



Standard Test Method for Solubility of Fixed Gases in Liquids¹

This standard is issued under the fixed designation D 2780; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the solubility of fixed gases in liquids. It is suitable for gases and liquids that do not react with each other and are compatible with borosilicate glass, mercury, stainless steel, PTFE (polytetrafluoroethylene), and FPM (vinylidene fluoride-hexafluoro propylene copolymer) under the conditions of the test. This test method also covers the determination of the concentration of fixed gases in solutions which are not saturated with the gas.

1.2 *This standard does not purport to address all of the safety concerns, if any, 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.* For specific hazard statements see 6.1, 6.2, 8.3, 8.4.2, and 9.3.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*²

D 831 Test Method for Gas Content of Cable and Capacitor Oils

D 2883 Test Method for Reaction Threshold Temperature of Liquid and Solid Materials

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E 260 Practice for Packed Column Gas Chromatography

3. Summary of Test Method

3.1 A specimen of the test liquid is saturated with a fixed gas under specified conditions of temperature and pressure. The saturation step may be eliminated if it is desired to determine the concentration of fixed gas in a liquid sample suitably

presented for analysis. A portion of the solution of gas in liquid is transferred to a gas extraction apparatus in which the fixed gas is quantitatively removed from the liquid. The separated gas is transferred to a gas buret in which its volume is determined.

4. Significance and Use

4.1 The solubility of fixed gases in liquids is an important engineering parameter in the design of hydraulic systems. It is a measure of the amount of gas which can be released from solution when a system undergoes changes in pressure and temperature. Theoretical considerations permit approximate values of gas solubility to be computed with reasonable accuracy. In this test method, dissolved gases are separated physically from a liquid and measured volumetrically. The test method permits subsequent analysis of separated gases by any appropriate method.

5. Apparatus

5.1 *Ambient Pressure Saturator*, suitable for the saturation of liquids with fixed gases at various temperatures at ambient pressure is shown in Fig. 1. The system comprises four parts:

5.1.1 *Gas Supply and Pressure Regulator*,

5.1.2 *Gas Dispersion Element*,

5.1.3 *Heating Mantle*, to fit 1000-mL separatory funnel (Fig. 1), and

5.1.4 *Temperature Measurement and Control Devices*.

NOTE 1—In the event that it is desired to saturate a liquid with a toxic or flammable gas, the use of this system is not recommended, unless suitable means are provided for the collection and disposal of the escaping gas.

5.2 *Elevated Pressure Saturator*, used to saturate liquids with gases at pressures other than ambient. A suitable vessel, usable at pressures up to 608 kPa (6 atm), is illustrated in Fig. 2. The vessel consists of a 2.5 L stainless steel bomb with a thermostatic control jacket. A valve at one end is connected to a pressure gage and gas supply. A valve at the other end is provided with a fitting that connects directly to the gas extraction apparatus.

5.2.1 *Thermostatic Control*, for jacket of saturator.

5.2.2 *Shaker*, reciprocating, horizontal.

5.2.3 *Vacuum Pump*, rotary.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

