# Recommended Practice on Thread Compounds for Casing, Tubing, and Line Pipe

API RECOMMENDED PRACTICE 5A3 FIRST EDITION, JUNE 1996

EFFECTIVE DATE: OCTOBER 1, 1996







One of the most significant long-term trends affecting the future vitality of the petroleum industry is the public's concerns about the environment. Recognizing this trend, API member companies have developed a positive, forward looking strategy called STEP: Strategies for Today's Environmental Partnership. This program aims to address public concerns by improving our industry's environmental, health and safety performance; documenting performance improvements; and communicating them to the public. The foundation of STEP is the API Environmental Mission and Guiding Environmental Principles.

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- To counsel customers, transporters and others in the safe use, transportation, and disposal of our raw materials, products, and waste materials.
- To economically develop and produce natural resources and to conserve those resources by using energy efficiently.
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- To commit to reduce overall emission and waste generation.
- To work with others to resolve problems created by handling and disposal of hazardous substances from our operations.
- To participate with government and others in creating responsible laws, regulations, and standards to safeguard the community, workplace, and environment.
- To promote these principles and practices by sharing experiences and offering assistance to others who produce, handle, use, transport, or dispose of similar raw materials, petroleum products and wastes.

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# **Exploration and Production Department**

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### FOREWORD

This standard shall become effective on the date printed on the cover but may be used voluntarily from the date of distribution.

Note: This is the first edition of this recommended practice and supersedes API Bulletin 5A2, *Bulletin on Thread Components for Casing, Tubing, and Line Pipe*, Sixth Edition, (May 31, 1988), American National Standard, ANSI/API BUL 5A2-88 (approved: July 12, 1993).

This recommended practice is under the jurisdiction of the API Subcommittee on Standardization of Tubular Goods. It has been determined that this recommended practice directly addresses safety and environmental issues and, therefore, has been reviewed in conformance with API's Environmental Mission and Guiding principles.

This recommended practice is for the convenience of users, purchasers and manufacturers in purchasing and producing thread compounds and is not intended to restrict or limit users, purchasers and manufacturers from purchasing and producing thread compounds meeting standards other than those contained herein.

*CAUTION:* Thread compounds may contain toxic or hazardous materials. The Material Safety Data Sheets (MSDS) for thread compounds should be read and observed. Store and dispose of containers, unused compound and other waste materials in accordance with federal, state and local regulations.

*CAUTION:* This recommended practice is not intended for the evaluation of compounds used on rotary shouldered connections. The testing of thread compounds used on rotary shouldered connections is described in API Recommended Practice 7A1 (RP 7A1).

*CAUTION:* It is generally recognized that performance test results, full-scale or otherwise, may not correlate with specific field service because of the variability of materials and coatings, field equipment and the service environment. Performance tests and criteria are specified herein to provide relative comparisons of thread compound performance under controlled conditions that may not indicate actual differences in service.

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# Recommended Practice on Thread Compounds for Casing, Tubing, and Line Pipe

## 1 Scope

### 1.1 PURPOSE

The purpose of this recommended practice is to provide means for evaluating the suitability of thread compounds, regardless of composition, for use on API round thread and buttress casing, tubing and line pipe connections in high-pressure service. The tests outlined herein are used to evaluate the critical performance properties of thread compounds under laboratory conditions. When evaluating the suitability of a thread compound, the user should consider full size connection test results and field experience in addition to the results of reduced scale (bench top) test methods ike those described herein.

#### **1.2 PERFORMANCE OBJECTIVE**

The performance objective of this recommended practice is to provide laboratory means with which to compare the performance of thread compounds to a standard that has demonstrated successful performance over a wide range of service conditions and a significant period of time.

The thread compound formulations specified in *obsolete* API Bulletin 5A2 (Sixth Edition, 1988) were originally developed by the Mellon Institute of Industrial Research, under an API sponsored research project, to meet the following performance objectives:

a. Adequate lubricating qualities to prevent galling in threaded connections during makeup.

b. No tendency to disintegrate nor undergo radical change in volume at temperatures up to 300° F (148.9° C).

c. No tendency to become excessively fluid at temperatures as high as 300° F (148.9° C).

d. Sealing properties sufficient to prevent leakage at temperatures as high as 300° F (148.9° C).

e. Absence of any deleterious instability and of any drier or hardener that will evaporate or oxidize, thereby changing the thread compound properties.

f. Resistance to water absorption.

g. Sufficient inert filler to prevent leakage of API roundthread casing and tubing joints under pressure as high as 10,000 psi (68.9 megaPascals).

h. Readily applicable by brush to pipe threads in cold weather.

The "API Modified Thread Compound" has been generally accepted by the industry for a wide range of service conditions over a period of many years. Therefore, the API Bulletin 5A2 modified formulation is utilized as the basis for the Reference Standard in this recommended practice and may be found in Appendix A. This recommended practice replaces API Bulletin 5A2. Another API sponsored research project, API PRAC Project 88, 89, 91–51, *Investigation of Pipe Thread Compounds*, indicates that limitation of particle size, solids content, grease thickeners and oil viscosity of API Bulletin 5A2 modified thread compound to the nominal range of tolerance is required in order to replicate performance properties. Therefore, in addition to the requirements found in API Bulletin 5A2, the Reference Standard is further limited to nominal parameters, as found in Section A.6, Appendix A, Reference Standard Formulation.

Note: It should be recognized that the service requirements for thread compounds can exceed the *obsolete* API Bulletin 5A2 requirements of "cold weather" application and 10,000 psi (68.9 megaPascals) and 300° F (148.9° C). Therefore, the extensive range of service conditions can require full size connection testing, field evaluation and engineering judgement to achieve acceptable performance.

### 1.3 METRIC CONVERSION

Metric conversion of US Customary units are provided throughout the text of this specification in parentheses, e.g., 6 in. (152.4 mm). The factors used for conversion of US Customary units to metric values are:

1 inch (in.)	=	25.4 millimeters (mm), exactly.
1 square inch (sq in.)	=	645.16 square millimeters
		(sq mm), exactly.
1 foot (ft)	=	0.3048 meters (m), exactly.
1 pound (lb)	=	0.454 kilograms (kg).
1 pound per sq in. (psi)	=	0.00689476 megaPascals
		(mPa), pressure
	=	0.00689476 Newtons/sq mm
		(N/sq mm), stress.
1 foot-pound (ft-lb)	=	1.35582 joules (J), impact en-
		ergy, or
		1.05500.01
	=	1.35582 Newton-meters ( $N \cdot m$ ),
		torque.

