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

Industries du pétrole et du gaz naturel — Évaluation et essais des graisses pour filetage utilisées pour les tubes de cuvelage, les tubes de production, les tubes de conduites et les éléments de garnitures de forage

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Contents

Page

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This third edition cancels and replaces the second edition (ISO 13678:2009), of which it constitutes a minor revision.

It is the intent of ISO/TC 67 that the second and third editions of ISO 13678 both be applicable, at the option of the purchaser, for a period of six months from the first day of the calendar quarter immediately following the date of publication of this third edition, after which period the second edition will no longer be applicable.

Introduction

This International Standard is based on API RP 5A3[9], second edition, July 2003, with errata and inclusion of all clauses of API RP 7A1 1)[13], first edition, November 1992, incorporated into Annex I.

This International Standard specifies requirements and gives recommendations for the manufacture, testing and selection of thread compounds for use on casing, tubing, line pipe and drill stem elements 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, line pipe, premium connections and rotary shouldered connections include:

a) consistent frictional properties that allow both proper and uniform connection engagement;

b) adequate lubrication properties to resist galling or damage of connection contact surfaces during make-up and breakout;

c) 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 seals, etc.) depending upon service requirements;

d) physical and chemical stability both in service and in expected compound storage conditions;

e) properties that allow effective application to the connection contact surfaces in expected service conditions and environment.

In addition, compounds for rotary shouldered connections provide:

— lubrication of the connection members during make-up to achieve the proper axial bearing stress;

— an effective seal between connection shoulders to prevent wash-out by drilling fluids;

— more uniform distribution of circumferential bearing stress if shoulders are not parallel;

— resistance to additional make-up down hole.

When evaluating the suitability of a thread compound, the user can define the service conditions and then consider field trials and field service experience in addition to laboratory test results. Appropriate supplementary tests can be utilized 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, are not recommended.

1) Obsolete. Incorporated into this International Standard.

Testing in compliance with this International Standard does not in 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 whether the thread compound/connection system in question meets the anticipated requirements of that particular field service application.

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

1 Scope

This International Standard provides requirements, recommendations and methods for the testing of thread compounds intended for use on threaded casing, tubing, and line pipe connections; and for thread compounds intended for use on rotary shouldered connections. The tests outlined 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 and are not applicable to some materials used for lubricating and/or sealing thread connections. It is recognized that many areas can have environmental requirements for products of this type. This International Standard does not include requirements for environmental compliance. It is the responsibility of the end user to investigate these requirements and to select, use and dispose of the thread compounds and related waste materials accordingly.

2 Conformance

2.1 Dual citing of normative references

In the interest of world-wide application of this International Standard, Technical Committee ISO/TC 67 has decided, after detailed technical analysis, that certain of the normative documents listed in Clause 3 and prepared by ISO/TC 67 or another ISO Technical Committee are interchangeable in the context of the relevant requirement with the relevant document prepared by the American Petroleum Institute (API), the American Society for Testing and Materials (ASTM) and the American National Standards Institute (ANSI). These latter documents are cited in the running text following the ISO reference and preceded by "or", for example "ISO XXXX or API YYYY". Application of an alternative normative document cited in this manner will lead to technical results different from those obtained from the use of the preceding ISO reference. However, both results are acceptable and these documents are thus considered interchangeable in practice.

2.2 Units of measurement

In this International Standard, data are expressed in both the International System (SI) of units and the United States Customary (USC) system of units. For a specific order item, it is intended that only one system of units be used, without combining data expressed in the other system.

Products manufactured to specifications expressed in either of these unit systems shall be considered equivalent and totally interchangeable. Consequently, compliance with the requirements of this International Standard as expressed in one system provides compliance with requirements expressed in the other system.

For data expressed in the SI system, a comma is used as the decimal separator and a space as the thousands separator. For data expressed in the USC system, a dot (on the line) is used as the decimal separator and a space as the thousands separator. In the text, data in SI units are followed by data in USC units in parentheses.

3 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2137, *Petroleum products and lubricants — Determination of cone penetration of lubricating greases and petrolatum*

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

ASTM D217, *Standard Test Methods for Cone Penetration of Lubricating Grease*

ASTM D2265, *Standard Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range*

ASTM D4048, *Standard Test Method for Detection of Copper Corrosion from Lubricating Grease*

4 Terms and definitions

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

4.1
API connection
pipe assembly consisting of two external threaded connectors (pins) and a coupling with two internal threaded connectors (box) or one pin and an integral box manufactured in accordance with ISO/API specifications

4.2
API modified thread compound
compound designated as “modified thread compound” in API BUL 5A2[8]

NOTE API BUL 5A2 is obsolete and has been replaced by API RP 5A3[9].

