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Petroleum products — Determination of vapour pressure — Reid method

Produits pétroliers — Détermination de la pression de vapeur — Méthode Reid

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FOREWORD

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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 3007 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	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Israel	Sweden
Canada	Mexico	Thailand
Chile	New Zealand	Turkey
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Germany	Portugal	U.S.S.R.

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

France
Netherlands

Petroleum products — Determination of vapour pressure — Reid method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the absolute vapour pressure (see note 1) of volatile crude oil and volatile non-viscous petroleum products, except liquefied petroleum gases (see note 2).

NOTES

1 Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the "Reid vapour pressure" is approximately the vapour pressure of the material at 37,8 °C in bars¹⁾ absolute. The "Reid vapour pressure" differs from the true vapour pressure of the sample owing to slight vaporization of the sample and the presence of water vapour and air in the confined space.

2 For determination of the vapour pressure of liquefied petroleum gases, reference should be made to ISO

2 REFERENCE

ISO . . . , *Petroleum products — Determination of vapour pressure of liquefied petroleum (LP) gases.*²⁾

3 PRINCIPLE

3.1 The gasoline chamber of the vapour pressure apparatus is filled with the chilled sample and connected to the air chamber at 37,8 °C or other temperature. The apparatus is immersed in a constant-temperature bath (37,8 ± 0,1 °C) and is shaken periodically until equilibrium is reached. The "manometer reading" corresponding to the pressure, read

on the gauge attached to the apparatus, suitably corrected (see table, clause 9) if the air chamber was initially at a temperature other than 37,8 °C, is the Reid vapour pressure.

3.2 This method provides for partial air saturation of products having a Reid vapour pressure below 1,8 bar (clauses 1 to 9, and 17), for no air saturation of products having a Reid vapour pressure above 1,8 bar (clauses 10 to 15, and 17) and for narrower tolerances in certain features for the measurement of the vapour pressure of aviation gasolines (clauses 16 and 17).

4 APPARATUS

The construction of the required apparatus is described in the annex. For samples having vapour pressures below 1,8 bar, use the gasoline chamber with one opening (see A.1.2) and for samples having vapour pressures above 1,8 bar, use the gasoline chamber with two openings (see A.1.3).

5 HANDLING OF SAMPLES

5.1 General

The general provisions in 5.2 to 5.6 shall apply to all samples for vapour pressure determinations, except as specifically excluded for samples having vapour pressures

1). 1 bar = 10⁵ N/m².

2) In preparation.

above 1,8 bar (see clause 10). The extreme sensitivity of vapour pressure measurements to losses through evaporation and to slight changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples.

5.2 Sampling procedure

The Reid vapour pressure determination is extremely sensitive to evaporation losses and to slight changes in the composition of the sample. Careless taking and handling of samples for Reid vapour pressure determinations can easily result in losses of the more volatile components, resulting in significant lowering of the determined vapour pressure. When obtaining, storing, or handling samples, observe all necessary precautions to ensure that the sample is truly representative of the product and is satisfactory for Reid vapour pressure testing.

5.3 Sample container capacity

The capacity of the sample container from which the vapour pressure sample is taken shall be not less than 1 l and not more than 8 l.

5.4 Sample handling temperature

In all cases, the sample container and its contents shall be cooled to a temperature of 0 to 4,5 °C before the container is opened.

5.5 Sample transfer

The Reid vapour pressure determination shall be the first test carried out on a sample. If it is necessary to transfer samples from larger sample containers or to withdraw samples for other tests, the method of transfer shown in figure 1 shall be used.

5.6 Care of samples

Samples shall be put in a cool place as soon as possible after they have been obtained and held there until the test has been completed. Samples in leaking containers shall not be considered for tests but shall be discarded and new samples obtained.

6 PREPARATION FOR TEST

6.1 Air saturation of sample in sample container

With the sample at a temperature of 0 to 4,5 °C, take the container from the water cooling-bath, unseal it, and examine it for its liquid content, which shall be between 70 and 80 % of the container capacity. After the correct liquid content has been assured, reseal the container, shake it vigorously, and return it to the water cooling-bath.

6.2 Preparation of gasoline chamber

Completely immerse the open gasoline chamber and the sample transfer connection in the water cooling-bath for a sufficient time to allow the chamber and connection to reach the bath temperature (0 to 4,5 °C).

6.3 Preparation of air chamber

In the preparation of the air chamber, the 37,8 °C procedure (see 6.3.1) and the ambient temperature procedure (see 6.3.2) give equivalent results on products to which they can be applied.

