INTERNATIONAL STANDARD

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Petroleum products — Determination of distillation characteristics at atmospheric pressure

Produits pétroliers — Détermination des caractéristiques de distillation à pression atmosphérique

Reference number
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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. 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3405 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 3405:1988), of which it constitutes a technically revision.

Annexes A and B form a normative part of this International Standard. Annexes C, D and E are for information only.

Petroleum products — Determination of distillation characteristics at atmospheric pressure

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a laboratory method for the determination of the distillation characteristics of light and middle distillates derived from petroleum with initial boiling points above 0 °C and end-points below approximately 400 °C, utilizing either manual or automated equipment, with the manual procedure being the referee method in cases of dispute, unless otherwise agreed.

NOTE The method is applicable to petroleum products incorporating a minor constitution of components from non-petroleum origin, but the precision data may not apply in all cases.

The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives important information on composition and behaviour during storage and use, and the rate of evaporation is an important factor in the application of many solvents. Limiting values to specified distillation characteristics are applied to most distillate petroleum product specifications in order to control end-use performance and to regulate the formation of vapours which may form explosive mixtures with air, or otherwise escape into the atmosphere as emissions (VOC).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 918:1983, *Volatile organic liquids for industrial use — Determination of distillation characteristics*.

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

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

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*.

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders*.

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1
decomposition point
thermometer reading (corrected) which coincides with the first indications of thermal decomposition of the liquid in the flask

NOTE Characteristic indications of thermal decomposition are an evolution of fumes and erratic thermometer readings which usually show a decided decrease after any attempt has been made to adjust the heat.

3.2
dry point
thermometer reading (corrected) that is observed at the instant the last drop of liquid evaporates from the lowest point in the flask; any drops or film of liquid on the side of the flask or on the thermometer are disregarded

NOTE The end-point (final boiling point), rather than the dry point is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end-point (final boiling point) whenever the sample is of such a nature that the precision of the end-point cannot consistently meet the requirements given in clause 12.

3.3
end-point
final boiling point
maximum thermometer reading (corrected) obtained during the test

NOTE This usually occurs after evaporation of all liquid from the bottom of the flask.

3.4
initial boiling point
thermometer reading (corrected) that is observed at the instant that the first drop of condensate falls from the lower end of the condenser tube

3.5
percent evaporated
sum of the percent recovered and the percent loss

3.6
percent loss
100 minus the total recovery

NOTE Sometimes called “front-end loss”; this is the amount of uncondensed material lost in the initial stages of the distillation.

3.7
corrected loss
percent loss corrected for barometric pressure

3.8
percent recovered
volume of condensate observed in the receiving graduated cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with a simultaneous temperature reading

3.9
percent recovery
maximum percent recovered, as observed in accordance with 9.10

3.10
percent residue
volume of residue measured in accordance with 9.11, and expressed as a percentage of the charge volume

3.11
percent total recovery
combined percent recovery and residue in the flask, as determined in accordance with 10.1

3.12
thermometer reading
temperature recorded by the sensor of the saturated vapour measured in the neck of the flask below the vapour tube, under the specified conditions of this test

3.13
temperature reading
thermometer or temperature-measurement device reading (3.12) which is corrected to 101,3 kPa barometric pressure

3.14
emergent stem effect
offset in temperature reading caused by the use of a total immersion mercury-in-glass thermometer in the partial immersion mode

NOTE The emergent part of the mercury column is at a lower temperature than the immersed portion, resulting in a lower temperature reading than that obtained when the thermometer was completely immersed for calibration.

3.15
temperature lag
offset in temperature reading between a mercury-in-glass thermometer and an electronic temperature-measurement device, caused by the different response time of the systems involved

4 Principle

The sample is assigned into one of five groups based on its composition and expected volatility characteristics, each group defining the apparatus arrangement, condenser temperature and operational variables. A 100 ml test portion is distilled under the specified conditions appropriate to the group into which the sample falls, and systematic observations of thermometer readings and volumes of condensate recovered are made. The volume of the residue in the flask is measured, and the loss on distillation recorded. The thermometer readings are corrected for barometric pressure, and the data are then used for calculations appropriate to the nature of the sample and the specification requirements.

5 Apparatus

5.1 General

Typical assemblies of the manual apparatus are shown in Figures 1 and 2.

5.2 Distillation flasks

The distillation flasks shall have a capacity of 100 ml or 125 ml and be constructed of heat-resistant glass, according to the dimensions and tolerances shown in Figures 3 and 4.

