

# Elastomer Life Estimation Testing Procedures

API TR 6J1  
FIRST EDITION, AUGUST 2000



American  
Petroleum  
Institute

**Helping You  
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**Upstream Segment**

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# Elastomer Life Estimation Testing Procedures

## 1 Scope

Estimating the service life of elastomeric sealing elements used in severe environments, such as encountered in energy exploration and oil/gas production industries, has been extremely difficult. Elastomeric sealing elements are frequently exposed to methane, hydrogen sulfide, and carbon dioxide gases, along with crude oil, water and corrosion inhibiting chemicals. The proposed procedure outlines a technique based on the Arrhenius principle of chemical reaction rates, which permits the life of an elastomeric material to be estimated when exposed to a severe service environment. The actual test procedure must be coordinated and agreed upon between the supplier of the equipment that incorporates the elastomer sealing elements and the end user. The procedure should be based upon a definition of the service conditions and requirements such as:

- a. *Temperature*—steady state or a high and low range of service.
- b. *Fluids and gases*—stagnant or flowing.
- c. *Pressure*—continuous or a low and high range.
- d. *Chemicals and additives*—inhibitors, descalers, acidizing, etc.
- e. *Mechanical requirements*—dynamic or static, torque, setting force.
- f. *Failure criteria*—pressure leakage, loss of mechanical function, inability to set or retrieve.

## 2 References

The following references are recommended as sources of additional information on the life prediction technique discussed above:

1. Vicic, J.C., *Testing of Polymers for Oil and Gas Applications*, American Chemical Society, Energy Rubber Group, 1984.
2. Abrams, P.I., Kennelley, K.J., Johnson, D.V., *A User's Approach to Qualification of Dynamic Seals for Sour Gas Environments*, American Chemical Society, Rubber Division, 1988.
3. Brady, J.E., Humiston, G.E., *General Chemistry Principles and Structure*, Third Edition, John Wiley & Sons, 1975.
4. Underwriters Laboratories Inc., *UL 746B Standard for Polymeric Materials—Long Term Property Evaluations*, 2nd Ed., 1979.
5. D. Janoff, J. Vicic, D. Cain, *Thermoplastic Elastomer Alloy, TPA, Subsea Hydraulic Seal Development for Service Including Water-Based Fluids*, Conference Papers,

International Conference on Oilfield engineering with Polymers, October 28–29, 1996, London, UK.

6. S. N. Zhurkov, *Intern. J. Fracture Mech.*, 1, 311, 1965.

## 3 Problem Statement

**3.1** Traditional methods of evaluating elastomers used for sealing elements involve the use of ASTM or other standard immersion-type tests. In these techniques, samples of the candidate elastomeric material are immersed in the anticipated environment for a specified time period in the free state. Immersion times can vary from hours, to weeks, to months. The samples may be in a pressurized or unpressurized environment. The physical properties before and after immersion are compared and a judgment is made as to the suitability of the elastomer for use in the service environment. The elastomeric material is generally not tested in its end use geometry (form) and not confined to a seal gland. In a properly designed seal gland, minimal seal surface area is exposed to the severe environment, and the gland physically limits the swell of the sealing element within the gland. The use of an immersion testing technique for retained physical properties does not answer the question of how long the elastomeric sealing element will function as a seal in a severe environment. Many sealing elements used in the energy exploration and oil/gas production industries are expected to remain serviceable (not leak) for up to 20 years in a severe service environment.

**3.2** Traditional immersion tests for retained physical properties have a role in the initial screening of suitable candidate elastomeric materials. A material would not be selected for service, which was severely attacked and deteriorated by the service environment in an immersion test. However, some degradation of physical properties (stress-strain) and volumetric swell can be tolerated. It should also be noted that certain elastomeric materials may sustain minimal property degradation in an immersion test, but they still may not be suitable for long-term sealing service. This is because they exhibit excessive creep or stress relaxation at high pressures and/or temperature.

## 4 Life Estimation Technique—Overview

**4.1** The elastomer life estimation technique described below is based on the Arrhenius principle of chemical reaction rates. This principle is concerned with chemical reaction rates and the effects of temperature on these rates. In general, for every 10°C (18°F) temperature increase, the chemical reaction rate doubles. Conversely for every 10°C (18°F) decrease in temperature, the chemical reaction rate is reduced by 1/2. A brief theoretical discussion of the

Arrhenius principle and its application to accelerated thermo-chemical aging follows:

#### 4.1.1 The Arrhenius equation has the basic form

$$k = A \exp(-Ea/RT)$$

where

$k$  = rate constant of a chemical reaction,

$A$  = proportionality constant related to collision frequency and orientation of molecules,

$Ea$  = activation energy,

$R$  = gas constant,

$T$  = absolute temperature.