The ambient temperature procedure is included as an alternative procedure for those laboratories not having a water bath of adequate size to immerse the air chamber.

6.3.1 37,8 °C procedure

After purging and rinsing the air chamber and pressure gauge in accordance with 7.5, connect the gauge to the air chamber. Immerse the air chamber to at least 25 mm above its top in the water bath maintained at $37,8 \pm 0,1$ °C (see note 1 to 7.5), for not less than 10 min just before coupling it to the gasoline chamber. Do not remove the air chamber from the bath until the gasoline chamber has been filled with sample as specified in 7.1.

6.3.2 Ambient temperature procedure

As an alternative to 6.3.1, the air chamber may be adjusted to ambient or other temperature which may be determined with an accuracy of at least 0,5 °C in the following manner: After purging and rinsing the air chamber and pressure gauge in accordance with 7.5, connect the gauge to the air chamber. Insert the thermometer into the chamber, supporting it by means of a loosely fitting (not airtight) stopper in the opening of the air chamber. Adjust the position of the thermometer so that it is aligned as closely as possible with the axis of the air chamber, and with the thermometer bulb located in the air chamber, about 230 mm from the opening. Leave the thermometer in position until the temperature reading has remained constant within 0,5 °C for a period of 5 min or more just before coupling the air chamber to the gasoline chamber. At this time, record the thermometer reading as the "initial air temperature".

7 PROCEDURE

7.1 Sample transfer

With everything in readiness, remove the chilled sample container from the bath, uncap it, and insert the chilled transfer connection and air tube (see figure 1). Quickly empty the chilled gasoline chamber and place it over the sample delivery tube of the transfer connection. Invert the entire system rapidly so that the gasoline chamber is finally in an upright position with the delivery tube extending to within 6 mm of the bottom of the gasoline chamber. Fill the gasoline chamber to overflowing. Lightly tap the gasoline chamber against the work bench to ensure that the sample is free of air bubbles. If any sample is displaced, refill the chamber to overflowing.

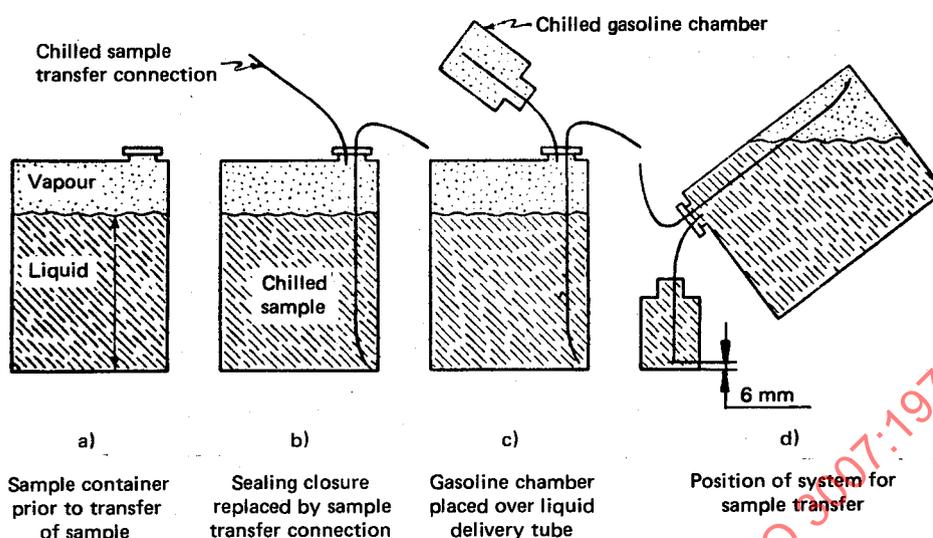


FIGURE 1 — Simplified sketches outlining method of transferring sample to gasoline chamber from open-type containers

7.2 Assembly of apparatus

Without delay, and as quickly as possible, attach the air chamber to the gasoline chamber. Do not take more than 20 s to complete the assembly of the apparatus after filling the gasoline chamber, using the following sequence of operations :

7.2.1 Add additional sample to the gasoline chamber to fill to overflowing.

7.2.2 Read the initial air temperature of the air chamber (see 6.3.2) or remove the air chamber from the $37,8^{\circ}\text{C}$ water bath (see 6.3.1).