The following formula was used to convert degrees Fahrenheit (°F) to degrees Celsius (°C):

 $^{\circ}$  C =  $^{5/9}$  ( $^{\circ}$  F minus 32)

# 2 References

**2.1** This standard includes by reference, either in total or in part, the latest editions of the following API, industry, and government standards, unless a specific edition is listed:

### API

Spec 5CT	Specification for Casing and Tubing
Spec 5L	Specification for Line Pipe
RP 5C1	Recommended Practice for Care and Use
	of Casing and Tubing
RP 5C5	Recommended Practice for Evaluation Pro-
	cedures for Casing and Tubing Connections
RP 7A1	Recommended Practice for Testing of
	Thread Compound for Rotary Shouldered
	Connections

Spec Q1 Specification For Quality Programs

PRAC Project 88, 89, 91–51, Investigation of Pipe Thread Compounds

#### ASTM1

B 117-90	Test Method of Salt Spray (Fog) Testing
C 561-91	Test Method for Ash in Graphite
D 217-88	Test Method for Cone Penetration
D 283-84	Method for Chemical Analysis of Dry
	Cuprous Oxide and Copper Pigments
D 521-90	Method for Chemical Analysis of Zinc
	Dust (Metallic Zinc Powder)
D 566-87	Test Method for Dropping Point of Lubri-
	cating Grease
D 1301-91	Method for Chemical Analysis for White
	Lead Pigments
D 1743-87	Test Method for Corrosion Preventative
	Properties of Lubricating Greases
D 2196-86	Standard Test Methods for Rheological
	Properties of Non-Newtonian Materials by
	Rotational (Brookfield) Viscometer
D 2265-88	Test Method for Dropping Point of Lubri-
	cating Grease Over Wide Temperature
	Range
D 4048-91	Test Method for Detection of Copper Cor-
	rosion from Lubricating Grease
E 11-87	Specification for Wire Cloth Sieves for

Deutsches Institut fur Normung<sup>2</sup>

DIN 51 802 Testing of the Corrosion Preventing Properties of Lubricating Greases

#### Federal Test Method

791B-321.2 Oil Separation and Leaching Tests

**Testing Purposes** 

#### 3 Definitions

For the purposes of this specification, the following definitions apply:

**3.1 shall:** Is used to indicate that a provision is mandatory.

**3.2 should:** Is used to indicate that a provision is not mandatory, but recommended as good practice.

**3.3 may:** Is used to indicate that a provision is optional.

**3.4 API modified thread compound:** A thread compound that is formulated in accordance with the requirements of obsolete API Bulletin 5A2, designated as the "modified thread compound."

**3.5 manufacturer:** The entity responsible for thread compound manufacture.

**3.6 purchaser:** The entity responsible for thread compound procurement from the manufacturer.

**3.7 Reference Standard:** A thread compound that is formulated in accordance with the requirements of Appendix A, *API Modified Thread Compound and Reference Standard*, to include the constituent limitations specified in Tables A–4, A–5 and A–6. It is not intended for general service.

**3.8 storage compound:** A substance that is applied to threaded oil field pipe connections to protect against corrosion during either shipment or storage or both.

*CAUTION:* Storage compounds alone are not intended for connection makeup.

**3.9 thread compound:** A substance that is applied to threaded oil field pipe connections prior to make-up to provide lubrication during assembly and disassembly and to aid in sealing against high internal and external pressure in service. Some thread compounds may also contain substances that provide storage compound properties.

**3.10 user:** The entity responsible for thread compound application.

### 4 Compound Characteristics

#### 4.1 PRODUCT CHARACTERIZATION

This recommended practice outlines tests that attempt to predict performance of thread compounds under service conditions, rather than specifying a formulation. Thus, the purchaser and the manufacturer should agree on the product characterization to be provided, such as:

- a. Appearance.
- b. Thickener type.
- c. Dropping point.
- d. Gas evolution.
- e. Rheology.

<sup>&</sup>lt;sup>1</sup>American Society for Testing and Materials, 100 Bar Harbor Drive, West Conshohocken, PA 19428.

<sup>&</sup>lt;sup>2</sup>Deutsches Institut fur Normung, Burggrafenstrasse 6, D-10787, Berlin, Germany

- f. Water resistance.
- g. Fluid type.
- h. Flash point.
- i. Evaporation.
- j. Oil separation.
- k. Specific gravity.

The thread compound manufacturer shall publish timely product bulletins of any change in formulation by the manufacturer which changes any performance characteristic. All documentation shall provide data that is representative of a typical production batch.

Test and inspection records generated under this recommended practice shall be retained by the manufacturer and shall be available to the purchaser for a period of three years after the date of manufacture.

#### 4.2 PHYSICAL/CHEMICAL CHARACTERISTICS

The physical/chemical characteristics of performance based thread compounds can vary widely and the formulation of many of the available compounds is proprietary. Therefore, the user should consider the performance properties and recommendations by compound manufacturers in addition to the physical/chemical characteristics outlined below.

#### 4.2.1 Dropping Point

Dropping point is a measure of thermal and chemical stability of thread compounds. API Bulletin 5A2 modified specifies a minimum dropping point of 190° F (88° C), but stated the need for service capabilities to 300° F (149° C). In order to meet current service requirements, the minimum dropping point temperature shall be 280° F (138° C) as measured in accordance with ASTM Specification D 566 or D 2265.

#### 4.2.2 Evaporation

Evaporation is an indicator of a compound's physical/chemical stability at elevated temperature, related to the base grease/oil or other additives. Evaporative loss shall be measured as a volumetric percentage. Due to the wide variation in density of thread compounds currently in service, weight percentage does not provide a reliable basis for comparison with API Bulletin 5A2 modified thread compound. Thus, the evaporation volumetric percentage was calculated from the weight percentage required in API Bulletin 5A2. The maximum evaporative loss, when evaluated in accordance with the test method in Appendix C, in 24 hours at 212° F (100° C) shall be 3.75 percent by volume.

Note: Volumetric percentage calculation—Determine the specific gravity of the test compound sample. The weight of the quantity of sample required for the test is determined either by direct measurement or by subtracting the tare weight of the test equipment containing the sample from the total weight of the sample plus equipment. The volume of the sample is then calculated by dividing the sample weight by its specific gravity. The oils or volatiles lost by separation or evaporation, if they are hydrocarbon based, can be assumed to have an approximate specific gravity of 0.9. The volume of the separated/evaporated material is calculated by dividing the measured weight loss by 0.9 (or the actual specific gravity). The volume percentage loss is then calculated by dividing the volume of the separated/evaporated material by the volume of the starting sample and multiplying by 100.

#### 4.2.3 Gas Evolution

Gas evolution is an indicator of a compound's chemical stability at elevated temperature. When evaluated in accordance with the test method in Appendix F, for 120 hours at  $150^{\circ}$  F (66° C), the volume of gas evolution shall be 20 cc, maximum.

