Designation: D4746 – 14$^{\varepsilon 1}$

# Standard Test Method for Determination of Quinoline Insolubles (QI) in Tar and Pitch by Pressure Filtration[1]

This standard is issued under the fixed designation D4746; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Editorial changes were made throughout the test method in September 2014.

## 1. Scope*

1.1 This test method covers the determination of the quinoline-insoluble matter (QI) in tar and pitch by pressure filtration and gives results comparable to those obtained by Test Method D2318.

1.2 Since this test method is empirical, strict adherence to all details of the procedure is necessary.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards are given in Section 7, 6.2, and 6.3.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D70 Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

D329 Specification for Acetone

D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials

D2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch

D4296 Practice for Sampling Pitch

E1 Specification for ASTM Liquid-in-Glass Thermometers

## 3. Summary of Test Method

3.1 The sample is digested in hot quinoline and filtered through a heated pressure filter. The insoluble material is washed with hot, fresh quinoline and with cold acetone, dried, and weighed.

## 4. Significance and Use

4.1 This test method is useful in evaluating and characterizing tar and pitch and as one element in establishing the uniformity of shipments and sources of supply.

## 5. Apparatus

5.1 *Pressure Filtration Vessel*—The pressure filtration vessel[3] (see Fig. 1) is a stainless steel (304) jacketed block heated by steam or cooled with water, sealed by a lid, flat gasket, and clamp. The interior of the block is designed to accept a 3A2 Berlin porcelain filtration crucible (see 5.4). The crucible is sealed within the block by the use of three O-rings, a crucible sealing collar, and an adjustable plunger.

5.1.1 The seal is accomplished when the sealing lid is placed on top of the block and clamped. The adjustable plunger applies a force to the crucible sealing collar, which in turn pushes downward on the crucible and simultaneously compresses O-ring gaskets, forming a seal between surfaces of the crucible and the wall of the block. The filtration of material is accomplished by nitrogen (10 psig to 30 psig) introduced through the lid of the pressure filter. The filtrate exits from the drain tube at the bottom of the block. The filtrate is disposed into a Buchner flask, attached to drain tube by a No. 7 rubber stopper. A vacuum hose is attached to the Buchner flask and placed in a 100 mL beaker containing water to indicate the exit of nitrogen when the filtration is completed.

5.2 *Beaker,* 50 mL.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The sole source of supply of the pressure filtration vessel known to the committee at this time is Koppers Inc., 436 Seventh Ave., Pittsburgh, PA 15219-1800. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

***A Summary of Changes section appears at the end of this standard**

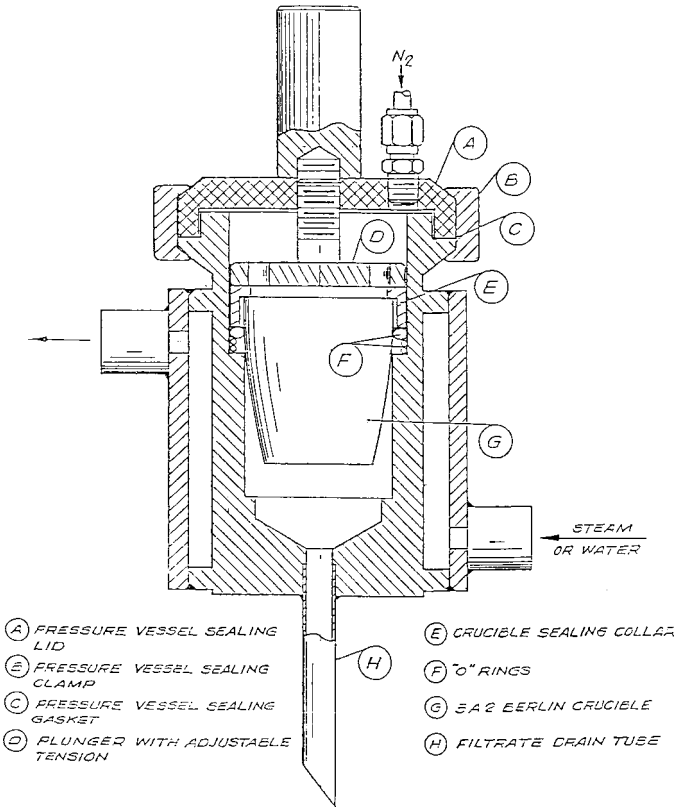



**FIG. 1 Pressure Filter**

5.3 *Buchner Flask,* 500.