7.2.3 Connect the air chamber to the gasoline chamber.

7.3 Introduction of apparatus into bath

Turn the assembled vapour pressure apparatus upside down to allow the sample in the gasoline chamber to run into the air chamber and shake vigorously in a direction parallel to the length of the apparatus. Immerse the assembled apparatus in the bath, maintained at $37,8 \pm 0,1^{\circ}\text{C}$, in an inclined position so that the connection of the gasoline and air chambers is below the water level and may be observed closely for leaks. If no leaks are observed, immerse the apparatus to at least 25 mm above the top of the air chamber. Observe the apparatus for leaks throughout the test. If a leak is detected at any time during the test, the sample shall be discarded and the test recommenced with a fresh sample.

NOTE — Liquid leaks are more difficult to detect than vapour leaks; because the much-used coupling device is normally in the liquid section of the apparatus, give it particular attention.

7.4 Measurement of vapour pressure

After the assembled vapour pressure apparatus has been immersed in the bath for 5 min, tap the pressure gauge lightly, and observe the reading. Withdraw the apparatus from the bath, invert it, shake it vigorously, and replace it in the bath in the shortest possible time to avoid cooling the apparatus. To ensure equilibrium conditions, repeat this agitation and gauge reading at least five times, at intervals of not less than 2 min, until the last two consecutive gauge readings are identical; this sequence of operations normally requires 20 to 30 min. Read the final gauge pressure to the nearest 0,002 5 bar for gauges with intermediate graduations of 0,005 bar and to the nearest 0,005 bar for gauges with graduations of 0,010 to 0,025 bar and record this value as the "uncorrected vapour pressure" of the sample under test. Immediately remove the pressure gauge and check its reading against that of the manometer, recording the value found as the Reid vapour pressure (under the procedure of 6.3.1), or as the "manometer reading" to be used in the calculations of clause 9 (under the procedure of 6.3.2).

7.5 Preparation of apparatus for next test

Disconnect the air chamber, gasoline chamber, and pressure gauge (see note 1). Displace trapped fluid from the Bourdon gauge in the following manner : Hold the gauge between the palms of the hands with the right hand on the face side and the threaded connection of the gauge forward. Extend the arms forward and upward at an angle of 45° with the coupling of the gauge pointing in the same direction. Swing the arms downward through an arc of about 135° so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three

times to expel all liquid. Purge the pressure gauge by directing a small jet of air into its Bourdon tube for at least 5 min.

Thoroughly purge the air chamber of residual sample by filling the air chamber with warm water (above 32 °C) and allowing it to drain (see note 2). Repeat this purging at least five times. After thoroughly removing the previous sample from the gasoline chamber, immerse the chamber in the ice-bath for the next test.

NOTES

1 In the case of crude oil, the Bourdon tube must be washed with a volatile solvent after each test.

2 If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top openings of the chambers closed as they pass through the surface of the water.

8 PRECAUTIONS

Gross errors are liable to be made in vapour pressure measurements if the prescribed procedure is not followed carefully. The following list emphasizes the importance of strict adherence to the precautions given in the procedure :

8.1 Checking the pressure gauge

Check all gauges against a manometer after each test in order to ensure higher precision of results (see 7.4). Ensure that gauges are in a vertical position before reading them.

8.2 Air saturation of sample

Open and close the sample container once after the contents have reached a temperature of 0 to 45 °C. Shake the container vigorously to ensure equilibrium of the sample with the air in the container (see 6.1).

8.3 Checking for leaks

Check all apparatus before and during each test for liquid and vapour leaks (see A.1.6 and note to 7.3).

8.4 Sampling

Because initial sampling and the handling of samples will greatly affect the final results, employ the utmost precaution and the most meticulous care to avoid losses through evaporation and slight changes in composition (see clause 5 and 7.1). In no case shall any part of the Reid apparatus itself be used as the sample container previous to actually conducting the test.

8.5 Purging the apparatus

Thoroughly purge the pressure gauge, the gasoline chamber, and the air chamber to be sure that they are free of residual sample. (This is most conveniently done at the end of the previous test; see 7.5.)

8.6 Coupling the apparatus

Carefully observe the requirements of 7.2.

8.7 Shaking the apparatus

Shake the apparatus "vigorously" as directed in 7.4 in order to ensure equilibrium conditions.

8.8 Temperature control

Carefully control the temperature at the time of air saturation and the temperature of the 37,8 °C bath (see annex, clauses A.3 and A.4, respectively). Be certain that the temperature of the air in the air chamber at the time of coupling with the gasoline chamber (see 7.2) has remained constant within 0,5 °C for a period of 5 min or more.

