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**Petroleum and natural gas industries —  
Evaluation and testing of thread  
compounds for use with casing, tubing and  
line pipe**

*Industries du pétrole et du gaz naturel — Évaluation et essai des graisses  
de filetage utilisées pour les tubes de cuvelage, les tubes de production et  
les tubes de conduites*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13678 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum and natural gas industries*, Subcommittee SC 5, *Casing, tubing and drill pipe*.

Annexes B to H and K form normative parts of this International Standard. Annexes A, I, J, L and M are for information only.

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## Introduction

This International Standard is based on API RP 5A3, first edition, June 1996.

This International Standard specifies requirements and gives recommendations for the manufacture, testing and selection of thread compounds for use on casing, tubing and line pipe based on the current industry consensus of good engineering practice.

It is intended that the words casing and tubing apply to the service application rather than to the diameter of the pipe.

The performance requirements of thread compounds for use with casing, tubing and line pipe include:

- consistent frictional properties that will allow both proper and uniform connection engagement;
- adequate lubrication properties to resist galling or damage of connection contact surfaces during make-up and break-out;
- adequate sealing properties for thread type seal connections and/or not inhibiting the sealing properties of non-thread sealing connections (e.g. metal-to-metal seals, polytetrafluoroethylene (PTFE) seals, etc.) depending upon service requirements;
- physical and chemical stability both in service and in expected compound storage conditions;
- properties that will allow effective application to the connection contact surfaces in expected service conditions and environment.

When evaluating the suitability of a thread compound, the user should define the service conditions, and then consider field trials and field service experience in addition to laboratory test results. Supplementary tests may be appropriate for specific applications which are not evaluated by the tests herein. The user and manufacturer are encouraged to discuss service applications and limitations of the compound being considered.

Representatives of users and/or other third party personnel are encouraged to monitor tests wherever possible. Interpolation and extrapolation of test results to other products, even of similar chemical composition, is not recommended.

It should be recognized by the user that testing in compliance with this International Standard does not in and of itself ensure adequate thread compound/connection system performance in field service. The user has the responsibility of evaluating the results obtained from the recommended procedures and test protocols and determining if the thread compound/connection system in question meets the anticipated requirements of that particular field service application.

Whether a thread compound meets local or global environmental legislation is outside the scope of this International Standard. However, it is the responsibility of the end user to be aware of the environmental requirements of the operating area and to select, use and dispose of the thread compound and related waste materials accordingly.



# Petroleum and natural gas industries — Evaluation and testing of thread compounds for use with casing, tubing and line pipe

## 1 Scope

This International Standard provides requirements, recommendations and methods for the manufacture, testing and selection of thread compounds for use on ISO/API round thread, buttress thread and proprietary casing, tubing and line pipe connections. The tests outlined within this International Standard are used to evaluate the critical performance properties, and physical and chemical characteristics of thread compounds under laboratory conditions.

These test methods are primarily intended for thread compounds formulated with a lubricating base grease. It is recognized that there may be materials used for the lubrication and/or sealing of threaded connections for which these test methods are not applicable.

This International Standard is not intended for the evaluation of compounds used with rotary shouldered connections. Such evaluation is described in API RP 7A1.

This International Standard does not address the environmental issues associated with the use and disposal of thread compounds.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2137:1985, *Petroleum products — Lubricating grease and petrolatum — Determination of cone penetration.*

ISO 2176:1995, *Petroleum products — Lubricating grease — Determination of dropping point.*

ISO 10400:1993, *Petroleum and natural gas industries — Formulae and calculation for casing, tubing, drill pipe and line pipe properties.*

## 3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

### 3.1

#### **seal**

barrier resisting the passage of fluids

### 3.2

#### **thread compound**

substance that is applied to threaded oilfield pipe connections prior to make-up to assist in their lubrication during assembly and disassembly and in their sealing against high internal and external pressures in service

NOTE Some thread compounds may also contain substances that provide storage compound properties.

**3.3 storage compound**

substance that is applied to threaded oilfield pipe connections to protect against corrosion during either shipment or storage or both

NOTE Compounds that are for storage only should not be used for connection make-up.

**3.4 API modified thread compound**

compound designated as “modified thread compound” in API BUL 5A2

**3.5 reference standard**

thread compound that is formulated in accordance with the requirements of annex B, to include the limitations and tolerances in Tables B.1, B.2 and B.3

NOTE The reference standard is not intended for general field service.

**3.6 connection**

pipe assembly consisting of two threaded male connection members (pins) and a coupling, or one pin and an integral female connection member (box)

**3.7 thread compound/connection system**

system consisting of the various critical threaded pipe connection components, including the specific connection geometry, and the individual connection materials and coatings combined with the thread compound

**4 Thread compound characteristics**

**4.1 Product characteristics**

This International Standard outlines tests to characterize the performance of thread compounds under service conditions, rather than specifying the formulation. Thus, the purchaser and the manufacturer should agree on the product characteristics to be provided, such as:

Thickener type	Rheological properties
Fluid type	Compound/copper reaction
Appearance	Extreme pressure properties
Dropping point	Fluid sealing properties
Mass density	Frictional properties
Oil separation	Corrosion inhibition
Flash point	Brushing/Adherence
Water-absorption resistance	Service applications
Gas evolution	Storage and service life limitations

The thread compound manufacturer should publish timely product bulletins when any modification in formulation is implemented which would result in a change of any performance characteristic. All documentation shall provide data that are representative of a typical production batch.

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

**4.2 Physical and chemical characteristics**

**4.2.1 General**

The physical and chemical characteristics of performance-based thread compounds are specified in Table 1. These properties can vary widely, and the formulation of many of the available compounds is proprietary.

Therefore, the user should consider the performance properties and recommendations given by the compound manufacturers, in addition to the physical and chemical characteristics outlined in Table 1.

**Table 1 — Thread compound control and performance tests**

Property	Test method	Performance value
Dropping point, °C (M)	ISO 2176	138 min. (S)
Evaporation, % volume fraction loss (M) 24 h at 100 °C	See annex D	3,75 max. (S)
Gas evolution, cm <sup>3</sup> (M) 120 h at 66 °C	See annex G	20 max. (S)
Oil separation, % volume fraction (M) 24 h at 100 °C (nickel gauze cone)	See annex E	10,0 max. (S)
Penetration, mm × 10 <sup>-1</sup> (M) Worked, 60 strokes at 25 °C Production acceptability range (min. to max.) Worked, 60 strokes at -7 °C	See annex C	≤ 30 (S) Report typical (R)
Mass density, % variance (M) From production mean value	Manufacturer's controls	± 5,0 max. (S)
Water leaching, % mass fraction loss (M) 2 h at 66 °C	See annex H	5,0 max. (S)
Application and adherence (M) Cold application Adherence at 66 °C, % mass fraction loss	See annex F	Applicable at -7 °C (R) 25 max. (R)
Corrosiveness (M) Specified corrosion level	ASTM D 4048	1B or better (R)
Corrosion inhibition, % area corrosion (I) 500 h at 38 °C	See annex L	< 1,0 % (R)
Compound stability, 12 months storage (M) Penetration change, mm × 10 <sup>-1</sup> Oil separation, % volume fraction	Manufacturer's controls See annex C See annex E	± 30 max. (R) 10,0 max. (R)
Compound stability, field service (I) 24 h at 138 °C, % volume fraction loss	See annex M	25,0 max. (R)
M = Mandatory I = Informative S = Specification R = Recommendation		
NOTE The values in this table are not intended to be consistent with annex A, Table A.3, which presents the original values and requirements of API BUL 5A2 (now obsolete). They have been revised to take into account the high-temperature requirements of current field operating conditions and the mass density variations between different proprietary thread compound formulations.		

