
International Standard



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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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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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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). This method does not apply to fuels containing oxygenated compounds miscible in water.

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 kilopascals (bars)¹⁾ 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 4265.

2 References

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling.*

ISO 4265, *Petroleum products — Determination of vapour pressure of liquefied petroleum (LP) gases.*

3 Principle

3.1 The liquid chamber of the vapour pressure apparatus is filled with the chilled sample and connected to the air chamber at 37,8 °C. The apparatus is immersed in a constant-temperature bath (37,8 ± 0,1 °C) and is shaken periodically until equilibrium is reached. The reading of the pressure gauge corrected for gauge error, or the reading of the mercury manometer, if the pressure is measured by this means, is the Reid vapour pressure.

3.2 This method provides for partial air saturation of products having a Reid vapour pressure below 180 kPa (1,8 bar) (clauses 4 to 9, and 17), for no air saturation of products having

a Reid vapour pressure above 180 kPa (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 annex A. For samples having vapour pressures below 180 kPa (1,8 bar), use the liquid chamber with one opening (see A.1.2) and for samples having vapour pressures above 180 kPa (1,8 bar), use the liquid chamber with two openings (see A.1.3). For samples having Reid vapour pressures below 180 kPa (1,8 bar), a prepressurized mercury manometer (annex B) may be used.

5 Samples and sampling

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 above 180 kPa (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

See annex C for sampling procedure.

5.3 Sample container capacity

The capacity of the sample container from which the vapour pressure sample is taken shall be 1 l. It shall be 70 to 80 % filled with the sample.

5.4 Sample handling temperature

The sample container and its contents shall be cooled to a temperature of 0 to 1 °C before the container is opened.

1) 1 kPa = 1 kN/m² = 0,01 bar

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

Place the sample, in its container, into the water cooling-bath.

With the sample at a temperature of 0 to 1 °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 or an equivalent refrigerator.

6.2 Preparation of liquid chamber

Completely immerse the open liquid chamber and the sample transfer connection in the water cooling-bath or an equivalent refrigerator for a sufficient time to allow the chamber and connection to reach the bath temperature (0 to 1 °C).

6.3 Preparation of air chamber

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 liquid chamber. Do not remove the air chamber from the bath until the liquid chamber has been filled with sample as specified in 7.1.

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 liquid chamber and place it over the sample delivery tube of the transfer connection. Invert the entire system rapidly so that the liquid chamber is finally in an upright position with the delivery tube extending to within 6 mm of the bottom of the liquid chamber. Fill the liquid chamber to overflowing. Lightly tap the liquid 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.

7.2 Assembly of apparatus

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

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

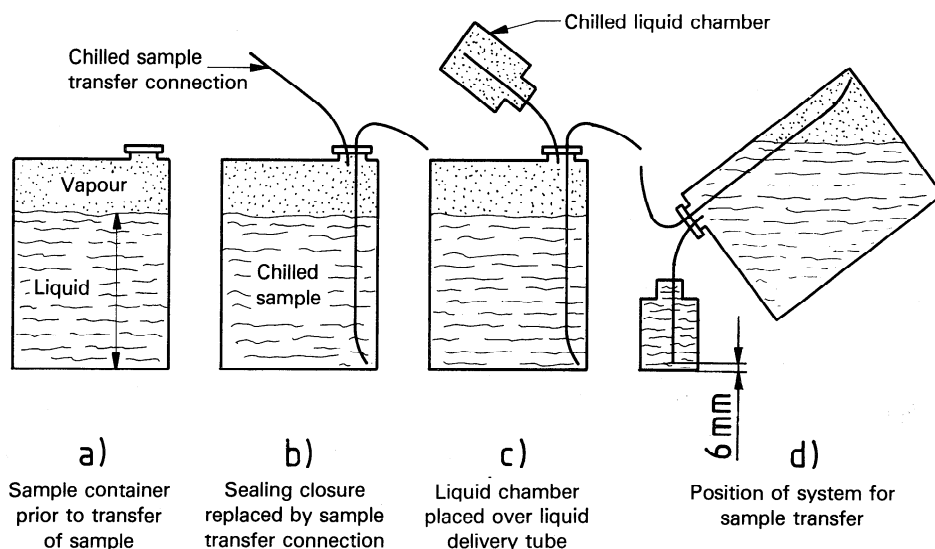


Figure 1 — Simplified sketches outlining method of transferring sample to liquid chamber from open-type containers

7.2.2 Remove the air chamber from the 37,8 °C water bath (see 6.3).

7.2.3 Connect the air chamber to the liquid chamber.

7.2.4 If a prepressurized mercury manometer is used, check the needle valve to be sure it is closed and connect the manometer hose to the coupling at the top of the air chamber.

