INTERNATIONAL STANDARD

ISO 9029

First edition
1990-12-15

Crude petroleum — Determination of water — Distillation method

Pétrole brut — Détermination de la teneur en eau — Méthode de distillation

Reference number
ISO 9029:1990(E)

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. Each 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9029 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annex A forms an integral part of this International Standard.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Crude petroleum — Determination of water — Distillation method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems 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.

1 Scope

This International Standard specifies a method for determining water in crude oil by distillation. The precision data have only been determined for water contents up to 1 % (*V*/*V*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

ISO 4259:1979, *Petroleum products — Determination and application of precision data in relation to methods of test.*

ISO 5280:1979, *Xylene for industrial use — Specification.*

3 Significance

A knowledge of the water content of crude oil is important in the refining, purchase, sale and transfer of products.

The amount of water as determined by this method is used to correct the volume involved in the custody transfer of oil.

4 Principle

A test portion is heated under reflux conditions with a water-immiscible solvent which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap. The water settles in the graduated section of the trap, and the solvent returns to the distillation flask.

5 Apparatus

Usual laboratory apparatus, together with the following:

5.1 General.

The recommended apparatus, shown in figure 1, consists of a glass distillation flask, a condenser, a graduated glass trap and a heater. Other types of apparatus may be used for this International Standard, provided it can be demonstrated that they operate within the precision established, in accordance with ISO 4259, with the preferred apparatus.

5.2 Distillation flask.

A 1 000 ml round-bottom glass distillation flask with a 24/39 conical ground-glass socket shall be used.

5.3 Trap.

A 5 ml graduated glass trap with 0,05 ml graduations and 24/39 ground-glass cone and socket shall be used.

5.4 Condenser.

The trap (5.3) shall be fitted with a 400 mm Liebig condenser.

5.5 Drying tube.

A drying tube filled with a self-indicating dessicant shall be placed at the top of the condenser (5.4).

NOTE 1 This tube is to prevent entry of atmospheric moisture.

5.6 Heater.

Any suitable gas or electric heater that can uniformly distribute heat to the entire lower half of the flask may be used. An electric heating mantle is preferred for safety reasons.

5.7 Jet-spray tube, for washing down the condenser inner tube, as shown in figure 2.

5.8 Pick, made of brass or bronze, or **scraper**, made of steel, with a PTFE tip, as shown in figure 2.

5.9 Calibration of the apparatus.

The assembled apparatus shall be calibrated and tested as described in clause 7 and the readings obtained shall be within the tolerances specified.

6 Solvent

Use xylene meeting the requirements of ISO 5280. The water content of the xylene is determined by carrying out a blank test (see 9.6).

7 Calibration and recovery test

7.1 General

Before initial use, calibrate the trap in accordance with 7.2. Before a series of tests, check the entire apparatus in accordance with 7.3.

7.2 Calibration

Before initial use, verify the accuracy of the graduation marks on the trap by adding 0,05 ml increments of distilled water, from a 5 ml microburette or a precision micro-pipette readable to the nearest 0,01 ml. If there is a deviation of more than 0,05 ml between the water added and water observed, reject the trap or recalibrate.

7.3 Recovery test

Test the overall recovery of water in the entire apparatus by introducing 400 ml of dry (0,02 % water maximum) xylene into the apparatus and proceeding as described in clause 9. When this initial run is complete, discard the contents of the trap and add 1,00 ml ± 0,01 ml of distilled water, from a burette or micro-pipette, directly to the distillation flask and proceed again in accordance with clause 9. Repeat this procedure again, but adding 4,50 ml ± 0,01 ml of distilled water to the xylene in the distillation flask.

The assembly of the apparatus is satisfactory only if trap readings are within the tolerances specified in table 1.

7.4 Malfunctions

A reading outside the limits suggests malfunctioning due to vapour leaks, too rapid boiling, inaccuracies in the graduations of the trap, or ingress of extraneous moisture. If such a malfunction can be identified, eliminate the malfunction and repeat the recovery test described in 7.3.

Table 1 — Tolerances on water recovery

Maximum capacity of trap at 20 °C ml	Volume of water added at 20 °C ml	Permissible limits for recovered water at 20 °C ml
5,00	1,00	1,00 ± 0,025
5,00	4,50	4,50 ± 0,025

8 Sampling (see annex A)

8.1 General

Sampling is defined as all steps required to obtain a representative sample of the contents of any pipe, tank or other system and to place the sample into the laboratory test container.

8.2 Laboratory sample

Only representative samples obtained as specified in ISO 3170 or ISO 3171 shall be used for this International Standard.

8.3 Preparation of test portions

The following sample-handling procedure shall apply in addition to those covered in 8.2.

