

Thread Compounds for Casing, Tubing, Line Pipe, and Drill Stem Elements

API RECOMMENDED PRACTICE 5A3
FOURTH EDITION, FEBRUARY 2023



American
Petroleum
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Thread Compounds for Casing, Tubing, Line Pipe, and Drill Stem Elements

1 Scope

This 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 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 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 errata or addenda) applies.

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*

ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*

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*

3 Terms and Definitions

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

3.1

API connection

A threaded assembly of tubular components manufactured in accordance with API 5B.

3.2

API modified reference standard formulation

A casing, tubing, and line pipe (CT and LP) 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 formulations are not intended for general field service.

3.3

API modified thread compound

A compound designated as modified thread compound is a compound that meets the material constituents and performance tests as stipulated in [Annex A](#).

3.4**bolt reference compound**

A reference thread compound formulated in accordance with [Annex I](#).

3.5**box**

A connector with internal threads.

3.6**casing and tubing, and line pipe****CT, LP**

Tubulars installed downhole in a well or on the surface for the purpose of constructing the well or service as a path for production or injection operations.

3.7**drill stem elements**

The components of the drilling assembly attached below the swivel or top drive.

NOTE Some examples include: kelly, drill pipe, subs, drill collars, stabilizers, reamers, and the drill bit; drilling assemblies often contain various tools with proprietary designs.

3.8**friction factor****FF**

A multiplier applied to connection make-up torque (MUT) to correct for the frictional property differences of the thread compound used and a reference compound as represented by: $MUT = \text{listed MUT} \times \text{friction factor}$.

3.9**pin**

A connector with external threads.

3.10**proprietary connection**

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

3.11**rotary shouldered connection****RSC**

A connection used on drill stem elements, which has threads and sealing shoulders.

3.12**seal**

A barrier resisting the passage of fluids.

3.13**storage compound**

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

3.14**thread compound**

The substance applied to threaded assemblies 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.

3.15

thread compound/connection system

A system consisting of the threaded connection components, including the connection geometry, individual connection materials and coatings combined with the thread compound.

3.16

tool joint

A forged or rolled steel component for drill-pipe designed to be welded to the drill-pipe body and having a rotary shouldered connection.

4 Thread Compound Characteristics

4.1 Product Characteristics

This 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
- Fluid type
- Appearance
- Dropping point
- Mass density
- Oil separation
- Flash point
- Water leaching
- Gas evolution
- Penetration
- Corrosion inhibition/copper corrosion
- Extreme-pressure properties
- Fluid sealing properties
- Frictional properties
- Corrosion inhibition
- Application and adherence
- Service applications
- Compound stability
- Evaporation

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 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.

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](#) that shall be satisfied to claim conformance to this standard. Other characteristics suggested for testing are included in [Table 2](#). These properties can vary widely and the formulation of many of the available compounds are proprietary. Therefore, the user should consider the performance properties and recommendations given by the thread compound manufacturers, in addition to the physical and chemical characteristics outlined in [Table 1](#) and [Table 2](#).

Table 1—Thread Compound Qualification, Physical and Chemical Characteristic Tests (Required)

Property	Test Method	Performance Value
Provide thickener-type	NA	Report
Dropping point, °C (°F)	ASTM D2265 or ISO 2176	138 (280) min.
Corrosion inhibition/Copper corrosion	ASTM D4048	—
Specified corrosion level	—	1B or better
Mass density, % variance	Manufacturer's controls	—
From production mean value	—	± 5.0 max.
Penetration, mm × 10 ⁻¹	See Annex C	—
Worked, 60 strokes at 25 °C (77 °F)	—	—
Production acceptability range (min. to max.)	—	± 15 max.
Worked, 60 strokes at -7 °C (19 °F)	—	Report typical
Evaporation, % volume fraction loss	See Annex D	—
24 h at 100 °C (212 °F)	—	3.75 max.
Oil separation, % volume fraction	See Annex E	—
24 h at 100 °C (212 °F) (nickel gauze cone)	—	10.0 max.
Compound stability, 12 months storage	Manufacturer's controls	—
Penetration change, mm × 10 ⁻¹	See Annex C	± 30 max.
Oil separation, % volume fraction	See Annex E	10.0 max.
Application and adherence	See Annex F	—
Cold application	—	at -7 °C (19 °F)
Adherence at 66 °C (151 °F), % mass fraction loss	—	25 max.
Gas evolution, cm ³	See Annex G	—
120 h at 66 °C (151 °F)	—	20 max.
Water leaching, % mass fraction loss	See Annex H	—
2 h at 66 °C (151 °F)	—	5.0 max.
Frictional property test	See Annex I	Value compared with reference compound
Galling resistance properties	See Annex J	Galling level 1–5
NOTE The values in this table are not intended to be consistent with Table A.3 , which presents the original values and requirements as informed in Annex A ; 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.		