#### 4.2.2 Dropping point

The dropping point is a measure of the tendency of grease to soften and to flow under the application of heat. Results of the dropping point test may be used as an indication of the maximum temperature to which a grease can be exposed without liquefaction or oil separation, for indication of the grease as to type, and for establishment of manufacturing or quality control limits for this characteristic. Results should not be considered as having any direct bearing on service performance unless such correlation has been established.

In the case of a thread compound, the dropping point is considered to be an indicator of the thermal stability of the base grease and other lubricant additives. Poor thermal stability could adversely affect thread compound

performance in high-temperature field service. In order to meet present-day requirements for high temperature service, the minimum dropping point temperature shall be 138 °C as measured in accordance with ISO 2176.

NOTE Extreme temperature field-service conditions may require a higher performance limit.

#### 4.2.3 Evaporation

Evaporation is an indicator of a compound's physical and chemical stability at elevated temperatures, related to the base grease/oil or other additives. Due to the wide variation in mass density of thread compounds currently in service, percentage mass fraction does not provide a reliable basis for comparison, therefore, evaporation loss shall be measured as a percentage volume fraction. The evaporative loss, when evaluated in accordance with the test method in annex D for a 24 h duration at a temperature of 100 °C, shall not exceed 3,75 % volume fraction.

#### 4.2.4 Gas evolution

Gas evolution is an indicator of a compound's chemical stability at elevated temperatures. When evaluated in accordance with the test method in annex G, the volume of gas evolution shall not exceed 20 cm<sup>3</sup>.

#### 4.2.5 Oil separation

Oil separation is an indicator of a compound's physical and chemical stability at elevated temperatures, related to the base grease/oil. Due to the wide variation in mass density of thread compounds currently in service, percentage mass fraction does not provide a reliable basis for comparison, therefore, oil separation loss shall be measured as a percentage volume fraction. In order to meet current requirements for high-temperature service, the maximum oil separation loss when evaluated in accordance with test method in annex E shall be 10,0 % volume fraction.

#### 4.2.6 Penetration

Penetration is a measure of the consistency, i.e. "thickness" or "stiffness", of a lubricating grease and relates to the ease of application or "brushability" of a thread compound. The compound manufacturer shall measure and record the penetration of each production batch of thread compound and report the mean value for that specific compound. When evaluated in accordance with the test method in annex C, the penetration acceptability range (minimum to maximum) at 25 °C shall not be greater than 30 cone penetration points. For information purposes, cold temperature (-7 °C) penetration should be reported as a typical value. Mass density will affect the values obtained from this procedure. Therefore, it is not a useful measurement for relative comparisons of materials with widely varying mass densities.

NOTE Brookfield viscosity (ASTM D 2196) is not substantially affected by material mass density, and therefore should provide a closer correlation to brushability than the cone penetration. The range below was determined using several different supplier samples of API modified thread compound 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 r/min and 25 °C, was 200 000 mPa·s to 400 000 mPa·s. A typical value for API modified thread compounds could range from 200 000 mPa·s to 240 000 mPa·s.

#### 4.2.7 Mass density

The mass density of a thread compound is determined by the type and quantity of the constituents utilized in the formulation. The range of mass density between production batches for a particular thread compound is an indication of the consistency of manufacture. The compound manufacturer shall measure and record the mass density of each production batch of thread compound and report the mean value for that specific compound. The mass density of a particular thread compound shall not vary more than 5,0 % from the manufacturer's established mean value.

#### 4.2.8 Water leaching

Water leaching is an indicator of the physical and chemical stability of compounds when exposed to water at elevated temperatures. When evaluated in accordance with the test method in annex H, the compound mass loss shall not exceed 5,0 %.

#### 4.2.9 Application and adherence properties

Thread compounds should be applied in a manner consistent with the manufacturer's recommendations and in sufficient quantity to provide effective lubrication and sealing characteristics for 8-round and buttress connections or effective lubrication characteristics for proprietary connections. The thread compound should be brushable and capable of adherence over a temperature range of  $-7\text{ }^{\circ}\text{C}$  to  $66\text{ }^{\circ}\text{C}$  without either agglomerating or sliding off the connector.

Laboratory tests for determining the thread compound application and adherence properties shall be performed and recorded. The laboratory test methods described in annex F are intended to provide a means for comparing thread compound performance, but may not be representative of field service.

#### 4.2.10 Corrosion inhibition and protection properties

Thread compounds are often utilized to provide shipping and storage corrosion protection on threaded connections, as well as lubrication and sealing properties. Certain field exposure conditions, particularly on offshore platforms and in-service conditions such as sour gas environments, require corrosion protection and inhibition. Therefore, the thread compound should provide an effective barrier against (and not contribute to) corrosive attack of connection threads and seals. The corrosion-inhibition properties of thread compounds depend on application variables such as the following:

- compound additive types and treatment levels;
- type and condition of threading process fluids and residue remaining on thread surfaces;
- compound application method and equipment utilized;
- type of thread protector and application method ("knock-on" or "screw-on");
- specific user application procedures and environmental conditions;
- compatibility with thread storage protection compound;
- galvanic differences between compound components, environment and connector material.

A laboratory test shall be performed and recorded to determine whether potentially corrosive components are present in the thread compound. A copper corrosion test should be carried out in accordance with the procedures in ASTM D 4048 or equivalent. Although copper is not typically utilized (other than as a thread surface plating) in the production of oilfield country tubular goods (OCTG) connections, it more readily reacts in the presence of reactive materials such as sulfur, chlorine, etc., which can also damage steel. Thread compounds should 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. It is, therefore, 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 annex L are generally accepted and utilized by lubricant test laboratories and users. They are intended to provide a means for the relative comparison of thread compound properties.

#### 4.2.11 Compound stability properties

Thread compound stability, both in storage and in service is an element essential 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. Excessive hardening in storage can adversely affect brushability and proper application of the compound onto the pipe thread surfaces.

The compound manufacturer shall keep production batch samples and evaluate them periodically for storage stability. Thread compound storage stability over a minimum of 12 months should be adequate to resist softening or hardening of more than 30 cone penetration points at 25 °C, when evaluated in accordance with the test method in annex C. Stratification or oil separation should not be greater than 10,0 % volume fraction over a minimum period of 12 months. The test described in annex M should also be performed and is intended to provide a means for the relative comparison of thread compound high-temperature stability.