8.3.1 The size of the test portion shall be selected as indicated in table 2, based on the expected water content of the sample.

If there is any doubt about the homogeneity of the mixed sample, determinations shall be made on the total volume of the sample if the sample size is compatible with the expected water content (see table 2). If this is not possible, a determination shall be made on at least three test portions. Include all these results in the test report and record their average as the water content of the sample.

8.3.2 To determine water on a volume basis, measure out mobile liquids in a cylinder of capacity equal to the test portion size selected in 8.3.1. Take care to pour the sample slowly into the graduated cylinder to avoid entrapment of air, and adjust the level as closely as possible to the appropriate graduation. Carefully pour the contents of the cylinder into the distillation flask and rinse the cylinder with a measured volume, consistent with the size of the cylinder, of xylene (see clause 6), in five portions, and add the rinsings to the flask. Drain the cylinder thoroughly to ensure complete test portion transfer.

8.3.3 To determine water on a mass basis, weigh out a test portion (see 8.3.1), pouring the test portion directly into the distillation flask. If it is necessary to use a transfer vessel (e.g. a beaker or a cylinder), rinse this vessel with five portions of xylene in the same way as described in 8.3.2 and add the rinsings to the flask, then calculate the mass of the test portion.

9 Procedure (see also annex A)

9.1 The precision of this International Standard can be affected by water droplets adhering to surfaces in the apparatus and therefore not settling into the water trap to be measured. To minimize the problem, chemically clean all apparatus, at least daily, to remove surface films and debris which hinder free drainage of water in the test apparatus. More frequent cleaning is recommended if the nature of the samples being run causes persistent contamination.

9.2 To determine water on a volume basis, proceed as indicated in 8.3.2. Add sufficient xylene to the flask to make the total volume 400 ml.

9.2.1 To determine water on a mass basis, proceed as indicated in 8.3.3. Add sufficient xylene to the flask to make the total volume 400 ml.

9.2.2 A magnetic stirrer is the most effective device to reduce bumping. Glass beads or other boiling aids, although less effective, have been found to be useful.

9.3 Assemble the apparatus as shown in figure 1, making sure all connections are vapour- and liquid-tight. It is recommended that glass joints are not greased. Circulate water, between 20 °C and 25 °C, through the condenser jacket.

Table 2 — Size of test portion

Expected water content % (*m*/*m*) or % (*V*/*V*)	Approximate test portion size g or ml
50,1 to 100,0	5
25,1 to 50,0	10
10,1 to 25,0	20
5,1 to 10,0	50
1,1 to 5,0	100
0,5 to 1,0	200
Less than 0,5	200

9.4 Apply heat to the flask. The type of crude oil being evaluated can significantly alter the boiling characteristics of the crude/solvent mixture. Apply heat slowly during the initial stages of the distillation (approximately half an hour to one hour) to prevent bumping and possible loss of water from the system (condensate shall not proceed higher than three-quarters of the distance up the condenser inner tube — point A in figure 1; to facilitate condenser wash-down, the condensate should be held as close as possible to the condenser outlet). After the initial heating, adjust the rate of boiling so that the condensate proceeds no more than three-quarters of the distance up the condenser inner tube. Collect the distillate in the trap at a rate of approximately 2 to 5 drops per second. Continue distillation until no water is visible in any part of the apparatus, except in the trap, and the volume of water in the trap remains constant for at least 5 min.

If there is a persistent accumulation of water droplets in the condenser inner tube, flush with xylene [a jet-spray washing tube (5.7) or equivalent device is recommended]. It is essential to shut off the heat at least 15 min prior to wash-down to prevent bumping. The addition to the xylene wash of an oil-soluble emulsion breaker at a concentration of 1 000 ppm helps dislodge the clinging water drops. After flushing, redistill for at least 5 min, applying the heat slowly to prevent bumping.

Repeat this procedure until no water is visible in the condenser and the volume of water in the trap remains constant for at least 5 min. If this procedure does not dislodge the water, use the pick or PTFE scraper (5.8), or an equivalent device, to cause the water to run into the trap.

9.5 When the carry-over of water is complete, allow the trap and contents to cool to room temperature. Dislodge any drops of water adhering to the sides of the trap with the pick or PTFE scraper and transfer them to the water layer. Read the volume of the water in the trap. The trap is graduated in 0,05 ml increments, but the volume is estimated to the nearest 0,025 ml.

9.6 Carry out a blank determination by placing 400 ml of solvent in the distillation flask and testing as outlined in 9.1 to 9.5.