9 EXPRESSION OF RESULTS

For the ambient temperature procedure specified in 6.3.2, calculate the "Reid vapour pressure" of the sample under test by applying to the "manometer reading" the correction given in the following table for the change in pressure of the water vapour and air in the chamber on heating from the "initial air temperature" to 37,8 °C.

TABLE — Corrections to be applied to "manometer readings" for calculating vapour pressure

Initial air temperature ¹⁾ °C	Barometric pressure ¹⁾ , bar					
	0,800	0,867	0,933	1,000	1,013	1,027
0	-0,17	-0,18	-0,19	-0,20	-0,20	-0,20
5	-0,15	-0,16	-0,17	-0,18	-0,18	-0,18
10	-0,13	-0,14	-0,15	-0,15	-0,15	-0,16
15	-0,11	-0,12	-0,12	-0,13	-0,13	-0,13
20	-0,09	-0,10	-0,10	-0,10	-0,10	-0,10
25	-0,07	-0,07	-0,07	-0,08	-0,08	-0,08
30	-0,04	-0,05	-0,05	-0,05	-0,05	-0,05
35	-0,02	-0,02	-0,02	-0,02	-0,02	-0,02
37,8	0	0	0	0	0	0
40	0,01	0,01	0,01	0,01	0,02	0,02

1) For other temperatures and pressures, the corrections may be calculated by means of the following equation :

$$c = \frac{(p - p_t)(t - 37,8)}{273 + t} - (p_{37,8} - p_t)$$

where

c is the correction, in bars;

p is the barometric pressure, in bars, at the time of the test;

p_t is the vapour pressure of water, in bars, at *t* °C;

t is the initial air chamber temperature, in degrees Celsius, at the beginning of the test;

p_{37,8} is the vapour pressure of water, in bars, at 37,8 °C, namely 0,065 6 bar.

Example : The pressure gauge gives an "uncorrected vapour pressure" reading of 0,689 bar. When the gauge is compared with a mercury manometer, a manometer reading of 0,682 bar is obtained when the pressure gauge reads 0,689 bar. If the initial air chamber temperature is 30 °C, the vapour pressure of water (*p_t*) would be 0,042 bar. If the observed barometric pressure is 0,900 bar, the correction *c* is then calculated to be -0,046 bar. The corrected vapour pressure is then :

$$0,682 - 0,046 = 0,636 \text{ bar}$$

MODIFICATIONS FOR PRODUCTS HAVING REID VAPOUR PRESSURES ABOVE 1,8 bar

10 GENERAL

With products having a vapour pressure over 1,8 bar (see note), the procedure specified in clauses 5 to 8 is hazardous and inaccurate. Consequently, the following clauses define changes in the apparatus and procedure for the determination of vapour pressures above 1,8 bar. Except as specifically stated, all the requirements of clauses 1 to 9 and clause 17 shall apply.

NOTE – When the question arises, the air-saturation method shall be used to determine whether or not a product has a vapour pressure above 1,8 bar.

11 APPARATUS

11.1 Bomb, as described in the annex, using the gasoline chamber with two openings.

11.2 Pressure-gauge calibration

A dead-weight tester (see A.7) may be used in place of the mercury manometer (see A.6) for checking gauge readings above 1,8 bar. In 7.4, 8.1, clause 9 and the table where the words "manometer" and "manometer reading" appear, include as an alternative "dead-weight tester" and "calibrated gauge reading," respectively.

12 HANDLING OF SAMPLES

12.1 The provisions of 5.3, 5.4 and 5.5 shall not apply.

12.2 The capacity of the sample container from which the vapour pressure sample is taken shall be not less than 0,5 l.

13 PREPARATION FOR TEST

13.1 The provisions of 6.1 and 6.2 shall not apply.

13.2 Any safe method of displacement of the test sample from the sample container that ensures filling the gasoline chamber with a chilled, unweathered sample may be employed. Displacement by self-induced pressure is described in 13.3 to 13.5 and clause 14.

13.3 Maintain the sample container at a temperature sufficiently high to maintain superatmospheric pressure, but not substantially over 37,8 °C.

13.4 Completely immerse the gasoline chamber, with both valves open, in the water cooling-bath for a sufficient length of time to allow it to reach the bath temperature (0 to 4,5 °C).

13.5 Connect a suitable ice-cooled coil to the outlet valve of the sample container.

NOTE – A suitable ice-cooled coil can be prepared by immersing a spiral of approximately 8 m of 6 mm copper tubing in a bucket of ice water.