7.3 Introduction of apparatus into bath

Turn the assembled vapour pressure apparatus upside down to allow the sample in the liquid 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 $37,8 \pm 0,1$ °C, in an inclined position so that the connection of the liquid 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, and 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 0,25 kPa (0,002 5 bar) for gauges with intermediate graduations of 0,5 kPa (0,005 bar) and to the nearest 0,5 kPa (0,005 bar) for gauges with graduations of 1,0 to 2,5 kPa (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.

7.5 Preparation of apparatus for next test

Disconnect the air chamber, liquid 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 liquid chamber, immerse the chamber in the ice-bath for the next test.

NOTES

1 In the case of crude oil, it is necessary to wash all the equipment with a volatile solvent, preferably toluene, 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.

7.6 Using a mercury manometer to measure the vapour pressure of products having a Reid vapour pressure less than 180 kPa (1,8 bar)

7.6.1 Sample transfer

Transfer the sample as specified in 7.1.

7.6.2 Assembly of apparatus

Assemble the apparatus as specified in 7.2, check that the needle valve on the air chamber is tightly closed, attach the manometer hose to the top of the air chamber and follow the sequence of operations in 7.2.

7.6.3 Introduction of apparatus into bath

Introduce the apparatus into the bath as specified in 7.3.

7.6.4 Prepressurizing the manometer

After the assembled vapour pressure apparatus has been immersed in the bath and checked for leaks as specified in 7.3, prepressurize the manometer and flexible hose to the expected vapour pressure of the sample (see the note), and record this value as the "initial manometer setting".

While the sample is being brought to equilibrium as specified in 7.6.5, observe the manometer occasionally to check for leaks in the manometer assembly. Any change in the "initial manometer setting" indicates a leak, in which case the vapour pressure apparatus should be detached and connected to an alternative manometer.

NOTE — For purposes of pressurizing and to avoid the necessity for re-running determinations, knowledge of the expected vapour pressure is very helpful. An indication of the expected vapour pressure level should be given, where possible, on the sample identification label. It is also helpful to maintain a list of current vapour pressures of samples being tested on a routine basis.

7.6.5 Measurement of vapour pressure

After the apparatus has been immersed in the bath for 5 min, and if no leaks have been observed, carefully withdraw the apparatus from the bath. Without opening the valve, invert the

apparatus, shake it vigorously along its length and replace in the bath in the shortest possible time to avoid cooling the apparatus.

Repeat this withdrawal and shaking operation after a further 5 min, again replacing the apparatus in the bath in the shortest possible time. After not less than 2 min, open the valve and read and record the manometer reading. Close the valve, withdraw the apparatus from the bath and repeat the agitation, the immersion, and the reading of the manometer at intervals of not less than 2 min until two consecutive manometer readings are constant to ensure that equilibrium has been attained. These operations normally require 20 to 30 min.

Read the final manometer pressure to the nearest 1 kPa (0,01 bar) and record the value as the "constant manometer reading" of the sample under test.

7.6.6 Appraisal of observations

For extremely accurate results, the "constant manometer reading" shall be within 10 kPa (0,1 bar) of the "initial manometer setting". If the difference is less than this amount, proceed as specified in clause 9. If the difference is greater, make a second determination using the first result as an approximation for prepressurizing the manometer. Repeat until the difference is within the prescribed limits.

7.6.7 Preparation of apparatus for next test

Disconnect the manometer hose, air chamber, and liquid chamber. Remove the connector and valve assembly from the air chamber and, with the valve open, purge with air for at least 5 min. Purge the air chamber by means of a jet of warm water for at least 1 min, or by filling with warm water and draining at least five times. After removing the previous sample from the liquid chamber, purge the chamber with cold water and immerse in the water cooling-bath for the next test.

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 1 °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 liquid 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

Ensure that the temperatures of the water cooling-bath (A.3), the baths and the water bath (A.4) are correct whenever the baths are required (see clause 6, and 7.2 and 7.3).

9 Expression of results

Report to the nearest 0,25 kPa (0,002 5 bar) or 0,5 kPa (0,005 bar) the final value recorded in 7.4 or 7.6.5, as the "Reid vapour pressure", in kilopascals (bars), without reference to temperature.

10 Modifications for products having Reid vapour pressures above 180 kPa (1,8 bar)

With products having a vapour pressure over 180 kPa (1,8 bar) (see the 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 180 kPa (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 180 kPa (1,8 bar).

11 Apparatus

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

11.2 Pressure-gauge calibration

A dead-weight tester (see clause A.7) may be used in place of the mercury manometer (see clause A.6) for checking gauge readings above 180 kPa (1,8 bar). In 7.4, 8.1 and clause 9 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 liquid 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 liquid 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 1 °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 800 mm 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 liquid chamber to the ice-cooled coil. With the 13 mm valve of the liquid chamber closed, open the outlet valve of the sample container and the 6 mm valve of the liquid chamber. Open the liquid chamber 13 mm valve slightly and allow the liquid 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 liquid chamber 6 mm valve. In the order named, close the liquid chamber 13 mm and 6 mm valves; and then close all other valves in the sample system. Disconnect the liquid chamber and the cooling coil.