#### 4.2.4 Oil Separation

Oil separation is an indicator of a compound's physical/chemical stability at elevated temperature, related to the base grease/oil. Oil separation loss is measured as a volumetric percentage. Due to the wide variation in density of thread compounds currently in service, weight percentage does not provide a reliable basis for comparison with API Bulletin 5A2 modified thread compound. API Bulletin 5A2 specifies a maximum oil separation loss of 5.0 percent by weight when evaluated in accordance with the test method in Appendix D (24 hours at 150° F (66° C) using a perforated nickel cone). The equivalent volume percent is approximately 10.3 percent. In order to meet current service requirements, the maximum oil separation loss shall be 10.0 percent by volume using a nickel gauze cone for 24 hours at 212° F (100° C).

Note: Volumetric percentage calculation—Determine the specific gravity of the test compound sample. The weight of the quantity of sample required for the test is determined either by direct measurement or by subtracting the tare weight of the test equipment containing the sample from the total weight of the sample plus equipment. The volume of the sample is then calculated by dividing the sample weight by its specific gravity. The oils or volatiles lost by separation or evaporation, if they are hydrocarbon based, can be assumed to have an approximate specific gravity of 0.9. The volume of the separated/evaporated material is calculated by dividing the measured weight loss by 0.9 (or the actual specific gravity). The volume percentage loss is then calculated by dividing the volume of the separated/evaporated material by the volume of the starting sample and multiplying by 100.

#### 4.2.5 Penetration

Penetration is a measure of the consistency (stiffness) of a lubricating grease and related to: (a) the ease of application (brushability) of a thread compound; (b) its tendency to stay in place after application, and (c) the likelihood of stratification (settling of solids) during storage. When evaluated in accordance with the test method in Appendix B, the acceptable range for the manufacture of a specific thread compound shall not be greater than 30 cone penetration points. Since material density affects this measurement, penetration is not recommended for relative comparison of materials with widely varying densities.

Note: Brookfield viscosity (ASTM D 2196) is not substantially affected by material density and therefore, should provide closer correlation to

brushability than the Cone Penetration specified in API Bulletin 5A2. The range below was determined using several different supplier samples of API Bulletin 5A2 modified thread compounds as well as proprietary thread compounds used currently with casing, tubing and line pipe connections. A specific spindle size, rotational speed and test temperature should be utilized to develop viscosity data for comparison. The Brookfield viscosity range, as measured with a #7 Spindle at 10 RPM and 77° F (25° C), was 200,000 to 400,000 centipoise (cp). A typical value for API Bulletin 5A2 Modified thread compounds could range from 200,000 to 240,000 cp.

#### 4.2.6 Specific Gravity

The specific gravity of a thread compound is determined by the constituents utilized in the formulation. The range of specific gravity for a particular thread compound is an indication of the consistency of manufacture. The specific gravity (gm/cc) of a particular thread compound shall not vary more than 5.0 percent from the manufacturer's established mean value.

#### 4.2.7 Water Leaching

Water leaching is an indicator of the physical/chemical stability of thread compounds when exposed to water at elevated temperature. When evaluated in accordance with Appendix G, in 2 hours at  $150^{\circ}$  F (66° C), the weight loss of the compound shall be 5.0 percent, maximum.

# 5 Compound Performance Properties

Note: The reduced scale (bench top) tests for the following compound performance properties may not correlate directly with full size connection tests or be representative of field service. They are not intended to exclude other methods, but to limit them to the performance property requirements discussed herein.

#### 5.1 FRICTIONAL PROPERTIES

A primary purpose of thread compound is to act as a lubricating material and to provide consistent and repeatable frictional properties between the mating connectors of a threaded connection. For a given amount of connection makeup (number of threads of engagement), the torque will vary in direct proportion to the coefficient of friction of the connection system. Changing frictional properties affects the following three important torque values:

- a. Torque required to make up the connection.
- b. Torque required to cause further make up.
- c. Torque required to break-out the connection.

The frictional properties of a thread compound in a connection depend on several factors external to the compound. These external factors include surface finish, relative surface speed of the mating surfaces, film thickness and surface contact pressure. Each of these parameters should be controlled when designing a test to determine the frictional properties and when using the compound in the field. A laboratory test for developing the thread compound frictional properties shall be performed and recorded. The laboratory test methods described in Appendix H are intended to provide a means for comparing thread compounds with the Reference Standard, described in Appendix A, A.6.

*CAUTION:* If different thread compounds are applied to opposite ends of a coupling, frictional differences can occur between the mill end connection and the field end connection, resulting in excessive engagement of the mill end prior to adequate engagement of the field end. The field torque required for proper connection assembly should be determined in accordance with the procedures found in API Recommended Practice 5C1, *Recommended Practice for Care and Use of Casing and Tubing*.

#### 5.2 EXTREME SURFACE CONTACT PRESSURE (GALL RESISTANCE) PROPERTIES

A primary purpose of thread compound is resistance to adhesive wear (metal galling) of mating connection surfaces when subjected to extreme surface contact pressure.

High surface contact pressure may be created in threaded connections during two different activities—manufacturing, and field service. The first includes product variations, such as geometric characteristics (thread length, pipe and coupling thickness) and process variations, such as machining (thread taper, lead, flank angles), surface finishing and coating. The second includes handling damage, contact surface contamination, inadequate or inconsistent application of thread compound, misalignment during stabbing or rotation and improper torque application.

An important consideration is the greater tendency of some materials toward connection galling than others. Galling tendency increases between two smooth metal surfaces along with an increasing similarity of composition, similarity of relative hardness, and decreasing actual hardness. For oil field tubular goods, the composition and hardness of the mating pair is virtually the same. Consequently, oil field tubulars are relatively gall-prone. Therefore, a coating for one of the connector pairs, such as zinc plate or iron phosphate, and API Bulletin 5A2 modified thread compound has traditionally provided adequate galling resistance.

The increasing use of quench-hardened alloys and the significantly greater tendency of martensitic chromium steels and nickel-based alloys to galling requires that all possible care be applied to every aspect of surface preparation, coating, handling, thread compound selection and application and connection assembly to achieve connection galling resistance.

A laboratory test for developing the thread compound extreme surface contact pressure properties (gall resistance) shall be performed and recorded. The laboratory test methods described in Appendix J are intended to provide a means for comparing thread compounds with the Reference Standard described in Appendix A, Section A.6.

*CAUTION:* Connections with inadequate surface preparation may not resist galling, regardless of handling or assembly technique. Conversely, connections with adequate surface preparation may be galled with inadequate handling or assembly technique. Each activity should be controlled to achieve repeatable extreme pressure properties. With this background, the combination of proper surface preparation, connection coating and thread compound selection and application should be established for each type of connection material, based on its tendency to gall, during both assembly and disassembly following service.