4.3
box
connector with internal threads

4.4
casing, tubing and line pipe
CT and LP
production and delivery tubulars

4.5
drill stem elements
components of the drilling assembly from the swivel or top drive to the bit, composed of the kelly, drill string, subs, drill collars and other down-hole tools such as stabilizers and reamers

4.6
pin
connector with external threads

4.7
premium connection
connection with or without metal-to-metal seal(s) that can provide greater clearance and/or higher performance properties when compared to the API connections

4.8
proprietary connection
connection, without published specifications, made and marketed by companies with exclusive rights to manufacture and/or sell

4.9
reference standard formulation
⟨casing, tubing and line pipe⟩ thread compound formulated, in accordance with the requirements of Annex B, to include the limitations and tolerances specified in Tables B.1, B.2 and B.3

4.10
reference standard formulation
⟨rotary shouldered connection⟩ thread compound formulated in accordance with the requirements of I.4.2.3

NOTE The reference standard formulations are not intended for general field service.

4.11
rotary shouldered connection
RSC
connection used on drill stem elements, which has threads and sealing shoulders

4.12
seal
barrier resisting the passage of fluids, gases and liquids

4.13
storage compound
substance applied to threaded pipe connections for protection against corrosion, during shipment and/or storage only, that is not used for connection make-up

4.14
thread compound
substance applied to threaded pipe connections prior to make-up for lubrication during assembly and disassembly and for assistance in sealing internal and external pressures

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

4.15
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

4.16
tool joint
threaded connector used to join sections of drill pipe

5 Thread compound characteristics

5.1 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 the following:

— thickener type;

— fluid type;

- appearance;
- dropping point;
- mass density;
- oil separation;
- flash point;
- water-absorption resistance;
- gas evolution;
- rheological properties;
- compound/copper reaction;
- extreme-pressure properties;
- fluid sealing properties;
- frictional properties;
- corrosion inhibition;
- brushing/adherence;
- service applications;
- storage and service-life limitations.

The thread compound manufacturer shall revise product bulletins when any modification in formulation is implemented which would result in a change of any critical performance characteristics. All documentation shall provide data which 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 of three years after the date of manufacture.

5.2 Physical and chemical characteristics

5.2.1 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 physical and chemical characteristics tests

Property[a]		Test method	Performance value[b]	
Dropping point, °C (°F)	M	ISO 2176 or ASTM D2265	138 (280) min.	S
Evaporation, % volume fraction loss	M	See Annex D		
24 h at 100 °C (212 °F)			3,75 max.	S
Gas evolution, cm^3	M	See Annex G		
120 h at 66 °C (151 °F)			20 max.	S
Oil separation, % volume fraction	M	See Annex E		
24 h at 100 °C (212 °F) (nickel gauze cone)			10,0 max.	S
Penetration, mm × 10^{-1}	M	See Annex C		
Worked, 60 strokes at 25 °C (77 °F)				
Production acceptability range (min. to max.)			±15 max.	S
Worked, 60 strokes at −7 °C (19 °F)			Report typical	R
Mass density, % variance	M	Manufacturer's controls		
From production mean value			±5,0 max.	S
Water leaching, % mass fraction loss	M	See Annex H		
2 h at 66 °C (151 °F)			5,0 max.	S
Application and adherence	M	See Annex F		S
Cold application			Applicable at −7 °C (19 °F)	R
Adherence at 66 °C (151 °F), % mass fraction loss			25 max.	R
Copper corrosion	M	ASTM D4048		
Specified corrosion level			1B or better	R
Corrosion inhibition, % area corrosion	I	See Annex L		
500 h at 38 °C (100 °F)			<1,0	R
Compound stability, 12 months' storage	M	Manufacturer's controls		
Penetration change, mm × 10^{-1}		See Annex C	±30 max.	R
Oil separation, % volume fraction		See Annex E	10,0 max.	R
Compound stability, field service	I	See Annex M		
24 h at 138 °C (280 °F), % volume fraction loss			25,0 max.	R

NOTE The values in this table are not intended to be consistent with Table A.3, which presents the original values and requirements of API BUL 5A2[8] (obsolete, replaced by API RP 5A3[9]). 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.

a M mandatory;
I informative.

b S specification;
R recommendation.

5.2.2 Dropping point

The dropping point test measures the tendency of grease to soften and flow when hot. 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, as a means of determining the type of grease and establishing manufacturing or quality control limits for this characteristic. Results are not 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 (280 °F), as measured in accordance with ISO 2176 or ASTM D2265.

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

5.2.3 Evaporation

The evaporation test indicates a thread compound's physical and chemical stability at elevated temperatures, which is 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 24 h at a temperature of 100 °C (212 °F), shall not exceed a 3,75 % volume fraction.

