



Designation: ~~E1066/E1066M~~—12 E1066/E1066M – 19

## Standard Practice for Ammonia Colorimetric Leak Testing<sup>1</sup>

This standard is issued under the fixed designation E1066/E1066M; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This ~~test method practice~~ 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  $4.5 \times 10^{-12}$  mol/s [ $1 \times 10^{-7}$  Std cm<sup>3</sup>/s]<sup>2</sup> or larger.

1.4 *Units*—The values stated in Std cm<sup>3</sup>/s or mol/s are to be regarded separately as standard. The values stated in each system ~~may~~ are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other. ~~Combining other, and~~ values from the two systems ~~may result in non-conformance with the standard; shall not be combined.~~

1.5 *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, health, and ~~health~~ environmental 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).

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

- 2.1 *ASTM Standards:*<sup>3</sup> <https://catalog.standards/sist/555f46ed-1c88-41e5-b023-e487490f5b4f/astm-e1066-e1066m-19>  
E1002 Practice for Leaks Using Ultrasonics  
E1316 Terminology for Nondestructive Examinations
- 2.2 *Other Documents:*<sup>4</sup>  
SNT-TC-1A Recommended Practice for Personnel Qualification and Certification in Nondestructive Testing  
ANSI/ASNT CP-189 ASNT Standard for Qualification and Certification of Nondestructive Testing Personnel

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, see Terminology E1316, Section E.

### 4. Summary of Practice

4.1 This ~~test method practice~~ 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

<sup>1</sup> This ~~test method practice~~ is under the jurisdiction of ASTM Committee E07 on Nondestructive Testing and is the direct responsibility of Subcommittee E07.08 on Leak Testing Method.

Current edition approved June 15, 2012 May 1, 2019. Published July 2012 July 2019. Originally approved in 1985. Last previous edition approved in 2006 2012 as E1066-95 E1066/E1066M-12 (2006). DOI: 10.1520/E1066-12.10.1520/E1066\_E1066M-19.

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

<sup>3</sup> 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.

<sup>4</sup> Available from American Society for Nondestructive Testing (ASNT), P.O. Box 28518, 1711 Arlington Ln., Columbus, OH 43228-0518, http://www.asnt.org.

1 and 100 % by volume at a ~~gage~~gauge pressure between 34.5 and ~~689.5 kPa~~ 689.5 kPa [5 and ~~100 psig~~ 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~~**Warning**—~~Ammonia in high concentrations can be hazardous. When working with ammonia it is recommended that an ammonia-sensitive badge be worn for safety.~~ ~~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~~ 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~~—~~Gauge~~—The ~~gage~~gauge must be able to withstand normal test pressures. The ~~gage~~gauge must be accurate to within 1 % of full scale. The ~~gage~~gauge must read at least 1.5× but not more than 4× the maximum test pressure to be used. The ~~gage~~gauge 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~~—*Gauges*—One or more test ~~gages~~~~gauges~~ must be connected to the system. If more than one ~~gage~~~~gauge~~ is used, one may be a recording ~~gage~~~~gauge~~. All ~~gages~~~~gauges~~ must have been calibrated within a specified time period. One indicating ~~gage~~~~gauge~~ 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]. 250 Pa [20 torr]. At this point water will boil off, indicated by a sudden halt in the vacuum ~~gage~~~~gauge~~ 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  $4.5 \times 10^{-8}$  mol/s [ $1 \times 10^{-3}$  Std cm<sup>3</sup>/s].<sup>2</sup>

#### 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.