International Standard 4626

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Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials

Liquides organiques volatils — Détermination de l'intervalle de distillation des solvants organiques utilisés comme matières premières

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ISO 4626-1980 (E)

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 4626 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in September 1976.

It has been approved by the member bodies of the following countries :

Australia	Iran	Poland
Austria	Israel	Romania
Brazil	Italy	South Africa, Rep. of
Bulgaria	Korea, Rep. of	Sweden
Canada	Mexico	Switzerland
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
France	Norway	Yugoslavia
India	Peru	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Germany
U.S.A.

This International Standard is one of three ISO publications dealing with the determination of distillation characteristics. The two others are :

- ISO/918, *Test method for distillation (distillation yield and distillation range).*
- ISO 3405, *Petroleum products – Determination of distillation characteristics.*

It is recognized that there may be some overlapping between these three documents which were developed in separate technical committees. In the absence of agreement on a general International Standard on the subject, it has however been thought necessary to publish them.

The completion in the future of such a general standard may therefore lead to the amendment or cancellation of this International Standard.

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SAFETY PRECAUTIONS

A Peroxide formation

Certain solvents and chemical intermediates, particularly but not only ethers and unsaturated compounds, may form peroxides during storage. These peroxides may present an explosion hazard when the product is distilled, especially as the dry point is approached.

When peroxide formation is likely, either because of the chemical nature of the product, the type of the product or its length of storage, the material should be analysed for peroxides and, if they are present, appropriate precautions should be taken, such as destruction of the peroxides before distillation, or protection of the operator.

Test for peroxides

Add 0,5 to 1,0 ml of the material to be tested to an equal volume of glacial acetic acid to which has been added about 100 mg of sodium or potassium iodide crystals.

Carry out a blank determination. A comparatively yellow colour indicates a low and a brown colour a high concentration of peroxide in the sample.

B Flammability

Most organic solvents and chemical intermediates are flammable. A fire hazard exists when a distillation is carried out and safety precautions should be taken.

Before starting the test, the flask should be examined for flaws and care should be taken that good seals are obtained where the side arm connects with the condenser and where the thermometer fits into the neck. Some solvents are liable to auto-ignition if distilled. Distillation of these products should be avoided. During the distillation, a suitable catch-pan and shield should be used to contain spilled liquid in the event of accidental breakage of the distillation flask.

Adequate ventilation should be provided to maintain the solvent vapour concentration below the explosive limit in the immediate vicinity of the distillation apparatus, and below the threshold limit value in the general work area.

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a method for determining the boiling range of liquids that boil between 30 and 300 °C at normal pressure, and that are chemically stable and do not corrode the apparatus during the distillation.

1.2 The method is applicable to organic liquids such as hydrocarbons, esters, alcohols, ketones, ethers and similar products.

NOTE — The method differs from that described in ISO/R 918 with respect to the volume of the distillation flask, the type of cooler and the distillation receiver.

The method differs from that specified in ISO 3405 with respect to the volume of the distillation flask and the diameter of the hole in the flask support.

2 REFERENCES

ISO 842, *Raw materials for paints and varnishes — Sampling.*

ISO/R 918, *Test method for distillation (distillation yield and distillation range).*

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling.*

ISO 3405, *Petroleum products — Determination of distillation characteristics.*

3 DEFINITIONS

3.1 initial boiling point : The temperature noted (corrected if required) at the moment when the first drop of condensate falls from the tip of the condenser during a distillation carried out under standardized conditions.

3.2 dry point : The temperature noted (corrected if required) at the moment of vaporization of the last drop of liquid at the bottom of the flask during a distillation carried out under standardized conditions, disregarding any liquid on the side of the flask and on the thermometer.

3.3 boiling range : The temperature interval between the initial boiling point and the dry point.

3.4 end point; final boiling point : The maximum temperature noted (corrected, if required) during the final phase of a distillation carried out under standardized conditions.

4 PRINCIPLE

Distillation of a 100 ml test portion under prescribed conditions which are equivalent to a simple batch distillation. Systematic observation of thermometer readings and volumes of condensate and calculation of the results from these data with correction to standard atmospheric pressure.

5 APPARATUS

The apparatus, a suitable form of which is shown in figures 1 to 4, shall comprise the following items :

5.1 Distillation flask, of heat-resistant glass, of capacity 200 ml, conforming to the dimensions shown in figure 1.

NOTE — Superheating of liquid in a new flask may be prevented by depositing a small amount of carbon in the bottom of the flask. This may be accomplished by heating and decomposing a pinch of tartaric acid in the bottom of the flask. The flask is then prepared for use by washing with water, rinsing with acetone, and drying.

An exception is made for diacetone alcohol : in order to avoid an erratic value for the initial boiling point, the distillation flask should be clean and free of any residual carbon deposit.

5.2 Thermometers, mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to the requirements in table 1.

NOTE — The thermometer should have been artificially aged by means of a suitable treatment before graduation, in order to ensure stability of the lowest point on the scale. This treatment should have been such that, after the procedure described below, the rise at a fiducial point is not greater than the maximum error specified, and the accuracy of the thermometer is within the limits specified.

Heat the thermometer to a temperature equal to its highest reading and keep it at this temperature for 5 min. Allow the thermometer to cool, either naturally in still air or slowly in the test bath (at a specified rate), to 20 °C above ambient temperature or to 50 °C, whichever is the lower, and then determine the lowest point on the scale. If rapid cooling is used, the lowest point on the scale shall be determined within 1 h. Heat the thermometer again to a temperature equal to its highest reading, keep it at this temperature for 24 h, allow the thermometer to cool to one of the two temperatures referred to above, at the same rate as at the start of the test, and re-determine the lowest point on the scale under the same conditions as before.

5.3 Draught screen

5.3.1 *For use with a gas burner*

The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in figure 2 and be made of sheet of metal of thickness approximately 0,8 mm.

Dimensions in millimetres

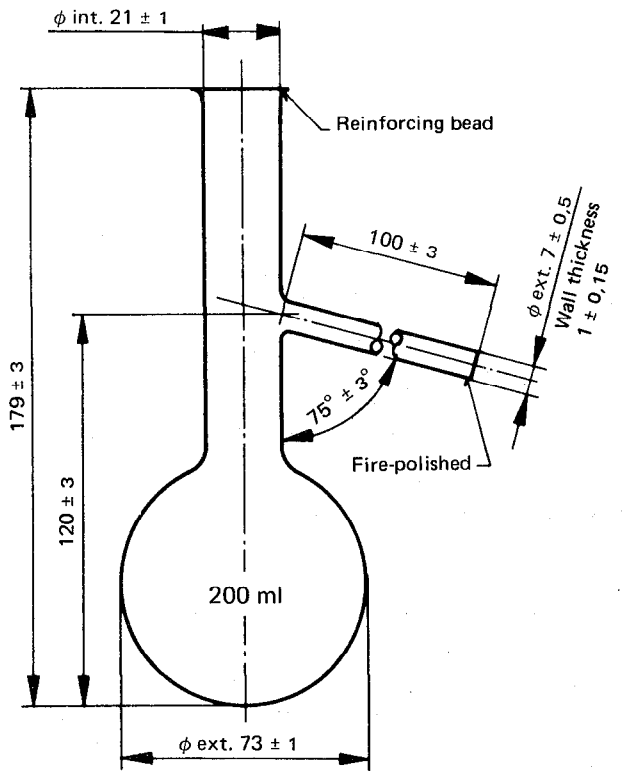

FIGURE 1 — **Distillation flask (5.1)**