14 PROCEDURE

14.1 The provisions of 7.1 and 7.2 shall not apply.

14.2 Connect the 6 mm valve of the chilled gasoline chamber to the ice-cooled coil. With the 13 mm valve of the gasoline chamber closed, open the outlet valve of the sample container and the 6 mm valve of the gasoline chamber. Open the gasoline chamber 13 mm valve slightly and allow the gasoline chamber to fill slowly. Allow the sample to overflow until the overflow volume is 200 ml or more.

Control this operation so that no appreciable drop in pressure occurs at the gasoline chamber 6 mm valve. In the order named, close the gasoline chamber 13 mm and 6 mm valves; and then close all other valves in the sample system. Disconnect the gasoline chamber and the cooling coil.

Cautions: Safe means for disposal of liquid and vapour escaping during this whole operation must be provided. To avoid rupture because of the liquid-full condition of the gasoline chamber, the gasoline chamber must be quickly attached to the air chamber and the 13 mm valve opened.

14.3 Immediately attach the gasoline chamber to the air chamber and open the gasoline chamber 13 mm valve. Do not take more than 25 s to complete the assembly of the apparatus after filling the gasoline chamber, using the following sequence of operations:

- 1) read the initial air temperature or remove the air chamber from the water bath,
- 2) connect the air chamber to the gasoline chamber, and
- 3) open the gasoline chamber 13 mm valve.

14.4 If a dead-weight tester is used instead of the mercury manometer (see 11.2), apply to the "uncorrected vapour pressure" the calibration factor, in bars, established for the pressure gauge at or near the "uncorrected vapour pressure," recording the value found as the "calibrated gauge reading" to be used throughout clause 9, in place of the "manometer reading".

15 PRECAUTION

The precaution specified in 8.2 shall not apply.

MODIFICATIONS FOR AVIATION GASOLINE OF ABOUT 0,5 bar REID VAPOUR PRESSURE

16 GENERAL

The following paragraphs define changes in apparatus and procedure for the determination of the vapour pressure of aviation gasoline. Except as specifically stated herein, all the requirements set forth in clauses 1 to 9 and clause 17 shall apply.

16.1 Ratio of volumes of air and gasoline chambers

The ratio of the volume of the air chamber to the volume of the gasoline chamber shall be between the limits of 3,95 and 4,05 (see note to A.1 in the annex).

16.2 Water cooling-bath

The water cooling-bath shall be held at a temperature of 0 to 1 °C.

16.3 Checking the pressure gauge

The gauge shall be checked at 0,5 bar against a mercury column before each vapour pressure measurement to ensure that it conforms to the requirements of clause A.2. This preliminary check shall be made in addition to the final gauge comparison specified in 7.4.

16.4 Air chamber temperature

The provisions of 6.3.1 shall be followed; the provisions of 6.3.2 shall not apply.

17 PRECISION

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows :

17.1 Repeatability

The difference between two 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 only in one case in twenty :

Range	Repeatability (Same operator and apparatus)
0 to 0,35 bar	0,007
0,35 to 1,1 bar (except aviation gasoline)	0,014
1,1 to 1,8 bar	0,021
Above 1,8 bar	0,028
Aviation gasoline (approximately 0,5 bar)	0,007

17.2 Reproducibility

The difference between 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 only in one case in twenty :

Range	Reproducibility (Different operator and apparatus)
0 to 0,35 bar	0,024
0,35 to 1,1 bar (except aviation gasoline)	0,021
1,1 to 1,8 bar	0,028
Above 1,8 bar	0,049
Aviation gasoline (approximately 0,5 bar)	0,010

18 TEST REPORT

The test report shall include the following particulars :

- a) a reference to this International Standard;
- b) which procedure has been used (37,8 °C procedure, ambient temperature procedure, modified procedure for products having Reid vapour pressure above 1,8 bar, or modified procedure for aviation gasoline of about 0,5 bar Reid vapour pressure);
- c) the result expressed as the "Reid vapour pressure" to the nearest 0,001 bar;
- d) any deviation, by agreement or otherwise, from the method described;
- e) full identification of the sample.

ANNEX

APPARATUS FOR REID VAPOUR PRESSURE TEST

A.1 REID VAPOUR PRESSURE BOMB

The bomb consists of two chambers – an air chamber (upper section) and a gasoline chamber (lower section) – conforming to the requirements given below.

