TC 28

International Standard ISO 6249

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Petroleum products — Gas turbine fuels — Determination of thermal oxidation stability — JFTOT method

Produits pétroliers — Carburéacteurs — Détermination de la stabilité à l'oxydation thermique — Méthode JFTOT

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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. Every 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4589 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

Petroleum products — Gas turbine fuels — Determination of thermal oxidation stability — JFTOT method

1 Scope and field of application

1.1 This International Standard specifies a procedure for rating the tendencies of gas turbine fuels to deposit decomposition products within the fuel system.

1.2 The test results are indicative of fuel performance during gas turbine operation and can be used to assess the level of deposits that form when liquid fuel contacts a heated surface that is at a specified temperature.

NOTE — The maximum heater control temperature (see 7.2.5) should be selected to suit each application of this method and specified when this method is called up.

2 Principle

This method for measuring the high temperature stability of gas turbine fuels uses the Jet Fuel Thermal Oxidation Tester (JFTOT), which subjects the test fuel to conditions which can be related to those occurring in gas turbine engine fuel systems. The fuel is pumped at a fixed volumetric flow rate through a heater after which it enters a precision stainless steel filter where fuel degradation products are retained.

The apparatus requires 600 ml of test fuel for a 150 min test. The essential data derived are the level of deposits on an aluminium heater tube, and the rate of plugging of a 17 µm nominal pore size precision filter located just downstream of the heater tube.

3 Apparatus

3.1 Jet Fuel Thermal Oxidation Tester[1] (JFTOT)

Either of two models can be used, Recording Model or Non-Recording Model. Both are the same size : 914 mm high, 762 mm wide, and 305 mm deep, and designed to sit upon a standard height chemical laboratory bench. Annex A gives a detailed description of the apparatus, which shall be used without modification.

NOTE — No attempt should be made to operate the JFTOT without first becoming acquainted with all components and the function of each.

3.2 Heater tube deposit rating apparatus

The level of deposits on the heater tube are rated by either the Mark 8A Tube Deposite Rater[1] or a Tuberator[1] and the Colour Standard for tube deposit rating.[1]

3.3 Materials, supplies and spares

3.3.1 The following items are supplied with each JFTOT :

3.3.1.1 Magnifying assembly for tuberator.

3.3.1.2 Tuberator adapter for heater tube.

3.3.1.3 AutoCal heater assembly.

3.3.1.4 Piston puller.

3.3.1.5 Cap seal.

3.3.1.6 Aeration tube.

3.3.1.7 Clear plastic tubing for aeration tube.

3.3.1.8 Aeration tube holder.

3.3.1.9 Funnel holder.

3.3.1.10 Nut driver, 12,7 mm.

3.3.1.11 Socket head (Allen) screw driver, 4 mm.

3.3.1.12 Power cord.

3.3.1.13 Constant-voltage transformer, 60 or 50 Hz.

3.3.1.14 Step-down transformer, 230-115 V.

NOTE — These are supplied only with 230 V-50 Hz (JFTOT).

3.3.1.15 Protector, sight glass.

1) Suitable apparatus is available commercially. Details of suppliers may be obtained from the Secretariat of ISO/TC 28 or from the ISO Central Secretariat.

3.3.2 The following items are required to be replaced with each test and therefore must be stocked in accordance with the volume of testing involved :

3.3.2.1 Heater tube and filter kit.

3.3.2.2 Pre-filter element.

3.3.2.3 Data sheets (see table 1).

3.3.2.4 General-purpose, retentive, qualitative filter paper.

3.3.3 The following supplies are spare parts needing periodic replacement as required and should therefore be stocked in accordance with the volume of testing involved.

3.3.3.1 Ceramic insulators (4/sets).

3.3.3.2 Lip seal, reservoir piston.

3.3.3.3 O-ring, reservoir.

3.3.3.4 O-ring, sight-glass.

3.3.3.5 O-ring, retention screw.

3.3.3.6 O-ring, line connections.

3.3.3.7 O-ring, pre-filter.

3.3.3.8 Thermocouple assembly.

3.3.3.9 Tin, 99,99 % pure pellets in capsules containing 1,6 ± 0,5 g.

3.3.3.10 Metering pump.

3.3.3.11 Chart paper, ΔP recorder.

3.3.3.12 Aeration tube.

3.3.4 The following additional items are not supplied with the JFTOT but are required for normal operation.

3.3.4.1 ALCOR Mark 8A Tube Deposit Rater.

3.3.4.2 EPPI Tuberator.

3.3.4.3 Nitrogen, compressed in a cylinder.

3.3.4.4 Pressure regulator, 0 to 7 MPa[1].

3.3.4.5 Solvent, may be methyl pentane, *n*-heptane or 2,2,4 trimethyl pentane, technical grades, 95 mol percent minimum purity.

