INTERNATIONAL STANDARD 3013

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

Aviation fuels — Determination of freezing point

Carburants aviation — Détermination du point de disparition des cristaux

First edition — 1974-06-01

UDC 665.733.3 : 665.743.3 : 536.421.4 **Ref. No. ISO 3013-1974 (E)**

Descriptors : petroleum products, aviation fuels, tests, physical tests, low temperature tests, melting points, determination.

Price based on 4 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

International Standard ISO 3013 was drawn up by Technical Committee ISO/TC 28, *Petroleum products,* and circulated to the Member Bodies in March 1973.

It has been approved by the Member Bodies of the following countries :

Australia	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Sweden
Bulgaria	Mexico	Thailand
Canada	Netherlands	Turkey
Chile	New Zealand	United Kingdom
Czechoslovakia	Norway	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	
Hungary	Romania	

No Member Body expressed disapproval of the document.

Printed in Switzerland

Aviation fuels — Determination of freezing point

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a procedure for the detection of separated solids in aviation reciprocating engine and turbine engine fuels at any temperature likely to be encountered during flight or on the ground.

2 DEFINITIONS

2.1 freezing point : That temperature at which crystals of hydrocarbons formed on cooling disappear when the temperature of the fuel is allowed to rise.

2.2 crystallization point : That temperature at which crystals of hydrocarbons first appear upon cooling the fuel.

3 APPARATUS (see figure 1)

3.1 Jacketed sample tube : a doublewalled, unsilvered vessel, similar to a Dewar flask, the space between the inner sample tube and the outer glass jacket being filled at atmospheric pressure with dry nitrogen or air. The mouth of the tube shall be closed with a cork stopper supporting the thermometer and packing gland through which the stirrer passes.

3.2 Gland, consisting of a brass tube of the design shown in figure 2. This tube must fit tightly into the cork stopper, the space between the brass tube and the stirring rod being filled with absorbent cotton. A packing gland is necessary to prevent condensation of moisture in the sample tube from the surrounding air at the low test temperatures used. (See also 3.3 for an alternative design.)

3.3 Collars, moistureproof, as shown in figures 3 and 4. These may be used instead of the gland (3.2) to prevent condensation of moisture.

3.4 Stirrer, made of 1,6 mm brass rod bent into a smooth three-loop spiral at the bottom.

3.5 Vacuum flask, unsilvered, having the minimum dimensions shown in figure 1. The capacity shall be sufficient to hold an adequate volume of cooling liquid and permit the necessary depth of immersion of the jacketed sample tube.

3.6 Thermometer, total immersion type, conforming to the following specification :

Range	− 80 to + 20 °C
Immersion	total
Graduation at each	0,5 °C
Longer lines at each	1 °C and 5 °C
Figured at each	5 °C
Scale error not to exceed	1 °C
Expansion chamber permitting heating to	45 °C
Overall length	300 ± 10 mm
Stem diameter	5,5 to 8,0 mm
Bulb length	8 to 16 mm
Bulb diameter	not greater than stem
Bulb shape	cylindrical
Length of graduated portion	170 to 210 mm
Distance from bottom of bulb to 0 °C line	220 mm max.
Top finish	plain or ring

NOTES

1 Toluene or other suitable liquid coloured red with a permanent dye shall be used as the actuating liquid. The filling above the liquid shall be gas under pressure.

2 The accuracy of this thermometer must be checked in accordance with ISO/R 386, *Principles of construction and adjustment of liquid-in-glass laboratory thermometers,* at temperatures of 0, − 40, − 60, and − 75 °C. Corrections shall be applied to test readings.