Thread compound stability test results shall be available in a product bulletin or certificate of conformance.

### 5 Thread compound performance properties

#### 5.1 General

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

#### 5.2 Frictional properties

A primary purpose of a thread compound is to act as a lubricating material and to provide consistent and repeatable frictional properties between the mating members of a threaded connection. For a given amount of connection engagement (a specific number of engaged threads), the torque required will vary in direct proportion to the apparent coefficient of friction of the thread compound/connection system. The frictional properties of the thread compound/connection system affect the following torque values:

- the torque required to make up the connection;
- the torque required to cause further make-up;
- the 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 connection geometry, machined surface finish, coating of the contact surfaces, relative surface speed (make-up revolutions per minute) of the connection members during make-up, compound film thickness and surface contact pressure. Each of these parameters should be taken into account when designing a test to determine frictional properties and when using a compound in the field.

A laboratory test for determining the thread compound frictional properties shall be performed and recorded. The laboratory test methods described in annex I are intended to provide a means for comparing thread compounds with the reference standard compound described in annex B.

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, and may result in excessive movement and engagement of the mill end prior to adequate engagement of the field end. The field torque required for proper assembly of ISO/API type connections should be determined in accordance with the procedures in ISO 10405 or as recommended by the connection manufacturer.

### 5.3 Extreme surface-contact pressure (gall resistance) properties

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

High surface-contact pressure in threaded connections can occur as a result of various factors during manufacturing and in field service. Manufacturing factors include product variations, such as geometric characteristics (thread length, pipe and coupling thicknesses) and process variations, such as machining (thread taper, lead, flank angles), surface finishing and coating. Field service factors include handling damage, contact-surface contamination, inadequate or inconsistent application of thread compound, misalignment during assembly and improper torque application.

An important consideration is the greater tendency of some materials towards connection galling than others. Galling tendency increases between two smooth metal surfaces along with increasing similarities of composition, similarities of relative hardness, and decreasing actual hardness. For OCTG, the composition and hardness of each component of the mating pair is virtually the same. Consequently, OCTG are relatively gall-prone. Therefore, a coating for one of the connection members, such as zinc plate or iron phosphate, and API modified thread compound has traditionally been utilized to provide 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, thread compound selection and application, handling and connection assembly to achieve connection galling resistance.

A laboratory test for determining the thread compound extreme surface-contact pressure properties (gall resistance) shall be performed and recorded. The laboratory test methods described in annex J are intended to provide a means for comparing thread compounds with the reference standard, described in annex B.

For specific service applications, the total thread compound connection system should be evaluated for galling resistance. This requires repeated assembly and disassembly tests on full-scale connections, preferably in the vertical mode, to simulate rig assemblies, with minimum and maximum amounts of thread compound. Such tests should be performed in accordance with the industry test methods referenced in annex J.

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. The combination of proper surface preparation, connection coating and thread compound selection and application should be established for each type of connection and material combination, based on their tendency to gall, during both assembly and disassembly following service.

### 5.4 Fluid sealing properties

A primary purpose of a thread compound, when used on thread sealing connections, is to provide fluid sealing for thread clearances, such as the helical root-to-crest clearances in ISO/API 8-round threads and the helical stab flank clearance in ISO/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 ensure the geometric integrity of the helical sealing passages. Contact pressure requirements are established for connection fluid pressure integrity and are given in ISO 10400.

A laboratory test for determining the thread-sealing properties of the thread compound shall be performed and recorded. The laboratory test methods described in annex K are intended to provide a means for comparing thread compounds with the reference standard, described in annex B.

For specific service applications, the total thread compound connection system should be evaluated for fluid-sealing integrity on full-scale connections. While it is important for a thread compound to provide fluid sealing for thread clearances on ISO/API connections, it is also important that the thread compounds do not inhibit the sealing

integrity of proprietary connections (metal-to-metal sealing). The solid particles that agglomerate may prohibit the designed mechanical seals (metal-to-metal) from efficiently contacting, resulting in a leakage path. Sealing tests should therefore be conducted on the thread compound/connection system, of which the thread compound is a part. Such tests shall be in accordance with the procedures defined in annex K.

## 6 Quality assurance and control

This International Standard is based on the concept that the function of a thread compound used with threaded connections for ISO/API casing, tubing and line pipe can 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 clauses 4 and 5.

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 shall have a comprehensive system of quality assurance to ensure that the represented properties are maintained throughout the range of variation of raw materials, manufacturing processes and application environment. Quality assurance activities shall be implemented within an appropriate management system. The manufacturer may be requested, by the purchaser, to furnish a certificate of conformity stating that the thread compound has been tested and evaluated in accordance with this International Standard and meets or exceeds the specified requirements.

## 7 Marking requirements

### 7.1 Marking

A thread compound manufactured and tested in conformance with the requirements of this International Standard shall be marked, on each container, with the manufacturer's identification, traceability identification and the following statement:

**"THIS THREAD COMPOUND CONFORMS WITH ISO 13678"**

### 7.2 Labelling

**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 labelled 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 shall be conspicuously labelled with the following cautionary statement:

**"STORAGE COMPOUND — NOT RECOMMENDED FOR MAKE-UP"**

**7.2.3** Each container shall be conspicuously labelled 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"**

## Annex A (informative)

### API modified thread compound

#### A.1 General

Clauses A.2 to A.6 of this annex are for information only, and are based on API BUL 5A2 (now obsolete), omitting all references to "silicone thread compound".

#### A.2 Compound

The compound is designated as the "modified thread compound". It is a mixture of metallic and graphite powders uniformly dispersed in a grease base. Proportions of solids and grease base are as listed in Table A.1.

**Table A.1 — Proportions of solids and grease base**

Component	Mass fraction %
Total solids	64,0 ± 2,5
Grease base	36,0 ± 2,5
Total	100,0

#### A.3 Composition of solids

The solids are 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.6.1 to A.6.4.

**Table A.2 — Proportions of solids**

Constituent	Mass fraction %	
	Total solids	Compound
Amorphous graphite	28,0	18,0 (±) 1,0
Lead powder	47,5	30,5 (±) 0,6
Zinc dust	19,3	12,2 (±) 0,6
Copper flake	5,2	3,3 (±) 0,3
Total	100,0	64,0

#### A.4 Grease base

Grease base for the modified thread compound is a grease which, when combined with the powdered metals and graphite, gives a compound which complies with the control and performance test requirements listed in Table A.3.

## A.5 Control and performance tests

The thread compound should 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 should comply with requirements listed in Table A.3 based on a test specimen which is representative of the entire contents of the container.

**Table A.3 — Modified thread compound control and performance tests**

Test	Requirement
Penetration, $\text{mm} \times 10^{-1}$ worked at 25 °C (NLGI <sup>a</sup> Grade No. 1) after cooling at -18 °C (see procedure annex C)	310 to 340 200 min.
Dropping point, °C (ASTM D 566)	88 °C min.
Evaporation, % mass fraction 24 h at 100 °C (see annex D)	2,0 max.
Oil separation, % mass fraction, nickel cone 24 h at 66 °C (see annex E)	5,0 max.
Gas evolution, $\text{cm}^3$ 120 h at 66 °C (see annex G)	20 max.
Water leaching, % mass fraction after 2 h at 66 °C (see annex H)	5,0 max.
Brushing ability (see annex E)	Applicable at -18 °C
<sup>a</sup> National Lubricating Grease Institute, 4635 Wyandotte Street, Kansas City, MO 64112-1596, USA.	
NOTE The information presented in this table applies only to the API modified thread compound formula.	