#### 5.3 FLUID SEALING PROPERTIES

A primary purpose of thread compound is to provide fluid sealing for thread clearances, such as the helical root-to-crest clearances in API round threads and the helical stab flank clearance in API buttress threads. Sealing is typically accomplished in a thread compound with solid particles that agglomerate to plug the thread clearances to prevent the contained fluid from passing through the connection.

Connection sealing also requires that positive contact pressure be maintained along the thread interface in order to insure the geometric integrity of the helical sealing passages. Contact pressure requirements are established for connection fluid pressure integrity and may be found in API Bulletin 5C3, Formulas and Calculations for Casing, Tubing, Drill Pipe and Line Pipe Properties.

A laboratory test for developing thread sealing properties of the thread compound shall be performed and recorded. The laboratory test methods described in Appendix K are intended to provide a means for comparing thread compounds with the Reference Standard, described in Appendix A, Section A.6.

#### 5.4 APPLICATION/ADHERENCE PROPERTIES

Thread compounds shall be applied in a manner consistent with the manufacturers recommendations and in sufficient quantity to provide effective lubrication and sealing characteristics for API connections. The thread compound shall be brushable and capable of adherence over a temperature range of 20° F to 150° F ( $-6.7^{\circ}$  C to  $66^{\circ}$  C) without either agglomerating or sliding off of the connector.

Laboratory tests for determining the thread compound application and adherence properties shall be performed and recorded. The laboratory test methods described in Appendix E, Application/Adherence Test, are intended to provide a means for the relative comparison of thread compound performance and may not be representative of field service.

### 5.5 CORROSION INHIBITION PROPERTIES

Thread compounds are utilized to provide shipping and storage protection on threaded connections, as well as lubrication and sealing properties. Therefore, the thread compound should provide an effective barrier against (and not contribute to) corrosive attack of connection threads. The corrosion inhibition properties of thread compounds will depend on the application variables, such as the following:

- a. Compound additive types and treatment levels.
- Type and condition of threading process fluids and residue.
- c. Compound application method and equipment.
- d. Type of thread protector and installation method.
- Specific user application procedures and ambient conditions.
- f. Compatibility with thread storage compound.
- g. Galvanic differences between compound components, thread protector, environment and connector material.

A laboratory test should be performed and recorded to determine whether potentially corrosive components are present in the thread compound. A generally accepted method is ASTM D 4048-91, *Test Method for Detection of Copper Corrosion from Lubricating Grease*. Although copper is not typically utilized in the production of OCTG connections, it more readily reacts in the presence of reactive materials such as sulfur, chlorine, et cetera, which can also damage steel. Thread compounds shall provide a level 1B or better by this method.

A laboratory test for determining the thread compound corrosion inhibition properties should be performed and recorded. Thread compounds vary as to the existence and treatment level of corrosion inhibition. Therefore, it is the purchaser's/user's responsibility to outline the necessary requirements with the compound manufacturer for products being utilized for storage or corrosive field applications. The methods listed in Appendix L are generally accepted and utilized by lubricant test facilities. They are intended to provide a means for the relative comparison of thread compound performance.

### 5.6 COMPOUND STABILITY PROPERTIES

Thread compound stability is an essential element to the provision of adequate sealing properties within an assembled connection. Instability in the form of excessive softening and separation can result in the development of leak passages over time or with temperature.

Thread compound stability should be adequate to resist softening of more than 30 cone penetration points during storage. Stratification or separation of greater than 10 percent by volume over a minimum period of 12 months would constitute failure. Thread compound stability test results shall be available in a product bulletin or certificate of conformance.

Laboratory tests for detecting the chemical stability properties of a thread compound should be performed. The laboratory test requirements described in 4.2, shall be performed and the data reported in a product bulletin. The test described in Appendix M should also be performed, and is intended to provide a means for the relative comparison of thread compound performance.

## 6 Quality

This document is based on the assumption that the function of API thread compound used with threaded connections for API casing, tubing and line pipe may be defined by performance properties that include, but are not limited to, friction, extreme surface contact pressure, thread sealing, adherence, and corrosion inhibition, as described in Section 5 of this document.

These performance properties are complex and sometimes interrelated and therefore difficult to quantify. Minor differences in product composition, manufacture, or application may result in significant changes in performance properties.

For these reasons, the manufacturer should have implemented a comprehensive system of quality assurance to insure that the represented properties are maintained throughout the range of variation of raw materials, manufacturing processes and application environment. This quality system should conform to a broadly recognized Quality Management System, such as API Specification Q1, *Specification For Quality Program*.

The manufacturer shall, upon request by the purchaser, furnish a certificate of compliance stating that the thread compound has been tested and evaluated in accordance with this recommended practice and has been found to meet all requirements.

### 7 Marking Requirements

#### 7.1 MARKING

Each container of thread compound manufactured and evaluated in conformance with the requirements of this recommended practice shall be marked with the manufacturer's identification, traceability identification and the following statement:

This thread compound was tested in conformance with the requirements of API Recommended Practice 5A3, *Recommended Practice on Thread Compounds For Casing, Tubing and Line Pipe.* 

#### 7.2 LABELING

**7.2.1** Unless a thread compound is dually applicable for both rotary shouldered connections and casing, tubing and line pipe connections, the container shall be conspicuously labeled with the following cautionary statement:

#### NOT RECOMMENDED FOR ROTARY SHOULDERED CONNECTIONS

**7.2.2** Unless a storage compound is dually applicable for both thread compound service and storage compound service, the container should be conspicuously labeled with the following cautionary statement:

#### STORAGE COMPOUND - NOT RECOMMENDED FOR MAKE UP

**7.2.3** Each container shall be conspicuously labeled with cautionary statements regarding storage, preparation or application required to achieve the characteristics disclosed in the product bulletin. Two examples are:

# STIR WELL BEFORE USING THREAD COMPOUND PLUS INLAND SHORT-TERM STORAGE

# APPENDIX A—API MODIFIED THREAD COMPOUND AND REFERENCE STANDARD

Note: The following four sections of Appendix A were transferred intact from API Bulletin 5A2 (superseded by Recommended Practice 5A3), omitting all references to "silicone thread compound." Section A.6 constitutes the Reference Standard, which is based on limiting the tolerances of API Bulletin 5A2 Modified constituents closer to nominal values.

# A.1 Compound

The compound shall be designated as the "modified thread compound." It shall be a mixture of metallic and graphitic powders uniformly dispersed in a grease base. Proportions of solids and grease base shall be as listed in Table A-1.