Caution: 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 liquid chamber, the liquid chamber must be quickly attached to the air chamber and the 13 mm valve opened.

14.3 Immediately attach the liquid chamber to the air chamber and open the liquid chamber 13 mm valve. Do not take more than 25 s to complete the assembly of the apparatus after filling the liquid 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 liquid chamber, and
- 3) open the liquid 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 kilopascals (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.

16 Modifications for aviation gasoline of about 50 kPa (0,5 bar) Reid vapour pressure

16.1 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.

16.2 Ratio of volumes of air and liquid chambers

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

16.3 Water cooling-bath

The water cooling-bath shall be held at a temperature of 0 to 1 °C (see clause A.3).

16.4 Checking the pressure gauge

The gauge shall be checked at 50 kPa (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.

16.5 Air chamber temperature

The provisions of 6.3 shall be followed.

17 Expression of results

17.1 Calculation

See clause 9.

17.2 Precision

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

17.2.1 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 values shown in the table below only in one case in 20.

Range, kPa (bar)	Repeatability (same operator and apparatus)
35 to 110 kPa (0,35 to 1,10 bar)	2,1 kPa

17.2.2 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 values shown in the table below only in one case in 20.

Range, kPa (bar)	Reproducibility (different operator and apparatus)
35 to 110 kPa (0,35 to 1,10 bar)	4,9 kPa

NOTE — The precision data above were developed in a 1981 co-operative testing programme involving 25 laboratories and 12 samples in duplicate covering the range of 5 to 16 psi Reid vapour pressure. The previous precision statement for other vapour pressure ranges was developed in the early 1950s, and is as follows:

Range, kPa (bar)	Repeatability
0 to 35 (0 to 0,35)	0,7
110 to 180 (1,10 to 1,80)	2,1
180 and above (1,80 and above)	2,8
Aviation gasoline (50 kPa) (0,5 bar)	0,7

Range, kPa (bar)	Reproducibility
0 to 35 (0 to 0,35)	2,4
110 to 180 (1,10 to 1,80)	2,8
180 and above (1,80 and above)	4,9
Aviation gasoline (50 kPa) (0,5 bar)	1,0

18 Test report

The test report shall contain at least the following information:

- a) the type and identification of the product tested;
- b) a reference to this International Standard;
- c) the result of the test;
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

Annex A

Apparatus for Reid vapour pressure test

(This annex forms an integral part of the Standard.)

A.1 Reid vapour pressure bomb

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

NOTE — **Caution:** To maintain the correct volume ratio between the air chamber and the liquid 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 Rp 1/4 gauge connection. In the other end of the air chamber, an opening approximately 13 mm in diameter shall be provided for coupling with the liquid chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

A.1.2 Liquid chamber (one opening)

The lower section or liquid 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 liquid chamber is between the limits of 3,95 to 4,05.

In one end of the liquid 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 liquid chamber shall be completely closed.

A.1.3 Liquid chamber (two openings)

For sampling from closed vessels, the lower section or liquid chamber, as shown in figure 2, shall be essentially the same as the liquid chamber described in A.1.2 except that a 6 mm valve shall be attached near the bottom of the liquid chamber and a 13 mm straight-through, full-opening valve shall be introduced in the coupling between the chambers. The volume of the liquid 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 liquid chamber (figure 2), the capacity of the liquid 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 liquid chamber shall be considered as part of the air chamber capacity.

A.1.4 Method of coupling air and liquid chambers

Any method of coupling the air and liquid chambers may be employed, provided that no liquid 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 liquid during assembly, it is desirable that the male fitting of a suitable coupling be on the liquid 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.

Caution: Some commercially available equipment does not make adequate provision for avoiding air compression effects. Before employing any apparatus, it shall be established that the act of coupling does not compress the air in the air chamber. This may be accomplished by tightly stoppering the liquid chamber opening and assembling the apparatus in the normal manner, utilizing the 0 to 35 kPa (0,35 bar) gauge. Any observable pressure increase on the gauge is an indication that the apparatus does not adequately meet the specifications of the method. If this problem is encountered, the manufacturer should be consulted for remedy.

A.1.5 Volumetric capacity of air and liquid chambers

In order to ascertain if the volume ratio of the chambers is between the specified limits of 3,95 to 4,05, measure a quantity of water greater than will be needed to fill the liquid and air chambers. The liquid chamber shall be completely filled with water, and the difference between the original volume and the remaining volume is the volume of the liquid chamber. Then, after connecting the liquid and air chambers, the air chamber shall be filled to the seat of the gauge connection with more of the measured water, and the difference in volumes shall be the volume of the air chamber.

A.1.6 Checking for freedom from leaks

Before placing new apparatus in service and as often as necessary thereafter, the assembled vapour pressure apparatus shall be checked for freedom from leaks by filling with air to 700 kPa (7 bar) gauge pressure and completely immersing in a water bath. Only apparatus which stands this test without leaking shall be used.