3.3.4.6 Disposable gloves.

3.3.4.7 Trisolvent, (equal parts by volume of toluene, acetone, and propan-2-ol, 99 % purity).

3.3.4.8 Wash bottle, polyethylene.

3.3.4.9 Cleaning pan, stainless steel (250 mm by 350 mm minimum).

3.3.4.10 Brush, polyamide 40 mm by 100 mm.

3.3.4.11 Brush, polyamide 15 mm by 75 mm.

3.3.4.12 Funnel, glass.

3.3.4.13 Thermometer, accurate to 1 °C with which temperatures between 15 and 32 °C can be read (e.g. ISO 1770 C or D).

3.3.4.14 Tweezers, flat bladed, non-serrated.

3.3.4.15 Rubber squeeze bulb.

3.3.4.16 Extractor for ceramic insulators.

3.3.4.17 Silicone grease.

3.3.4.18 Paper tissues.

3.3.4.19 Aluminium foil, about 450 mm wide.

3.3.4.20 Graduated cylinder, capacity 100 ml.

4 Sampling

Take a representative sample of the product to be tested according to ISO 3170 or ISO 3171 (or other relevant method).

5 Standard operating conditions

Standard conditions of test are as follows :

5.1 Test portion volume, 600 ml.

5.2 Preparation of test portion.

Filter through a single layer of filter paper (3.3.2.4) followed by a 6 min aeration at 1,5 l/min air flow rate.

5.3 Fuel system pressure, 3,45 MPa[1] gauge.

5.4 Maximum heater tube temperature, preset as specified for the fuel under test.

5.5 Fuel flow rate, 3,0 ml/min.

1) 1 MPa = 10 bar.

5.6 Test duration, 2 h 30 min.

6 Preparation for test

6.1 Disassembly

All the steps for disassembly of the test section, if required, are given in clause 8.

6.2 Calibration of the heater tube temperature controller

6.2.1 The AutoCal Calibrator is used for calibrating the heater tube temperature controller. This calibrator is a nickel-plated heater tube which has a small well containing pure tin. This method utilizes the freezing point of tin, 232 °C, as the calibration temperature.

6.2.2 Install the AutoCal Calibrator by placing the hollow end of the calibrator flush with the top surface of the upper fixed bus-bar and tighten both socket head (Allen) screws.

6.2.3 Secure the plugged end of the calibrator by raising the lower floating bus-bar to the upper limit of its travel and tighten both socket head (Allen) screws.

6.2.4 Lower the thermocouple through the upper section of the calibrator and coat the thermocouple tip with silicone grease to prevent sticking to the tin.

6.2.5 Lower the thermocouple into the well to press lightly against the upper surface of the solid tin.

6.2.6 Set digital readout on HEATER TUBE TEMPERATURE CONTROL at 232 °C.

6.2.7 Set the POWER CONTROL at zero.

6.2.8 Switch the CONTROL MODE to MANUAL position.

6.2.9 Switch the POWER to ON.

6.2.10 Check that the water pressure indicator is in the green arc. Adjust the WATER FLOW CONTROLLER so that the indicator is in the green arc (this corresponds to 38 ± 8 l/h).

6.2.11 Switch the AUTOCAL to ON.

6.2.12 Switch HEATER to ON.

6.2.13 Set POWER CONTROL to 75 to 80 setting.

NOTE — A higher setting may be required for some instruments. First ensure good contact at low voltage connections and then, if required, adjust removable stop at rear of power controller (see A.4.2).

6.2.14 Wait at least 2 min before proceeding with the calibration check to allow for temperature stabilization and warm-up of the temperature controller.

6.2.15 Depress the AUTOCAL pushbutton for 3 s and observe the deviation meter needle of the HEATER TUBE TEMPERATURE CONTROL. Repeat at short intervals until the deviation meter needle swings to full right. This indicates that the tin is molten.

6.2.16 While the temperature deviation meter needle is deflected full right, carefully lower the thermocouple to the bottom of the well, noting the total distance of travel on the thermocouple positioning indicator and then raise it 2,5 mm. Ensure that the thermocouple is centred in the well.

If the travel of the thermocouple is not at least 5 mm, refill the well with a new charge of tin pellets (3.3.3.9) in accordance with the instructions in annex D and repeat as above, starting with 6.2.2.

6.2.17 If the temperature deviation meter needle is now less than full right, depress the AUTOCAL button until the needle is again full right scale deflection and then release. The deviation meter needle will slowly move from right to left, stop, and then abruptly reverse to the right and pause for about 3 to 5 s. During the time the needle is stationary, adjust the digital readout to centre the needle. The pause period constitutes the freezing point, the change of state from liquid to solid tin. The drop in temperature below freezing point and abrupt reversal is due to the super-cooling characteristic of tin (see figure 1). If the deviation meter needle does not remain completely stationary for a minimum of 3 s, the tin is contaminated and needs replacement. See annex D for replacement instructions.