NOTE For tests specifying the dry point, especially selected flasks with bottoms and walls of uniform thickness are desirable.

5.3 Condenser tube and cooling bath

Typical types of condenser and cooling bath are illustrated in Figures 1 and 2.

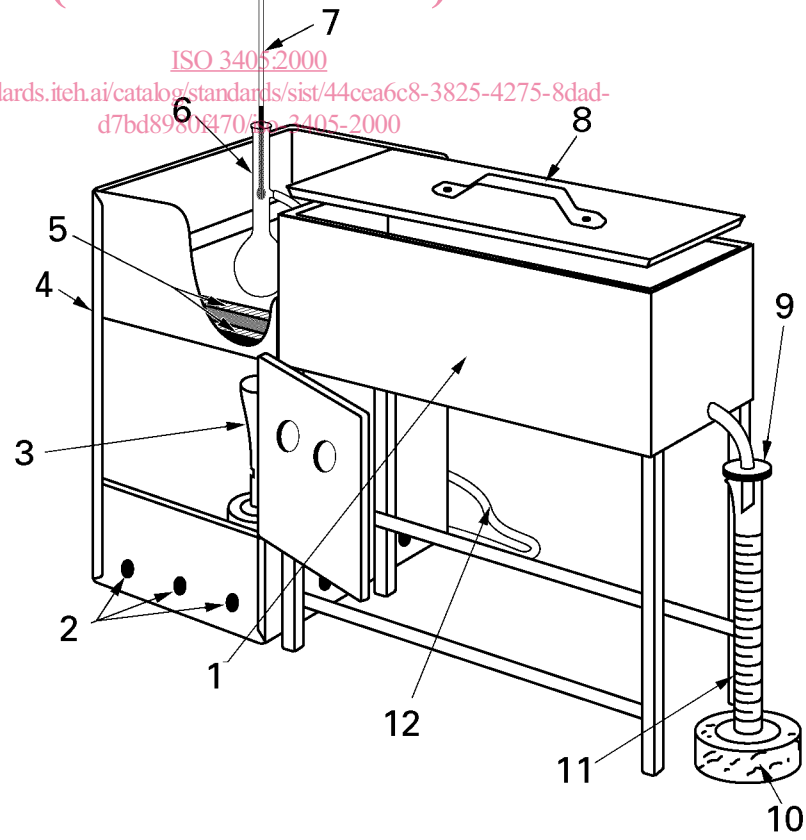
NOTE Other types of apparatus may be used, provided that the test results obtained by their use are such as to correlate with the results obtained with those illustrated, and to satisfy the precision criteria given in clause 12.

5.3.1 The condenser shall be made of seamless non-corrosive metal tubing, 560 mm ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0,8 mm to 0,9 mm.

NOTE Brass or stainless steel are suitable materials.

5.3.2 The condenser shall be set so that 393 mm ± 3 mm of the tube is in contact with the cooling medium, with 50 mm ± 3 mm outside the cooling bath at the upper end, and 114 mm ± 3 mm outside at the lower end. The portion of tube projecting at the upper end shall be set at an angle of 75° to the vertical. The portion of the tube inside the cooling bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15° ± 1° with respect to the horizontal, and no 100 mm section shall have a gradient outside a 15° ± 3° range. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end cut off at an acute angle. Provisions shall be made to enable the flow of distillate to run down the side of the receiving graduated cylinder. Figure 5 gives an illustration of the lower end of the condenser tube.