## A.6 Component material requirements

### A.6.1 Graphite

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

**Composition (ASTM C 561)**

Ash, % mass fraction	28 min., 37 max.
----------------------	------------------

**Particle size (ASTM E 11)**

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.6.2 Lead powder**

Lead powder should conform to the following requirements.

**Composition (ASTM D 1301)**

Free metal content, % mass fraction, min.	95,0
Lead oxide content, % mass fraction, max.	5,0

**Particle size (ASTM E 11)**

Pass No. 50 sieve, % mass fraction, min.	100,0
On No. 100 sieve, % mass fraction, max.	2,0
Pass No. 325 sieve, % mass fraction	30 min., 92 max.

**A.6.3 Zinc dust**

Zinc dust should be homogeneous. The zinc dust should be so constituted that the finished thread compound can meet the gas evolution test requirements of Table A.3. It should conform to the following requirements.

**Composition (ASTM D 521)**

Total zinc, calculated as Zn, % mass fraction, min.	98,0
Metallic zinc, % mass fraction, min.	95,0
Iron, lead and cadmium, % mass fraction, max.	1,0
Calcium calculated as CaO, % mass fraction, max.	0,5
Moisture and other volatile matter, % mass fraction, max.	0,1
Zinc oxide (ZnO)	Remainder

**Particle size (ASTM E 11)**

Pass No. 100 sieve, % mass fraction, min.	100,0
Pass No. 325 sieve, % mass fraction, min.	90,0

#### A.6.4 Copper flake

Copper flake should conform to the following requirements.

##### Composition (ASTM D 283)

Copper, % mass fraction, min.	97,0
Grinding and polishing compound, % mass fraction, max.	0,25

##### Particle size (ASTM E 11)

Pass No. 200 sieve, % mass fraction, min.	100,0
Pass No. 325 sieve, % mass fraction, min.	99,0
Thickness > 5 $\mu\text{m}$ , % mass fraction, max.	5,0

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## Annex B (normative)

### Reference standard formulation

The following reference standard formulation for thread compound is based on tightening the tolerances of API modified thread compound constituents closer to nominal values. In order to provide the replication required for a reference standard, the annex A tolerances and ranges have been tightened as shown in Tables B.1, B.2 and B.3.

**Table B.1 — Reference standard composition and tolerances**

Component	Mass fraction %
Grease base	36,00 ± 1,05
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 of Table B.2.

**Table B.2 — Grease base requirements**

Property	Requirement
Consistency	NLGI Grade "0"
Worked penetration, mm × 10 <sup>-1</sup> (ISO 2137, 60 strokes)	365 to 385
Thickener, % mass fraction, lithium 12-hydroxystearate	2,0 to 4,5
Petroleum oil viscosity, mm <sup>2</sup> /s	115 to 170 @ 40 °C 9,5 to 14,0 @ 100 °C

**NOTE** API BUL 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 the discrete test data. Full-scale test data from a combined API/Joint industry research project (API 1997 [8]) indicate 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 N80 tubing exceeded 150 % 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, CT, USA) at the rate of 2,0 % (by mass) 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) of 250 kg and a Timken OK Load (ASTM D 2782) of approximately 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 of Table B.3.

**Table B.3 — Reference standard constituent limitations, % mass fraction**

Test parameter	Amorphous graphite	Copper flake	Lead powder	Zinc dust
Ash	30 to 36	Not available	Not available	Not available
Moisture, max.	1,0	0,1	0,1	0,1
Metallic content, min.	Not available	97,0	95,0	95,0
Metal/Other oxides, max.	Not available	3,0	5,0	5,0
<u>Particle size distribution</u>				
Pass No. 50 sieve, min.	100,0	100,0	100,0	100,0
On No. 100 sieve, max.	0,3	0,0	1,0	0,0
On No. 200 sieve	10,0 to 18,0	0,0	5,0 to 25,0	2,0 max.
On No. 325 sieve	20,0 to 31,0	1,0	14,0 to 55,0	5,0 max.
Pass No. 325 sieve	50,0 to 70,0	99,0	40,0 to 80,0	93,0
NOTE This reference standard compound is not intended for general service.				

## Annex C (normative)

### Penetration test

#### C.1 General

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

#### C.2 Apparatus

**C.2.1 Penetrometer.**

**C.2.2 Cone** (full-scale).

**C.2.3 Grease worker.**

**C.2.4 Spatula.**

**C.2.5 Cooling chamber**, capable of maintaining  $-7\text{ °C} \pm 1,1\text{ °C}$ .

#### C.3 Procedure

Prepare two samples for worked penetration. After working (60 strokes), determine penetration of the first sample at 25 °C in accordance with ISO 2137.

After working the second sample at 25 °C, mound excess compound on top of penetration cup and place in cooling chamber along with penetrometer cone for 3 h at  $-7\text{ °C}$  (API BUL 5A2:  $-18\text{ °C}$ ). After the 3 h 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.

## Annex D (normative)

### Evaporation test

#### D.1 General

This annex describes a procedure for measuring losses of volatile materials from thread compound at a temperature of 100 °C under static conditions.

#### D.2 Apparatus

**D.2.1 No. 3 Coors porcelain evaporating dish**, shallow form or its equivalent.

**D.2.2 Gravity convection oven**, capable of maintaining a test temperature of 100 °C ± 1,1 °C.

**D.2.3 Precision balance**.

**D.2.4 Desiccator**.

#### D.3 Procedure

Weigh a 30 cm<sup>3</sup> (approximate) sample into the tared evaporating dish (D.2.1). Place the assembly in an oven (D.2.2) at 100 °C ± 1,1 °C for 24 h. Then place sample in a desiccator (D.2.4), cool, weigh (D.2.3), and report material loss as percent evaporation loss, calculated as percent volume fraction<sup>1)</sup> (API BUL 5A2: percent mass fraction).

---

1) The *percent volume fraction* is calculated by first determining (if not already known) the mass density (kg/m<sup>3</sup>) of the test compound sample. The mass of the amount required for the test is determined either by direct measurement or by subtracting the tare mass of the test equipment containing the sample from the total mass of the sample plus equipment. The volume of the sample in cubic centimetres is then calculated by dividing the sample mass in grams by its mass density and multiplying by 1000. The oils or volatiles lost by separation or evaporation can be assumed to have an approximate mass density of 900 if they are hydrocarbon based. The volume of the separated/evaporated materials is calculated by dividing the measured mass loss in grams by 900 (or the actual mass density if known to be different) and multiplying by 1000. The *percent volume fraction* loss is then calculated by dividing the volume of the separated/evaporated material by the volume of the starting sample and multiplying by 100.