5.4 *Filtering Crucible,* porcelain, with a medium-porosity bottom, top outer diameter 1¾ in. (45 mm), bottom outer diameter 1.2 in. (30 mm), height 2 in. (50 mm).

5.5 *Analytical Balance.*

5.6 *Desiccator.*

5.7 *Thermometer,* 0 °F to 300 °F (150 °C).

5.8 *Drying Oven,* maintained at 221 °F (105 °C).

5.9 *Mortar and Pestle.*

5.10 *Two Wash Bottles,* 500 mL.

5.11 *Steam Bath.*

## 6. Reagents

6.1 *Quinoline*—Refined, meeting the following requirements:

6.1.1 The quinoline shall be distilled from 5 % to 95 % within a range of 2 °F (1 °C) that shall include the temperature of 459.3 °F (237.4 °C) after corrections for barometric pressure and emergent steam have been applied. The distillation shall be carried out in accordance with Method D850 using a total-immersion thermometer with a range from 383 °F to 581°F (195 °C to 305°C), graduated in 1 °F (0.5 °C) and conforming to the requirements for thermometer 69C as described in Specification E1. Temperature measuring devices such as precision thermocouples, resistance temperature detectors (RTDs), and liquid-in-glass thermometers with equal or better accuracies in the appropriate temperature range may be used.

6.1.2 The quinoline shall have a specific gravity at 15.5/15.5 °C of 1.092 to 1.098, as determined by Test Method D70, or other method of equivalent accuracy.

6.1.3 The quinoline shall be clear and light in color and shall contain less than 0.5 volume percent of water as determined by Test Method D95. If not, redistill the quinoline in an all-glass apparatus, discarding the first 5 % and collecting the next 90 %. If the quinoline contains suspended matter but is clear, light in color, and contains less than 0.5 % water, filter the quinoline through a crucible containing 5 g of celite filter aid.

6.1.4 Store the quinoline in a tightly closed, dark bottle.

6.2 *1 + 1 Hydrochloric Acid Solution*—Add equal volume concentrated hydrochloric acid to distilled water. (**Warning—** Corrosive.)

6.3 *Acetone,* meeting Specification D329. (**Warning—** Flammable (Health Hazard).)

## 7. Hazards

7.1 Fumes of the solvents should be removed by means of proper hoods from all working areas. The working area should be kept free of sparks and flames. Quinoline fumes should not be inhaled, and prolonged contact with skin should be avoided.

7.2 Observe proper laboratory procedures for handling and diluting hydrochloric acid.

## 8. Bulk Sampling

8.1 Samples from shipments shall be taken in accordance with Practice D4296 and shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

## 9. Dehydration of Sample

9.1 *Hard Pitch*—If the solid bulk sample contains free water, air dry a representative portion in a forced draft oven at 122 °F (50 °C).

9.2 *Soft Pitch* (*Softening Point <140 °F* (60 °C))—If the presence of water is indicated by surface foam on heating, maintain a representative portion of the bulk sample at a temperature between 257 °F and 302 °F (125 °C and 150 °C) in an open container until the surface is free of foam. Take care not to overheat, and remove the heat source immediately when the foam subsides.

9.3 *Tar*—A wet tar sample can either be dehydrated or used as received as long as conditions stated in 9.3.1 and 9.3.2 are met.

9.3.1 Dehydrate a representative portion of the bulk sample.

9.3.2 As an alternative to dehydration, the water content of the tar is determined by Test Method D95 and, if the water content is less than 10 weight percent, the QI content is corrected to a dry-tar basis (see 13.2). This alternative method applies only to stable emulsions of water in tar, that is, no water separates when the tar sample is left undisturbed for 24 h at room temperature.

## 10. Preparation of Working Sample

10.1 Pitch that is sufficiently hard at room temperature shall be finely pulverized in a mortar. Crush this sample so that all

of it will pass the 250 µm (No. 60) sieve. Soft pitches and tars shall be warmed and well agitated before sampling.

10.2 The amount of sample to be taken for the test shall be such that the final quantity of quinoline insoluble (QI) shall be not less than 0.1 g. As a guide, the following sample sizes are recommended:

| | Sample Size, g |
|---|---|
| Crude coke oven tar | 5.0 |
| Horizontal retort tars | 2.5 |
| Fiber pitch | 5.0 |
| Other pitches | 2.5 |
| Distilled tars | 2.5 |
| Vertical retort tars | 10.0 to 25.0 |

10.2.1 When the sample contains 15 % QI or if the softening point is greater than 248 °F (120 °C), the amount used for testing should be reduced, but to not less than 0.5 g.