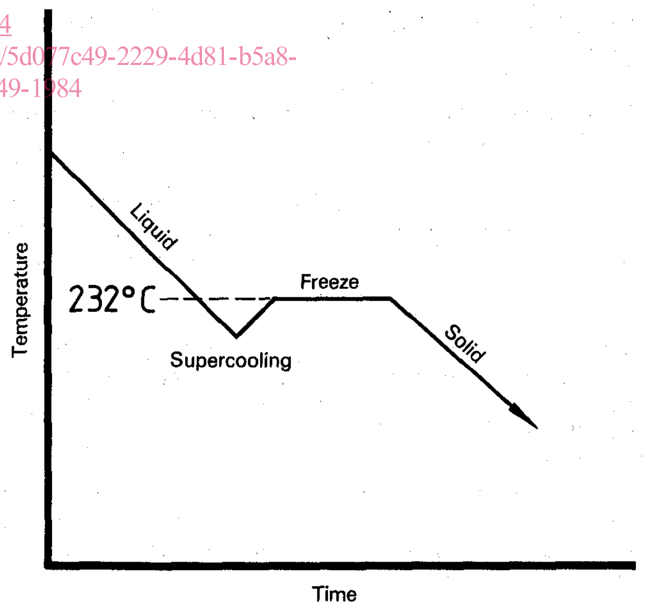


Figure 1 — Freezing characteristics of tin

6.2.18 Repeat this procedure as necessary until the deviation meter needle is centred during the pause without the need for adjustment of the controller digital setting. Observe and record on a data sheet (3.3) the "indicated freezing point of tin", that is, the digital setting to centre the needle during the pause.

NOTE — Although the calibration procedure involves a determination of "freezing point", the value obtained is reported as "melting point" because this term is traditionally used on the data sheets (see table 1).

6.2.19 When the "indicated freezing point of tin" has been satisfactorily determined, refreeze the tin by depressing the AUTOCAL pushbutton to obtain a full right scale deflection of the temperature deviation meter needle. Then release the pushbutton and immediately raise the thermocouple tip so as to be accessible and remove all residual traces of silicone and tin by wiping the tip with paper tissue. Inspect the tip closely for cleanliness.

6.2.20 Switch the HEATER to OFF.

6.2.21 Switch the AUTOCAL to OFF.

6.2.22 Raise the thermocouple to the extreme upper limit and remove the AutoCal calibrator.

6.3 Inspection of components

6.3.1 Inspect the reservoir cover O-ring and all O-rings used on the line fittings including the nitrogen and fuel return lines for cuts, abrasion and excessive swelling and replace as necessary.

6.3.2 Inspect the ceramic insulators and replace if they are cracked or chipped.

6.3.3 Inspect all stainless steel components for damage and replace as necessary.

6.4 Inspection and testing of reservoir piston lip seal

6.4.1 Inspect the lip seal for cuts, abrasion, or excessive swelling and replace as necessary.

6.4.2 When required to install a lip seal on the piston, ensure that the inner lip is properly placed under the retaining shoulder of the piston. See figure 2 for the correct lip seal assembly position.

6.4.3 With the thumbs, gently push the sealing edge of the lip seal outward from the centre of the reservoir piston while slowly rotating the piston in the hands. This will minimize leaks past the seal.

6.4.4 Attach the piston puller (3.3.1.4) to the piston. Wet the lip seal and reservoir wall with fuel and insert the piston so that the top of the lip seal is about 25 mm from the top of the reservoir.

6.4.5 Close the outlet of the reservoir with the cap seal (3.3.1.5).

6.4.6 Pour fuel on top of the piston to a depth of 6 mm.

6.4.7 Press downward on the piston puller until air leaks past the lip seal as evidenced by the appearance of air bubbles.

6.4.8 Release the pressure and observe whether air leakage past the lip seal stops. Change to a new lip seal if the air leakage does not stop immediately.

6.4.9 Remove the cap seal from the outlet and observe whether the piston moves downward with the piston puller in place. Change to a new lip seal if the piston does not move downward and repeat the lip seal test procedure, commencing with 6.4.2.

6.5 Cleaning

6.5.1 Wear protective gloves (3.3.4.6) because of the possibility of skin irritation from solvents.

6.5.2 Position the cleaning pan (3.3.4.9) to catch solvent during cleaning operations.

6.5.3 Place a new piece of aluminium foil (3.3.4.19) about 450 mm square on the bench on which to place all test section components after cleaning.