Table A–1—Proportions of Solids and Grease	Base
--	------

Component	Percent by Weight	
Total Solids Grease Base Total	64.0, ±2.5 36.0, ±2.5 100.0	

# A.2 Composition of Solids

The solids shall be a mixture of amorphous graphite, lead powder, zinc dust, and copper flake in the proportions listed in Table A–2 and as specified in A.5.1 to A.5.4.

Table A–2—Propor	tions of	Solids
------------------	----------	--------

	Percent b	y Weight
Constituent	Total Solids	Compound
Amorphous Graphite	28.0	18.0, ±1.0
ead Powder	47.5	$30.5, \pm 0.6$
inc Dust	19.3	12.2, ±0.6
Copper Flake	5.2	3.3, ±0.3
fotal	100.0	64.0

# A.3 Grease Base

Grease base for the modified thread compound shall be a grease which, when combined with the powdered metals and graphite, will comply with the control and performance test requirements listed in Table A–3.

## A.4 Control and Performance Tests

The thread compound shall be subjected to control and performance tests for penetration, dropping point, evaporation, oil separation, gas evolution, water leaching, and brushing ability as designated in Table A–3. The thread compound shall comply with requirements listed in Table A–3 based on a test specimen which represents the entire contents of the container.

#### Table A–3—Modified Thread Compound Control and Performance Tests

Test	Requirement
Penetration (See Appendix B) Worked at 77°F (NLGI <sup>a</sup> Grade No. 1) After cooling at 0°F	310-340 200 minimum
Dropping point (° F) (ASTM D566)	190 minimum
Evaporation, percent (See Appendix C) 24 hr at 212°F	2.0 maximum
Oil separation, percent, nickel cone (See Appendix D) 24 hr at 150° F	5.0 maximum
Gas evolution, cc at 150° F (See Appendix F) 120 hr	20 maximum
Water leaching, percent loss in weight (See Appendix G) After 2 hr at 150° F	5.0 maximum
Brushing Ability (Appendix E)	Applicable at 0° F

<sup>a</sup> National Lubricating Grease Institute, 4635 Wyandotte Street, Kansas City, Missouri 64112-1596, USA. Tel: 816-931-9480.

# A.5 Component Material Requirements

**A.5.1** Graphite should be a natural amorphous type, free of powdered coal, lamp black, carbon black, oil, grease, abrasives or other deleterious materials. It should conform to the following requirements:

Ash, (ASTM C561)	28% min., 37% max.
Particle size: (ASTM E11)	
Pass No. 50 sieve, min.	100.0 %
On No. 100 sieve, max.	1.0 %
On No. 200 sieve, min.	10.0 %
Pass No. 325 sieve	30% min., 80% max.

**A.5.2** Lead powder should conform to the following requirements:

Free metal content, (ASTM D13	95.0% min.
Lead oxide content, max, (ASTI	5.0%
Particle size: (ASTM E11)	
Pass No. 50 sieve, min.	100.0%
On No. 100 sieve, max.	2.0%
Pass No. 325 sieve	30% min., 92% max.

**A.5.3** Zinc dust should be homogenous. The zinc dust should be so constituted that the finished thread compound can meet the gas evalution test requirements of Table A–3. It should conform to the following requirements:

Composition: (ASTM D521)	
Total Zinc, calculated as Zn, min.	98.0%
Metallic Zinc, min.	95.05%
Iron, lead, cadmium, max.	1.0%
Calcium, calculated as CaO, max.	0.5%
Moisture, other volatiles, max.	0.1%
Zinc oxide, ZnO	Remainder
Particle size: (ASTM E11)	
Pass No. 100 sieve, min.	100.0 percent
Pass No. 325 sieve, min.	90.0 percent

**A.5.4** Copper flake should conform to the following requirements:

Copper, percent (ASTM D283)	97.0%
Grinding/polishing compound, max.	0.25%
(ASTM D283)	
Particle size: (ASTM E11)	
Pass No. 200 sieve, min.	100.0%
Pass No. 325 sieve, min.	99.0%
Thickness > 5 microns, max.	5.0%

### A.6 Reference Standard Formulation

In order to provide the replication required for a Reference Standard, the above tolerances for constituent particle size, solids content, grease thickeners and oil viscosity have been limited as shown in Table A–4.

Table A–4—Reference Standard Content Tolerances

Component	Percent by Weight	
Grease Base	36.00, ±1.05	
Amorphous Graphite	18.00, ±0.30	
Lead Powder	30.50, ±0.50	
Zinc Dust	12.20, ±0.20	
Copper Flake	3.30, ±0.05	

The grease base shall conform to the requirements in Table A–5.

#### Table A-5—Requirements of Grease Base

Property	Requirement	
Consistency	NLGI Grade "0"	
Worked penetration (ASTM D 217-60 strokes)	365–385	
Thickener (lithium 12-hydroxystearate, wgt %)	2.0-4.5	
Petroleum oil viscosity	115–170 cSt at 40° C 9.5–14.0 cSt at 100° C	

Technical Note: API Bulletin 5A2 did not specify requirements for the "extreme pressure" performance properties of the base grease utilized in the formulation of API Modified Thread Compound. Commercial formulations however, have included extreme pressure additives because of their recognized benefit in resisting the galling and wear of opposing contact surfaces under high bearing pressures. The additives used by commercial manufacturers however, can vary substantially in quality and performance. Therefore, the Reference Standard formulation was specified to exclude those and other additives that may introduce a variable that would adversely affect the direct comparison of discreet test data. Full scale test data from a combined API/Joint Industry research project (continuation of API PRAC XX-51: Environmentally Acceptable Pipe Thread Compounds: Evaluation and Establishment of Performance Standards, Phase 1) indicates that it may be necessary to include an extreme pressure additive in the formulation of the grease base specified for the Reference Standard. The average break-out torque for 3.5 in. N-80 tubing exceeded 150 percent of the make up torque when using the Reference Standard formulated as specified without extreme pressure additives. There was also a high incidence of galling of the test specimen connection members. These problems were addressed by the addition of a commercial formulation of antimony dialkyldithiocarbamate (available as "Vanlube 73" through R.T. Vanderbilt Company, Inc., Norwalk, Connecticut) at the rate of 2.0 percent (by weight) to the base grease. This particular extreme pressure additive was chosen because of its wide use in the lubricant industry and its ready availability. The base grease with the addition of the extreme pressure additive exhibited a Four-Ball Weld Point (ASTM D 2596: Test Method for Measurement of Extreme-Pressure Properties of Lubricating Grease) of 250 kg and a Timken OK Load (ASTM D 2782: Test Method for Measurement of Extreme-Pressure Properties of Lubricating Fluids) of approximately 20 lb. (9.08 kg). It is recommended that if antimony dialkyldithiocarbamate is not available, that the extreme pressure additive that is utilized be added at a rate that will yield similar results as in the ASTM test methods cited.