#### 4.1.2 Rewriting the equation using natural logs gives:

$$\ln k = -Ea/RT + \ln A$$

If we let

$$\ln k = y$$

$$\ln A = b$$

$$-Ea/R = m$$

$$1/T = x$$

It can be seen that the equation represents a straight line,  $y = mx + b$ , where  $-Ea/R$  is the slope. If the times to failure for various temperatures are converted to natural logs, the experimental data can be plotted on semi-log graph paper. Regression analysis gives the best straight line fit through the experimentally determined data points. If the correlation coefficient is at or near 1.0, the line can be extended and time to failure for other temperatures extrapolated with confidence.

#### 4.2 Examples of accepted industrial procedures that utilize Arrhenius aging techniques are:

ASTM D3045—*Heat Aging of Plastics Without Load*.

ASTM D2990—*Tensile, Compressive, and Flexural Creep and Creep Rupture of Plastics*.

Underwriters Laboratories Inc., UL 746B, *Standard for Polymeric Materials—Long Term Property Evaluations*.

#### 4.3 To approximate the life of an elastomeric material for use in a severe service environment, tests should be conducted in the specified environment under accelerated temperature and/or pressure conditions. Without some type of accelerated testing, it may be difficult to quantify the service life of an elastomer component. Elevated temperature and/or pressure testing can provide a useful method for estimating elastomeric material capabilities under realistic conditions.

Life estimation testing may be considered as the best estimate of long term service life to evaluate the long-term performance of an elastomer in a severe service environment. The basic technique involves collecting time to failure data at elevated temperatures (higher than the maximum anticipated service temperature) and plotting the results on semi-log graph paper. The vertical scale is the log of time to failure and the horizontal scale is the reciprocal of the absolute temperature. Figure 1 shows a typical life estimation plot. Alternately, the time to failure at the service temperature also can be calculated from the appropriate mathematical formula.

4.4 Certain precautions should be exercised when performing accelerated temperature and/or pressure tests. It should be verified experimentally that the failure mechanism (and activation energy) does not change with elevated temperatures or pressures. In addition, it must be recognized gas diffusion may occur through an elastomer seal at an accelerated rate and this must be properly accounted for if this is used as failure criteria. It also may be helpful to test an elastomer material with known field performance as a reference for comparison. Stagnant fluids and gases may give better or worse life estimation than if the fluids are periodically refreshed.

## 5 Procedure For Life Estimation Testing Of Elastomers

5.1 The proposed procedure requires the use of an autoclave (a high temperature pressure vessel) to collect time to failure data. Various autoclave and fixture designs can be used. Figure 2 illustrates one design for a life estimation autoclave sealed with standard size O-rings made from the candidate elastomer. The autoclave should be capable of operation, with a proper safety factor, up to the maximum temperature, pressure and test environment needed for the accelerated test. The internal volume should be appropriately sized to avoid depletion of the test environment during the test; the minimum internal volume should be equal to or exceed 100 cc. The main body and end closures contain O-ring glands that are fabricated from an appropriate alloy. Typically, a corrosion resistant alloy is used to fabricate the test fixture. Since thermo-chemical degradation of the elastomeric sealing element is of interest, thermo-mechanical effects should be minimized. Therefore, clearances between the end closure and the test vessel bore are minimized to eliminate extrusion (thermo-mechanical type failure) of the candidate elastomer.

If additional mechanical protection is required for the O-ring seal, an anti-extrusion ring (back-up ring) of suitable material can be used. In life estimation testing, only the thermo-chemical effects of a severe environment on a candidate elastomer are evaluated. Actual geometry and thermo-mechanical effects are best-evaluated using full scale testing.



