



Standard Test Method for Ammonia Colorimetric Leak Testing¹

This standard is issued under the fixed designation E 1066; 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 reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the testing of large single- and double-walled tanks, pressure and vacuum vessels, laminated, lined- or double-walled parts, complex piping systems, flexible containers (such as aircraft fuel tanks), glass-to-metal seals in hybrid packages, and systems that inherently contain or will contain ammonia (such as large tonnage refrigeration systems and fertilizer storage systems).

1.2 This method can be used on piping, valves and containers with welded, fitted, or laminated sections that can be sealed at their ends or between their outer and inner walls and that are designed for internal pressures of 34.5 kPa (5 psig) or greater.

1.3 Basic procedures are described based on the type of inspection used. These procedures should be limited to finding leakage indications of 4.5×10^{-12} mol/s (1×10^{-7} Std cm³/s)² or larger.

1.4 *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 more specific safety precautionary information see 7.4, 8.2, 9.4.1, and 10.3.1).

2. Referenced Documents

2.1 ASTM Standards:

E 1002 Test Method for Leaks Using Ultrasonics³

E 1316 Terminology for Nondestructive Examinations³

2.2 Other Documents:

SNT-TC-1A Recommended Practice for Personnel Qualification and Certification in Nondestructive Testing⁴

ANSI/ASNT CP-189 ASNT Standard for Qualification and Certification of Nondestructive Testing Personnel⁴

¹ This test method is under the jurisdiction of ASTM Committee E-7 on Nondestructive Testing and is the direct responsibility of Subcommittee E 07.08 on Leak Testing.

Current edition approved Sept. 10, 1995. Published November 1995. Originally published as E 1066 – 85. Last previous edition E 1066 – 94.

² The gas temperature is referenced to 0°C. To convert to another gas reference temperature, T_{ref} , multiply the leak rate by $(T_{ref} + 273)/273$.

³ *Annual Book of ASTM Standards*, Vol 03.03.

⁴ Available from American Society for Nondestructive Testing, 1711 Arlington Plaza, P.O. Box 28518, Columbus, OH 43228-0518.

3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, see Terminology E 1316, Section E.

4. Summary of Test Method

4.1 This test method consists of testing a container already filled with ammonia or of introducing an anhydrous ammonia or an ammonia-nitrogen mixture into a container or system so that the final ammonia percentage achieved is between 1 and 100 % by volume at a gage pressure between 34.5 and 689.5 kPa (5 and 100 psig). The ammonia flows through leaks existing in welds and connections and reacts with a developer that is applied outside of the container producing a visible indication.

4.2 Two basic developer procedures are described:

4.2.1 Smoke-producing developers.

4.2.2 Color-change developers.

4.3 Methods of introducing ammonia into unfilled systems are described, together with methods of estimating the concentration and pressure needed to achieve specific detectable leak rates.

4.4 Procedures for testing large tanks and systems are described.

4.5 Ultrasonic pretesting for gross leaks is described.

5. Personnel Qualification

5.1 It is recommended that personnel performing leak testing attend a dedicated training course on the subject and pass a written examination. The training course should be appropriate for NDT level II qualification according to Recommended Practice No. SNT-TC-1A of the American Society for Nondestructive Testing or ANSI/ASNT Standard CP-189.

6. Significance and Use

6.1 This method is useful for locating and measuring the size of gas leaks either as a quality-control test or as a field-inspection procedure. It can be used to test critical parts or containers that will hold toxic or explosive gases or liquids or as a quick test for other containers.

7. Interferences

7.1 The interior and exterior welds and joints where leaks are often found must be free of oil, grease, flux, slag, paint, or other contaminants that might temporarily block or mask leakage. New containers should not be painted prior to test. Smoking during the test may cause false indications.

7.2 Pure ammonia gas is likely to attack brass or copper metals in a humid environment. It will not if there is no water vapor present.

7.3 Ammonia gas attacks wood fibers when the wood contains a high moisture content. Dry wood tolerates ammonia concentrations below 30 %.