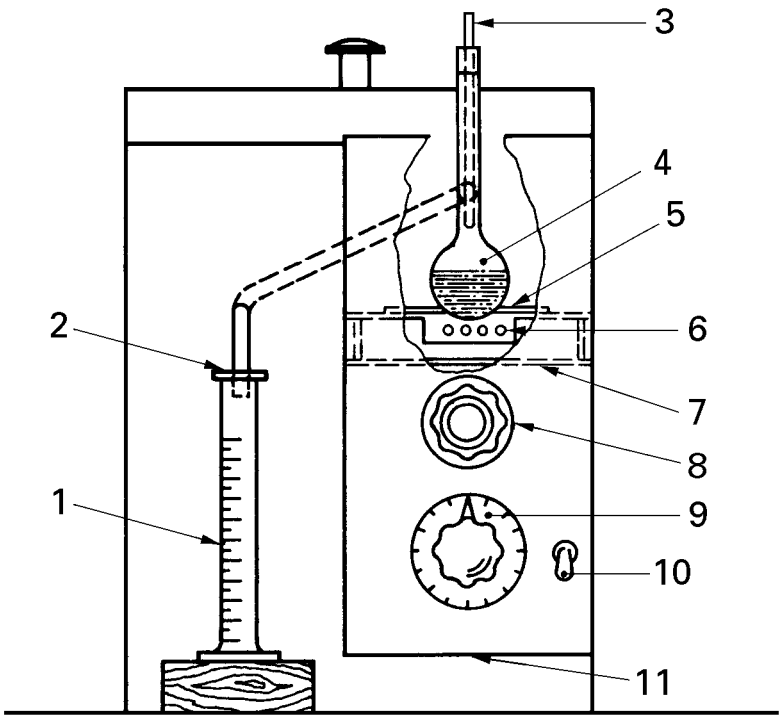
NOTE The flow of distillate down the side of the graduated cylinder may be accomplished either by using a drip-deflector which is inserted in the receiver, or by having the downward length of the condenser tube curve slightly backwards so as to ensure contact with the wall of the graduated cylinder at a point 25 mm to 32 mm below the top of the graduated cylinder when it is in position to receive distillate.

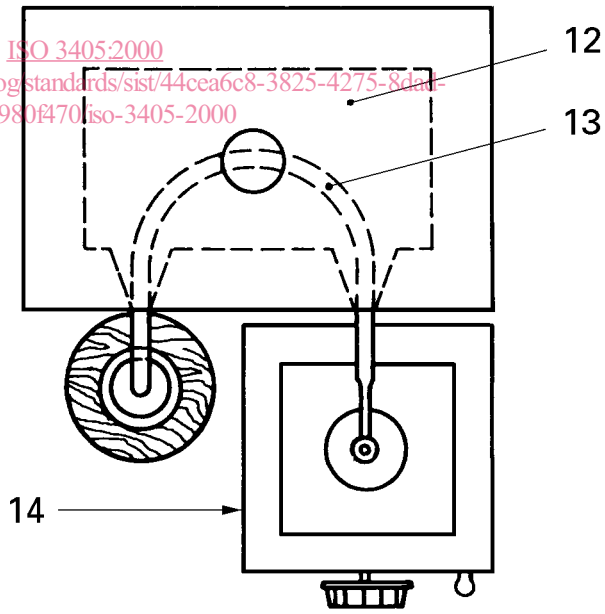


Key

1 Cooling bath
2 Air vents
3 Burner
4 Shield
5 Heat-resistant boards
6 Distillation flask
7 Thermometer
8 Bath cover
9 Blotting paper
10 Support
11 Graduated cylinder
12 Gas line

Figure 1 — Apparatus assembly using a gas burner



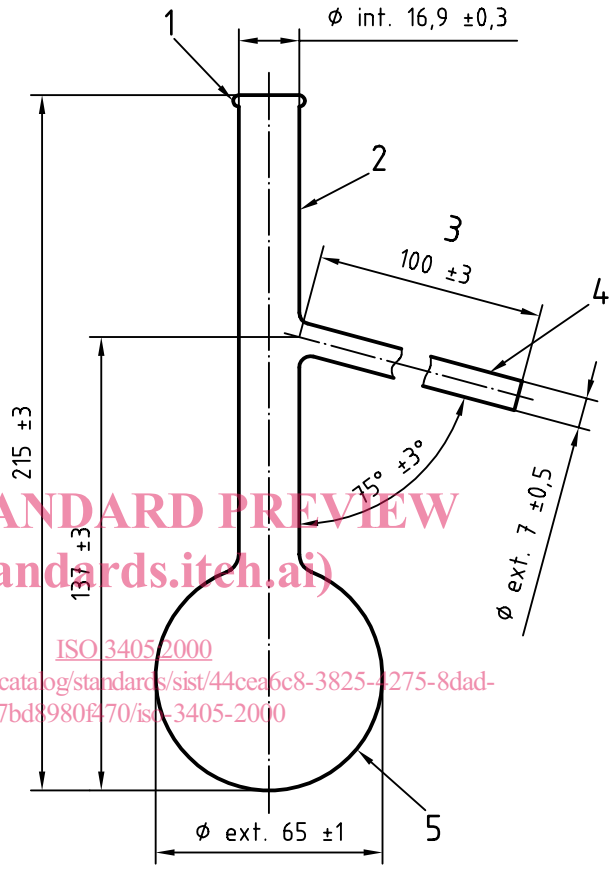


Key

1 Graduated cylinder
2 Blotting paper
3 Thermometer
4 Distillation flask
5 Flask-support board
6 Electric heating element
7 Flask-support platform
8 Flask-adjusting knob
9 Indicating dial
10 Switch
11 Open bottom shield
12 Cooling bath
13 Condenser tube
14 Shield