6.5.4 Using the wash bottle (3.3.4.8) filled with solvent (3.3.4.5), flush the reservoir cover O-ring.

6.5.5 Flush all inside surfaces of the reservoir with solvent while scrubbing the surfaces with a brush (3.3.4.10).

6.5.6 Flush all inside surfaces of the reservoir with solvent without brushing, and place the reservoir upside down on the aluminium foil.

6.5.7 Using the squeeze bulb (3.3.4.15), blow out the reservoir fuel outlet exit fitting on the bottom of the reservoir to remove the remaining solvent.

6.5.8 Handling the reservoir piston with a piston puller, repeat 6.5.4 and 6.5.5 for the reservoir piston, being careful not to brush or damage the lip seal.

6.5.9 Flush the reservoir cover assembly with solvent.

6.5.10 Flush the heater tube fuel supply line and heater tube fuel outlet line with solvent and thoroughly blow dry using the squeeze bulb.

6.5.11 Flush the pre-filter components with solvent.

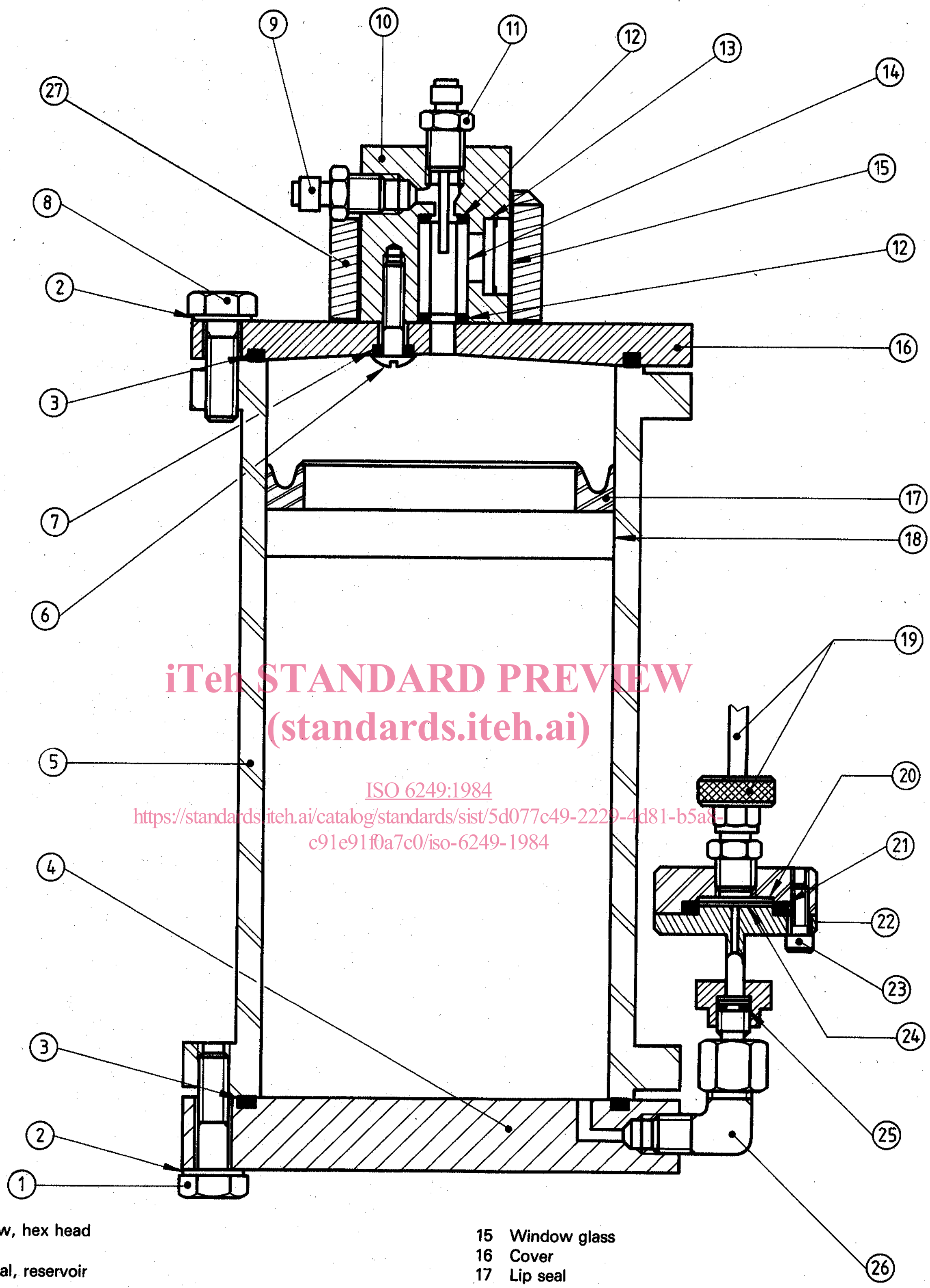
6.5.12 Using a polyamide brush (3.3.4.11) saturated with the trisolvent (3.3.4.7) brush the inside surfaces of the heater tube housing.