## Annex E (normative)

### Oil separation test

#### E.1 General

This annex describes a procedure for measuring the tendency of thread compound to separate oil at 100 °C under static conditions.

#### E.2 Apparatus

**E.2.1 Nickel filter cone**<sup>2)</sup> or equivalent, approximately 38 mm diameter and with sides forming an angle of 60 °. The cone shall be perforated with approximately 200 holes of 0,8 mm diameter.

NOTE An acceptable alternative is a 60 mesh nickel gauze cone from Federal Test Method Standard 791B-321.2.

**E.2.2 50 ml beaker**, cut to a height of 41,0 mm.

**E.2.3 Gravity convection oven**, capable of maintaining 100 °C ± 1,1 °C.

**E.2.4 Precision balance**.

**E.2.5 Desiccator**.

#### E.3 Procedure

Weigh approximately 11 cm<sup>3</sup> of sample into the nickel filter cone. Take care to avoid formation of air pockets within the compound. The exposed surface of the compound should be smooth and convex to prevent trapping free oil. Suspend the cone in a tared beaker so that the cone tip is approximately 9,5 mm from bottom of beaker. Place the cone-beaker assembly in the oven for 24 h at 100 °C and then weigh. Remove the cone from the beaker; cool the beaker in a desiccator and weigh. Calculate the gain in mass of the beaker as percent oil separation, expressed as percent volume fraction<sup>3)</sup> (API BUL 5A2: percent mass fraction).

2) Fisher Scientific Company Cat. No. 9-760 or equivalent. The Fisher Scientific Company cone is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

3) The *percent volume fraction* is calculated by first determining (if not already known) the mass density (kg/m<sup>3</sup>) of the test compound sample. The mass of the amount required for the test is determined either by direct measurement or by subtracting the tare mass of the test equipment containing the sample from the total mass of the sample plus equipment. The volume of the sample in cubic centimetres is then calculated by dividing the sample mass in grams by its mass density and multiplying by 1000. The oils or volatiles lost by separation or evaporation can be assumed to have an approximate mass density of 900 if they are hydrocarbon based. The volume of the separated/evaporated materials is calculated by dividing the measured mass loss in grams by 900 (or the actual mass density if known to be different) and multiplying by 1000. The *percent volume fraction* loss is then calculated by dividing the volume of the separated/evaporated material by the volume of the starting sample and multiplying by 100.

## Annex F (normative)

### Application/adherence test

#### F.1 General

This annex describes a method for evaluating the “brushability” and adherence of a thread compound.

#### F.2 Apparatus

- F.2.1 **Sample can**, suitable to hold approximately 450 cm<sup>3</sup> of test compound.
- F.2.2 **Paint brush** with short (3 cm), stiff bristles, 3 cm to 5 cm wide.
- F.2.3 **Pin-end**, cut off from ISO/API 2-7/8 inch-OD threaded tubing.
- F.2.4 **Cooling chamber**, capable of maintaining  $-7\text{ °C} \pm 1,1\text{ °C}$ .
- F.2.5 **Gravity convection oven**, capable of maintaining a temperature of  $66\text{ °C} \pm 1,1\text{ °C}$ .

#### F.3 Procedure

##### F.3.1 Cold application and adherence

Place approximately 450 cm<sup>3</sup> of thread compound into a suitable sample can (F.2.1). Refrigerate compound sample, brush (F.2.2) and pin-end (F.2.3) to  $-7\text{ °C}$  (F.2.4) until temperature stabilizes (minimum 2 h).

After thermal stabilization, brush the compound onto the threaded area of the pin-end. Evaluate whether the brushability and adherence are such that the compound can be applied without agglomeration or significant voids in a smooth, uniform layer of approximately 2 mm thickness.

Record and report results and observations.

##### F.3.2 Elevated temperature adherence

Place approximately 450 cm<sup>3</sup> of thread compound into a suitable sample can (F.2.1). Weigh and record, to the nearest 0,1 g, the combined total mass of the compound sample, container and application brush (F.2.2). Weigh and record, to the nearest 0,1 g, the mass of the ISO/API tubing pin-end (F.2.3).

Brush the compound onto the threaded area of the pin-end in a smooth, uniform layer of approximately 2 mm thickness. Reweigh and record, taking care not to disturb or remove the compound applied to the tubing specimen. Verify by difference that the amount of compound applied to the pin-end is consistent with the amount removed from the container.

Place the coated pin-end, suspended or supported horizontally over a collection tray, in the 66 °C oven (F.2.5) for 12 to 17 h. Weigh and record to the nearest 0,1 g the mass of the pin-end with remaining adhering compound.

Calculate the mass loss of compound from the tubing pin-end as percent mass fraction. The mass loss of compound should not exceed 25 %.

Record and report test results and observations.

## Annex G (normative)

### Gas evolution test

#### G.1 General

This annex describes a procedure for measuring the gas evolved from a thread compound at a specified temperature.

#### G.2 Apparatus

**G.2.1 Apparatus**, as shown in Figure G.1, Figure G.2 or equivalent.

#### G.3 Procedure

- a) Fill bomb 1 to within 15,0 mm of the top edge with test compound, being careful to avoid air pockets. The use of a vibrator is helpful. Smooth top surface flush.
- b) Seal the bomb, close needle valve 2 and insert the bomb assembly in an oil bath preset at the test temperature.
- c) Attach the bomb assembly by means of tubing 3 to the gas bottle 4 and manometer 5.
- d) Open the needle valve 2.
- e) At the end of 15 min, 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.
- f) Open valves 10 and measure displaced water by means of a graduate. Record. Close valves 10.
- g) Repeat step f) at periodic intervals over a 5-day test period.
- h) Calculate evolution of gas as follows: Determine displacement, in cubic centimetres, caused by normal expansion of air and compound at the test temperature. Subtract from total displacement. Remainder is displacement caused by gas evolution.

#### G.4 Sample test data

a)	Oil bath temperature	66 °C
b)	Room temperature	25 °C
c)	Temperature difference	41 °C
d)	Coefficient of expansion of air (change in volume per unit volume per °C)	0,003 67
e)	Diameter of container ( $D = 2r$ )	50,0 mm
f)	Air depth ( $h$ )	15,0 mm

## G.5 Calculations

- a) Volume  $V$  of air in bomb above compound at 25 °C

$$V = \pi r^2 h = 3,1416 \times (2,50 \text{ cm})^2 \times 1,50 \text{ cm}$$
$$= 29,45 \text{ cm}^3 \text{ (initial air volume)}$$

- b) Expansion of the volume of air in bomb at 66 °C

$$V_2 = 29,45 \times 0,00367 \times 41$$
$$= 4,43 \text{ cm}^3 \text{ (expansion of the volume of air in bomb is the volume of air displaced)}$$

$$4,43/29,45 \times 100 = 15,04 \% \text{ (volume change)}$$

- c) Contraction of initial air volume displaced from bomb due to cooling to 25 °C