7.4 Ammonia in high concentrations can be hazardous. When working with ammonia it is recommended that an ammonia-sensitive badge be worn for safety.

7.4.1 The lower explosive limit (LEL) for ammonia and air is 15 %. The upper explosive limit (UEL) is 28 %.

7.4.2 The ceiling for limited exposure to ammonia (1 h) is about 500 ppm.

7.5 If the container to be tested has parts made of stainless steel, nickel, or chromium alloys, the color-change developer residue should have a sulfur and halogen content of under 500 ppm of each.

8. Apparatus

8.1 *Apparatus for Precleaning:*

8.1.1 *Gloves.*

8.1.2 *Vacuum Pump or Heat Gun*, if necessary to dry container.

8.1.3 *Spray Gun*, if aerosol cleaner not used.

8.1.4 *Ammonia-Sensitive Monitor*, to test area contamination.

8.2 *Safety Apparatus:*

8.2.1 *Mask*, covering mouth and nose.

8.2.2 *Ammonia Monitors.*

8.2.3 *Gas Mask*, if personnel work inside the tank or with high concentrations of ammonia during a test.

8.3 *Apparatus for Injecting Ammonia Gas:*

8.3.1 *Pressure Gage*—The gage must be able to withstand normal test pressures. The gage must be accurate to within 1 % of full scale. The gage must read at least 1.5× but not more than 4× the maximum test pressure to be used. The gage must be in current calibration.

8.3.2 *Pressure-Relief Valve*, if high pressures are to be used.

8.3.3 *Pressure-Reducing, -Control, and -Mixing Valves*, for the ammonia and nitrogen circuits if not already mixed.

8.3.4 *High-Conductance Injection Line and Exhaust Line.*

8.4 *Apparatus for Applying Colorimetric Developer:*

8.4.1 *Spray Gun*, if an aerosol is not used.

8.4.2 *Temperature-Controlled Heat Gun*, if a water-based developer is used.

8.5 *Apparatus for Post Cleaning and Inspection:*

8.5.1 *Brush or Vacuum Sweeper*, for developer powder.

8.5.2 *Tape*, for marking and sealing leaks.

9. General Procedures for Test Objects not Already Containing Ammonia

9.1 *Openings:*

9.1.1 Seal all openings using plugs or covers that can withstand the test pressure and can be completely removed after the test.

9.1.2 Locate the test gas inlet on the bottom of the test object with the trapped air vent at the highest point.

9.1.3 Components rated at pressures below the test pressure must be isolated.

9.2 *Gages*—One or more test gages must be connected to the system. If more than one gage is used, one may be a recording gage. All gages must have been calibrated within a specified time period. One indicating gage must be easily visible to the operator controlling the pressure throughout the pressurizing cycle.

9.3 *Pre-Test Inspection:*

9.3.1 Before pressurizing is begun, inspect the outside (and inside if possible) of the test object to verify that it is dry, free of oils, greases, smoke deposits, or slag and that all welds and connectors are exposed.

9.3.2 An ultrasonic pretest (Section 12) can be used to locate gross leaks.

9.4 *Vacuum Drying:*

9.4.1 If the test object contains wood or copper parts that will be exposed to ammonia, and if the vessel is designed to be safe under vacuum, it may be necessary to vacuum-dry the inside of the object.

9.4.2 Moisture begins to evaporate at a pressure of about 3 kPa (25 torr). Bring the test object down to a pressure of 250 Pa (20 torr). At this point water will boil off, indicated by a sudden halt in the vacuum gage needle. When the needle starts to go down indicating a lower pressure it can be assumed that all but trace amounts of water have been eliminated.

9.5 *Pressurizing*—Gradually increase the pressure in the system to 50 % of test pressure during which time frequent checks should be made for leakage. Thereafter, slowly increase the pressure to the final test pressure. The test pressure usually is between 75 and 150 % of design pressure and should not violate any applicable codes. If large leaks are expected and an ultrasonic pretest has not been conducted, stop the pressurization at 6.9 kPa (1 psig) and repair any leaks found before continuing.