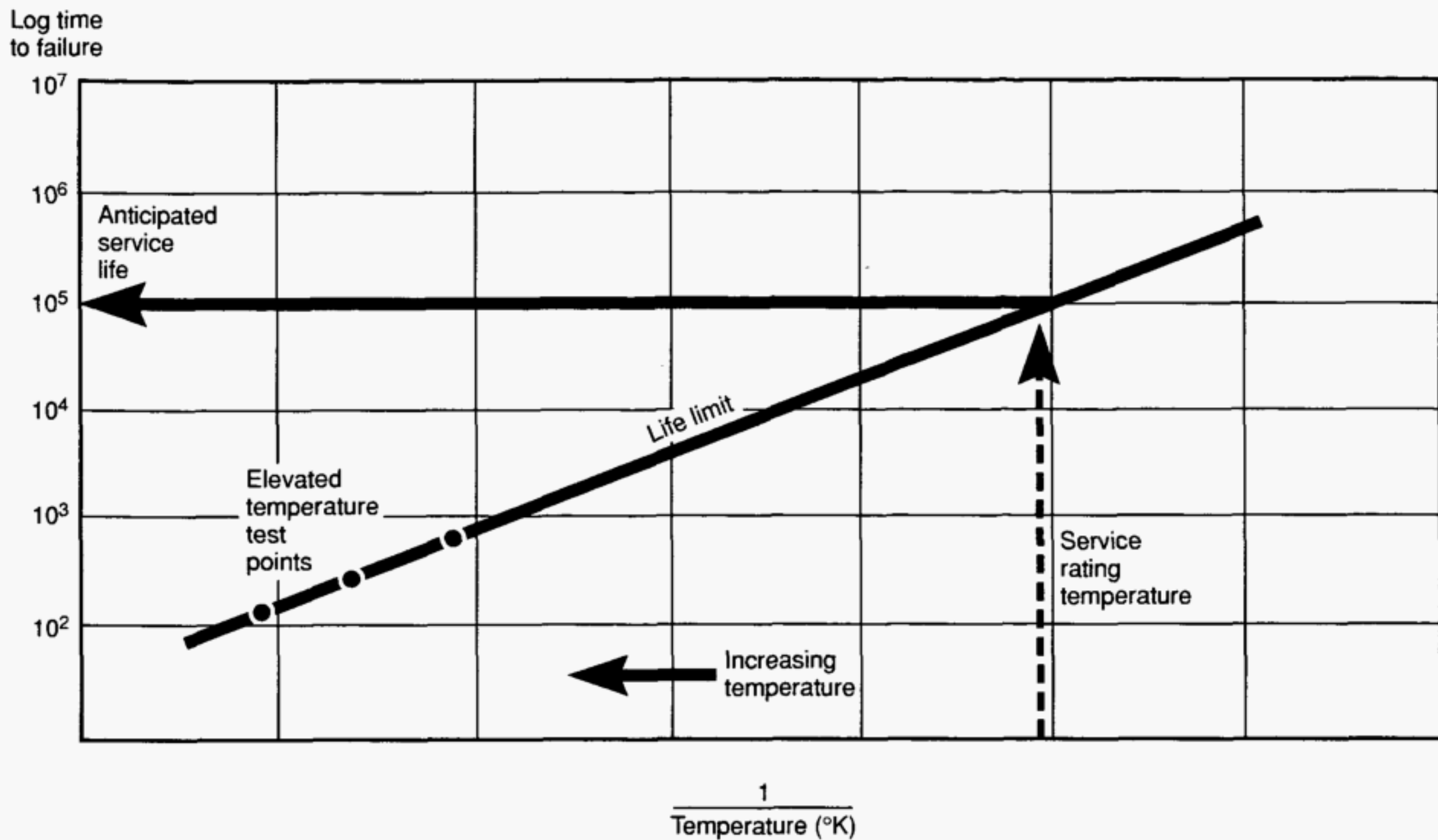


Figure 1—Typical Life Estimation Plot

**5.2** The severe service environment is introduced into the test chamber formed by the two end closures. The test vessel is pressurized and heated to a predetermined temperature during each test cycle. The length of the test cycle is established by the testing protocol, i.e., steady state temperature for downhole components or alternating low and high temperature cycles for surface wellhead equipment. In this example for a surface wellhead application, a 72-hour (3-day) test cycle is used. Figure 3 shows how the 3-day test cycle is conducted. The objective of the test sequence is to establish the rate of chemical degradation as a function of temperature.

**5.3** The selection of a starting temperature for a life estimation experiment is somewhat arbitrary. A good starting point is an elevated temperature that will consistently give a failure in one or two test cycles. Some experimentation may be required to establish this maximum test temperature. Once the maximum test temperature is determined, lower test temperatures can be selected, usually in  $10^\circ\text{C}$  ( $18^\circ\text{F}$ ) increments. For example, if  $450^\circ\text{F}$  is determined to be the maximum test temperature where only one test cycle can be consistently completed, the next lower test temperature would be  $432^\circ\text{F}$ . If the experiment follows the Arrhenius relation, two or more test cycles should be completed at  $432^\circ\text{F}$ . If two or more test cycles are not achieved at  $432^\circ\text{F}$ , the test temperature would be lowered by another  $18^\circ\text{F}$  until at least two or more test cycles are achieved consistently. At each subsequent test tem-

perature, sufficient test runs should be done to obtain test data that are statistically significant. A minimum of three different test temperatures should be used, but preferably, five tests or more should be done with some replicates.

