Elastomer Life Estimation Testing Procedures

API TR 6J1
FIRST EDITION, AUGUST 2000

American Petroleum Institute

Helping You Get The Job Done Right.SM

Elastomer Life Estimation Testing Procedures

Upstream Segment

API TR 6J1
FIRST EDITION, AUGUST 2000

American Petroleum Institute

Helping You Get The Job Done Right.℠

SPECIAL NOTES

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed.

API is not undertaking to meet the duties of employers, manufacturers, or suppliers to warn and properly train and equip their employees, and others exposed, concerning health and safety risks and precautions, nor undertaking their obligations under local, state, or federal laws.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the material safety data sheet.

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. Sometimes a one-time extension of up to two years will be added to this review cycle. This publication will no longer be in effect five years after its publication date as an operative API standard or, where an extension has been granted, upon republication. Status of the publication can be ascertained from the API Upstream Segment [telephone (202) 682-8000]. A catalog of API publications and materials is published annually and updated quarterly by API, 1220 L Street, N.W., Washington, D.C. 20005.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this standard or comments and questions concerning the procedures under which this standard was developed should be directed in writing to the general manager of the Upstream Segment, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the general manager.

API standards are published to facilitate the broad availability of proven, sound engineering and operating practices. These standards are not intended to obviate the need for applying sound engineering judgment regarding when and where these standards should be utilized. The formulation and publication of API standards is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

FOREWORD

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to assure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any federal, state, or municipal regulation with which this publication may conflict.

Suggested revisions are invited and should be submitted to the general manager of the Upstream Segment, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

CONTENTS

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 ½. 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 maybe 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.

