stacked specimens rotate the top specimen over and take additional three readings from the bottom side of the top specimen. The readings are taken 1 sec after the pin make s contact with the rubber.

8.4.3.1 After this set of 6 measurements, rotate the bottom specimen to the top and the top specimen to the bottom. Take another set of 6 readings as above.

8.4.3.2 After this set of 6 measurements, rotate the bottom specimen to the top and the top to the bottom. Take another set of 6 readings as above. Record all 18 measurements from this stack of three specimens.

8.4.3.3 Repeat the set of 18 hardness readings for the second set of 3 rectangular specimens. Calculate an average for the original hardness from all 36 readings.

8.5 *Initiating the Immersion Tests* - All immersion tests for a test series, shall be conducted concurrently in the same heated immersion test block.

8.5.1 Fill the immersion test tubes with 150 +- 5 cm^3 of the candidate or reference oil as appropriate. Four test tubes are required for each compound - oil combination. In each tube suspend three rectangular specimens or three dumbbell specimens from a stainless steel wire hanger, see Figure 1. Inert (to oil or rubber) spacers should be located between each test specimen to prevent specimens from touching each other or the test tube wall. See Figure 2. Each test tube shall be covered with a stopper as specified in 6.1.3.

8.5.2 Insert the test tubes into the heating block that has been set to the temperature required for the evaluation, on a random basis and age for a period of 336 +- 0.5 hrs. Insure that no specimen touches another specimen or the test tube wall. Such an occurrence will invalidate the test. To insure that aging conditions are equal for candidate and reference oil tests, the final endpoint of the 336 hr aging for either oil should be the same with an allowed tolerance or difference of 8 hrs.

8.6 *Terminating the Immersion Tests* - At the end of the aging period, remove the specimens form the test tubes and place them (while on the wire hanger) on a clean absorbent towel or surface. Allow for a cooling period while on the hanger of no more than 30 minutes.

8.6.1 Remove the specimens from the wire hanger and place them on a new clean absorbent towel or surface. Remove the excess oil with a clean absorbent towel. Begin the 'after immersion' testing approximately 30 to 60 minutes after removal from the test tube. Complete the final 'after immersion' testing within 2 hrs from time of removal from the test tube.

8.7 *Physical Testing: After Immersion Measurements* - The response of the immersed compound test specimens to the effects of the oil or liquid is assessed on the basis of the change in properties, before immersion Vs after immersion, expressed as a percent with the exception of hardness which is expressed in points. Using the procedures as set forth in 8.4.1, 8.4.2 and 8.4.3 repeat all of the stress-strain, dimensional property mass measurements (in air and water or other suitable liquid) and hardness measurements. Record these measurements as described above in 8.4. It is suggested that the hardness measurement be taken before the volume measurement to insure that the Durometer tip is kept free of moisture.

8.8 *Percent Change in Properties* - The percent change in stress strain properties is given by Equations 1, and 2 for elongation at break and tensile strength. The percent change in the volume of the test specimen is given by Equation 4 and the percent change in hardness is given by Equation 5.

$$\%\Delta E = [(E_A - E_O) / E_O] 100 \qquad (1)$$

$\%\Delta E$ = percent change in elongation at break
E_O = original elongation at break

E_A = aged (after immersion) elongation at break

$$\% \Delta TS = [(TS_A - TS_O) / TS_O] 100 \quad (2)$$

$\% \Delta TS$ = percent change in tensile strength
TS_O= original tensile strength
TS_A = aged (after immersion) tensile strength

Equation 3 is given for test specimen weighing in air and in water, which has a density of 1.00, for liquids other than water use alternative Equation 3A.

$$\% \Delta V = [\{ (M3 - M4) - (M1 - M2) \} / (M1 - M2)] 100 \quad (3)$$

$\% \Delta V$ = percent change in volume
M1 = original mass in air, g
M2 = original mass in water, g
M3 = mass in air after immersion test, g
M4 = mass in water after immersion test, g

$$\% \Delta V = [(M3 - M1) / \{ d (M1 - M2) \}] 100 \quad (3A)$$

M1 = original mass in air, g
M2 = original mass in liquid, g
M3 = mass in air after immersion test, g
d = density of liquid, g/cm^3

$$\Delta H = (H_A - H_O) \quad (4)$$

ΔH = point change in hardness
H_O= original hardness
H_A = aged (after immersion) hardness