**5.4** Use of the Arrhenius principle in estimating the life of an elastomeric component requires that the chemical process that controls degradation remains constant. If test temperatures are excessive, other reactions may occur and data obtained may lead to erroneous life estimation. Once sufficient data have been accumulated, a least squares regression analysis is done and the data plotted to look for any non-linearity in the life estimation curve. If a single degradation reaction occurred during testing, the best-fit line should approximate a straight line. For a valid life estimation, the least squares regression analysis on the test data should indicate a correlation coefficient greater than 0.90. Once satisfactory test data have been generated, the life estimation line (best fit to data) may be extended to the specified maximum service temperature. An estimate of service life can be read from the vertical scale of the life estimation plot or it can be calculated from the appropriate mathematical formula.

**5.5** Proper simulation of the chemical reactions that occur between candidate elastomers and the severe service environment requires a sufficient volume of chemicals must be present to prevent depletion of the reactants. A three (3) day



test cycle is used so that the candidate elastomeric material is regularly exposed to fresh chemicals. In service, the elastomer may be constantly exposed to a steady stream of fresh chemicals and/or produced fluids/gases or it may only be exposed to stagnant conditions. At the end of the 3-day test cycle, the test vessel is typically rapidly depressurized and purged of the liquid and gas phases. Other decompression cycles can be used with agreement of all concerned parties. Fresh liquid and gas are added and the candidate sealing elements are pressure tested at ambient temperature. For specific applications, other temperatures below ambient can be used. If the seals hold pressure for one hour without leakage, the test vessel is heated up to the test temperature for another 3-day test cycle. This is repeated until failure is observed.

**5.6** Some examples of typical failure modes observed for elastomers in life estimation testing are excessive compression set, hardening, cracking and chemical softening.

## 6 Summary

The life estimation procedure outlined above provides a cost effective technique to evaluate the long term effects of a chemical environment on a elastomer component. Use of the Arrhenius principle of chemical reaction rates allows an accelerated estimation of the thermochemical degradation of the elastomer in a severe service environment. This evaluation technique for studying the long-term effects of an environment on an elastomer compound is an alternative to full scale, long term testing in the field.