The solid components shall conform to the requirements in Table A–6.

Test Parameter	Amorphous Graphite	Copper Flake	Lead Powder	Zinc Dust
Ash (wgt percent)	30-36	NA	NA	NA
Moisture, maximum (percent)	1.0	0.1	0.1	0.1
Metallic content, minimum (percent)	NA	97.0	95.0	95.0
Metal/other oxides, maximum (percent)	NA	3.0	5.0	5.0
Particle Size Distribution				
Through #50 sieve, minimum (percen	t) 100	100	100	100
On #100 sieve, maximum (percent)	0.3	0.0	1.0	0.0
On #200 sieve, range (percent)	10-18	0	5-25	2 maximum
On #325 sieve, range (percent)	20-31	1	14-55	5 maximum
Through #325 sieve, range (percent)	50-70	99	40-80	93

### Table A-6—Reference Standard Constituent Limitations

Note: This Reference Standard is not intended for general service.

# **APPENDIX B—PENETRATION TEST**

# B.1 Scope

This section describes a procedure for measuring consistency (stiffness) of thread compound.

# **B.2** Apparatus

- a. Penetrometer.
- b. Cone (full-scale).
- c. Grease worker.
- d. Spatula.

# **B.3** Procedure

Prepare two samples for worked penetration. After working, determine penetration of the first sample at 77° F (25° C) in accordance with ASTM D 217.

After working the second sample at 77° F ( $25^{\circ}$  C), mound excess compound on top of penetration cup and place in cooling chamber along with penetrometer cone for 3 hr at 0° F (minus 17.8° C). After the 3-hr soaking period, remove cup and strike excess compound flush with the top of cup. Place cup and sample in cooling chamber for an additional hour. Without further working, determine penetration as quickly as possible.

# APPENDIX C-EVAPORATION TEST

# C.1 Scope

This section describes a procedure for measuring losses of volatile materials from thread compound at a temperature of  $212^{\circ}$  F (100° C) under static conditions.

# C.2 Apparatus

a. No. 3 Coors porcelain evaporating dish, shallow form or its equivalent.

b. Gravity convection oven capable of maintaining a test temperature of  $212^{\circ}$  F,  $\pm 2^{\circ}$  F (100° C,  $\pm 1.1^{\circ}$  C).

- c. Precision balance.
- d. Desiccator.

# C.3 Procedure

A 50-gram sample is weighed into the tared evaporating dish. The assembly is then placed in an oven at  $212^{\circ}$  F,  $\pm 2^{\circ}$  F (100° C,  $\pm 1.1^{\circ}$  C), for 24 hr. Sample is then placed in a desiccator, cooled, weighed, and weight loss reported as percent evaporation loss.

# APPENDIX D—OIL SEPARATION TEST

## D.1 Scope

This section describes a procedure for measuring the tendency of thread compound to separate oil at  $150^{\circ}$  F (66° C) under static conditions.

# **D.2** Apparatus

a. Nickel Filter Cone (Fisher Scientific Company Cat. No. 9-760 or equivalent). The cone is 38 mm diameter and the sides have a 60° angle. The cone is perforated with approximately 200 holes of 1/32 in. (0.79 mm) diameter. An acceptable alternative is a 60 mesh nickel gauze cone from Federal Test Method Standard 791B-321.2.

Note: The nickel gauze cone may result in a greater percent weight loss than the nickel filter cone. Reference data may be obtained from the API Exploration and Production Department.

b. 50 ml beaker cut to a height of  $1^{5}/_{8}$  in. (41.28 mm).

c. Gravity convection oven capable of maintaining  $150^{\circ}$  F,  $\pm 2^{\circ}$  F (66° C,  $\pm 1.1^{\circ}$  C).

- d. Precision balance.
- e. Desiccator.

# **D.3** Procedure

A 20 gram sample is weighed into the nickel filter cone. Care should be taken to avoid formation of air pockets within the compound. Exposed surface of the compound should be smooth and convex to prevent trapping free oil. The cone is then suspended in a tared beaker so that the cone tip is approximately  $^{3}/_{8}$  in. (9.5 mm) from bottom of beaker. The cone-beaker assembly is then placed in the oven for 24 hr at  $150^{\circ}$  F (66° C) and weighed. The cone is then removed from the beaker; the beaker is cooled in a desiccator and weighed. The gain in weight of the beaker is calculated as percent oil separation.

# APPENDIX E—APPLICATION/ADHERENCE TEST

# E.1 Scope

This section describes a method for evaluating the brushability and adherence of a thread compound.

# E.2 Procedure

**E.2.1** Place 1 pint (473 cc) of thread compound into a quart (liter) plug-top sample can. Refrigerate (or heat) sample, stiff (short bristle) brush and API EU tubing sample to at least  $20^{\circ}$ F (-6.9°C), until temperature stabilizes.

**E.2.2** Weigh the compound coated brush before and after application of the compound sample to the threads of the API pipe specimen.

**E.2.3** After thermal stabilization, brush the compound onto the threads of the API specimen. The adherence shall be such that at least 75 percent of the compound is deposited to the threaded surfaces, to include thread roots, without more than three applications. Verify percentage by weight measurements as described in E.2.2 above.

**E.2.4** Clean the specimen and brush and repeat the procedure utilizing  $150^{\circ}$  F (66° C) in a controlled oven to condition the specimen, brush and compound.

**E.2.5** Record and report test results.

# APPENDIX F—GAS EVOLUTION TEST

# F.1 Scope

This section describes a procedure for measuring the gas evolved from a thread compound.

# F.2 Apparatus

Apparatus as shown in Figure F-1, Figure F-2 or equivalent.

### F.3 Procedure

1. Fill bomb A to within 5/8 in. (15.9 mm) of the top edge with test compound, being careful to avoid air pockets. The use of a vibrator is helpful. Smooth flush top surface.

2. Seal the bomb, close needle valve B and insert the bomb assembly in an oil bath preset at the test temperature.

3. Attach the bomb assembly by means of tubing C to the gas bottle D and manometer E.

4. Open the needle valve B.

5. At the end of 15 minutes, observe the pressure increase indicated by the manometer. This increase is caused ordinarily by the normal expansion of air in the system and of the compound itself.

6. Open valves F and G and measure displaced water by means of a graduate. Record. Close valves F and G.

7. Repeat step f. at periodic intervals over a 5-day test period.

8. Calculate evolution of gas as follows: Determine displacement in cc caused by normal expansion of air and compound at the test temperature. Subtract from total displacement. Remainder is displacement caused by gas evolution.