Table 2—Compound Qualification, Physical and Chemical Characteristic Tests (Suggested)

Property	Test Method	Performance Value
Fluid sealing properties (thread sealing service)	See Annex K	Pass/Fail
Corrosion inhibition properties	See Annex L	Report passing hours
Compound stability, field service 24 h at 138 °C (280 °F), % volume fraction loss	See Annex M —	— 25.0 max.
NOTE The values in this table are not intended to be consistent with Table A.3 , which presents the original values and requirements as informed in Annex A ; 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 (ASTM D2265, ISO 2176)

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, for indication of the type of grease and for establishment of manufacturing or quality control limits for this characteristic.

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. To meet requirements for high temperature service, the minimum dropping point temperature shall be 138 °C (280 °F), as measured in accordance with ASTM D2265 or ISO 2176. The measured dropping point shall be reported.

NOTE Extreme-temperature field-service conditions may require a higher minimum dropping point limit.

4.2.3 Corrosion Inhibition Properties (ASTM D4048 and [Annex L](#))

Thread compounds are often used 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;
- type of surface treatment applied after threading;
- compound application method and equipment used;
- 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 performed in accordance with the procedures in ASTM D4048 or equivalent. Although copper is not typically used (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 using thread compounds with metallic zinc, 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 reported.

The methods listed in [Annex L](#) are generally intended to provide a means for the relative comparison of thread compound corrosion-inhibition properties. Thread compounds vary as to the existence and treatment level of corrosion inhibition. It is, therefore, the responsibility of the purchaser/user to outline the necessary requirements with the compound manufacturer for products being used for storage or corrosive field applications.

4.2.4 Mass Density (Manufacturer's Controls)

The mass density test result of a thread compound depends on the type and quantity of the constituents used in the formulation. The range of mass density between production batches for a 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 more than 5.0 % from the manufacturer's established mean value. The measured mass density and the deviation % from the production mean shall be reported.

4.2.5 Penetration ([Annex C](#))

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 acceptability range (minimum to maximum) at 25 °C (77 °F) shall not exceed 30 cone penetration points (i.e. mean value $\pm$ 15). For information purposes, cold temperature penetration, at -7 °C (19 °F), is reported as a typical value.

4.2.6 Evaporation ([Annex D](#))

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 a 24-hour duration at a temperature of 100 °C (212 °F), shall not exceed 3.75 % volume fraction. The measured evaporative loss shall be reported as a volume fraction.

4.2.7 Oil Separation ([Annex E](#))

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. 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 not exceed 10.0 % volume fraction. The measured oil separation shall be reported as a volume fraction.

4.2.8 Application and Adherence Properties ([Annex F](#))

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 adherence and application properties shall be performed in accordance with [Annex F](#). Cold application and adherence and elevated temperature adherence observations and results shall be reported.

4.2.9 Gas Evolution ([Annex G](#))

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 cm³. The measured volume of gas evolution shall be reported.

4.2.10 Water Leaching ([Annex H](#))

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 %. The percentage of mass loss during the water leaching test shall be reported.

4.2.11 Compound Stability Properties ([Annex C](#), [Annex E](#), and [Annex M](#))

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 (see test described in [Annex E](#)) 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 should be reported.

5 Thread Compound Performance Properties

5.1 Frictional Property Test ([Annex I](#))

A thread compound acts as a lubricant during make-up and break-out 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 that are required to:

- make-up the connection;
- cause further make-up;
- 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 considered when designing a test to determine frictional properties and when using a compound in the field.

The frictional properties test for thread compounds shall be conducted according to the requirements specified in [Annex I](#). The methods described in [Annex I](#) are intended to provide a means of comparing thread compounds to the specified bolt reference compound. The small-scale (bench top) test described in [Annex I](#) compares the friction properties of a test compound to a non-lead-based reference compound formulated for laboratory use. Small-scale tests might not correlate directly with full-scale connection tests or be truly representative of field service so full-scale tests should still be performed. [Annex I](#) provides a small-scale test procedure that was developed and validated using the RSC compounds commonly used in field applications. This test method has limited usefulness for determining friction factors for use on API 8-round and buttress connections.

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 appropriate procedures described in API 5C1, e.g. field make-up or such sections, or as recommended by the connection manufacturer.

5.2 Galling Resistance Properties ([Annex J](#))

A thread compound provides resistance to metal galling of the mating connection surfaces subjected to extreme surface-contact pressure.

High surface-contact pressure in threaded connections can occur because 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 toward 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 OCTG (oil country tubular goods), 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 for one of the connection members, such as zinc phosphate and manganese phosphate and API modified thread compound, has traditionally been used 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 or full-scale test, such as described in [Annex J](#), for determining the total thread compound/connection system extreme surface-contact pressure properties (gall resistance) shall be performed and the results recorded. The method described in [Annex J](#) is intended to provide a means for comparing thread compounds with the reference standard, described in [Annex I](#).