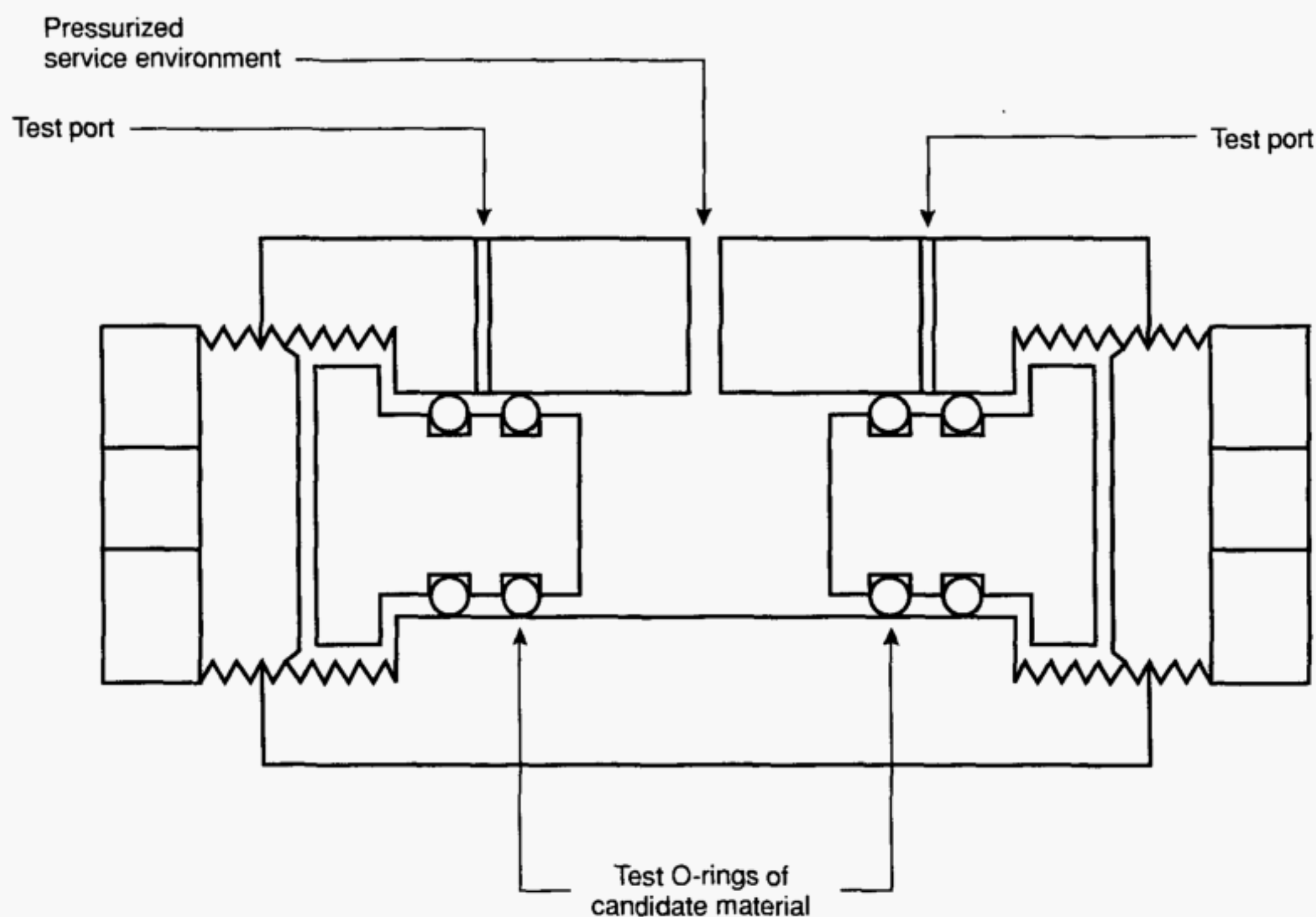


Figure 2—O-Ring Test Fixture



Figure 3—Typical Test Cycle

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Designation: D6104 – 97 (Reapproved 2017)<sup>ε1</sup>

## Standard Practice for Determining the Performance of Oil/Water Separators Subjected to Surface Run-Off<sup>1</sup>

This standard is issued under the fixed designation D6104; 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.

<sup>ε1</sup> NOTE—The Keywords Section was added editorially in December 2017.

### 1. Scope

1.1 This practice covers the procedure, any necessary related apparatus, and the sampling technique to be used in determining the performance characteristics of oil/water separators subjected to contaminated run-off.

1.2 This practice does not address the determination of the performance characteristics of an oil/water separator subjected to the sudden release of a relatively large quantity of hydrocarbons that may appear, in pure form or at high concentration, in the influent to the separator. In this case, refer to Practice **D6157**.

1.3 This practice does not address the determination of the performance characteristics of an oil/water separator subjected to a mechanically emulsified influent such as provided by a pump.

1.4 This practice does not investigate the ability of the separator to handle debris or suspended solids, that is, grit or tree leaves.

1.5 While the effluent may meet code requirements for total oil and grease content, this practice does not address the presence of soluble organics, that is, benzene, toluene, ethylbenzene, and xylene (BTEXs) which may be detected in the effluent. It also does not make any provisions for the effects of detergents, surfactants, soaps, or any water soluble matter (that is, salts), or any portion of an essentially insoluble matter that may be found in solution on separation. (Effects of certain water soluble chemicals or solids may be investigated by adding them to the water at predetermined constant concentrations.)

1.6 In order to estimate the effect of water temperature on the performance of the separator, the tests described in this practice must be performed at two water temperatures. The

selected temperatures must be at least 10°C (18°F) apart, with the temperature ranging from a minimum of 0°C (32°F) to a maximum of 50°C (122°F).

1.7 This practice does not make any provisions for the variation of pH or temperature during a test run. Refer to **Appendix X1** for further detail.

1.8 This practice can be used with a variety of hydrocarbons. It adopts No. 2 fuel oil with a density<sup>2</sup> of 845 kg/m<sup>3</sup> (52.73 lb<sub>m</sub>/ft<sup>3</sup>) and a viscosity<sup>2</sup> of 1.9 to 4.1 centistokes at 40°C (104°F) and SAE 90 lubricating oil with a density<sup>2</sup> of 930 kg/m<sup>3</sup> (58 lb<sub>m</sub>/ft<sup>3</sup>) at 15.5°C (60°F) and a viscosity (see SAE J313) of 13.5 to < 24 centistokes at 100°C (212°F) as the comparative testing media. It is understood that the results obtained from this practice are only directly applicable to No. 2 fuel oil and SAE 90 lubricating oil for the tested concentrations and only careful interpolation or extrapolation, or both, is allowed to other hydrocarbons. Low viscosity or high density hydrocarbons or hydrocarbons that contain a larger fraction of highly soluble compounds may need to be tested separately.

NOTE 1—No extrapolation outside the range of the tested influent or effluent oil concentrations is allowed as performance may not be linear. Hence, to establish performance at a higher or lower concentration, the separator shall be tested for that specific condition. In addition, linearity must be established prior to using linear interpolation.

1.9 Since regulations are based on effluent total hydrocarbon content, this practice does not set forth any lower limits on oil particle size for the evaluation of separator efficiency. However, a standardized means for mixing oil and water shall be specified to ensure repeatability. It must be noted however that smaller particles, having a greater surface area to volume ratio, rise at a slower rate than their larger counterparts. (Guide **F933** requires that 20 % of all oil particles be smaller than or equal to 50 μm and IMO MEPC 60 (30) does not mention any particle size requirements but asks the user to avoid emulsion causing chemicals.)