## 11. Crucible Preparation

11.1 If the crucible, after thorough cleaning (see 11.2), has been used for less than six determinations, clean it as follows. Remove the mat, wash the crucible with distilled water, dry, and ignite in a muffle furnace for 1 h at about 1472 °F (800 °C). Cool the crucible slowly by placing it in a drying oven for 1 h after removal from the furnace to prevent cracking and place it in a desiccator while still warm.

11.2 After the crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1 + 1 hydrochloric acid solution. Refer to 6.2. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as in 11.1.

## 12. Procedure

12.1 Weigh the recommended amount of sample (usually 2.5 g) into a 50 mL beaker and record weight ($W_1$).

12.2 Add 2.0 mL of refined quinoline per gram of sample to beaker.

12.3 Place thermometer 0 °F to 300 °F (150 °C) (6 in. length) into solution and heat on steam bath to 167 °F ± 10 °F (75 °C ± 5 °C) for not less than 15 min nor more than 20 min. Closely inspect the thermometer and bottom of the beaker for undissolved pitch and stir so that all of the pitch is brought into solution.

12.4 Place gaskets into the pressure vessel.

12.5 Start to heat block with steam (see 5.1 and Fig. 2).

12.6 Weigh a 3A2 Berlin crucible and record mass ($W_2$).

12.7 Place the preweighed crucible in the pressure vessel and then put crucible collar on (see 5.1 and Fig. 3).

12.8 When the quinoline-pitch solution has completed the heating cycle (see 12.3), pour the solution into the crucible, decanting it alongside the thermometer. Wash down the thermometer and beaker with 5 to 10 mL of quinoline and place on the steam bath.

12.9 Place the lid of the pressure filter with the adjustable plunger on the pressure vessel. The plunger shall be adjusted so that there is about ⅛ to ¼ in. gap between the lid and the flat gasket (see Fig. 1) of block. To close, force down by hand and attach clamp.

NOTE 1—Sealing of the crucible is a trial-and-error process. Excessive pressure could cause the crucible to be pushed past the O-rings during filtration or not allow complete sealing of the lid. Insufficient pressure of the plunger could cause leaks around the O-rings.

12.10 Add 10 psig of nitrogen using the pressure controller to pressurize the vessel to 10 psig with nitrogen. One may see slight bubbling from the indicator before the filtrate can be seen coming from the drain. Once the filtrate is flowing, pressure can be increased to 30 psig gradually. Nitrogen may bubble slowly (caused by volume displacement in the collecting flask) through the water seal and may bubble fast when filtration is completed. When filtrate stops coming out of bottom, shut off nitrogen and remove lid.

NOTE 2—In some cases, nitrogen stops bubbling although there is no draining of the filtrate. The filtration is actually completed, the quinoline insolubles have compacted on the bottom (filter cake) of the crucible and do not let nitrogen pass through. By increasing the pressure, nitrogen will eventually break through the filter cake.

12.11 Continue filtering the sample as described in 12.8 to 12.10 until the filtrate is clear. This usually will take about three washings of the beaker. When the third or last washing is poured into the crucible, rinse the beaker with the wash bottle, allowing the solution to run into the crucible to remove any carbon deposits. Filter the rinses.

12.12 When the last filtration is complete, shut the steam and nitrogen off. Remove the lid and start cooling the block with water (it takes approximately 5 min to cool the block and crucible).

12.13 Once cool, fill the crucible to about three quarters of its volume with acetone. Place lid on and follow 12.10 to filter. Perform this step three times.

12.14 When acetone rinses are complete, shut off the nitrogen, remove the lid, remove the crucible, and wipe sides with a paper towel containing acetone to remove any traces of quinoline. Place crucible in an oven at 221 °F (105 °C) for a minimum of 30 min, preferably 1 h. Remove, place in a desiccator, and allow to cool for 30 min.

12.15 Weigh the crucible containing quinoline insolubles and record the mass ($W_3$).

NOTE 3—For pitches yielding slow-filtering quinoline insolubles, a higher pressure may be used of up to 50 but not to exceed 75 psig. Under these conditions, a stainless steel disk with a ⅛ in. hole is added to the bottom of the block. Under high pressure, the crucible can be forced through O-rings. The stainless steel plate reduces the distance the crucible will travel to a minimum and thus avoids breakage and sample loss.