## F.4 Sample Test Data

- a. Oil bath temperature:  $65.56^{\circ}$ C ( $150^{\circ}$ F).
- b. Room temperature:  $25^{\circ}$  C (77° F).
- c. Temperature difference: 40.56°C (73°F).

d. Coefficient of expansion of air (change in volume per unit volume per degree C): 0.00367.

- e. Diameter of container (D=2r): 2 in. (50.8 mm).
- f. Air depth (*h*):  $\frac{5}{8}$  in. (15.9 mm).
- g. Compound sample: 275 grams.

### F.5 Calculations

1. Volume of air in bomb above compound at 25° C (77° F)

 $V = r \times h = 3.1416 \times 1 \times 0.625 \times 16.387$  cc/in.

=32.176 cc (initial air volume).

2. Expansion of the volume of air in bomb at 65.56° C (150° F)

$$V = 32.176 \times 0.00367 \times 40.56$$

= 4.79 cc (expansion of the volume of air bomb is the volume of air displaced) × 100

= 14.89 percent (volume change)

3. Contraction of initial air volume displaced from bomb due to cooling to  $77^{\circ}$  F (25° C)  $4.79 \times 14.89$  percent = 0.71 cc.

4. Corrected expansion of air displaced from the bomb.

5. Evolved gas = total displaced volume – corrected displaced air expansion volume.



Figure F–1—Gas Evolution Test Apparatus—A



Figure F–2—Gas Evolution Test Apparatus—B

### G.1 Scope

This section describes a procedure for determining the ability of thread compound to resist the washing action of water.

# G.2 Apparatus

Apparatus as shown in Figure G–1, Figure G–2 or equivalent. Apparatus includes a 50 mm Coors No. 3 porcelain filter cone; or a 60 mesh nickel gauze cone as specified in Federal Test Method Standard 791B-321.2 (see note); 100 ml glass beaker with 6 equidistant  $^{1}/_{4}$  in. (6.35 mm) holes located  $^{1}/_{16}$  in. (1.59 mm) from the bottom of the beaker; Chromel triangle to support the cone and 100 ml beaker; 1000 ml beaker with side arm extension at the bottom;  $^{1}/_{16}$  in. (1.59 mm), mesh screen; 6 in. brass cylinder with a diameter of 3 in. (76.2 mm), wall thickness of  $^{1}/_{4}$  in. (6.35 mm) from the top, and  $^{1}/_{8}$  in. (3.18 mm) orifice centered in the bottom; a centrifugal pump capable of circulating 1 liter of 150° F (66° C) water per minute; two pieces of  $^{1}/_{4}$  in. (6.35 mm) ID connecting hose; ring stand; and heater.

Note: The nickel gauze cone may result in a greater percent weight loss than the nickel filter cone. Reference data may be obtained from the API Exploration and Production Department.

### G.3 Procedure

A tared porcelain filter cone is filled with approximately 17 grams of sample, smoothed and a slight concavity of about  $1/_{32}$  in. (0.79 mm) made with a spatula. The cone is supported in the 100 ml beaker and both, in turn, are supported by a triangle in the 1000 ml beaker. The entire glass assembly is placed on the hot plate which is elevated from the pump. The brass cylinder is supported by a clamp from the ring stand. The mesh screen baffle is placed equidistant between the brass cylinder and the upper rim of the cone which are 1/2 in. (12.7 mm) apart. At least 500 ml of distilled water previously heated to 150° F (66° C) in the 1000 ml beaker are recycled from the 1000 ml beaker to the brass cylinder is so regulated by a screw clamp that the head of water is just up to the overflow level. Any water washed through the grease escapes via the holes in the small beaker into the large beaker where it has been noted that it clings to the side. The water is recirculated for 2 hr for which time the temperature remains between 140–150° F (60–66° C). After the test has been completed, the apparatus is disassembled, the cone and contents dried for a period of 24 hr at 150° F (66° C), and the weight loss is calculated as a percent of the original sample.



Figure G-1—Water Leaching Test Apparatus—A



Note: Constant temperature bath located in gas evolutation test apparatus (See Figure F-2).

Figure G-2-Water Leaching Test Apparatus-B

# APPENDIX H—FRICTIONAL PROPERTIES TEST

#### H.1 Full Size Connection Test

A full size test to determine frictional properties should be performed. At least two tests are required; one for tubing, preferably  $3^{1/2}$  in. (88.9 mm) OD, and one for casing, preferably  $9^{5/8}$  in. (244.5 mm) OD. Since the coefficient of friction can vary with thread form variation, care should be exercised to insure uniformity of test sample, thread form and fit.

Technical Note: The full scale test procedure referenced in these sections specifies a certain number of turns past a low "Reference Torque" for the make up of 8-round test specimens. Full scale test data from a combined API/Joint Industry research project (continuation of API PRAC Project XX-51, Environmentally Acceptable Pipe Thread Compounds: Evaluation and Establishment of Performance Standards, Phase 1) demonstrated that if thread compounds have substantially different friction properties and/or composition (e.g. solid component type or particle size, volume percent solids) there can be a significant difference (one full turn or greater) in the initial engaged position or stand-off of the test specimen connection members when using a Standard Reference torque. This difference in the initial stand-off will result in a similar difference in the final engaged position. It is essential that any comparative testing of thread compounds, either frictional properties or fluid sealing properties, be done to the same final engaged position within the allowable API tolerances. The reason being that the amount of engagement of the connection members determines both the pull-out strength and the leak-tightness of the connection. In the research project referenced above, an initial Reference Standoff was established using the Reference Standard. The connection test specimens for all subsequent compounds tested were initially made-up to the Reference Stand-off and then made-up to the specified number of turns to the final engaged position.

# H.2 Laboratory Test

A laboratory test should be conducted to determine frictional properties. An example is the procedure described in API Recommended Practice 7A1. This procedure utilizes a shouldered fixture with cylindrical threads. It does not entrap the thread compound, allowing the film thickness to be very thin. The surface contact pressure is relatively high, between 30,000 psi (206.8 megaPascals) and 60,000 psi (413.7 mega-Pascals). This compares to 5,000 psi (34.5 megaPascals) to 20,000 psi (137.9 megapascals) for API casing, tubing and line pipe connections.

The coefficient of friction determined by API Recommended Practice 7A1 generally ranges 40 percent (+/-) around a value of 0.08. However, when full scale tests are performed on API 8-round connections, the same compounds can indicate frictional coefficients as low as 0.02–0.04. Such low coefficients may be caused by the substantial differences in configuration between various connections and the RP 7A1 specimen. In API tubing and casing connections, low coefficients can result from reduced surface contact pressure between connectors as they "float" together on a thick layer of thread compound during make up. Thus, an apparent coefficient of friction is indicated that is a function of both the compound application and the connection configuration. A test method should be selected to control such variables as the volume of compound applied.