9.6 *Leak Test:*

9.6.1 At the completion of test pressure holding time, examine the system for leakage. Examination of leakage shall be made of all welds, joints, and connections.

9.6.2 The inspector shall mark all accessible leaks found on the equipment using a nondeleterious distinctive tape. The magnitude of leak shall be described in terms of the diameter of the color-change indication or the apparent density of the smoke produced.

9.7 *Depressurizing*—After inspection, slowly release the pressure by venting the ammonia-nitrogen mixture to atmosphere or into water. Ammonia is very soluble; 1 L of water can absorb between 800 and 2000 L of gaseous ammonia. A vacuum pump may be used to help exhaust the remaining ammonia or the tank may be purged with nitrogen or compressed air.

9.8 *Removing the Color-Change Developer*—Remove the test developer from the test object by brushing it from the surface and cleaning with a dust-remover.

10. Smoke-Developer Method

10.1 *Sensitivity*—This test is the least sensitive and least calibratable of the developer methods. Its sensitivity with pure ammonia at 6.9 kPa (1 psig) is approximately 4.5×10^{-8} mol/s (1×10^{-3} Std cm³/s).²

10.2 Application:

10.2.1 This test is used primarily on systems that already contain ammonia or as a quick pre-test before applying the color-change developer.

10.2.2 Where small volumes need to be pressurized and pure ammonia may be used, this method can provide rapid leak location.

10.3 Smoke-Developer Types:

10.3.1 Sulfur candles produce a hazardous gas (sulfur dioxide) and for this reason are not recommended. This gas produces a visible smoke upon contact with ammonia.

10.3.2 Some dilute acid vapors produce a visible smoke with ammonia. The liquids that give rise to these vapors can be applied by wet swab or by aerosol. An aerosol spray is the preferred technique.

10.4 Process:

10.4.1 If the test object does not contain ammonia, it should be pressurized slowly to between 6.9 and 69 kPa (1 and 10 psig) with pure ammonia. If leak location rather than the leakage rate is desired, a cloth saturated with ammonium hydroxide can be placed in the pressurized space. Move the developer vapor slowly over areas of possible leaks. A white cloud will be produced at the leak location.

10.4.2 Mark and seal leak locations with tape whenever possible.

11. Color-Change Method

11.1 Sensitivity:

11.1.1 By varying the ammonia concentration, the test pressure and the development time, any leakage rate from 4.5×10^{-11} to 4.5×10^{-12} mol/s (1 to 1×10^{-7} Std cm³/s)² can be detected. The equation in 11.1.2 is usually applied by specifying the smallest leakage rate required (MDLR), as well as the maximum test pressure allowable. Depending on which is the more important remaining variable, test time or ammonia concentration, the less critical variable is solved for.

11.1.2 *Leakage Rate Calculation*—The following equation allows precise calculation of the test time in seconds (t), percentage of ammonia tracer (c), and absolute pressure in pounds per square inch (psia) within the test vessel (p) for any minimum detectable leakage rate (MDLR). This equation is based on laboratory data using the time in seconds required to generate a 1-mm reaction spot as the diameter of the minimum development area. Leakage rates are expressed in atm-cm³/s.

$$\text{MDLR} = \frac{2.534}{(15p^2 - 3241)(c^{2/3})(t)} \quad (1)$$

where:

t = time in seconds,

c = % ammonia,

p = psia, and

MDLR = atm-cm³/s.

11.1.3 The equation can be recast to solve for any of the variables. For example, a company wishing to test for leakage of 4.5×10^{-11} mol/s (1×10^{-6} Std cm³/s)² could proceed as follows: Assuming that 345 kPa (50 psia) was the maximum pressure their system could tolerate, it would take 4 min and 30 s to develop a reaction spot of 1 mm using a 50 % concentration of ammonia.