Dimensions in millimetres

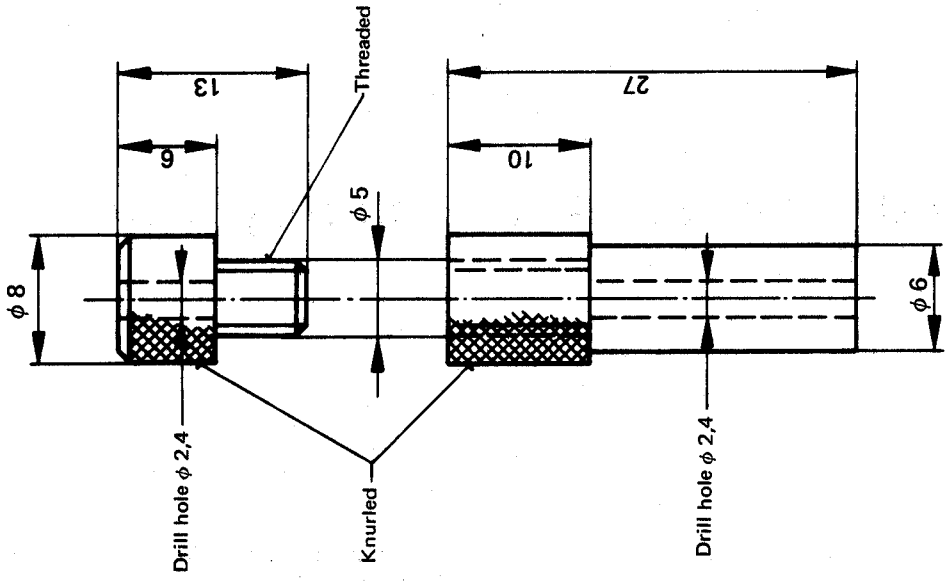


FIGURE 2 – Brass packing gland for the stirrer

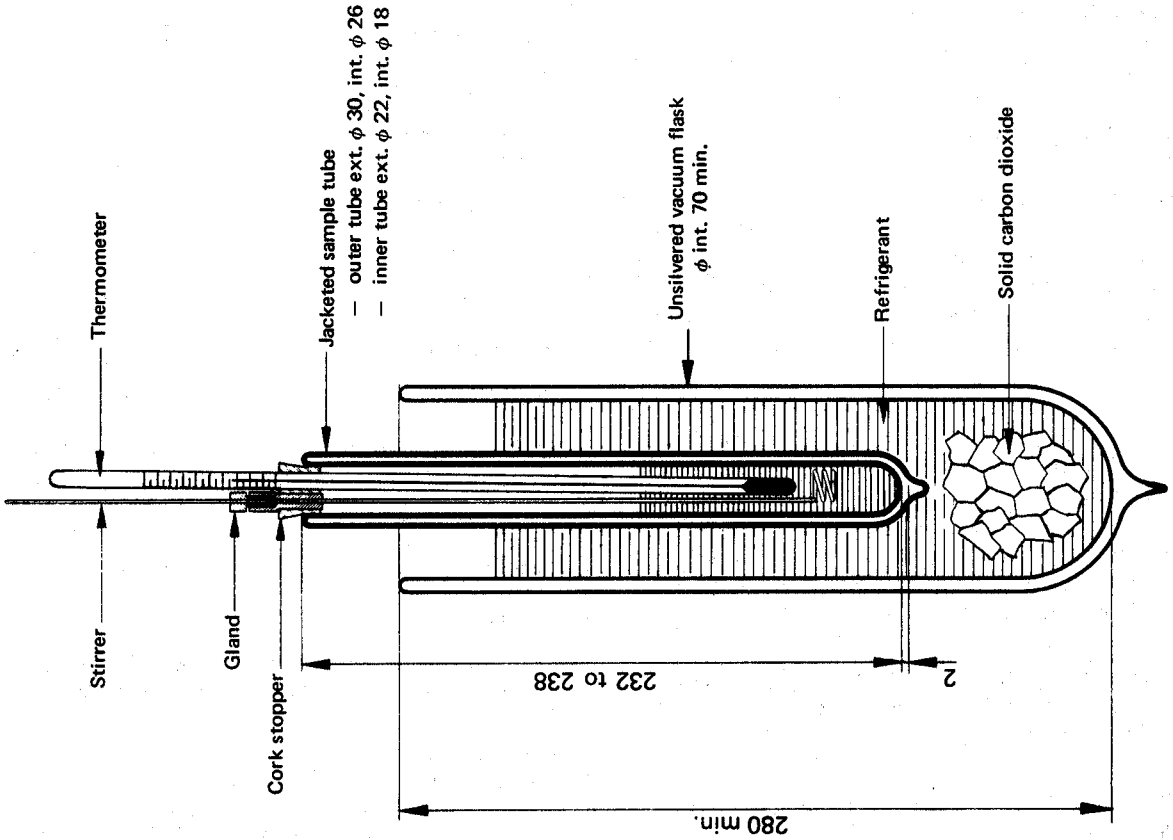
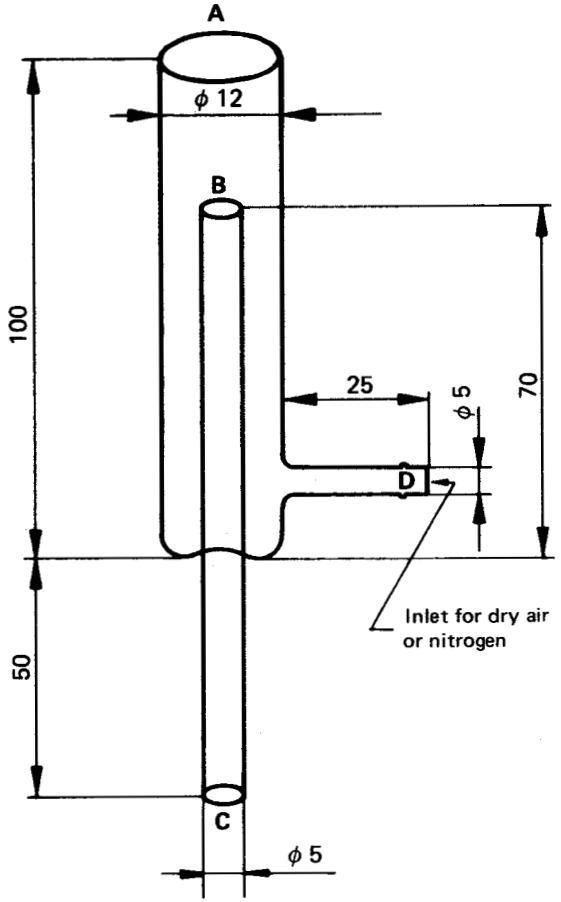