5.2.4 Gas evolution

The gas evolution test indicates a thread 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.

5.2.5 Oil separation

The oil separation test indicates a compound's physical and chemical stability at elevated temperatures, which is 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 the test method in Annex E shall be a 10,0 % volume fraction.

5.2.6 Penetration

The penetration test measures 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 (77 °F) shall not be greater than 30 cone penetration points. An acceptability range for penetrations is used because thread compounds with penetrations between 265 and 385 can be used for different applications. For information purposes, cold temperature penetration, at –7 °C (19 °F), is reported as a typical value. Mass density affects the values obtained from this procedure. Therefore, it is not a useful measurement for relative comparisons of materials with widely varying mass densities.

NOTE 1 Brookfield viscosity (ASTM D2196[27]) is not substantially affected by material mass density and can therefore 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. It is appropriate that a specific spindle size, rotational frequency and test temperature 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.

NOTE 2 The SI unit of viscosity is the pascal second (Pa·s). The pascal second is rarely used in scientific and technical publications today. The most common unit of viscosity is the dyne second per square centimetre (dyne·s/cm^2), which is given the name poise (P) after the French physiologist Jean-Louis Poiseuille (1799-1869). Ten poise equal one pascal second (Pa·s) making the centipoise (cP) and millipascal second (mPa·s) identical.

— 1 pascal second = 10 poise = 1 000 millipascal second

— 1 centipoise = 1 millipascal second

5.2.7 Mass density

The mass-density test result of a thread compound depends on the type and quantity of the constituents utilized in the formulation. The range of mass densities 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 batch shall not vary by more than 5,0 % from the manufacturer's established mean value.

5.2.8 Water leaching

The water-leaching test indicates 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 %.

5.2.9 Application and adherence properties

Thread compounds should be applied in a manner consistent with the compound manufacturer and thread manufacturer's recommendations and in sufficient quantity to provide effective lubrication and/or sealing characteristics for threaded connections. The thread compound shall be brushable and capable of adherence over a temperature range of –7 °C (19 °F) to 66 °C (151 °F) 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 it is possible for them not to be representative of field service.

5.2.10 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 compounds with corrosion protection shall 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 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 D4048 or equivalent. Although copper is not typically utilized (other than as a thread surface plating) in production connections, it more readily reacts in the presence of reactive materials such as sulfur and chlorine, which can also damage steel. Thread compounds should provide a level 1B or better by this method. For RSCs, if thread compounds with metallic zinc are used, it is recommended that active sulfur be limited to less than 0,3 %.

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/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.

5.2.11 Compound stability properties

Thread compound stability, both in storage and in service, is a property essential to adequate sealing performance 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 changes in 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 is adequate to resist softening or hardening of more than 30 cone penetration points at 25 °C (77 °F), 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.

6 Thread compound performance properties

6.1 Small-scale test

The small-scale (bench top) test described in I.4 compares the friction properties of a test compound to a lead-based reference compound formulated for laboratory use. There is a possibility that small-scale tests might not correlate directly with full-scale connection tests or be truly representative of field service. Annex I [formerly API RP 7A1[13] (obsolete)] covers a small-scale test procedure that was developed and validated utilizing the metal-based RSC compounds that were commonly used in field applications in the early 1990s. Subsequent industry test programmes utilizing non-metallic RSC compounds have shown limited correlation of small-scale test frictional properties with full-scale test results. Therefore, this test method has limited usefulness for determining friction factors for non-metallic compounds for use on any type of connection.

6.2 Frictional properties

A thread compound acts as a lubricant during make-up and breakout and provides 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 varies 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 also 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, such as described in Annex I, for determining the thread compound frictional properties should be performed and recorded. The laboratory test methods described in Annex I are intended to provide a means of comparing thread compounds with the specified reference standard formulations.

In the case of casing, tubing and line pipe, 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 can 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 connections should be determined in accordance with the procedures described in ISO 10405[2] or API RP 5C1[10] or as recommended by the connection manufacturer.

6.3 Extreme surface contact pressure (gall resistance) properties for casing, tubing and line pipe

A thread compound provides 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 and 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 with increasing similarities of composition, similarities of relative hardness and decreasing actual hardness. For Oil Country Tubular Goods (OCTG), the composition and hardness of each component of the mating pair is virtually the same. Consequently, OCTG are relatively prone to galling. Therefore, a coating such as zinc phosphate and manganese phosphate and API modified thread compound, for one of the connection members, 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, duplex stainless 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 such as that described in Annex J for determining the total thread compound/connection system extreme surface contact pressure properties (gall resistance) should be performed and the results 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.