9. Report

9.1 For each of the percent change in property parameters and point change for hardness as evaluated in 8.8, calculate the average and standard deviation. Appendix X1 contains recommended data forms to report the results of the immersion testing in the evaluation of candidate oils. These may be modified (1) for use with compounds other than the four reference compounds as listed and (2) for use in evaluating candidate rubber or elastomer compounds with selected reference oils or candidate oils.

9.2 The following data and information should be in the report for each liquid or oil evaluated:

9.2.1 Rubber or elastomer (batch, date and code), test date, test number
9.2.2 Test temperature, deg C
9.2.3 Test duration, hrs
9.2.4 Percent volume change (D471)

9.2.5 Change in Durometer hardness, points (D2240)
9.2.6 Percent change in tensile strength, (D412)
9.2.7 Percent change in elongation at break, (D412)
9.2.8 Aging block or bath identification
9.2.9 If requested, report to the TMC the data and information on the reference materials from the test

10. Precision and Bias

10.1 The precision and bias for this method has not been evaluated. A program to evaluate the precision will be undertaken as soon as possible after the adoption of this standard.

11. Key Words

11.1 compatibility, rubber, elastomer, automotive oil, heavy duty engine oil, seal,

Annex A1

A1.1 *Reference Materials* - Reference oils may be obtained from the ASTM Test Monitoring Center or TMC located at 6555 Penn Avenue, Pittsburgh PA 15206; phone 412-365-1010; Fax 412-365-1047. In order to receive reference oils individual laboratories shall agree to furnish the TMC with immersion test result data developed with these reference oils

Note: Unless otherwise specified, these oils are not Committee D11 IRMs.

A1.2 *Reference Rubbers or Elastomers* - Cured prepared sheets of reference rubbers or elastomers may be obtained from an organization known as the Parts Distributor or PD. The company currently functioning in this capacity is; OH Technologies Inc., Attn Jason Bowden, PO Box 5039, 9300 Progress Parkway, Mentor OH 44060; phone 440-354-7007, Fax 440-354- 7080; email: jhbowden@ohtech.com

Note: Unless otherwise specified, these oils are not Committee D11 IRMs

Annex A2

Elastomer materials, formulations, and expected properties of cured materials:

Elastomer	Ingredients	Parts	Points Hardness	Tensile, Mpa	Elongation, %	Specific Gravity
Fluoroelastomer (FKM)	Viton A-275C or Fluorel FC-2123	100.00	71	13.3 min	270	1.84
	Maglite D	3.00				
	N-990 Carbon Black	30.00				
	Calcium Hydroxide - Reagent Grade	6.00				
	Press Cure:	10 min. @ 177°C				
	Post Cure:	16 hrs. @ 232°C				

	Viton is a registered trademark of Dupont Dow Elastomers, Fluorel is a registered trademark of 3M					
Polyacrylate (ACM)	HyTemp 4051 EP	100.00	66	11.9	175	1.31
	N-550 Carbon Black	65.00				
	Stearic Acid	1.00				
	Naugard 445	2.00				
	TE-80	2.00				
	Sodium Stearate	4.00				
	HyyTemp NPC-50	2.00				
	Press Cure:	12 min. @ 170°C				
	HyTemp is a registered trademark of Zeon Chemicals					
Silicone (VMQ)	Dow Corning Product ID.24122V-BLK					
	Cure:	vulcanized 5 minutes @ 370°F				
	Pots Cure:	4 hours @ 200°C				
Nitrile (NBR)	Nipol DN3350	100.00	68	19.6	290	1.25
	Zinc Oxide	5.00				
	Stearic Acid	2.00				
	Stangard 500	2.00				
	N-774 Carbon Black	70.00				
	Thiokol TP-95	5.00				
	Varox DCP40KE	3.00				
	Press Cure:	12min. @ 170°C				
	Nipol is a registered trademark of Zeon Chemicals					
Manufacturer shall mark each elastomer sheet to designate the direction of the grain of the material.						