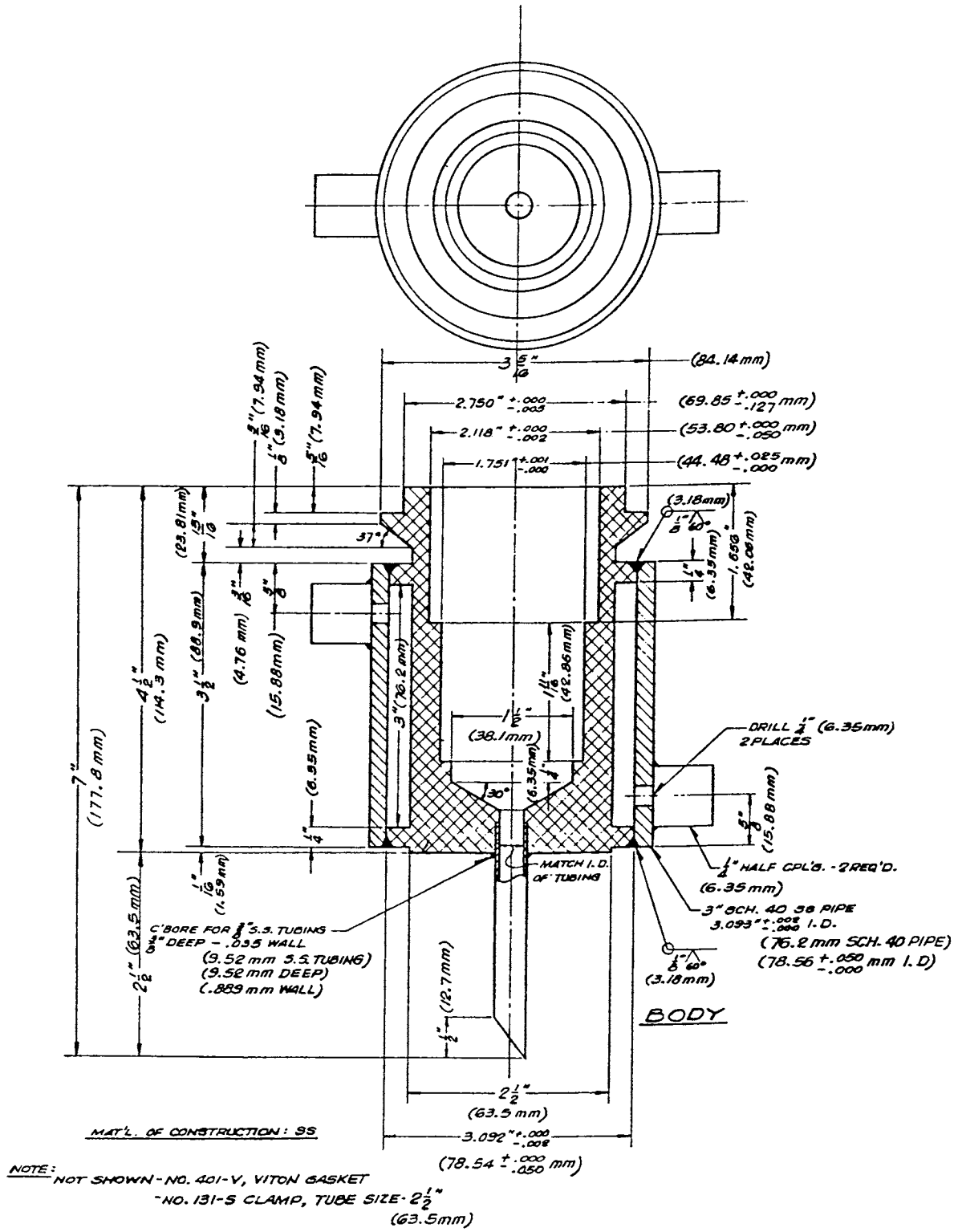
## 13. Calculation

13.1 Calculate the quinoline-insoluble (QI) content as follows:

$$QI,\ \% = [(W_3 - W_2)/W_1] \times 100 \quad (1)$$

*where:*

$W_3$ = mass of crucible with QI, g;
$W_2$ = mass of the crucible, g; and
$W_1$ = mass of the sample, g.