Figure 2 — Apparatus assembly using an electric heater

Dimensions in millimetres

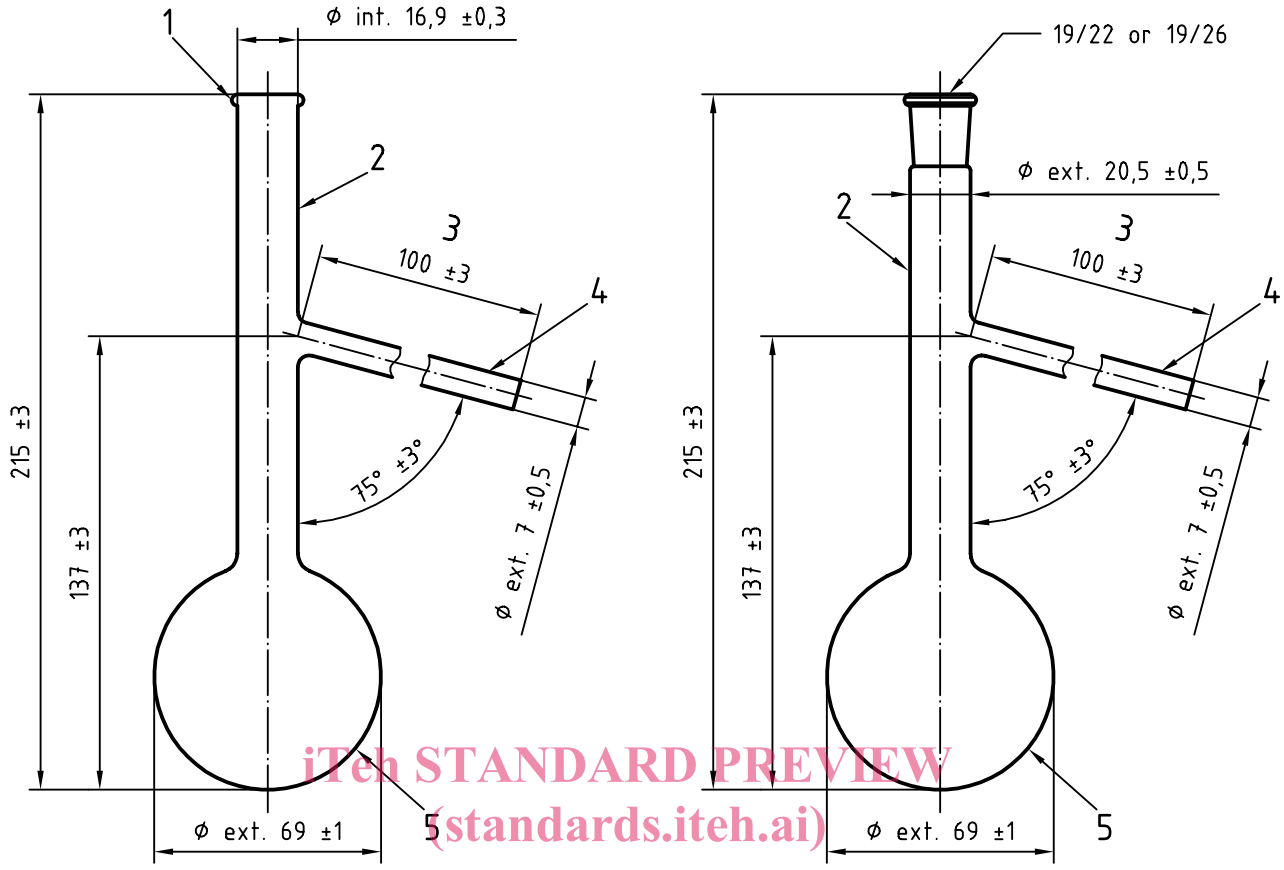


Key

1 Reinforcing bead
2 Wall 1,8 ± 0,2
3 Fire polished 100 ± 3
4 Wall 1,15 ± 0,15
5 Wall 1,5 ± 0,5

Figure 3 — 100 ml flask

Dimensions in millimetres

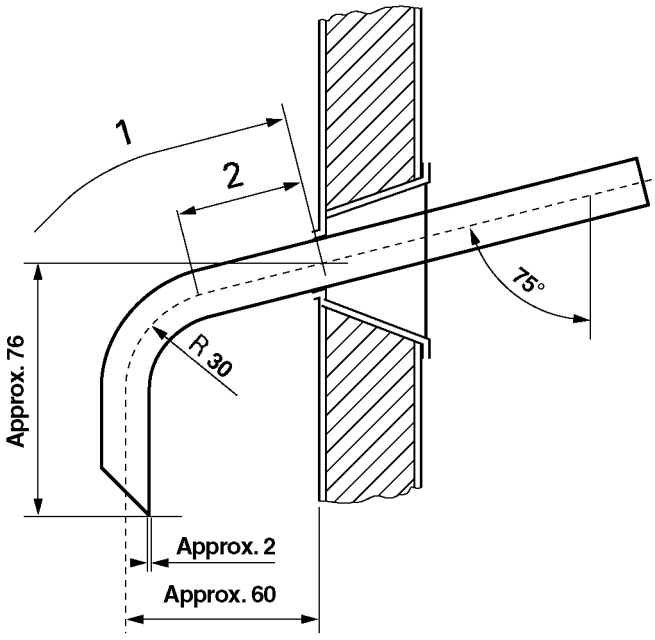


Key

1 Reinforcing bead
2 Wall 1,8 ± 0,2
3 Fire polished 100 ± 3
4 Wall 1,15 ± 0,15
5 Wall 1,5 ± 0,5

Figure 4 — 125 ml flasks – Alternative neck designs

Dimensions in millimetres



Key

1 Total length 114 ± 2
2 Linear part
Approx. 38

Figure 5 — Lower end of condenser tube

5.3.3 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.

5.4 Metal shield or enclosure for flask (manual apparatus only)

Shields shall be provided to protect the operator from damage to the unit during operation, and to protect the distillation flask from draughts. They shall allow easy access to the distillation during operation, and be provided with at least one window to observe the dry point at the end of the distillation.

NOTE 1 A typical shield for a unit fitted with a gas burner would be 400 mm high, 280 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (see Figure 1).

NOTE 2 A typical shield for a unit fitted with an electric heater would be 440 mm high, 200 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (see Figure 2).

5.5 Heat source

5.5.1 Gas burner (see Figure 1), capable of bringing over the first drop from a cold start within the time specified, and continuing the distillation at the specified rate. A sensitive regulating valve and gas pressure governor to give complete control of heating shall be provided.

5.5.2 Electric heater (see Figure 2), of low heat retention and adjustable from 0 W to 1 000 W.

5.6 Flask-support

5.6.1 Type 1 for use with gas burner (see Figure 1). Either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield shall be used.