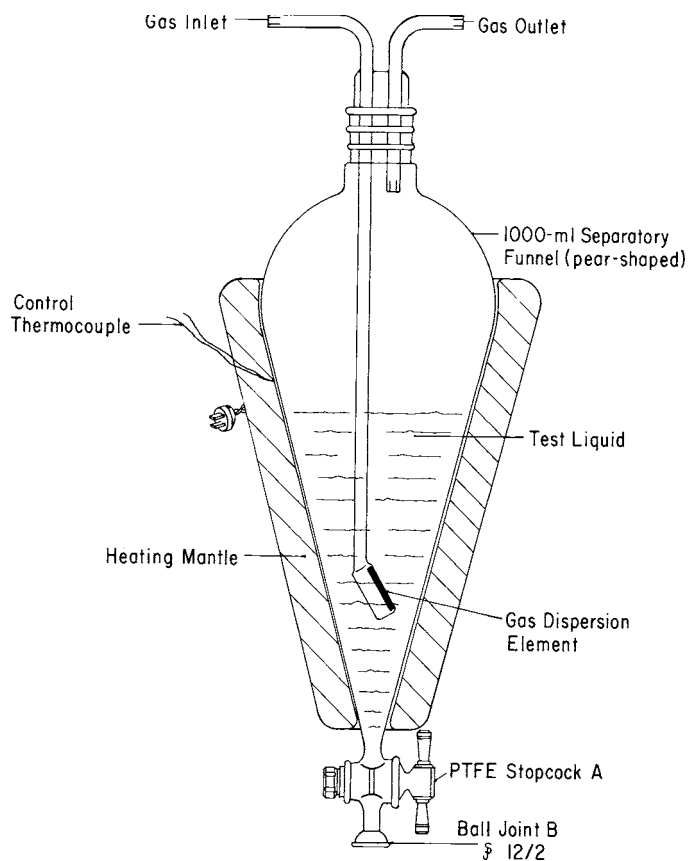


FIG. 1 Ambient Pressure Saturator

5.2.4 *Transfer Line*, with two male socket joint 12/2 fittings.

5.3 *Gas Extraction System*,³ as shown schematically in Fig. 4.

3. A detailed drawing of the extraction chamber is shown in Fig. 4. The apparatus provides for the separation of dissolved gases from a liquid by repeatedly forcing the liquid containing gas to pass through a narrow annular passage under reduced pressure. Gas removed in this manner is stored and measured in a gas buret. Provision is made for heating the extraction chamber by means of a condensing vapor bath. The gas buret is jacketed. Cooling water may be circulated through the jacket if it is necessary to reduce the temperature of the contents of the buret. A manometer is attached to the manifold connecting the saturation system, gas extractor, and gas buret. Grease-free stopcocks and ball joints are used throughout the system (Note 2). All tubing and connections are 1 mm inside diameter.

NOTE 2—PTFE stopcocks are satisfactory for most purposes. However, for greatest precision construct the apparatus with stopcocks and joints which are fitted with O-ring seals.

6. Reagents and Materials

6.1 *Mercury*, triple-distilled, instrument-grade, sufficient amount to fill extraction apparatus, gas buret, and leveling bulbs. (Warning—Poison can be harmful if inhaled or swallowed. Vapor harmful, emits toxic fumes when heated. Vapor

pressure at normal room temperature exceeds threshold limit value for occupational exposure. See A1.1.)

6.2 *Compressed Gases*, as required for saturating liquids to be studied. (Warning—Compressed gas under high pressure. Gas reduces oxygen available for breathing. See A1.2.)

7. Sampling

7.1 To obtain specimens for total gas solubility measurements, collect samples in accordance with Practice D 4057. For the determination of the concentration of fixed gases in solutions which are not saturated with the gas, take samples in accordance with the procedure described in Section 3 of Test Method D 831.

8. Procedure A

8.1 Procedure A covers the determination of the solubility of fixed gases in liquids at ambient pressure.

8.2 Add to the ambient pressure saturator (Fig. 1) a sufficient amount of the liquid to cover the gas dispersion element with at least 50 to 80 mm of liquid. Bring the cell to temperature equilibrium at whatever temperature is desired for the determination.

8.3 Saturate the liquid with the test gas (Warning—See 6.2) by bubbling the gas through the liquid. Adjust the gas flow rate so that the gas stream causes thorough but not violent agitation of the liquid. If saturation is to be carried out at an elevated temperature, it may be necessary to reestablish temperature equilibrium after the start of gas flow. (Warning—Be certain

³ The gas extraction system is similar to that described by J. H. D. Hooper, API Proceedings, 1948.