NOTE – Caution : To maintain the correct volume ratio between the air chamber and the gasoline chamber, the units shall not be interchanged without recalibrating to ascertain that the volume ratio is within satisfactory limits.

A.1.1 Air chamber

The upper section or air chamber, as shown in figure 2, shall be a cylindrical vessel 51 ± 3 mm in diameter and 254 ± 3 mm in length, inside dimensions, with the inner surfaces of the ends slightly sloped to provide complete drainage from either end when held in a vertical position. On one end of the air chamber, a suitable gauge coupling with an internal diameter not less than 5 mm shall be provided to receive the 6 mm gauge connection. In the other end of the air chamber, an opening approximately 13 mm in diameter shall be provided for coupling with the gasoline chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

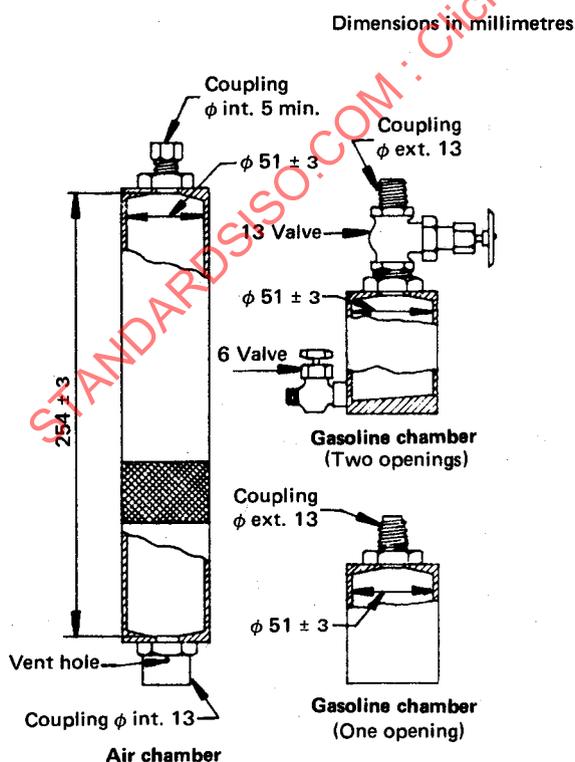


FIGURE 2 – Vapour pressure bomb

A.1.2 Gasoline chamber (one opening)

The lower section or gasoline chamber, as shown in figure 2, shall be a cylindrical vessel of the same inside diameter as the air chamber and of such volume that the ratio of the volume of the air chamber to the volume of the gasoline chamber is between the limits of 3,8 and 4,2 (see note).

In one end of the gasoline chamber, an opening approximately 13 mm in diameter shall be provided for coupling with the air chamber. The inner surface of the end containing the coupling member shall be sloped to provide complete drainage when inverted. The other end of the gasoline chamber shall be completely closed.

NOTE – The ratio for units to be used for aviation gasoline testing shall be 3,95 to 4,05.

A.1.3 Gasoline chamber (two openings)

For sampling from closed vessels, the lower section or gasoline chamber, as shown in figure 2, shall be essentially the same as the gasoline chamber described in A.1.2 except that a 6 mm valve shall be attached near the bottom of the gasoline chamber and a 13 mm straight-through, full-opening valve shall be introduced in the coupling between the chambers. The volume of the gasoline chamber, including only the capacity enclosed by the valves, shall fulfil the volume ratio requirements as set forth in A.1.2.

NOTE – In determining capacities for the two-opening gasoline chamber (figure 2), the capacity of the gasoline chamber shall be considered as that below the 13 mm valve closure. The volume above the 13 mm valve closure including the portion of the coupling permanently attached to the gasoline chamber shall be considered as part of the air chamber capacity.

A.1.4 Method of coupling air and gasoline chambers

Any method of coupling the air and gasoline chambers may be employed, provided that no gasoline is lost during the coupling operation, that no compression effect is caused by the act of coupling, and that the assembly is free from leaks under the conditions of the tests. To avoid displacement of gasoline during assembly, it is desirable that the male fitting of a suitable coupling be on the gasoline chamber. To avoid compression of air during the assembly of a suitable screw coupling, a vent hole may be used to ensure atmospheric pressure in the air chamber at the instant of sealing.

A.1.5 Volumetric capacity of air and gasoline chambers

In order to ascertain if the volume ratio of the chambers is between the specified limits of 3,8 to 4,2 (see note to A.1.2) measure a quantity of water greater than will be