PRESSURE FILTER

**FIG. 2 Pressure Filter Dimensions**

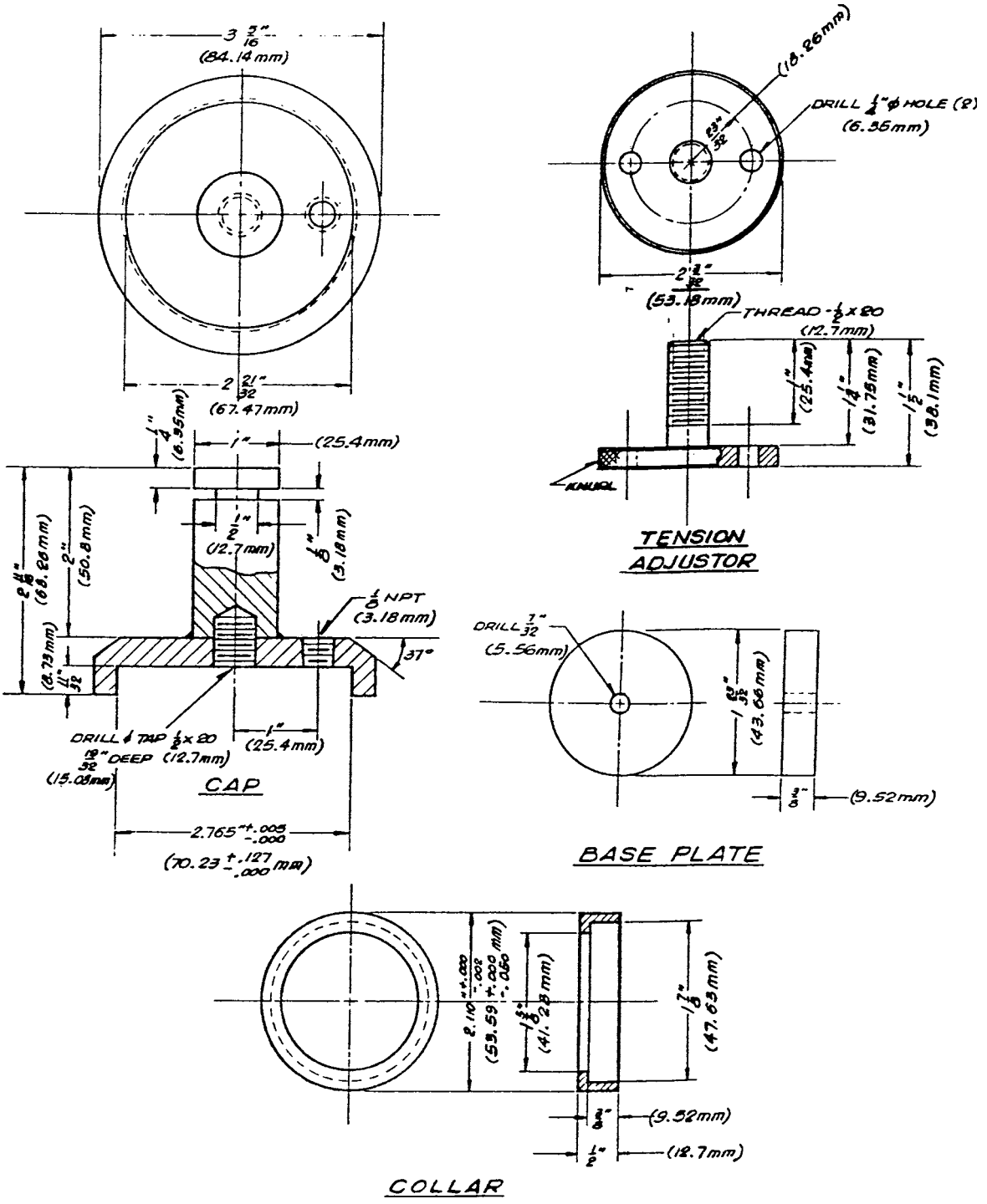



**FIG. 3 Pressure Filter Tension Adjuster, Collar, and Cap**

13.2 If the QI was determined on a wet tar sample (see 9.3.2), correct the QI value determined in 13.1 to a dry-tar basis as follows:

$$\text{QI, mass \% (dry basis)} = \frac{\text{QI, mass \% (wet basis) from 13.1}}{(100 - \text{water content of tar, mass \%})} \times 100 \quad (2)$$

## 14. Report

14.1 Report the quinoline insoluble (QI) content to the nearest 0.1 %.

## 15. Precision and Bias[4]

15.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless they differ by more than 0.2 %.

15.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless they differ by more than 0.3 %.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D08-1002.

## 16. Keywords

16.1 coal-tar insolubles; pitch; pressure filtration; QI; quinoline-insolubles; tar

## SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D4746 – 98 (2013)) that may impact the use of this standard. (Approved May 1, 2014.)

*(1)* Revised subsection 6.1.1.