Figures

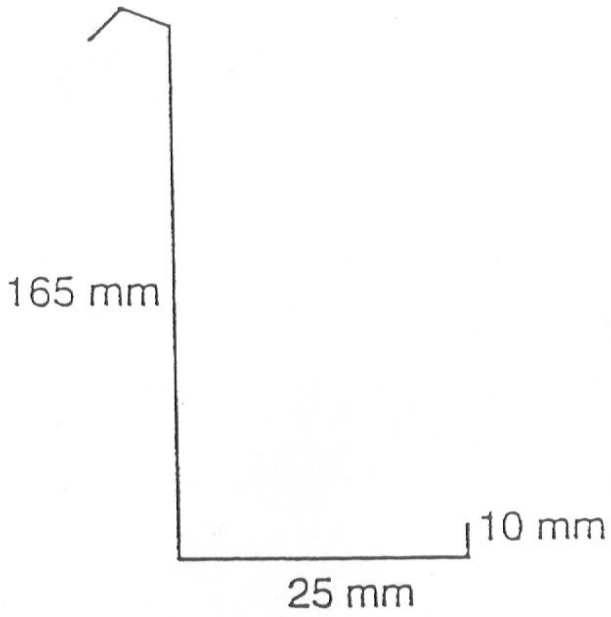

FIG. 1 Wire Hanger

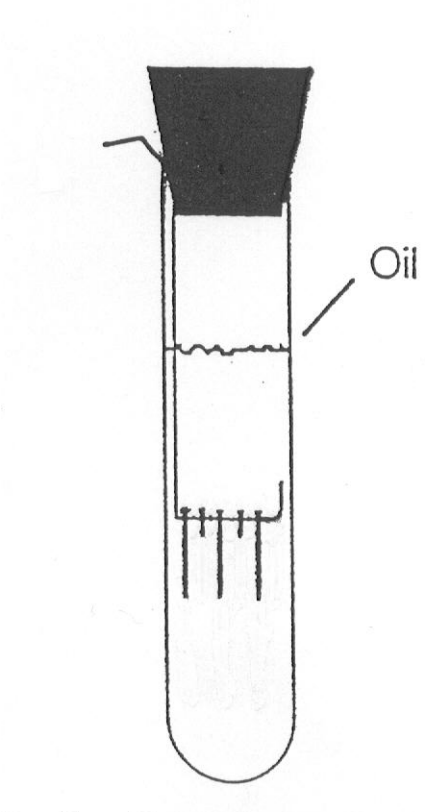

FIG. 2 Test Tube Arrangement

Appendix

X1 Recommended Report Forms

REPORT FORMS

PC-9 OIL SEAL COMPATIBILITY TEST

Candidate Oil Test

Sample Code:

Rubber or Elastomer (Batch)	Test Temperature, °C	Test Duration, Hours	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
Nitrile (NBR) (NBR0500S)	100	336				
Average						
Standard Deviation						
Polyacrylate (ACM) (ACM1199S)	150	336				
Average						
Standard Deviation						
Fluoroelastomer (FKM) (FKM1199S)	150	336				
Average						
Standard Deviation						
Silicone (VMQ) (SIL1199S)	150	336				
Average						
Standard Deviation						

Signature Block

PC-9 OIL SEAL COMPATIBILITY TEST

Reference Oil Test

Reference Oil Code:

Rubber or Elastomer (Batch)	Test Temperature, °C	Test Duration, Hours	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
Nitrile (NBR) (NBR0500S)	100	336				
Average						
Standard Deviation						

Reference Oil Code:						
Polyacrylate (ACM) (ACM1199S)	150	336				
Average						
Standard Deviation						

Reference Oil Code:						
Fluoroelastomer (FKM) (FKM1199S)	150	336				
Average						
Standard Deviation						

Reference Oil Code:						
Silicone (VMQ) (SIL1199S)	150	336				

Average						
Standard Deviation						

Signature Block