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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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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.

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^3 (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^{-1} (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^{-1} 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^3.

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 °C to 66 °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