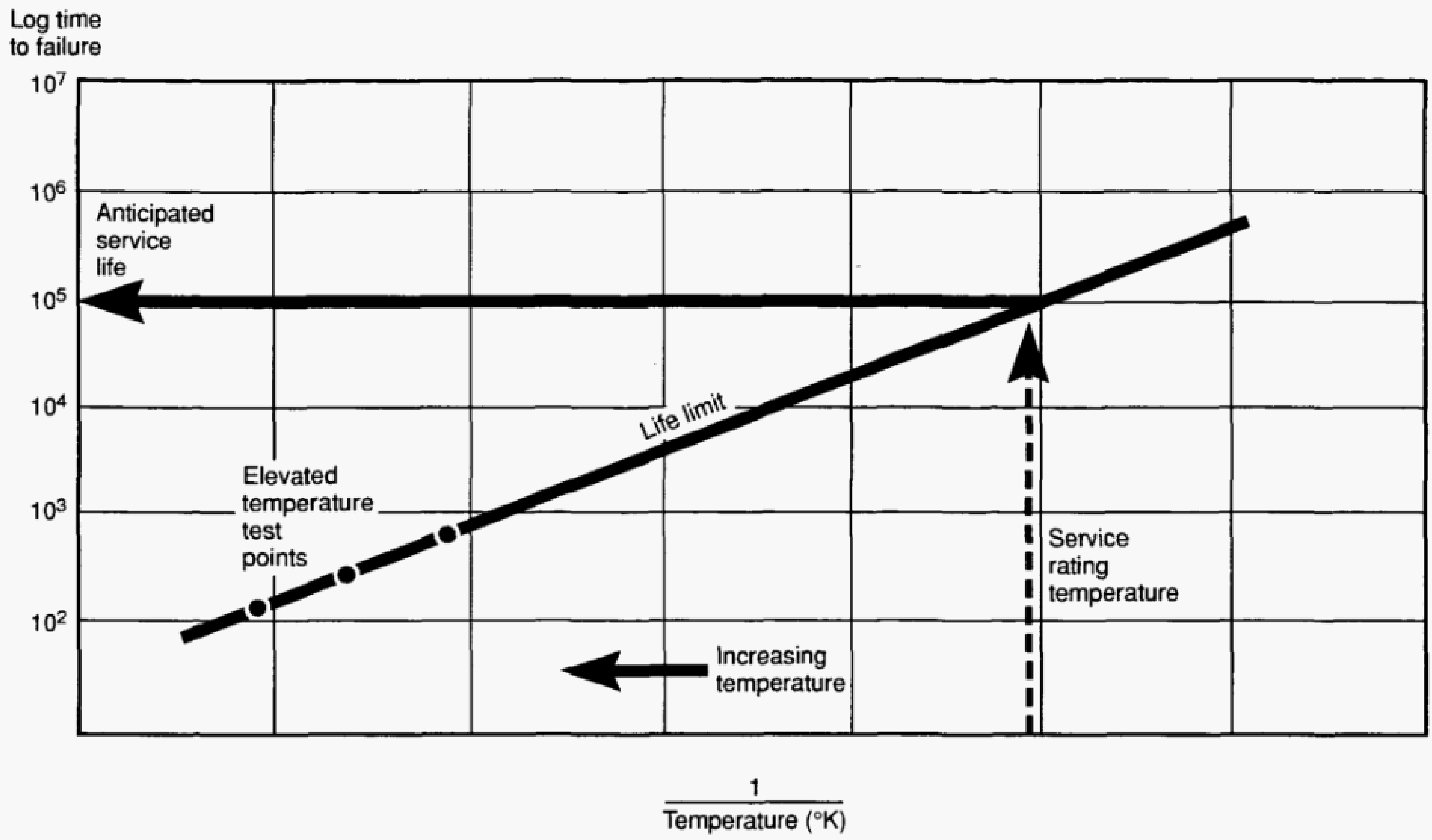

Figure 1—Typical Life Estimation Plot

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°C (18°F) increments. For example, if 450°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°F. If the experiment follows the Arrhenius relation, two or more test cycles should be completed at 432°F. If two or more test cycles are not achieved at 432°F, the test temperature would be lowered by another 18°F until at least two or more test cycles are achieved consistently. At each subsequent test temperature, 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.

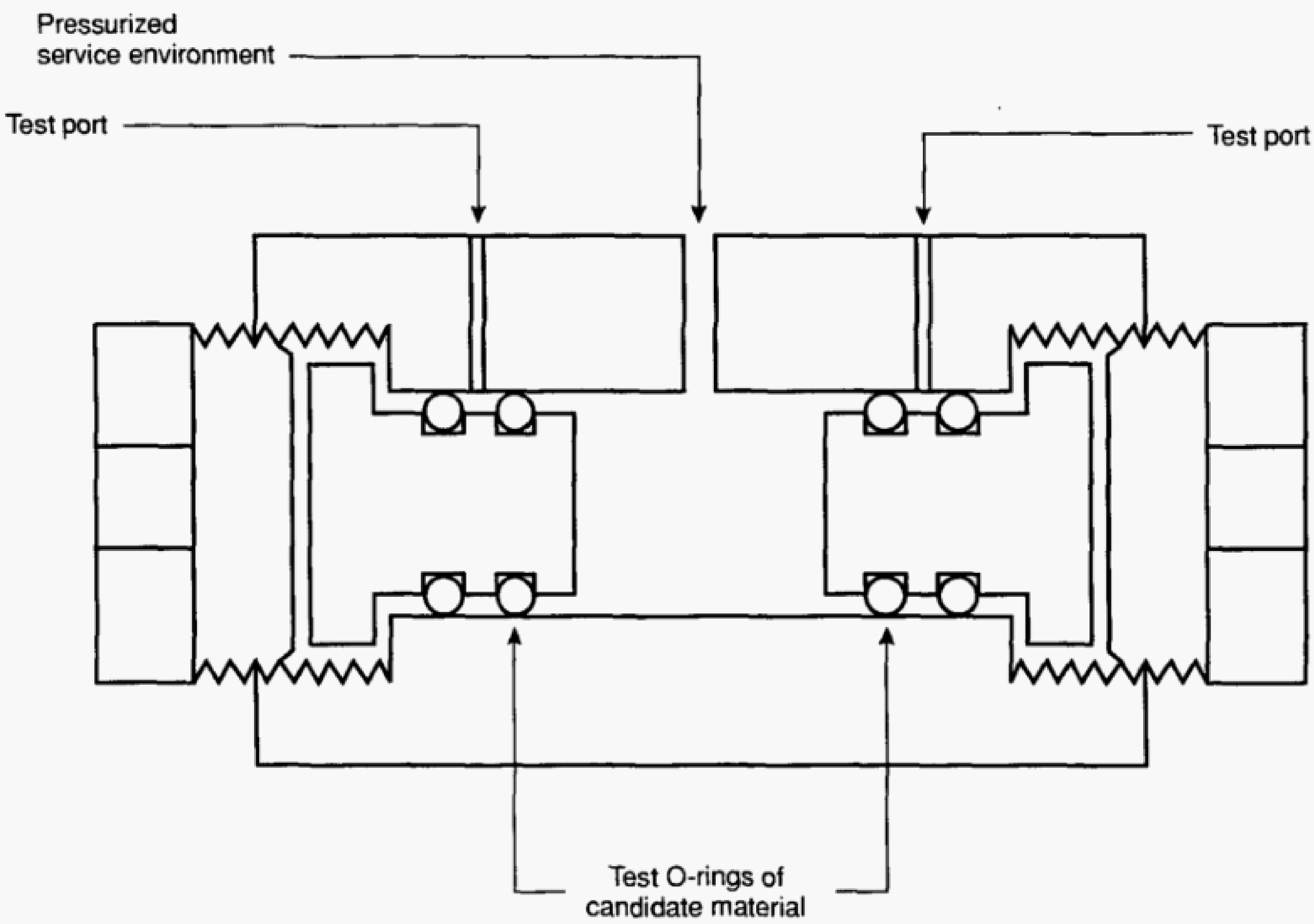

Figure 2—O-Ring Test Fixture

Figure 3—Typical Test Cycle

API Related Publications Order Form - 2000

Date: ____________ *(Month, Day, Year)*

❑ **API Member** *(Check if Yes)*

Invoice To – ❑ Check here if same as "Ship To"