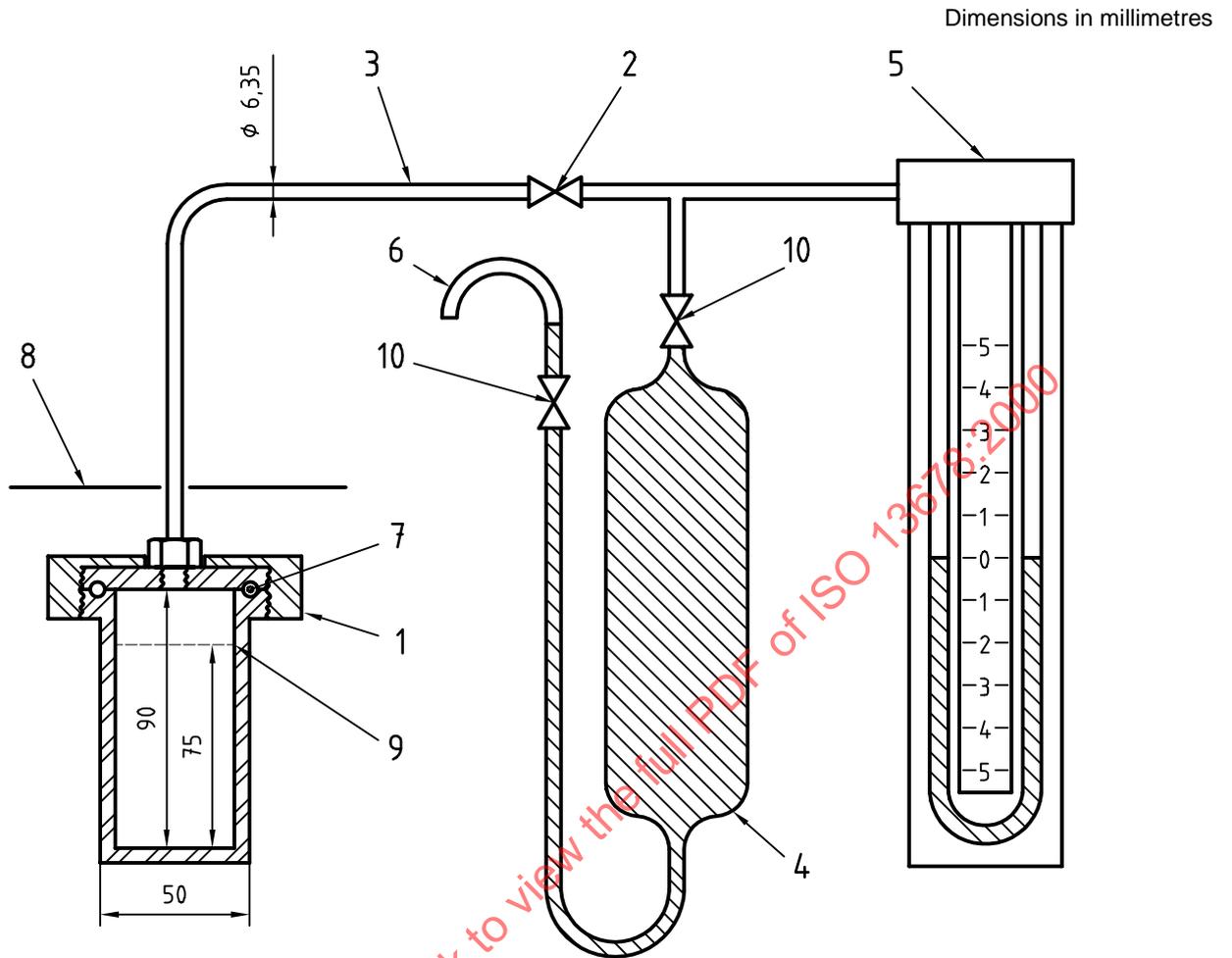
$$4,43 \times 15,04 \% = 0,67 \text{ cm}^3$$

- d) Corrected expansion of air displaced from the bomb

$$4,43 - 0,67 = 3,76 \text{ cm}^3$$

- e) Evolved gas = total displaced volume minus corrected displaced air expansion volume

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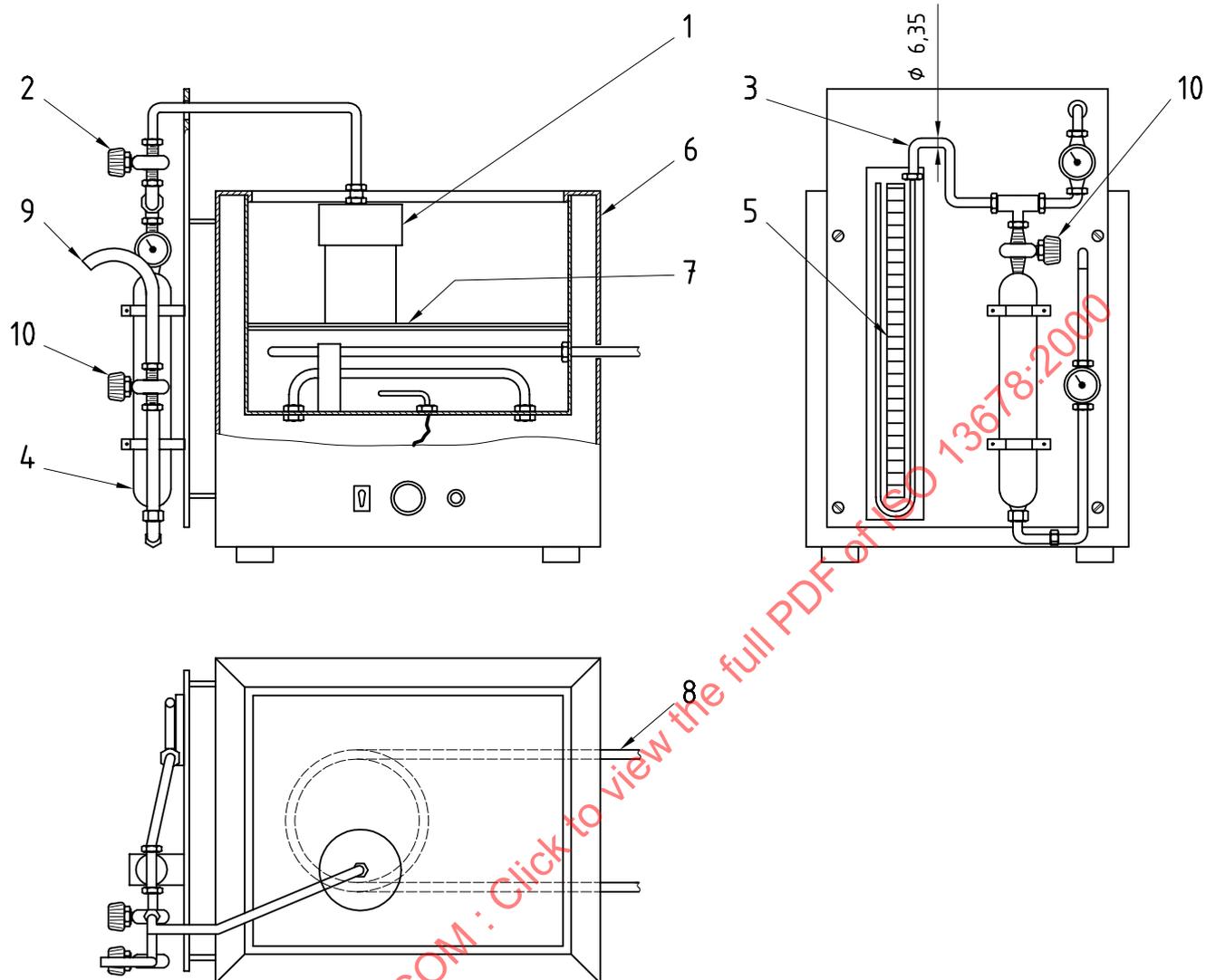