1.10 Although the tests described in this practice intend to simulate contaminated storm water run-off separation

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee **D19** on Water and is the direct responsibility of **D19.06** on Methods for Analysis for Organic Substances in Water

Current edition approved Dec. 15, 2017. Published January 2018. Originally approved in 1997. Last previous edition approved in 2011 as D6104 – 97 (2011). DOI: 10.1520/D6104-97R17E01.

<sup>2</sup> Bolz, R. E., and Tuve, G. L., *CRC Handbook of tables for Applied Engineering Science*, 2nd Edition, CRC Press, 1981.



requirements, they do not cover all possible applications. It is the end user's responsibility to determine whether his separation requirements are within the scope of this practice.

1.11 A product different from the general description herein may be tested and found to be in compliance with the performance criteria set forth.

1.12 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.13 *This practice does not purport to address all the environmental hazards, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate environmentally responsible practices and to determine the applicability of regulatory limitations prior to use.*

1.14 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.15 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

**D1129 Terminology Relating to Water**

**D3370 Practices for Sampling Water from Closed Conduits**

**D4281 Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination (Withdrawn 2012)<sup>4</sup>**

**D6157 Practice for Determining the Performance of Oil/Water Separators Subjected to a Sudden Release**

**F933 Guide for Evaluation of Oil Water Separation Systems for Spilled Oil Recovery Applications (Discontinued 2001) (Withdrawn 2001)<sup>4</sup>**

### 2.2 EPA Standards:<sup>5</sup>

**EPA-413.1 "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, revised March 1983**

**EPA-413.2 "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, revised March 1983**

**EPA-1664 H-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons) EPA-821-B-94-004B, April 1995**

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>5</sup> Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

### 2.3 SAE Standards:<sup>6</sup>

**SAE J306 Axle and Manual Transmission Lubricant Viscosity Classification**

**SAE J313 Surface Vehicle Recommended Practice (R) Diesel Fuels**

## 3. Terminology

### 3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminology **D1129**.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration, n*—the certified evaluation of the accuracy of a measuring instrument as performed by its manufacturer or an independent licensed or accredited third party.

3.2.2 *contaminated run-off, n*—rain water which has collected oily contaminants from the surfaces it came in contact with and which may appear in the influent to a separator. Unlike a release, the level of contamination in this case is much lower.

3.2.3 *effluent, n*—the aqueous release from a separator.

3.2.4 *flow totalizer, n*—a counter, usually attached to a flow meter, that evaluates the total volume of the fluid that has flowed through over a given time period.

3.2.5 *influent, n*—the oily aqueous input to a separator.

3.2.6 *oily discharge, n*—any release of oily contaminants into the environment that exceeds the allowable limit.

3.2.7 *re-entrainment, n*—the condition in which the level of contamination of the effluent water of a separator containing oil is higher than the influent contamination level due to internal remixing. This definition usually applies to situations where clean water passes through a separator that already contains hydrocarbons stored within and atop the water so as to form an interface.

3.2.8 *release, n*—any sudden discharge of an oily substance from vessels that are specifically designed to store, contain, or transfer oily products such as storage tanks, pipelines, diked areas, and transfer equipment and which may appear in the influent to a separator.

3.2.9 *separator, n*—a flow through primary treatment device the primary purpose of which is to separate oil from water.

## 4. Summary of Practice

4.1 The practice evaluates a separator's ability to reduce the total hydrocarbon content of contaminated run-off. For this, an influent is supplied at the separator's rated flow for the selected hydrocarbon content (either 350 or 1000 mg/L). The corresponding effluent hydrocarbon content is determined by obtaining and analyzing grab samples.

4.2 The practice also evaluates the effluent of a separator at rated oil storage capacity in relation to a non-contaminated influent and its corresponding rated flow in order to establish its re-entrainment characteristics.

<sup>6</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096, <http://www.sae.org>.



4.3 The data generated in this practice are considered valid for the separators tested only. However, the results of these tests may be extrapolated to smaller or larger size separators provided that applicable geometric and dynamic similitude are maintained. Where the use of extrapolation is not applicable, that size unit must be subjected to testing.

4.4 The flow rate for these tests must equal the manufacturer's rated flow for the given separator at the given influent contamination level and for the selected effluent peak contamination concentration.

4.5 For the purpose of this test, the water temperature should be between 10°C (50°F) and 21.1°C (70°F) and the pH of the water between 6 and 9.