Currently, the most reliable test method is typically full-scale connection tests (repeated make-and-break of connection assemblies) such as described in API 5C5. These provide information on test methods for thread compound extreme surface-contact pressure (galling) properties and a full-scale connection test procedure, which includes the evaluation of compound galling-resistance properties for API connections.

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 cannot resist galling, regardless of handling or assembly technique. Conversely, connections with adequate surface preparation can 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.

5.3 Fluid Sealing Properties for Casing, Tubing, and Line Pipe ([Annex K](#))

When used on thread seal connection designs, a thread compound provides fluid sealing for thread clearances, such as the helical root-to-crest clearances in API 8-round threads and the helical stab flank clearance in API

buttress threads. Sealing is typically accomplished in a thread compound with solid particles that agglomerate to plug the thread clearances to prevent the contained fluid from passing through the connection.

Connection sealing also requires that positive contact pressure be maintained along the thread interface to ensure the geometric integrity of the helical sealing passages. Contact pressure requirements are established for connection fluid pressure integrity and given in API 5C3.

A laboratory test shall be performed, and the results recorded for the qualification of thread compounds intended to provide thread-sealing properties. The laboratory test method described in [Annex K](#) is intended to provide a means for comparing thread compounds with the CT and LP reference standard formulation, 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 API connections, it is also important that the thread compounds do not inhibit the sealing integrity of connections with metal-to-metal seals. The solid particles that agglomerate can 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 should be in accordance with the procedures defined in [K.3](#).

6 Quality Assurance and Control

This standard is based on the concept that the function of a thread compound used with threaded connections for API casing, tubing, line pipe and drill stem elements 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 [Section 4](#) and [Section 5](#).

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

For these reasons, the manufacturer should 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. It is possible for the purchaser to request that the manufacturer furnish a declaration of conformity stating that the thread compound has been tested and evaluated in accordance with this 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 standard should be marked, on each container, with the manufacturer's identification, traceability identification, manufacture date, shelf-life ending date, and one of the following statements.

- "THIS THREAD COMPOUND CONFORMS WITH API 5A3 FOR USE WITH CASING, TUBING, AND LINE PIPE"
- "THIS THREAD COMPOUND CONFORMS WITH API 5A3 FOR USE WITH ROTARY SHOULDERED CONNECTIONS"
- "THIS THREAD COMPOUND CONFORMS WITH API 5A3 FOR USE WITH CASING, TUBING, LINE PIPE, AND ROTARY SHOULDERED CONNECTIONS"

7.2 Labeling

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

— “STORAGE COMPOUND—NOT RECOMMENDED FOR MAKE-UP”

7.2.2 Each container shall be conspicuously labeled with cautionary statements regarding storage, preparation or application required to achieve the characteristics disclosed in the product bulletin, including any special thread compound manufacturer’s conditions required for storage before use.

EXAMPLE “STIR WELL BEFORE USING”, “THREAD COMPOUND PLUS INLAND SHORT-TERM STORAGE”, etc.

Annex A (informative)

API Modified Thread Compound

A.1 General

This annex is for information only and is based on API Bulletin 5A2 omitting all references to silicone thread compound.

NOTE API Bulletin 5A2 has been superseded by API Recommended Practice 5A3.

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 ± 0.5	18.0 ± 1.0
Lead powder	47.5 ± 1.0	30.5 ± 0.6
Zinc dust	19.3 ± 0.2	12.2 ± 0.6
Copper flake	5.2 ± 0.3	3.3 ± 0.3
Total	100.0	64.0

A.4 Grease Base

Grease base for the modified thread compound is thickened petroleum oil 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	Reference
Dropping point, °C (°F)	88 °C (190 °F) min.	ASTM D566 ^a
Penetration, mm × 10 ⁻¹ worked at 25 °C (77 °F) (NLGI) ^b (Grade No. 1) after cooling at -18 °C (0 °F)	— 310–340 200 min.	See Annex C — —
Evaporation, % mass fraction 24 h at 100 °C (212 °F)	— 2.0 max.	See Annex D —
Oil separation, % mass fraction, nickel cone 24 h at 66 °C (151 °F)	— 5.0 max.	See Annex E —
Brushing ability	Applicable at -18 °C (0 °F)	See Annex F
Gas evolution, cm ³ 120 h at 66 °C (151 °F)	— 20 max.	See Annex G —
Water leaching, % mass fraction after 2 h at 66 °C (151 °F)	— 5.0 max.	See Annex H —
NOTE The information presented in this table applies only to the API modified thread compound formula.		
^a ASTM D2265 may be used in place of ASTM D566.		
^b National Lubricating Grease Institute, 4635 Wyandotte Street, Kansas City, MO 64112-1596, USA.		