This result is calculated as follows:

$$t = \frac{2.534p}{(15p^2 - 3241)(c^{2/3})(\text{MDLR})} \quad (2)$$

or

$$t = \frac{2.534 (50)}{(15 \times 50^2 - 3241)(50^{2/3})(1 \times 10^{-6})} \quad (3)$$

11.1.4 It has been determined experimentally for wall thicknesses of 1 to 50 mm that for any given pressure differential, the observed leakage rate varies inversely with the thickness of the wall being tested. Although the relationship is not linear, the decrease in observed leakage can be approximated by adding the following numbers to the exponents of the theoretical MDLR you wish to detect.

11.1.4.1 For wall thicknesses of 2 to 10 mm (0.08 to 0.4 in.) add nothing.

11.1.4.2 For wall thicknesses between 10 and 25 mm (0.4 to 1 in.) add – 1.

11.1.4.3 For wall thicknesses between 25 and 50 mm (1 to 2 in.) add – 2.

11.2 *Application*—This method can be used on containers or systems that are filled or unfilled with ammonia as long as the atmosphere surrounding the test area is not contaminated with ammonia vapors.

11.3 Process Steps:

11.3.1 Pre-clean and Dry:

11.3.1.1 Welds and test connections must be clean of smoke, soil, weld slag, grease, etc., that could block access of the tracer or contaminate the developer. Even water vapor can obscure leaks smaller than 4.5×10^{-10} mol/s (1×10^{-5} Std cm³/s).² Wear gloves to avoid contaminating the surface with skin oils especially where the developer is to be applied.

11.3.1.2 Test the color-change developer on the surface of the test object to verify that the developer dries to the correct color.

11.3.1.3 If the developer color is not correct, check the atmosphere in the test area with an ammonia-sensitive monitor to see if it is contaminated with excessive ammonia vapors. If the atmosphere is contaminated, it must be cleared or a smoke developer used. If the atmosphere is not contaminated, passivate or more thoroughly clean the developer side of the test object.

11.3.1.4 Use a low-residue volatile cleaner. If some welds cause a slight discoloration of the developer, an acidic cleaner can be used to clean and passivate the weld. If the cleaner is water based it should be dried at a low temperature of less than 200°F with a heat gun.

11.3.1.5 For locating leaks smaller than 4.5×10^{-11} mol/s (1×10^{-6} Std cm³/s)², vacuum clearing of the inside of the container may be helpful. By drawing a slight vacuum on the inside, water vapor will be removed and a uniform ammonia mixture can be introduced.

11.3.2 *Apply Developer:*

11.3.2.1 The developer can be sprayed on by aerosol or in bulk with an ordinary paint sprayer. If the inside of a container is being coated, a water-based developer may prevent irritating vapor problems. An aerosol spray can be used to touch spots missed by a bulk sprayer. Use an impregnated-filter paper to cover complex connections that can not be easily sprayed or viewed.

11.3.2.2 Spray the developer by holding the spray gun or aerosol 25 to 60 cm away from the area to be coated. Apply the developer coat thinly, as thick coats take longer to be developed and may result in self-discoloration.

11.3.2.3 Use a hot-air gun to dry water-based developers immediately after their application, especially when the humidity is high. It may also be valuable to preheat the area to be developed to drive away any moisture. Hot air applied over 200°C may harm the developer.

11.3.3 *Inject Tracer:*

11.3.3.1 For leaks larger than 4.5×10^{-10} mol/s (1×10^{-5} Std cm³/s)² where the concentration of ammonia will be less than 15 % or more than 28 %, oil-free dry air can be substituted for nitrogen.

11.3.3.2 Calculate the pressure and concentration of ammonia needed according to the equation in 11.1.3.

11.3.3.3 If a mixing valve is not available, the ammonia concentration can easily be approximated by interpolating between the following procedures:

(a) *25 % Ammonia-75 % Nitrogen Mixture:*

- (1) Fill the nitrogen to atmosphere.
- (2) Fill with ammonia to a gage pressure of 96.5 kPa (14 psig).
- (3) Pressurize with nitrogen up to a gage pressure of 289.6 kPa (42 psig).