## 5. Significance and Use

5.1 The Clean Water Act promulgated the implementation of water quality standards and contamination limits for a wide range of pollutants including oil and grease. Specifically, the EPA prohibits "the discharges of oil that cause a film or sheen upon or cause discoloration of the surface of the water." Several state and local agencies have adopted this statement in addition to setting concentration limits, that is, 15 mg/L or even 5 mg/L. The purpose of this practice is to evaluate the performance of a separator in regards to the regulations and user requirements.

5.2 Another purpose of this practice is to establish that a separator containing oil at its rated capacity would still be capable of meeting the above criteria when subjected to run-off.

5.3 This practice is not applicable if the influent to a separator contained a sudden release as much higher concentrations would be expected. For this case, see Practice [D6157](#).

5.4 This practice is not applicable if the influent to a separator is conveyed by a pumping means.

5.5 The data generated in this method is valid for the separators tested only. The results of these tests may be extrapolated to smaller or larger size separators provided that applicable geometric and dynamic similitude are maintained. Where sound engineering method limits the use of extrapolation, that size unit must be subjected to testing.

5.6 The flow rate for all the tests must equal the manufacturer's total rated flow for the given separator at a given influent contamination level and for the selected effluent peak contamination concentration.

## 6. Test Set-Up and Apparatus

6.1 *Water Supply*—The water supply can be either a water main, a water reservoir and a pump, or an elevated storage tank capable of providing the volume and flow rate of water necessary for a test run as described in the procedure. If either a storage tank or reservoir is used, the volume shall be at least three times the liquid volume of the separator.

6.1.1 *Flow Totalizer or Sight Glass*—The water supply should be equipped with a calibrated means of indicating the total volume of water dispensed, that is, a flow totalizer or a sight glass. The selected device should be within 5 % accuracy.

6.1.2 *Flow Rate Indicator*—The water supply must also be equipped with a calibrated means of controlling and indicating the flow rate, that is, throttling valve and flow meter, orifice plates or, venturis. The means used for controlling the flow rate must be capable of maintaining the flow within 5 % of the desired value.

6.2 *Oil Supply*—The oil supply should be large enough to store the quantity required for the larger concentration test and for its entire duration. A minimum estimate could be based on three separator liquid volumes.

6.2.1 *Flow Totalizer or Sight Glass*—The oil storage tank should be equipped with a calibrated sight glass or flow totalizer. The selected device should be within 5 % accuracy.

6.2.2 *Flow Rate Indicator*—The oil supply should also be equipped with a calibrated means of controlling and indicating the flow rate, that is, throttling valve and flow meter, orifice plates or, venturis. The means used for controlling the flow rate must be capable of maintaining the flow within 5 % of the desired value.

6.3 *Separator*—A separator with an outlet pipe extending far enough to allow grab sampling as described in Practices [D3370](#).

6.4 *Mixer*—A means for mixing the hydrocarbons with the water consisting of a commercially available horizontal PVC pipe section with a minimum surface roughness of 0.00015 cm (0.000005 ft) having a length of at least 20 diameters with one end connected directly to the inlet of the separator. An oil injection port shall be provided at the other end of the pipe and at its bottom portion and shall not extend into the pipe more than one third its diameter in order to prevent stratification.<sup>7</sup> The pipe diameter shall be selected such that it runs full and at a Reynolds number, based on the hydraulic diameter, in excess of 70 000 and a velocity in excess of 1 m/s (3.28 ft/s). The injection port diameter shall be sized to provide, at the higher test concentration, an injection velocity approximately equal to 1 m/s.

6.5 *Influent Sampling Port*—An influent sampling port for temperature and pH reading. (If on-line temperature and pH readers are not available, a small sample should be extracted and the temperature read immediately at the beginning of every test. pH analysis may be performed at a later time.)

## 7. Procedure

7.1 *Test A—Investigation of Re-Entrainment at Rated Oil Storage Capacity:*

7.1.1 Fill the separator with oil to the manufacturer's rated oil storage capacity.

7.1.2 Allow fresh water to enter the separator at its rated flow until at least three volume changes are achieved and the effluent concentration reaches steady-state. Take an effluent grab sample at every one third ( $\frac{1}{3}$ ) of the separator volume change. Samples must be gathered and handled in accordance with Practices [D3370](#).

<sup>7</sup> Perry, R. H., and Green, D., *Perry's Chemical Engineer's Handbook*, 6th Edition, McGraw-Hill, 1984.



NOTE 2—Steady-state means that, when analyzed, the last three samples shall depict a “constant” oil and grease concentration with respect to the other samples within the accuracy of the accepted sample analysis method. If this condition cannot be attained within three volume changes then the total volume of water necessary shall be increased until this condition is met.

7.1.3 Each sample container shall be labeled with a serial number and a run number, the date of the test and the initials of the person performing the test.