Two hard boards, made of ceramic or other heat-resistant material not containing asbestos, 3 mm to 4 mm in thickness, shall rest upon the ring or the platform, whichever is used. The board immediately above the ring or platform shall have a central opening 76 mm to 100 mm in diameter, and outside line dimensions slightly smaller than the inside boundaries of the shield.

The second, or flask-support board, shall be slightly smaller in outside dimensions than the first board and shall have a central opening conforming to the dimensions given in Table 2. It shall be 3 mm to 4 mm in thickness at the central hole rim. The flask-support board may be moved slightly in accordance with the directions for positioning the distillation flask so that direct heat is applied to the flask only through the opening in this board. The position of the flask is set by adjusting the length of the side-arm inserted into the condenser.

5.6.2 Type 2 for use with an electric heater (see Figure 2). The flask-support is a platform on top of the electric heater and adjustable from the outside of the shield. The two hard boards described in 5.6.1 are mounted on this support. Provision shall be made for moving the upper (flask-support) board slightly in the horizontal plane to ensure that direct heat is applied only through the specified opening in this board. The flask-support assembly shall be able to move vertically to ensure contact of the flask-support board with the bottom of the distillation flask during the distillation, and to allow for easy mounting and removal of the distillation flask from the unit.

5.7 Graduated cylinders

5.7.1 Receiving cylinder, of 100 ml capacity, generally in accordance with ISO 4788. It shall be graduated at intervals of 1 ml and have a graduation at the 100 ml mark. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° to the horizontal. Construction details and tolerances for the graduated cylinder are shown in Figure 6.

For automated apparatus, the cylinder shall conform to the physical specifications described in this subclause, with the exception of all graduations but that at 100 ml. Graduated cylinders for use in automated units may also have a metal base.