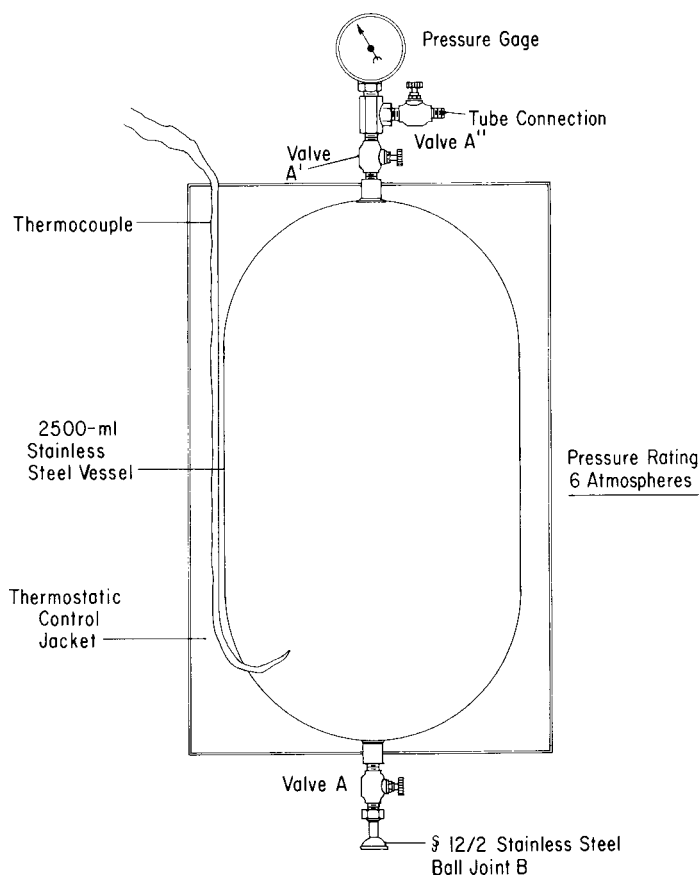


FIG. 2 Elevated Pressure Saturator

that the test gas does not react with the sample under the conditions of the test. For example, oxygen may react with certain unsaturated hydrocarbons even at room temperature.)

NOTE 3—To saturate liquids at higher temperatures, it may be necessary to preheat the saturating gas to avoid cooling the liquid. It may also be necessary to provide the saturating cell with a reflux capability if it is desired to study volatile liquids at elevated temperatures.

8.4 Attach the saturator containing the saturated liquid to the assembled gas extraction system (Fig. 3). Maintain temperature equilibrium of the saturator continuously in order to prevent any change in gas content. Evacuate the system in the following manner:

8.4.1 With stopcock *A* closed, adjust stopcocks *L*, *M*, and *N* to vent the gas extraction chamber to the atmosphere through *S*.

8.4.2 Fill the gas extraction chamber with mercury by raising leveling bulb *H* until mercury level reaches stopcock *L*. (**Warning**—Poison can be harmful if inhaled or swallowed. Vapor harmful, emits toxic fumes when heated. Vapor pressure at normal room temperature exceeds threshold limit value for occupational exposure. See A1.1.)

8.4.3 Turn stopcock *L* to connect the saturator to manifold.

8.4.4 Adjust stopcock *M* to connect the gas buret *P* to the atmosphere through *S*.

8.4.5 Fill the gas buret with mercury by raising leveling bulb *Q* until mercury level reaches stopcock *M*.

8.4.6 Turn stopcock *M* to connect manifold to stopcocks *L* and *N*.

8.4.7 Adjust stopcock *N* to connect the manometer *R* to the atmosphere through *S*.

8.4.8 Attach the vacuum pump to *S* and evacuate the manometer.

8.4.9 Turn stopcock *N* to connect *S* with manifold and evacuate the manifold up to stopcock *A* on the gas saturator.

8.4.10 Close stopcock *N* and disconnect vacuum pump.

8.5 The following procedure is used to determine the volume of the dissolved gases present in a measured quantity of a liquid suitably presented for analysis (Note 3) or saturated in the manner prescribed in 8.3.

NOTE 4—An excellent procedure for presentation of such samples is given in Section 3 of Test Method D 831.

8.5.1 Lower leveling bulb *H* until mercury level in the gas extraction chamber falls well below the glass umbrella *F*.

8.5.2 Adjust stopcock *L* to connect the gas extraction chamber to the saturator through ball joint *B* and stopcock *A*.

8.5.3 Open stopcock *A* and permit approximately 50 mL of the sample liquid to flow into the gas extraction chamber.

8.5.4 Close stopcock *A*.

8.5.5 As the liquid flows into the gas extraction chamber some dissolved gas immediately breaks out of solution. To transfer this gas to the buret turn stopcocks *L* and *M* so as to connect the gas extraction chamber to the buret, raise leveling bulb *H* and lower leveling bulb *Q*.