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 in [Table A.4](#).

Table A.4—Modified Thread Compound Component Requirements (Graphite)

Materials	% mass fraction
Composition (ASTM C561)	—
Ash	28 min., 37 max.
Particle size (ASTM E11)	—
Pass No. 50 sieve	100.0 min.
Retained on No. 100 sieve	1.0 max.
Retained on No. 200 sieve	10.0 min.
Pass No. 325 sieve	30 min., 80 max.

A.6.2 Lead Powders

Lead powder should conform to the following requirements in [Table A.5](#).

Table A.5—Modified Thread Compound Component Requirements (Lead Powders)

Materials	% mass fraction
Composition (ASTM D1301)	—
Free metal content	95.0 min.
Lead oxide content	5.0 max.
Particle size (ASTM E11)	—
Pass No. 50 sieve	100.0 min.
Retained on No. 100 sieve	2.0 max.
Pass No. 325 sieve	30 min., 92 max.

A.6.3 Zinc Dust

Zinc dust should be homogeneous and 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 in [Table A.6](#).

Table A.6—Modified Thread Compound Component Requirements (Zinc Dust)

Materials	% mass fraction
Composition (ASTM D521)	—
Total zinc, calculated as Zn	98.0 min.
Metallic zinc	95.0 min.
Iron, lead, and cadmium	1.0 max.
Calcium calculated as CaO	0.5 max.
Moisture and other volatile matter	0.1 max.
Zinc oxide (ZnO)	Remainder
Particle size (ASTM E11)	—
Pass No. 100 sieve	100.0 min.
Pass No. 325 sieve	90.0 min.

A.6.4 Copper Flake

Copper flake should conform to the following requirements in [Table A.7](#).

Table A.7—Modified Thread Compound Component Requirements (Copper Flake)

Materials	% Mass Fraction
Composition (ASTM D283)	—
Copper	97.0 min.
Grinding and polishing compound	0.25 max.
Particle size (ASTM E11)	—
Pass No. 200 sieve	100.0 min.
Pass No. 325 sieve	99.0 min.
Thickness > 5 μm	5.0 max.
NOTE Copper flake conformity was based on ASTM D283, which has been withdrawn and may be replaced by ASTM E478.	

Annex B (informative)

Casing, Tubing, and Line Pipe API Modified Reference Standard Formulation

The following casing, tubing, and line pipe (CT and LP) reference standard formulation for thread compounds is based on tightening the tolerances of API modified thread compound constituents closer to nominal values. To provide the replication required for a reference standard, the CT and LP reference standard formulation tolerances and ranges are shown in [Tables B.1](#), [B.2](#), and [B.3](#).

The grease base shall conform to the requirements of [Table B.2](#).

The solid components shall conform to the requirements of [Table B.3](#).

COMMENTARY—API Bulletin 5A2 did not specify requirements for the extreme pressure performance properties of the base grease utilized in the formulation of API modified thread compound. Commercial formulations 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 can 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) indicate that it would possibly 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 Label 1: 3 ½ inch 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 antimony dialkyldithiocarbamate 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 D2596) of 250 kg and a Timken OK Load (ASTM D2509) of approximately 9.08 kg. If antimony dialkyldithiocarbamate is not available, the extreme pressure additive that is utilized is added at a rate that yields equivalent results in the ASTM test methods cited.

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

Table B.2—Grease Base Requirements

Property	Requirement
Consistency Worked penetration, mm × 10 ⁻¹ ASTM D217 (ISO 2137), 60 strokes	NLGI No. "0" — 365–385
Thickener, % mass fraction, lithium 12-hydroxystearate	— 2.0–4.5
Petroleum oil viscosity, mm ² /s	115–170 at 40 °C (104 °F) 9.5–14.0 at 100 °C (212 °F)
Antimony dialkyldithiocarbamate	1.9–2.1 %

Table B.3—Reference Standard Constituent Limitations, Percent Mass Fraction

Test Parameter	Amorphous Graphite	Copper Flake	Lead Powder	Zinc Dust
Ash	30–36	NA	NA	NA
Moisture, max.	1.0	0.1	0.1	0.1
Metallic content, min.	NA	97.0	95.0	95.0
Metal/Other oxides, max.	NA	3.0	5.0	5.0
Particle size distribution	—	—	—	—
Pass No. 50 sieve, min.	100.0	100.0	100.0	100.0
Retained on No. 100 sieve, max.	0.3	0.0	1.0	0.0
Retained on No. 200 sieve	10.0–18.0	0.0	5.0–25.0	2.0 max.
Retained on No. 325 sieve	20.0–31.0	1.0	14.0–55.0	5.0 max.
Pass No. 325 sieve	50.0–70.0	99.0	40.0–80.0	93.0
NOTE This reference standard compound is not intended for general service.				