Company:

Name/Dept.:

Address:

City: State/Province:

Zip: Country:

Customer Daytime Telephone No.:

Fax No.:

(Essential for Foreign Orders)

Ship To – (UPS will not deliver to a P.O. Box)

Company:

Name/Dept.:

Address:

City: State/Province:

Zip: Country:

Customer Daytime Telephone No.:

Fax No.:

(Essential for Foreign Orders)

❑ **Payment Enclosed $** ____________

❑ **Payment By Charge Account:**

❑ MasterCard ❑ VISA ❑ American Express

Account No.:

Name (As It Appears on Card):

Expiration Date:

Signature:

❑ **Please Bill Me**

P.O. No.:

Customer Account No.:

State Sales Tax – The American Petroleum Institute is required to collect sales tax on publications mailed to the following states: AL, AR, CT, DC, FL, GA, IL, IN, IA, KS, KY, ME, MD, MA, MI, MN, MO, NE, NJ, NY, NC, ND, OH, PA, RI, SC, TN, TX, VT, VA, WV, and WI. Prepayment of orders shipped to these states should include applicable sales tax unless a purchaser is exempt. If exempt, please print your state exemption number and enclose a copy of the current exemption certificate.

Exemption Number: State:

Quantity	Order Number	Title	SO*	Unit Price	Total
	G03230	**Bull 6J, *Testing of Oilfield Elastomers (A Tutorial)***		**$ 50.00**	
	G06A17	**Spec 6A, *Wellhead and Christmas Tree Equipment***		**$ 150.00**	

Shipping and Handling – All orders are shipped via UPS or First Class Mail in the U.S. and Canada. Orders to all other countries will be sent by Airmail. U.S. and Canada, $5 per order handling fee, plus actual shipping costs. All other countries, $15 per order handling fee, plus actual shipping costs.

Rush Shipping Charge – FedEx, $10 in addition to customer providing FedEx account number: ____________. UPS Next Day, $10 plus the actual shipping costs (1-9 items). UPS Second Day, add $10 plus the actual shipping costs (1-9 items).

Rush Bulk Orders – 1-9 items, $10. Over 9 items, add $1 each for every additional item. *NOTE: Shipping on foreign orders cannot be rushed without FedEx account number.*

Returns Policy - Only publications received in damaged condition or as a result of shipping or processing errors, if unstamped and otherwise not defaced, may be returned for replacement within 45 days of the initiating invoice date. A copy of the initiating invoice must accompany each return. Material which has neither been damaged in shipment nor shipped in error requires prior authorization and may be subject to a shipping and handling charge. **All returns must be shipped prepaid using third class postage. If returns are due to processing or shipping errors, API will refund the third class postage.**

Subtotal	
State Sales Tax *(see above)*	
Rush Shipping Charge *(see left)*	
Shipping and Handling *(see left)*	
Total *(in U.S. Dollars)*	

****To be placed on Standing Order for future editions of this publication, place a check mark in the space provided.***

Pricing and availability subject to change without notice.

Mail Orders: **American Petroleum Institute, Order Desk, 1220 L Street, NW, Washington, DC 20005-4070, USA**

Fax Orders: **202-962-4776**

Phone Orders: **202-682-8375**

To better serve you, please refer to this code when ordering: **L A 4 5 0 9 0 2 0 0**

The American Petroleum Institute provides additional resources and programs to industry which are based on API Standards. For more information, contact:

• Training/Workshops	Ph:	202-682-8490
	Fax:	202-962-4797
• Inspector Certification Programs	Ph:	202-682-8161
	Fax:	202-962-4739
• American Petroleum Institute Quality Registrar	Ph:	202-682-8574
	Fax:	202-682-8070
• Monogram Licensing Program	Ph:	202-962-4791
	Fax:	202-682-8070
• Engine Oil Licensing and Certification System	Ph:	202-682-8233
	Fax:	202-962-4739

To obtain a free copy of the API Publications, Programs, and Services Catalog, call 202-682-8375 or fax your request to 202-962-4776. Or see the online interactive version of the catalog on our web site at www.api.org/cat.

American Petroleum Institute

Helping You Get The Job Done Right.℠

01.21.00

8/00—6C

Additional copies available from API Publications and Distribution:
(202) 682-8375

Information about API Publications, Programs and Services is available on the World Wide Web at: http://www.api.org

American Petroleum Institute
1220 L Street, Northwest
Washington, D.C. 20005-4070
202-682-8000

Order No. G06J11