8.5.5.1 Care shall be taken during the gas transfer process to avoid mercury surges, which can force the sample liquid into manifold or gas buret *P*.

8.5.6 Close stopcock *L*. Lower leveling bulb *H* until the sample liquid is drawn in a thin film through the annular space *E* between the umbrella *F* and the wall *G* of the gas extraction chamber.

8.5.7 Raise the leveling bulb *H* to force the liquid upward through check valve *D*.

8.5.8 Repeat 8.5.6 and 8.5.7 several times, and then transfer the liberated gas to the gas buret as described in 8.5.5.

8.5.9 Measure the volume and pressure of the gas collected in the buret with stopcocks *M* and *N* turned to connect the buret to the manometer *R*. For this measurement it is convenient to adjust the pressure as read on manometer *R* in the buret to 1 atm with leveling bulb *Q*.

8.5.10 Repeat 8.5.5-8.5.9 until the volume of gas collected is constant.

8.5.11 Close stopcock *L* and disconnect the extraction chamber from the rest of the system at ball joint *C*. Allow the extraction chamber to cool if necessary.

NOTE 5—For studies of the solubility of gases in viscous liquids it may be necessary to heat the gas extraction chamber in order to achieve proper operating conditions. A condensing vapor jacket *J* together with boiler *I* and refluxing condenser *K* are provided for this purpose. Water is a suitable reflux liquid. In most cases, however, no additional heating is necessary.

8.5.12 Tilt the chamber slightly and carefully raise leveling bulb *H* to force the liquid sample out of the chamber into beaker.