10 Expression of results

Calculate the water content of the sample using one of the following expressions as appropriate:

a) $$\% (V/V) = \frac{V_2 - V_0}{V_1} \times 100$$

b) $$\% (V/V) = \frac{V_2 - V_0}{m/\rho} \times 100$$

c) $$\% (m/m) = \frac{V_2 - V_0}{m} \times 100$$

where

V_0 is the volume, expressed in millilitres, of water in the trap (rounded to the nearest 0,025 ml) when measurement is made on the solvent blank;

V_1 is the volume, expressed in millilitres, of the test portion;

V_2 is the volume, expressed in millilitres, of water in the trap (rounded to the nearest 0,025 ml);

m is the mass, expressed in grams, of the test portion;

ρ is the density, expressed in grams per millilitre, of the sample.

It is assumed that the density of water is 1 g/ml.

Volatile water-soluble material, if present, may be measured as water.

Report the result as the water content rounded to the nearest 0,025 %.

11 Precision

The precision of the method as obtained by statistical examination of inter-laboratory test results in the range from 0,01 % to 1,0 % is as follows:

11.1 Repeatability

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

from 0,0 % (V/V) to 0,1 % (V/V) water: see figure 3;

from 0,1 % (V/V) to 1,0 % (V/V) water: 0,08 % (V/V).

11.2 Reproducibility

The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

from 0,0 % (V/V) to 0,1 % (V/V) water: see figure 3;

from 0,1 % (V/V) to 1,0 % (V/V) water: 0,11 % (V/V).

12 Test report

The test report shall contain at least the following information:

a) all details necessary for the identification of the product tested;

b) a reference to this International Standard;

c) the result of the test (see clause 10);

d) the individual results for each test portion, if more than one test portion is examined (see 8.3);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) the date of the test.