NOTE — This is the only component that requires cleaning with trisolvent.

6.5.13 Flush the heater tube housing and filter bypass line with solvent and blow dry with the squeeze bulb. Visually inspect the inner surfaces of the heater tube housing for cleanliness and repeat 6.5.12 as required to remove all deposits.

6.5.14 Flush the four ceramic insulators and heater tube housing nuts with solvent.

6.5.15 Flush the glass filter funnel (3.3.4.12) and glass aeration tube with solvent.



1 Cap screw, hex head
2 Washer
3 O-ring seal, reservoir
4 Base
5 Cylinder
6 Screw, truss head
7 O-ring seal, ret screw
8 Cap screw, hex head
9 Nitrogen inlet fitting
10 Housing, drop flow indicator
11 Drop tube fitting
12 O-ring seal, sight glass
13 Retaining ring
14 Sight glass
15 Window glass
16 Cover
17 Lip seal
18 Piston
19 Heater tube fuel supply line assembly
20 Filter back-up screen
21 O-ring seal, pre-filter
22 Housing, pre-filter
23 Cap screw, Allen head
24 Membrane filter
25 O-ring seal
26 Reservoir fuel outlet fitting
27 Protector, sight glass

Figure 2 — Reservoir and pre-filter assembly

6.6 Assembly of heater tube section

6.6.1 A new heater tube, test filter, and three new high temperature O-rings are required for each test.

6.6.2 During assembly, be sure to have CLEAN hands or wear clean, dry gloves.

6.6.3 Hold the heater tube at one end and insert it carefully, open end upwards, into the heater housing, the lower end can be recognized by the presence of the drive screw which closes it. (See figure 3 for correct assembly of the heater tube and heater tube housing.)

NOTE — If the centre test section is touched, reject the tube as it will affect the deposit-forming characteristics on the tube.

6.6.4 Onto one end of the heater tube, sequentially install a flared ceramic insulator (flared end out), high temperature O-ring, shoulder ceramic insulator (large end first), and hexagon nut. Lightly finger tighten the nut with the heater tube approximately centred in the housing (see figure 3).

6.6.5 Repeat the above procedure for the other end of the heater tube.

6.6.6 Observe the heater tube through the fuel discharge hole of the heater tube housing. Align heater tube shoulder with the centre of the fuel discharge hole (see figure 4). Tighten the hexagon nuts firmly with fingers only. DO NOT USE A WRENCH.

6.6.7 Using clean tweezers (3.3.4.14), install the test filter, RED-COLOURED SIDE OUT, in the outlet chamber of the heater tube housing.

6.6.8 Place a new O-ring on top of the test filter, pushing the O-ring in until it bottoms against the filter.

6.6.9 Connect the fuel outlet line assembly to the heater tube housing outlet. Finger tighten lightly.

6.6.10 Using paper tissue (3.3.4.18), wet with solvent (3.3.4.5), clean the contact areas of the bus-bars.

6.6.11 Raise the thermocouple to the uppermost position.

6.6.12 Place the heater tube section between the bus-bars. Check the alignment, connect and tighten the heater tube fuel outlet and bypass lines to the bulkhead fittings on the rear wall of the test section, being sure the O-rings on these fittings are in place.

6.6.13 If the bus-bar caps have been removed, check for proper mating. Numbers are stamped on inside faces and these must be the same and must face each other.

NOTE — Normally the caps are not removed entirely from their bus-bars during disassembly.

6.6.14 Tighten both socket head (Allen) screws of the upper fixed bus-bar cap, after making sure that the upper end of the heater tube is flush with the top surface of the upper fixed bus-bar.

6.6.15 Raise the lower floating bus-bar until it touches the lower ceramic insulator of the heater tube test section and tighten both socket head (Allen) screws of the lower floating bus-bar cap.

6.6.16 Check the thermocouple for correct position by raising the position indicator to the thermocouple reference line, (see figure 5). The thermocouple tip must be flush with the top of the heater tube and the top of the upper fixed bus-bar. If the thermocouple tip is not flush, see annex D.