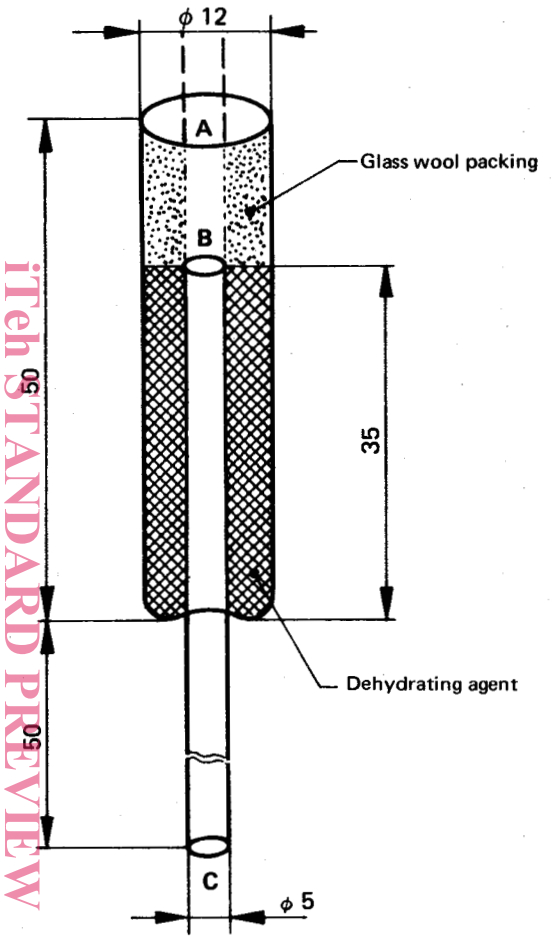
FIGURE 1 – Apparatus for determination of the freezing point of aviation fuels