**Key**

- |   |  |    |                                  |
|---|--|----|----------------------------------|
| 1 | Bomb   | 6  | Line to measure displaced volume |
| 2 | Valve  | 7  | Silicone rubber "O" ring         |
| 3 | Stainless steel tubing                       | 8  | Oil bath level                   |
| 4 | 250 cm <sup>3</sup> gas bottle, water-filled | 9  | Compound sample level            |
| 5 | 25 cm mercury manometer                      | 10 | Valves                           |

**Figure G.1 — Example of gas evolution test apparatus**

Dimensions in millimetres



**Key**

- |   |  |    |  |
|---|--|----|--|
| 1 | Bomb   | 6  | Constant-temperature bath                |
| 2 | Valve  | 7  | Shelf                                    |
| 3 | stainless steel tubing                       | 8  | 66 °C water supply (see 6 in Figure H.2) |
| 4 | 250 cm <sup>3</sup> gas bottle, water-filled | 9  | Line to measure displaced volume         |
| 5 | 25 cm mercury manometer                      | 10 | Valves                                   |

**Figure G.2 — Example of gas evolution test apparatus**

## Annex H (normative)

### Water leaching test

#### H.1 General

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

#### H.2 Apparatus

As shown in Figure H.1, Figure H.2 or equivalent, including as follows.

**H.2.1 Coors No. 3 porcelain filter cone**, 50 mm diameter.

**H.2.2 100 ml glass beaker**, with six equidistant 6 mm holes located 1,6 mm from the bottom.

**H.2.3 Chrome triangle**, to support the cone.

**H.2.4 100 ml beaker**.

**H.2.5 1 000 ml beaker**, with side-arm extension at the bottom.

**H.2.6 Copper screen**, 1,6 mm mesh.

**H.2.7 Brass cylinder**, length 150 mm with a diameter of 75 mm, wall thickness approximately 6 mm, overflow 20 mm from the top, 3,0 mm orifice centred in the bottom.

**H.2.8 Centrifugal pump**, capable of circulating 1 litre of 66 °C water per minute.

**H.2.9 Connecting hose**, two pieces, of 6,35 mm ID.

**H.2.10 Ring stand**.

**H.2.11 Heater**.

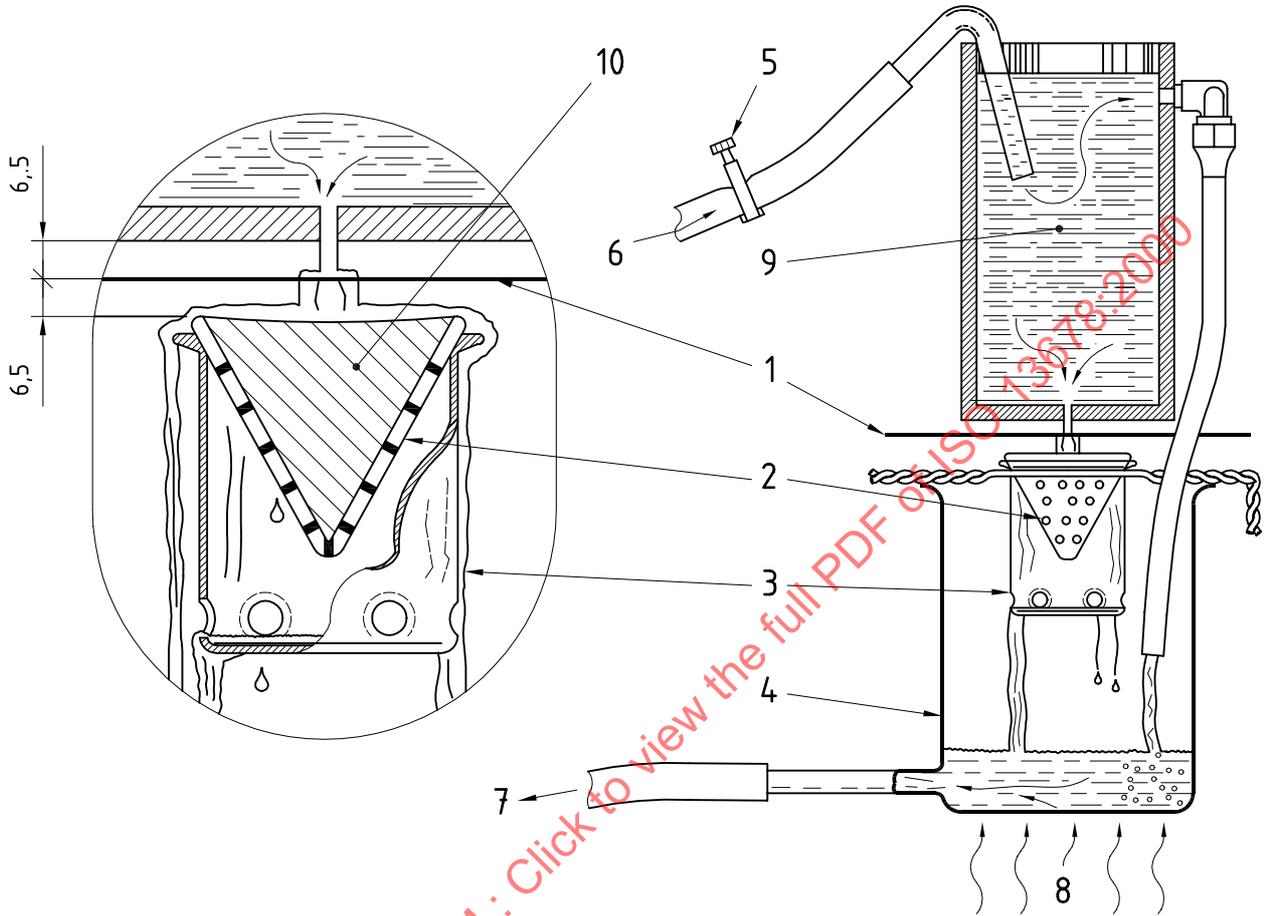
#### H.3 Procedure

Fill a tared porcelain filter cone with approximately 17 g of sample, smooth the top and make a slight concavity of about 1 mm with a spatula. Suspend the cone in the 100 ml beaker and support both on a triangle in the 1 000 ml beaker. Place the entire glass assembly on the hot plate which is elevated from the pump. Support the brass cylinder by a clamp from the ring stand. Place the mesh screen baffle equidistant between the brass cylinder and the upper rim of the cone, which are located 13,0 mm apart. Recycle from the 1 000 ml beaker to the brass cylinder at least 500 ml of distilled water, previously heated to 66 °C in the 1 000 ml beaker, and regulate by a screw clamp so 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.