7.1.4 The run number, date, water temperature and pH, the number of samples taken, the flow rate, influent oil concentration and the total volume of water, the model number of the separator, and a description of any ancillary equipment shall be recorded and the data sheet signed by a registered or licensed third party present during the test.

7.1.5 Samples shall be analyzed by an independent testing laboratory certified for the selected testing method in accordance with Test Method **D4281**, EPA 413.1, EPA 413.2, EPA-1664 or other EPA approved standard. The analytical standard used must be specified.

## 7.2 Test B—Contaminated Run-Off Tests:

7.2.1 Make necessary adjustments to the testing apparatus in order to obtain a 350-mg/L oil grease concentration in the influent.

7.2.2 Fill the separator with oil to its rated capacity minus the estimated amount of oil that would be added to the separator at the end of the run. For example, consider a separator having a 1000-L liquid capacity and 100-L oil storage capacity based on three volume changes at 350-mg/L concentration of No. 2 diesel with 0.83 specific gravity, the volume of No. 2 Diesel that would be added to the separator is  $(350 \times 10^{-6}) \times 3000/0.83 = 1.265$  L. Therefore, the separator shall be filled with 98.735 L of oil (100 to 1.265).

7.2.3 Allow the mixture to enter the separator at its rated flow for the given test conditions until at least three volume changes are achieved or the effluent concentration reaches steady-state (see **Note 2**). Take an effluent grab sample at every one third volume change. Samples must be gathered and handled in accordance with Practices **D3370**.

7.2.4 Each sample container shall be labeled with a serial number and a run number, the date of the test, and the initials of the person performing the test.

7.2.5 The run number, date, water temperature and pH, the number of samples taken, the flow rate, influent oil concentration and the total volume of water, the model number of the separator, and a description of any ancillary equipment shall be

recorded and the data sheet signed by a registered or licensed third party present during the test.

7.2.6 Samples shall be analyzed by a certified independent testing laboratory in accordance with Test Method **D4281**, EPA 413.1, EPA 413.2, EPA 1664 or other EPA approved standard. The analytical standard used must be specified.

7.2.7 Repeat **7.2.1** through **7.2.6** at an influent concentration of 1000 mg/L.

## 7.3 General Notes:

7.3.1 All measuring instruments, metering pumps, and other auxiliary equipment must be calibrated and certified prior to testing.

7.3.2 In the case of custom built equipment, the method of calibration must be clearly described and attached to the report and such calibration must be performed by a certified or registered independent third party.

## 8. Report

8.1 The report shall clearly indicate the tested separator make and model as well as the manufacturer’s description, including all standard ancillary equipment.

8.2 The report shall include a copy of all the laboratory sample analysis reports including the analysis method. The report must bear enough pertinent information in order to correlate it to the particular test that was performed as well as the signature of an official laboratory representative.

8.3 Representation of the data shall contain the separator make and model, the initial volume of oil stored within the unit, the total number of volume changes, the date the test was performed, the water temperature and pH, the type of oil used, its specific gravity, viscosity and temperature, the flow rate, the name and signature of person who performed the test, the type of test, that is, Test A, the run starting time, the time each sample was taken, its number, and its corresponding laboratory analysis.

8.4 In the case a type B test was performed, the tabular representation must also include the influent oil concentration and the total volume of oil injected into the system.

8.5 All tables shall have the peak and average effluent contamination values listed.

## 9. Keywords

9.1 No. 2 fuel oil; oil/water separators; performance; SAE 90 lubricating oil; surface run-off



**APPENDIXES****(Nonmandatory Information)****X1. EFFECTS OF pH AND TEMPERATURE ON SEPARATION**

X1.1 Elevated pH levels may reduce the separation efficiency of a separator whereas reduced pH levels may enhance separation. Similarly, changes in temperature may also affect separation efficiency. For example, a reduction in temperature from 18°C to 10°C (65 to 50°F) may cause an increase in the specific gravity of the water of only 0.12 %. However, it causes

a 25 % increase in the dynamic viscosity of the water. Hence, the net effect of a decrease in temperature is adverse on separation efficiency. The same series of tests may be repeated at different pH levels or temperatures in order to determine their effect on separation.

**X2. PARTICLE SIZE AND SEPARATION**

X2.1 Finer dispersions of oil in the water entering a separator will result in reduced separator efficiency. In order to predict the performance of a separator in a given application, the oil droplet size distribution during the test must compare to the oil droplet size distribution in the field. To generate finer dispersions, the Reynolds number in the mixing pipe may be

raised, a pipe with greater surface roughness may be selected or an orifice plate, or a static mixer may be used. As previously mentioned, this practice relies on a standardized mixing device to obtain a datum for comparison as opposed to using relatively expensive and often unreliable particle size measurement equipment.

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