8.5.13 Lower leveling bulb *H* and allow any excess sample to drain from the chamber walls.

8.5.14 Repeat 8.5.12.

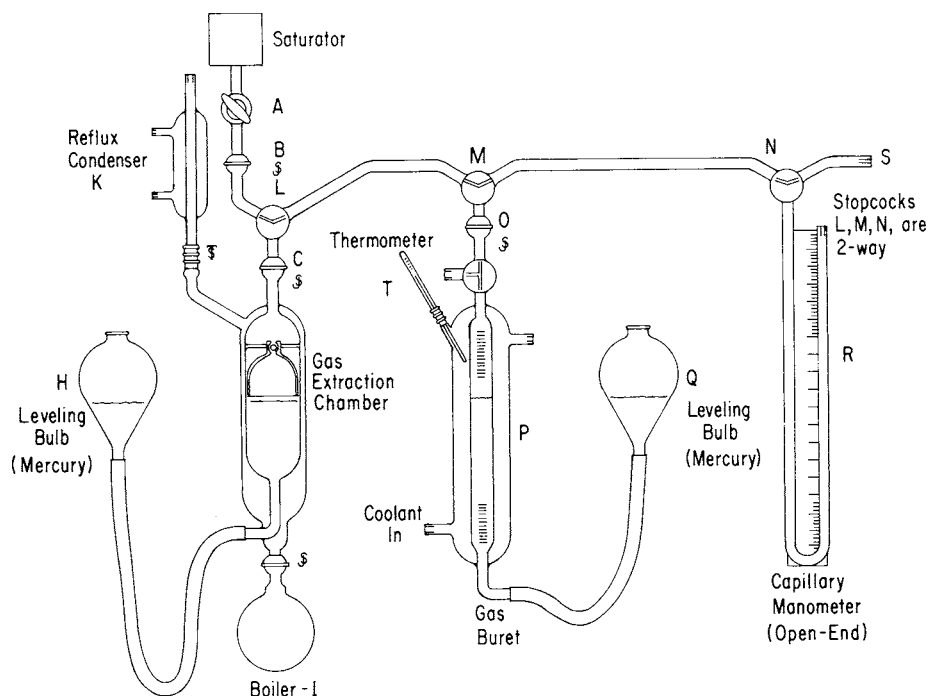


FIG. 3 Gas Extraction System

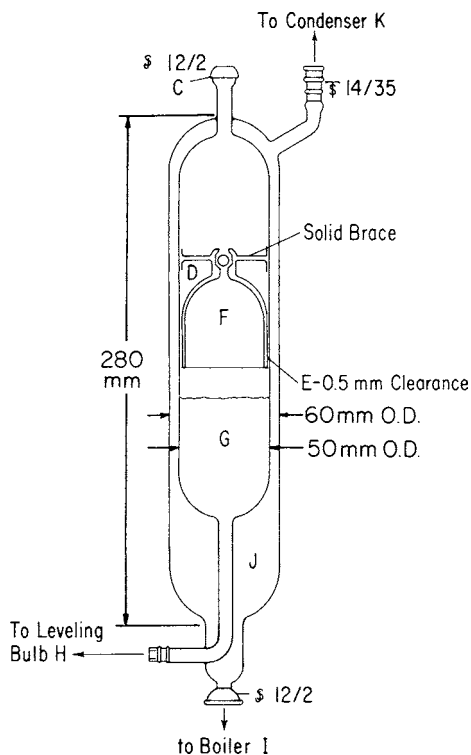


FIG. 4 Gas Extraction Chamber

NOTE 6—When testing the solubility of gas mixtures, such as air, analysis of the separated gas for the individual constituents by gas chromatography or mass spectroscopy may be desirable to determine the actual percentage of each dissolved gas in the liquid; see Practice E 260.

9. Procedure B

9.1 Procedure B covers the determination of the solubility of fixed gases in liquids at elevated pressure.

9.2 Place in the elevated pressure saturator (Fig. 2) 300 to 500 mL of the sample liquid that has been saturated with the test gas (Warning—See 6.2) in the ambient pressure saturator (Fig. 1) in the manner described in 8.3 to free it from dissolved air. The transfer line, described in 5.2.4, is used. If flowing the liquid by gravity is too slow, evacuate the elevated pressure saturator partially prior to starting the transfer, and close valve A. Bring the saturator to temperature equilibrium at the temperature desired for the determination.

NOTE 7—Should solubility measurements at subambient temperatures be desired, the saturation cell can be modified by the addition of a jacket through which a suitably thermostated cooling fluid may be circulated.

9.3 Charge the saturation cell with the test gas at the desired pressure through valves A' and A'' with valve A closed. Then close valves A' and A'' and shake the cell for 5 min. Recharge the cell with gas to restore pressure after the initial saturation step. Shake again. Repeat the process of charging and shaking until constancy of pressure indicates that the liquid has been completely saturated. Allow the sample to stand at constant pressure and temperature for at least 30 min to permit the release of entrained bubbles. (Warning—At elevated pressures the reactivity of samples with gases is greatly enhanced. Some samples in contact with oxygen or air may form mixtures capable of autoignition at room temperature or below. Do not attempt the use of oxygen at elevated pressures under any circumstances. Studies with air under moderate pressure may

8.5.15 Weigh the collected sample.

8.5.15.1 For studies of the solubility of gases in volatile liquids, provision must be made for that portion of the collected gas which consists of organic vapors. In some cases these can be reduced to a negligible amount by cooling the gas buret by passing a cold fluid through its liquid jacket. If this is not adequate, use the alternative procedure described in 10.3.1.

be attempted provided that the autoignition temperature (reaction threshold temperature) of the sample in the presence of air at the test pressure is not exceeded. See Test Method **D 2883**.)

9.4 Attach the saturator containing the saturated liquid to the assembled gas extraction system. Evacuate the gas extraction system by the procedure described in **8.4**. Then extract the gas from a measured portion of the sample by the procedure described in **8.5**.

10. Calculation

10.1 Calculate the volume of gas dissolved in the sample at the specified temperature and pressure of test in the following manner:

$$V_{\text{ost}} = V_B \times (P_B/P_O) \times [(T_o + 273.2)/(T_B + 273.2)] \quad (1)$$

where:

V_{ost} = volume of extracted gas at temperature and pressure of test, mL,

V_B = volume of gas in gas buret, mL,

P_B = pressure of gas in gas buret, Pa (or torr),

P_O = test pressure, Pa (or torr),

T_B = temperature of gas in gas buret, ° C, and

T_o = test temperature, ° C.