### H.3 Industry Test Methods

API PRAC Project 88, 89, 91–51 *Investigation of Pipe Thread Compounds*, November 1992, Section 4.0, "Small Scale Friction Test Development," provides a development history of test methods for thread compound frictional properties. Section 6.0, describes a full size connection test procedure that includes the evaluation of compound frictional properties.

Note: The thread compound manufacturer is responsible for frictional performance of the thread compound.

Note: For more information, including a comparison of the performance of the Reference Standard to a commercial formulation of API Modified, it is recommended that the summary report of the referenced research project be reviewed.

# APPENDIX J-EXTREME SURFACE CONTACT PRESSURE (GALLING) TEST

# J.1 Industry Test Methods

API PRAC Project 88, 89, 91–51, *Investigation of Pipe Thread Compounds*, November 1992, Section 5.0, "Galling Test," provides a development history of test methods for thread compound extreme surface contact pressure (galling) properties.

Note: Since industry consensus has not been achieved, manufacturers and users are encouraged to further develop galling test methods until such acceptance occurs. The thread compound manufacturer is responsible for the extreme surface contact pressure performance of the thread compound.

# APPENDIX K—FLUID SEALING TEST

### K.1 Industry Test Methods

API PRAC Project 88, 89, 91–51, *Investigation of Pipe Thread Compounds*, November 1992, Section 6.0 describes a full size connection test for the evaluation of thread compound fluid sealing properties.

Numerous attempts have been made to design laboratory tests for evaluating the sealing ability of tubing and casing thread compounds. At least eleven attempts have been made between 1957 and 1993, to develop a small scale test for thread compound sealing ability. Of these eleven different methods, none has achieved industry acceptance or been adopted by either thread compound manufacturers or users. From these past efforts it is clear that designing a small scale test to determine the sealing ability of thread compounds will require extensive additional effort.

API PRAC Project 88, 89, 91–51, Section 3.0 utilized a spiral groove test apparatus that closely approximated API BTC thread clearance and leak path length. Some inconsistency was found and may be attributed as much to particle size variation among compounds as to the test method itself. The potential for positive capability to discriminate between compounds is indicated. However, the influence of particle shear properties on sealing was not accounted for.

Note: Since industry consensus has not been achieved, manufacturers and users are encouraged to further develop sealing test methods until such acceptance occurs. The thread compound manufacturer is responsible for the fluid sealing performance of the thread compound.

Technical Note: The full scale test procedure referenced in these sections specifies a certain number of turns past a low Reference Torque for the makeup of 8-round test specimens. Full scale test data from a combined API/Joint Industry research project (continuation of API PRAC Project XX-51, Environmentally Acceptable Pipe Thread Compounds: Evaluation and Establishment of Performance Standards, Phase 1) demonstrated that if thread compounds have substantially different friction properties and/or composition (e.g. solid component type or particle size, volume percent solids) there can be a significant difference (one full turn or greater) in the initial engaged position, or stand-off of the test specimen connection members when using a Standard Reference Torque. This difference in the initial stand-off will result in a similar difference in the final engaged position. It is essential that any comparative testing of thread compounds, either frictional properties or fluid sealing properties, be done to the same final engaged position within the allowable API tolerances. The reason is that the amount of engagement of the connection members determines both the pull-out strength and the leak-tightness of the connection. In the research project referenced above, an initial Reference Standoff was established using the Reference Standard. The connection test specimens for all subsequent compounds tested were initially made-up to the Reference Stand-off and then made-up to the specified number of turns to the final engaged position.

Note: For more information, including a comparison of the performance of the Reference Standard to a commercial formulation of API Modified, it is recommended that the summary report of the referenced research project be reviewed.

# K.2 Recommended Test Parameters

Any test selected to satisfy the requirements of this recommended practice, should include the following process, as a minimum.

**K.2.1** Test pressure shall be applied with dry nitrogen gas.

**K.2.2** Maximum test pressure shall be 12,000 psi (82.7 megaPascals), or as agreed between purchaser and manufacturer.

**K.2.3** Leak detection shall be by the bubble method as shown in API Recommended Practice 5C5. Leak rate may be by visual recording of observed off-gas over time, or similar means, to yield a rate measured in cc/minute.

**K.2.4** Pressure shall be applied as in Figure K–1. Starting at 1000 psi (6.9 megaPascals), pressure shall be applied in 1000 psi (6.9 megaPascals) increments with each increment held for a minimum of 15 minutes. The maximum test pressure shall be held for a minimum of 8 hours (which should be observed continuously for the initial 15 minutes).

**K.2.5** A test log shall be maintained as shown in Figure K-2 and a continuous strip chart of the test pressure shall be recorded over the complete test duration.

**K.2.6** Reference pressure tests shall be performed using the reference compound prior to qualifying thread compound tests for record.

**K.2.7** Both test results shall be reported. The log and strip chart shall be made a part of the test report.



Figure K-1-Small-Scale Sealability Test Procedure

Time	Pressure	Off-gas (cc)	Comments

### SMALL SCALE SEALABILITY TEST LOG

Figure K–2—Small-Scale Sealability Test Log

# **APPENDIX L—CORROSION INHIBITION TESTS**

## L.1 Recommended Performance Criteria

Thread compounds should provide corrosion inhibition for API Specification 5CT Grade L80 material for 500 hours, minimum, at 100° F (38° C), when measured in accordance with ASTM B-117 Salt Fog (Spray) Test (5 percent NaCl, pH neutral), with an 80 cm collection rate of 1.0 to 2.0 ml per hour.

# L.2 Alternate Test Methods

Alternately, thread compounds may be evaluated by other test methods, including the following:

a. ASTM D 1743-87, *Test Method for Corrosion Preventative Properties of Lubricating Greases* (48 hours).

b. DIN 51 802, *Testing of the Corrosion Preventing Properties of Lubricating Greases* (164 hours).

c. SPE 11396, A Test Program for the Evaluation of Oil field Thread Protectors, Dale, B.A., Moyer, M.C. & Sampson, T.W., 1983; (40 days).

d. NACE 134, Evaluation of the Corrosion Inhibition Property of Storage Compounds, Choi, H.J. & Jones, S.B., 1990; (40 days).

# APPENDIX M—COMPOUND STABILITY TEST

# M.1 Recommended Performance Criteria

A representative sample of the thread compound shall be evaluated by baking at a minimum temperature of 280°F (138°C) for a minimum of 24 hours. The total loss due to base grease/oil separation or component evaporation shall not exceed 25 percent by volume.

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