Recirculate the water for 2 h during which time the temperature remains between 60 °C to 66 °C.

After the test has been completed, disassemble the apparatus, dry the cone and contents for a period of 24 h at 66 °C and calculate the mass loss as a percent mass fraction.

Dimensions in millimetres

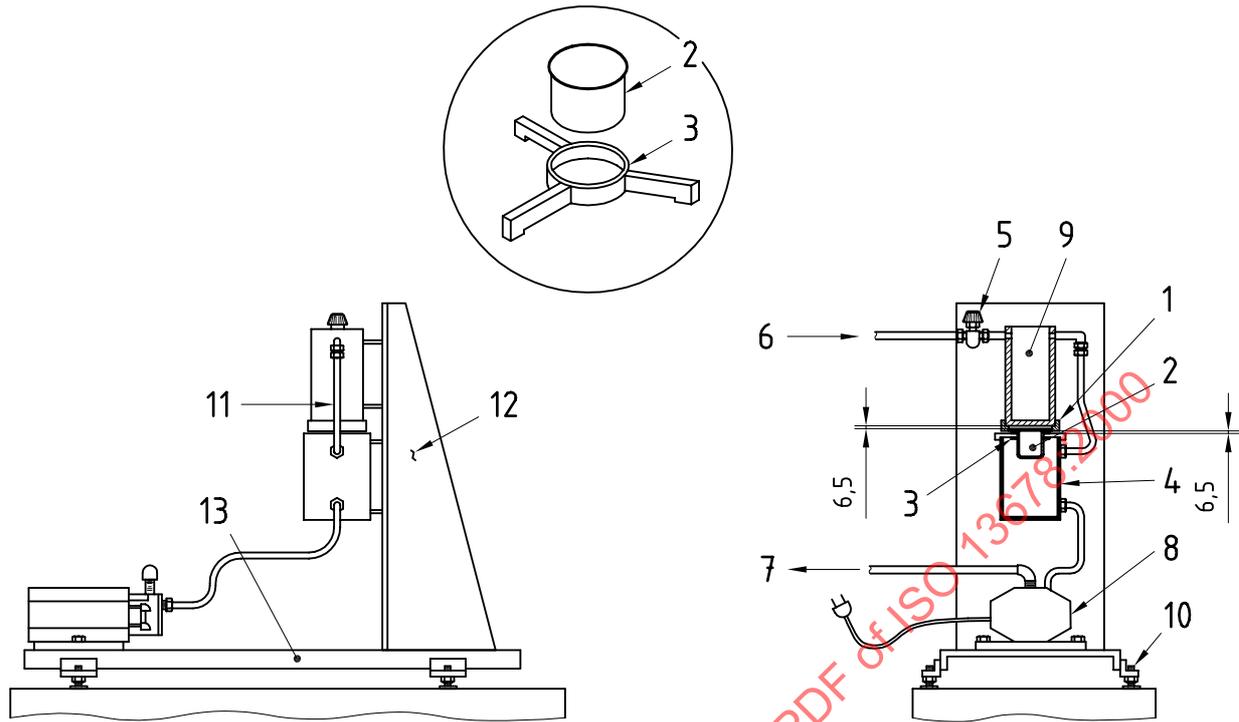


**Key**

- |   |                            |    |  |
|---|----------------------------|----|--|
| 1 | Copper screen, 1,6 mm mesh | 6  | Flow from pump                           |
| 2 | Porcelain cone             | 7  | Flow to pump                             |
| 3 | 100 ml beaker, modified    | 8  | Applied heat                             |
| 4 | 1 000 ml beaker, modified  | 9  | Water supply under uniform pressure head |
| 5 | Flow-regulating valve      | 10 | Compound sample                          |

**Figure H.1 — Example of water leaching test apparatus**

Dimensions in millimetres



**Key**

- |   |   |    |  |
|---|---|----|--|
| 1 | Threaded ring and copper screen                         | 7  | Flow to constant-temperature bath        |
| 2 | Compound sample cup (see inset)                         | 8  | Pump                                     |
| 3 | Holder (see inset)                                      | 9  | Water supply under uniform pressure head |
| 4 | Receiver cylinder                                       | 10 | Levellers                                |
| 5 | Flow-regulating valve                                   | 11 | Overflow tube                            |
| 6 | Flow from constant-temperature bath<br>(see Figure G.2) | 12 | Vertical support                         |
|   |   | 13 | Support platform                         |

NOTE Constant-temperature bath located in gas-evolution test apparatus (see Figure G.2).

**Figure H.2** — Example of water leaching test apparatus

## Annex I (informative)

### Frictional properties test

#### I.1 General

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

#### I.2 Industry test methods

The API has carried out investigations of pipe thread compounds (API PRAC Project 88, 89, 91-51:1992). Section 4.0 "Small Scale Friction Test Development" provides a history of development of test methods for thread compound frictional properties. Section 6.0 describes the procedure for a full-scale connection test which includes the evaluation of compound frictional properties for ISO/API type connections. ISO 13679 describes test procedures for any threaded connection.

#### I.3 Full-scale test

At least two test protocols utilizing multiple-connection test samples should be performed; one for tubing, preferably 88,9 mm (ISO/API 3½ in) outside diameter, and one for casing, preferably 244,5 mm (ISO/API 9⅝ in) outside diameter. Since the apparent coefficient of friction of the thread compound/connection system can vary with thread form, seal variation and material grade and finish, care should be exercised to ensure uniformity of connection test sample variables.

**NOTE** The full-scale API 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 (API 1997 [8]) demonstrated that if thread compounds have substantially different friction properties and/or compositions (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 to the test specimen connection members when using a standard reference torque. This difference in the initial stand-off results in a similar difference in the final engaged position. It is essential that any comparative testing of thread compounds, either for frictional properties or fluid sealing properties, be done to the same final engaged position within the allowable API tolerances. This is because 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 stand-off was established using the reference standard. The connection test specimen for all subsequent compounds tested was initially made-up to the reference stand-off and then made-up to the specified number of turns to the final engaged position.

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.

#### I.4 Small-scale test

An example of a small-scale test is the procedure described in API RP 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 200 MPa and 425 MPa. This compares to 35 MPa to 140 MPa for ISO/API casing, tubing and line pipe connections.

The coefficient of friction of typical tubing and casing compounds determined using API RP 7A1 ranges from 0,067 to 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 to 0,04. This difference may be caused by reduced surface contact pressure between the full-scale connection-members as they "float" together on a thick layer of thread

compound, as well as the significant difference in the connection variables described above. Thus, an apparent coefficient of friction is indicated. A test method should be selected to take into account such variables, including volume of compound applied.

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