6.6.17 Insert the thermocouple into the upper end of the heater tube and lower to the 38,7 mm position.

6.7 Assembly and installation of pre-filter

6.7.1 For each test, use a new 0,45 µm nominal pore size membrane filter element of 25 mm diameter.

6.7.2 Using clean tweezers (3.3.4.14), install the filter element back-up screen in the pre-filter housing recess.

6.7.3 Using clean tweezers, place the 0,45 µm nominal pore size filter element on the back-up screen.

6.7.4 Install the O-ring on the other half of the pre-filter housing.

6.7.5 Assemble the two housing sections, insert the three screws and tighten.

6.7.6 Connect the pre-filter assembly to the reservoir outlet and finger tighten firmly.

6.7.7 Connect the heater tube fuel supply line to the pre-filter and finger tighten firmly.

6.7.8 Install the cap seal at the end of the heater tube fuel supply line.

6.8 Preparation of test portion

6.8.1 Place a filter paper (3.3.2.4) into a glass funnel and set the funnel (3.3.4.12) into the funnel holder (3.3.1.9) that attaches to the reservoir.

6.8.2 Measure the test portion of 600 ml of the laboratory sample using a clean graduated cylinder (3.3.4.20).

NOTE — Because it is necessary to have the test portion at a temperature between 15 and 32 °C after filtration, it is desirable to have it within this temperature range at this time.