(b) *1 % Ammonia-99 % Nitrogen Mixture:*

- (1) Fill with nitrogen to atmosphere.
- (2) Fill with ammonia to a gage pressure of 4.14 kPa (0.6 psig).
- (3) Pressurize with nitrogen up to a gage pressure of 289.6 kPa (42 psig).

11.3.3.4 If the container being tested is a pressure vessel that will not be under steady pressure when in use, three successive increases and decreases in pressure during the test may be more successful in finding intermittent small leaks than severe over-pressurization.

11.3.3.5 If the container or part is double-walled or lined, ammonia can be introduced between the walls and the outside of both walls developed.

11.3.4 *Inspect:*

11.3.4.1 After the appropriate development time, all color indications 1 mm or larger indicate unacceptable leaks. In case of doubt, circle suspected areas and allow 1 to 2 min additional development time.

11.3.4.2 As leaks develop, dust off developer and cover leak with adhesive tape. The tape not only marks the point for repair, but prevents tracer leakage from contaminating the atmosphere.

11.3.5 *Repair and Reinspect*—If extensive welding is required, it may be necessary to exhaust the tracer and rinse the container with nitrogen before beginning repairs.

11.3.6 *Post Clean:*

11.3.6.1 Flush mixture to the outside or into water and purge with nitrogen or compressed air to remove all traces of ammonia.

11.3.6.2 The developer can be brushed, vacuumed, or washed off.

11.4 *Field Testing Open Containers:*

11.4.1 Containers that need to be tested while open can be tested in their unfilled condition with a sensitivity of approximately 4.5×10^{-9} mol/s (1×10^{-4} Std cm³/s).²

11.4.2 To locate leaks in installed tank bottoms, the developer can be applied to the inside of the tank and ammonia then injected underneath the tank. The ground must be dry during this test.

11.4.3 To locate leaks in tank seams above ground, the developer can be applied to the inside seams and ammonia then sprayed from a cylinder or aerosol along the outside seams.

12. Ultrasonic Pretest to Locate Gross Leaks

12.1 This test can be applied to the system prior to colorimetric testing to locate leaks larger than 4.5×10^{-6} mol/s (1×10^{-1} Std cm³/s)² and as a precautionary procedure to save time.

12.2 *Test Methods:*

12.2.1 *Pressure Test:*

12.2.1.1 The gas used as a test medium should be air, nitrogen, or other nonflammable gas.

12.2.1.2 The system should be pressurized with the gas gradually to 50 % of design pressure. 172 kPa (25 psig) is a minimum pressure for this test.

12.2.2 *Transmitter Test*—Instead of pressurizing, one or more ultrasonic sound transmitters matched to the ultrasonic detector can be placed inside the test object. The transmitter saturates the area with ultrasound which penetrates small holes and cracks.

12.3 *Ultrasonic Test*—Inspect all joints and connections and welds with an acoustic leak detector capable of hearing airborne audio frequencies in the range of 40 to 50 kHz. (See Test Method E 1002.)

13. Reporting Test Results

13.1 The following data should be included in a report on test results:

- 13.1.1 Date tested.
- 13.1.2 Conditions (temperature, pressure, relative humidity).
- 13.1.3 Test gas mixture.
- 13.1.4 Test pressure.
- 13.1.5 Test time.
- 13.1.6 Leak locations.
- 13.1.7 Leakage rate.
- 13.1.8 Signature of tester.

14. Precision and Bias

14.1 This procedure is believed to represent the best available practice. It has been confirmed by laboratory results, but no round-robin tests have been made to determine the precision and reliability of this method. The accuracy of the test is dependent on the operational procedure used and the training and qualification of the operators.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

15. Keywords

15.1 chemical based leak testing; colorimetric leak testing; leak testing; smoke leak testing