Dimensions in millimetres

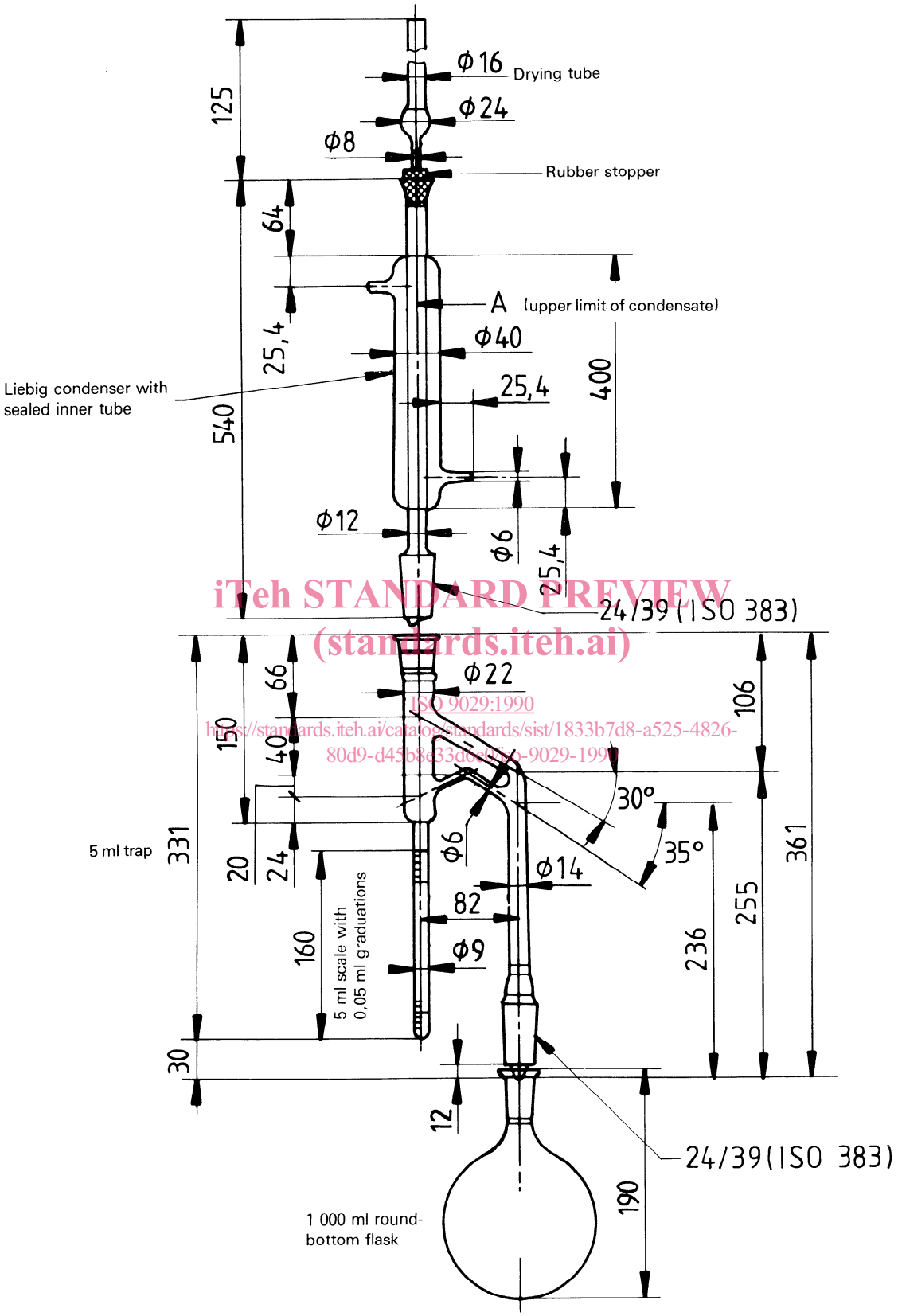

Figure 1 — Distillation apparatus

Dimensions in millimetres

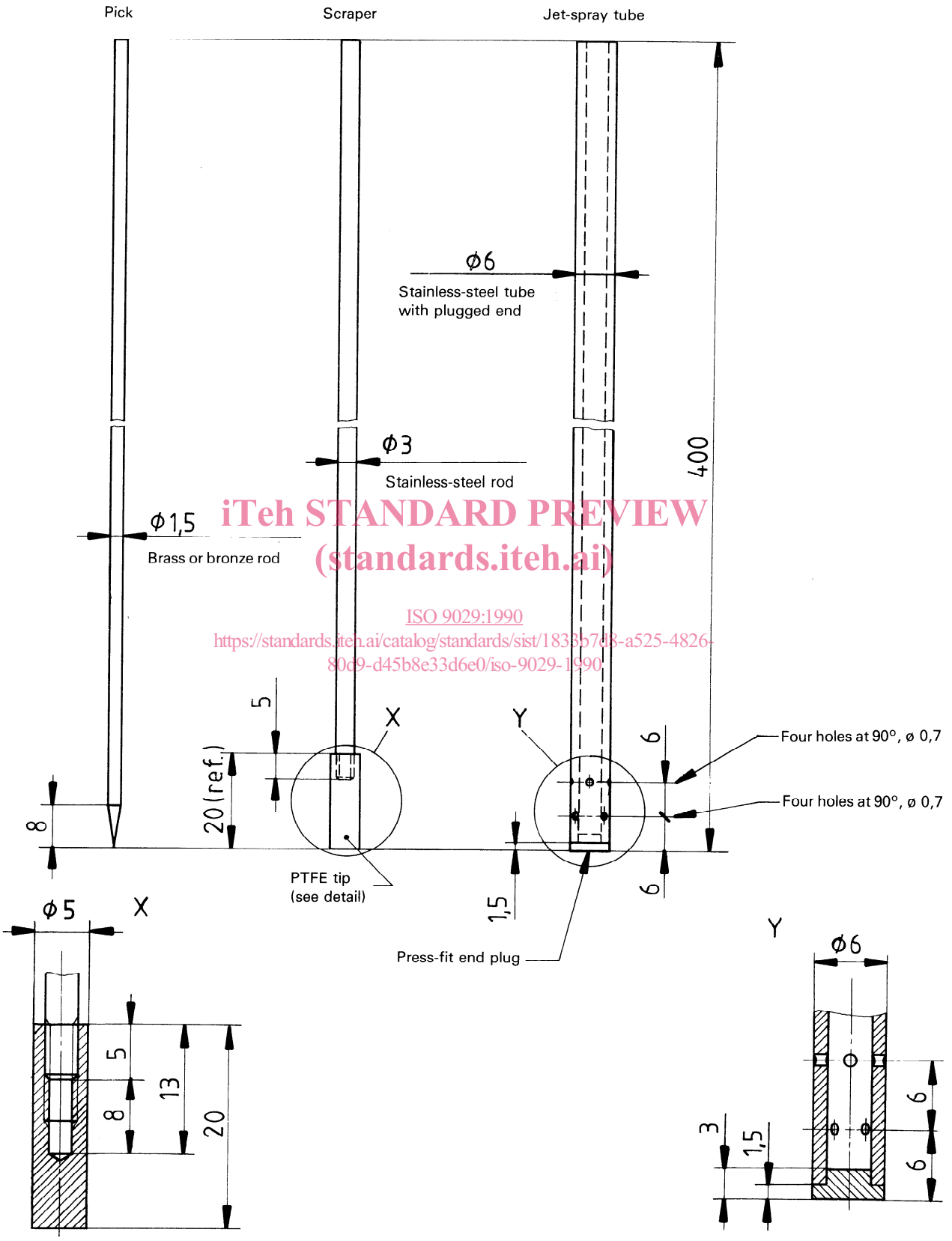

Figure 2 — Typical pick, scraper and jet-spray tube for distillation apparatus