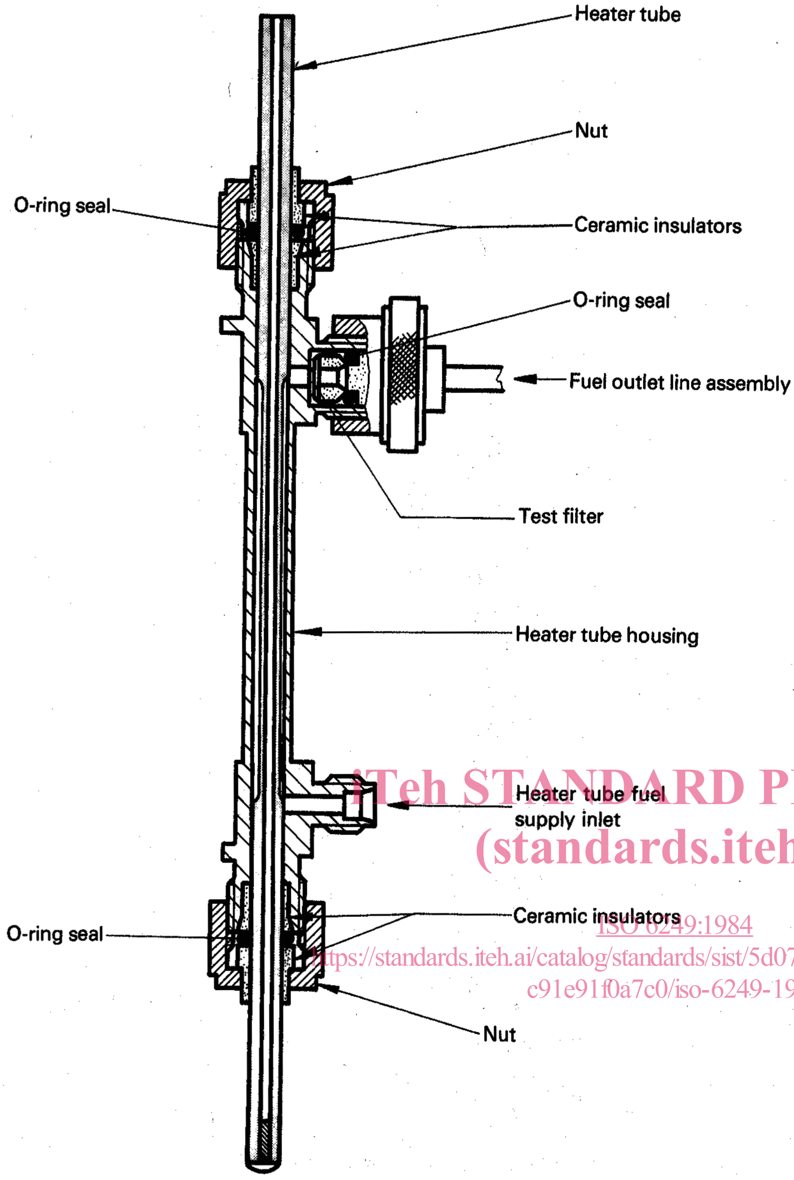


Figure 3 — Assembly drawing of heater tube section

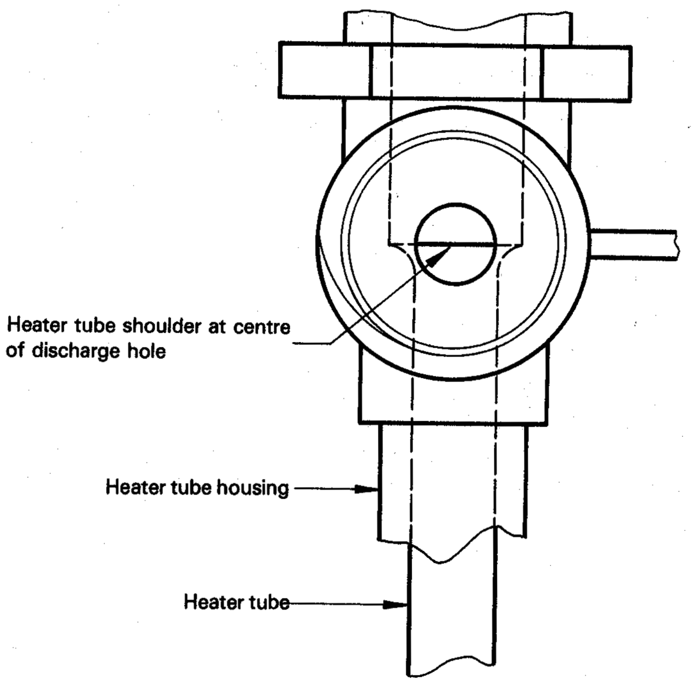
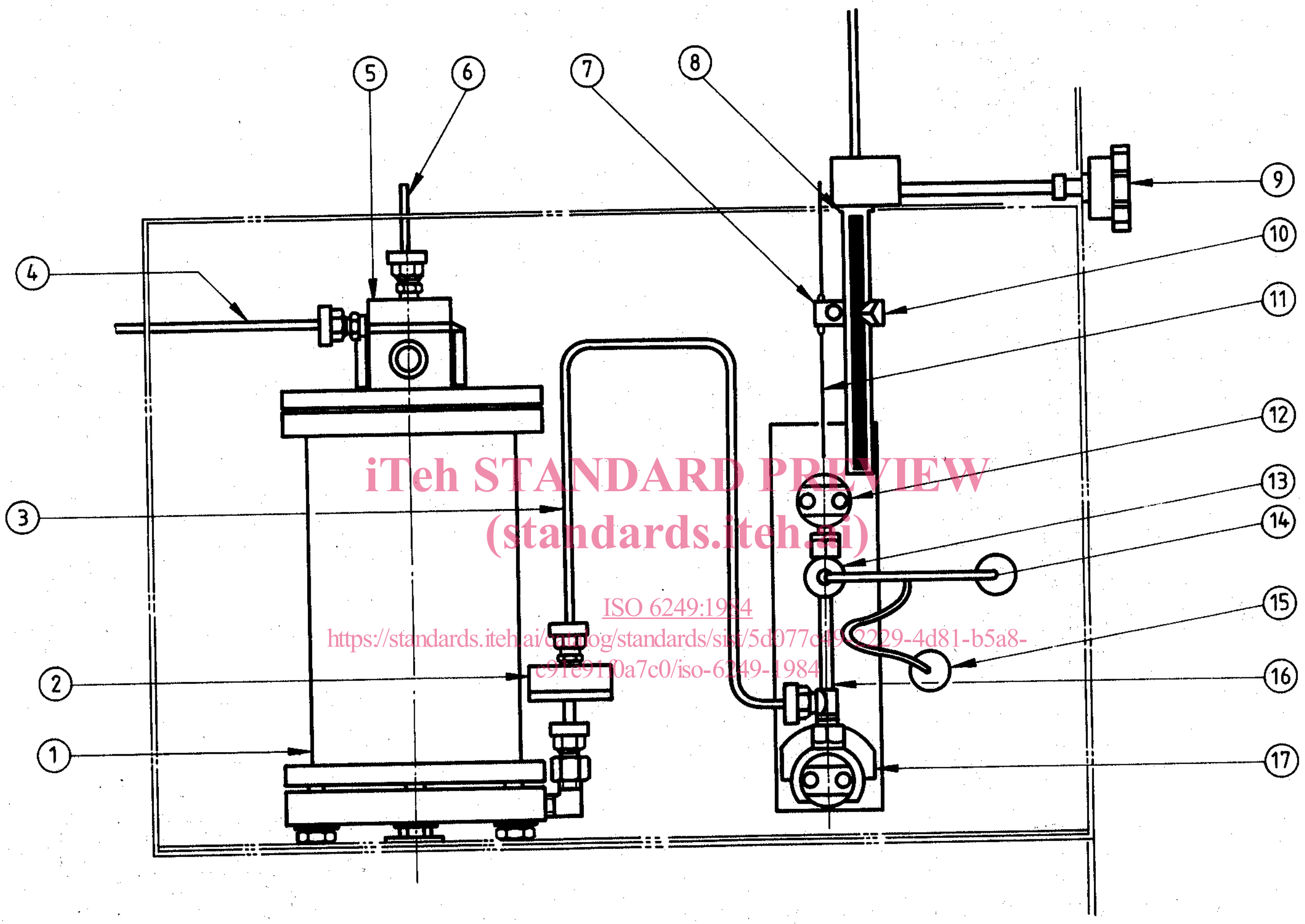