NOTE — The collar of borosilicate glass is inserted at C into the two-hole cork stopper that holds the thermometer. Next, the stirrer is passed through tube CB and extends beyond A. This assembly is attached to the freezing point tube. Before the tube is lowered into the freezing bath, the collar is flushed with dry air or nitrogen, introduced at D and leaving at A. The air effectively dried by passing through U-tubes connected in series, one packed with dehydrating agent such as anhydrous calcium sulphate or silica gel, and a second packed with glass beads coated with phosphorus pentoxide. The passing of air through the collar in this fashion is continued during the entire determination. Nitrogen gas of low moisture content is usually more convenient to use.

FIGURE 3 — **Moistureproof collar, Type A**

Dimensions in millimetres



NOTE — This collar of borosilicate glass is packed with a dehydrating agent such as anhydrous calcium sulphate or silica gel having a mesh diameter of approximately 1,7 mm (No. 12 mesh) at the lower part up to within 5 mm of the upper end (B) of the tube BC. Then, with the stirrer in place, a collar of glass wool impregnated with the same size dehydrating agent is pressed snugly over the joint up to A. The glass wool packing should be replaced after every third or fourth run.

FIGURE 4 — **Moistureproof collar, Type B**

4 PROCEDURE

4.1 Transfer 25 ml of the fuel to be tested to the clean, dry jacketed sample tube. Close the tube tightly with the cork holding the stirrer and thermometer and adjust the thermometer position so that its bulb is in the centre of the fuel sample. Run 1 drop of alcohol down the stirring rod to wet the packing gland and tighten the gland as much as possible consistent with permitting smooth movement of the stirrer without using undue force.

4.2 Clamp the jacketed sample tube so that it extends as far as possible into the vacuum flask containing the cooling medium (see note). Add solid carbon dioxide as necessary throughout the test to maintain the coolant level in the vacuum flask well above the level of the test sample.

NOTE – Any convenient liquid cooled with solid carbon dioxide may be used. Acetone and alcohol are suitable. Liquid nitrogen may also be used as a coolant instead of liquids cooled with solid carbon dioxide. Mechanical refrigeration units may also be used.

4.3 Stir the fuel vigorously and continously, except when making observations, taking care that the stirrer loops remain below the fuel surface at all times (see note 1). Disregard any cloud which appears at approximately $-10\ ^{\circ}C$ and which does not increase in intensity as the temperature is lowered, since this is due to water (see note 2). Record as the crystallization point the temperature at which crystals of hydrocarbon appear. Remove the jacketed sample tube from the coolant and allow the sample to warm up slowly, stirring the fuel continously. Record as the freezing point the temperature at which the hydrocarbon crystals completely disappear. If the difference between the two temperatures is greater than $3\ ^{\circ}C$, repeat the cooling and warming until the difference is less than $3\ ^{\circ}C$.

NOTES

1 When the approximate freezing point is known, it is sufficient to provide occasional stirring until the temperature is within $10\ ^{\circ}C$ of the expected freezing point, but vigorous stirring must be maintained thereafter. A mechanical stirring device may be used.

2 If a cloud appears due to dissolved water that interferes with the observation of hydrocarbon crystals, the sample can be dried over anhydrous sodium sulphate before filling the sample tube.

5 PRECISION

The following criteria shall be used for judging the acceptability of results (95 % confidence level) :

5.1 Repeatability

Duplicate results by the same operator shall be considered suspect if they differ by more than $0,7\ ^{\circ}C$.

5.2 Reproducibility

The results submitted by each of two laboratories shall be considered suspect if the two results differ by more than $2,6\ ^{\circ}C$.

6 TEST REPORT

Apply any correction established for the thermometer used (see note 2 to 3.6). Report the corrected temperature of crystal disappearance to the nearest $0,5\ ^{\circ}C$ as the freezing point and make reference to this International Standard.