10.2 Calculate the volume of the liquid sample at the test temperature as follows:

$$V_L = \text{weight liquid}/d_T \quad (2)$$

where:

V_L = volume liquid at the test temperature, mL, and

d_T = density of liquid at the test temperature, g/mL.

10.3 Calculate the gas solubility, V_c as follows, reporting the result as the Ostwald coefficient:

$$V_c = V_{\text{ost}}/V_L \quad (3)$$

10.3.1 If the gas dissolved in the sample contains more than one component or if the gas contains an appreciable fraction of vapors from the liquid sample, connect the calculation of the Ostwald coefficient given in **10.3** by multiplying the calculated coefficient by the mol fraction of the desired component in the extracted gas. Thus:

$$V_{c1} = V_c \times N_1, V_{c2} = V_c \times N_2, \dots, V_{ci} = V_c \times N_i \quad (4)$$

where:

$V_{c1} \dots V_{ci}$ = Ostwald coefficients of components 1 through i , and

$N_1 \dots N_i$ = mole fraction of components 1 through i .

10.4 Alternatively, the gas solubility may be calculated as a Bunsen coefficient, B . In order to do this it is necessary to determine the volume of extracted gas at STP.

$$V_{\text{STP}} = V_B \times (P_B/760) \times [273.2/(T_B + 273.2)] \quad (5)$$

where:

V_{STP} = volume of extracted gas at STP, mL,

V_B = volume of gas in gas buret, mL,

P_B = pressure of gas in gas buret, torr, and

T_B = temperature of gas in gas buret, °C.

10.5 Calculate the volume of the liquid sample at 288.7 K (15.5°C) (60°F) as follows:

$$V_{15.5} = \text{weight liquid}/d_{15.5} \quad (6)$$

where:

V = volume of liquid at 288.7 K (15.5°C) (60°F), and

$d_{15.5}$ = density and liquid at 288.7 K (15.5°C) (60°F).

10.6 Calculate the Bunsen coefficient, B , as follows:

$$B = V_{\text{STP}}/V_{15.5} \quad (7)$$

10.6.1 If the gas dissolved in the sample contains more than one component or if the gas contains an appreciable fraction of vapors from the liquid sample, the calculation of the Bunsen coefficient given in **10.6** must be corrected by multiplying the calculated coefficient by the mole fraction of the desired component in the extracted gas. Thus:

$$B_1 = B \times N_1, B_2 = B \times N_2, \dots, B_i = B \times N_i \quad (8)$$

where:

$B_1 \dots B_i$ = corrected Bunsen coefficients of components 1 through i , and

$N_1 \dots N_i$ = mole fractions of components 1 through i .

11. Report

11.1 Report the solubility of a gas in a liquid sample expressed as the Ostwald coefficient. Indicate the temperature and pressure at which the liquid was saturated with the gas.

11.2 If the method has been used to determine the concentration of gas in a gas-liquid solution which is not saturated, indicate that fact, and report the Ostwald coefficient.

11.3 If the solubility of concentration of a gas in an unsaturated solution is to be reported as a Bunsen coefficient, that fact should be clearly stated in the report.

12. Precision and Bias

12.1 Because of the complex nature of Test Method D 2780 for solubility of fixed gases in liquids, there is not a sufficient number of volunteers to permit a cooperative laboratory program for determining the precision and bias. If the necessary volunteers can be obtained, a program will be undertaken at a later date.

13. Keywords

13.1 gases; liquids; solubility

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Mercury

Warning—Poison. Can be harmful or fatal if inhaled or swallowed. Vapor harmful; emits toxic fumes when heated. Vapor pressure at normal room temperature exceeds threshold limit value for occupational exposure.

Do not breathe vapor.

Keep container closed.

Use with adequate ventilation.

Do not take internally.

Cover exposed surfaces with water, if possible, to minimize evaporation.

Do not heat.

Keep recovered mercury in tightly sealed container prior to sale or purification.

Do not discard in sink or in rubbish.

A1.2 Compressed Gases

Warning—Compressed gas under high pressure. Gas reduces oxygen available for breathing.

Keep cylinder valve closed when not in use.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received.

Do not mix gases in cylinders.

Never drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinders.

For technical use only. Do not use for inhalation purposes.

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