Figure 4 — Alignment of heater tube



1 Reservoir
2 Pre-filter
3 Heater tube fuel supply line
4 Nitrogen inlet line
5 Drop flow indicator
6 Spent fuel return line
7 Thermocouple clamp
8 Thermocouple reference line
9 Thermocouple position control
10 Thermocouple position indicator
11 Thermocouple
12 Upper fixed bus-bar
13 Test filter housing
14 Heater tube fuel outlet line
15 Filter bypass line
16 Heater tube housing
17 Lower floating bus-bar

Figure 5 — Test section compartment

6.8.3 Pour the test portion into the filter and allow it to flow into the reservoir.

6.8.4 Remove the funnel holder.

6.8.5 Insert a clean thermometer (3.3.4.13) and measure the temperature of the test portion. The temperature of the test portion shall be between 15 and 32 °C. If the fuel temperature is outside these limits, a suitable way to change the temperature is to set the reservoir containing the filtered test portion into a hot or cold water bath as required to bring the test portion temperature within limits.

6.8.6 Insert into the reservoir the sintered glass aeration tube (3.3.16) attached to the aeration tube holder (3.3.18). Position the diffuser so that it is touching the bottom of the reservoir.

6.8.7 Using clean, dry flexible plastics tubing, connect the aeration tube to the METERED AIR outlet on the left side of the JFTOT.

6.8.8 Open the AIR FLOW FLOATING INDEX INDICATOR control valve approximately 1/8 of a turn to avoid possible excessive pressure build-up.

6.8.9 Switch the POWER to ON.

6.8.10 Turn the AERATION TIMER control to 6 min.

6.8.11 Using the AIR FLOW valve, adjust the air flow so that the indicator float is in the green range on the flowmeter (this corresponds to approximately 1,5 l/min).

6.8.12 Record on the data sheet the clock time at which aeration is complete. No more than 1 h should elapse between this time and the time at which the heater switch is turned on.

6.8.13 When the AERATION TIMER has shut off the air flow, remove the aeration tube and its holder from the reservoir. Use the test portion dripping from the aeration tube to wet the piston lip seal in 6.9.1.

6.9 Assembly of reservoir section

6.9.1 With the reservoir piston puller (3.3.1.4) attached to the piston, wet the lip seal with filtered test portion dripping from the aeration tube in accordance with 6.8.13.

6.9.2 Insert the piston into the reservoir.

6.9.3 Push down on the piston puller, applying a gentle rocking motion, moving the piston downward until air leakage past the seal is replaced by fuel leakage; that is, eliminate all air below the piston and lip seal. Loosen the cap seal at the end of the heater tube fuel supply line. Apply a slight downward pressure on the piston puller until fuel appears at the cap seal, and retighten the cap seal. Remove the piston puller.

6.9.4 Wet the reservoir O-ring with any fuel and place the O-ring into the cover groove.

6.9.5 Place the reservoir cover on top of the fuel reservoir, taking care that the O-ring stays in the groove. Turn the cover so that the nitrogen inlet fitting on the drip flow indicator is diametrically opposite to the pre-filter fitting.

Ensure that the PROTECTOR SIGHT GLASS is mounted properly over the drip flow housing.

6.9.6 Insert the cap screws into the reservoir cover holes and tighten uniformly with the nut driver (3.3.1.10).

6.9.7 Flush the outside of the reservoir with solvent (3.3.4.5) to remove fuel.

6.9.8 Place the reservoir assembly into the cabinet with the pre-filter section turned toward the heater tube section so that the fore and aft bottom cap screw heads drop into positioning recesses.

6.9.9 Connect the nitrogen inlet line to the side of the drip flow indicator and finger tighten firmly.

6.9.10 Connect the fuel return line to the top of the drip flow indicator and finger tighten firmly.

6.9.11 Remove the cap seal from the heater tube fuel supply line and immediately connect the line to the heater tube housing inlet. The time between removal of the cap seal and connecting should be a minimum to reduce loss of test portion.

6.9.12 Recheck all eight knurled fittings to be sure they are tightened firmly. Recheck that the thermocouple is in the 38,7 mm position.

6.9.13 The apparatus is now ready for the test.

7 Test procedure

7.1 Fuel system pressurization

7.1.1 Check that the NITROGEN PRESSURIZE valve is CLOSED.

7.1.2 Check that the NITROGEN BLEED valve is CLOSED.

7.1.3 Check that the MANOMETER BYPASS valve is OPEN.

7.1.4 Adjust the nitrogen supply pressure to 3,45 MPa[1] on the pressure regulator control gauge.

7.1.5 Open and close the NITROGEN PRESSURIZE valve to obtain a pressure of approximately 0,2 to 0,3 MPa[1]. Immediately check the test section for any obvious fitting leaks.

1) 1 MPa = 10 bar.