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Document Name: CGA G-2.2: Guideline Method for Determining Minimum of 0.2% Water in Anhydrous Ammonia

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REAFFIRMED 1997

GUIDELINE METHOD FOR DETERMINING MINIMUM OF 0.2% WATER IN ANHYDROUS AMMONIA

SECOND EDITION

COMPRESSED GAS ASSOCIATION, INC.

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1 Introduction

1.1 DOT regulations

Anhydrous ammonia shipped in DOT Specification MC-330 or MC-331 cargo tanks constructed of quenched and tempered steel (QT) *must* have a minimum water content of 0.2 percent by weight. Shippers or carriers, except as provided for in the regulations, are required to perform periodic analyses for the prescribed water content in the ammonia. See 49 CFR 173.315(a) Note 14 and 173.315(1). [1] [1]

1.2 Purpose of publication

CGA G-2.2 is intended to provide shippers and carriers with a guideline method of analysis to determine the presence in anhydrous ammonia of the prescribed minimum water content of 0.2 percent by weight as required by the DOT regulations. Lack of the appropriate percentage of water in single loads of ammonia has been found by experience to have resulted in extensive stress corrosion damage to the QT cargo tanks. This method is intended for field use and thus the equipment and procedure selected may vary slightly from that used under laboratory conditions. Other proven methods of determining water content are acceptable.

1.3 Ammonia properties

At room temperatures and atmospheric pressure, anhydrous ammonia is a pungent, colorless gas. Ammonia vapor at a pressure of 1 atmosphere (101.325 kPa) and a temperature of 32 °F (0 °C) is lighter than air, having a relative density of 0.5970. The sharp, pungent odor of ammonia serves as a warning signal such that very small concentrations of ammonia vapor in air are readily detectable.

1.4 Safety considerations

1.4.1 Physiological effects

Ammonia is not a cumulative metabolic poison. However, depending upon the concentration in the immediate atmosphere, it can have an irritating or corrosive effect on human tissues and can cause laryngeal and bronchial spasm, edema, and asphyxia. Liquid ammonia may cause severe injury upon contact with the skin, resulting in frostbite and caustic burns.

1.4.2 Personal protective equipment

Anyone handling anhydrous ammonia shall wear the following protective equipment as a minimum:

1.4.2.1

Protective gloves impervious to ammonia;

1.4.2.2

Chemical splash goggles; and

1.4.2.3

Full face shield over the chemical splash goggles as an option, but not as a substitute.

1.4.3 Personnel safety

Individuals working with anhydrous ammonia must be trained in safe handling and operating practices, and be knowledgeable of appropriate actions to take, including applicable first aid procedures, in the event of an ammonia release emergency. Information regarding these matters is given in CGA G-2, *Anhydrous Ammonia,* and

[1] References in this document are shown by bracketed numbers and are listed in the order of appearance. See Section 6. References.

in ANSI K61.1/CGA G-2.1, *American National Standard Safety Requirements for the Storage and Handling of Anhydrous Ammonia.* [2] and [3]

2 Reagents and equipment required

2.1 Reagents

Calcium sulfate, anhydrous, 8 mesh, for use as a desiccant, Fisher Scientific Company Cat. No.7-577-3 or equivalent.

2.2 Equipment

2.2.1

Glass residue tube, 100 ml, graduated; SGA Scientific Inc. Print 571293 or equivalent. See Fig. 1.

2.2.2

Ammonia sampling device. See Fig. 2.

2.2.3

Cap device. See Fig. 3.

2.2.4

Desiccant tube, 200 mm long x 16 mm ID with adapter tips for 9.5 mm ID tubing; Fisher Scientific Company Cat. No. 9-242C or equivalent.

2.2.5

Tygon® tubing, 9.5 mm ID, compatible with anhydrous ammonia, Fisher Scientific Company Cat. No. 14-169-3E or equivalent.

2.2.6

Valve, steel bar stock, 1/4-inch FPT, Aloyco Figure No. 40-N or equivalent.

2.2.7

Flow regulator assembly with 25 mil orifice; Spraying Systems Company type 4908-1/4TT4916-25SS or equivalent. See Fig. 4.

2.2.8

Residue tube rack. See Fig. 5.

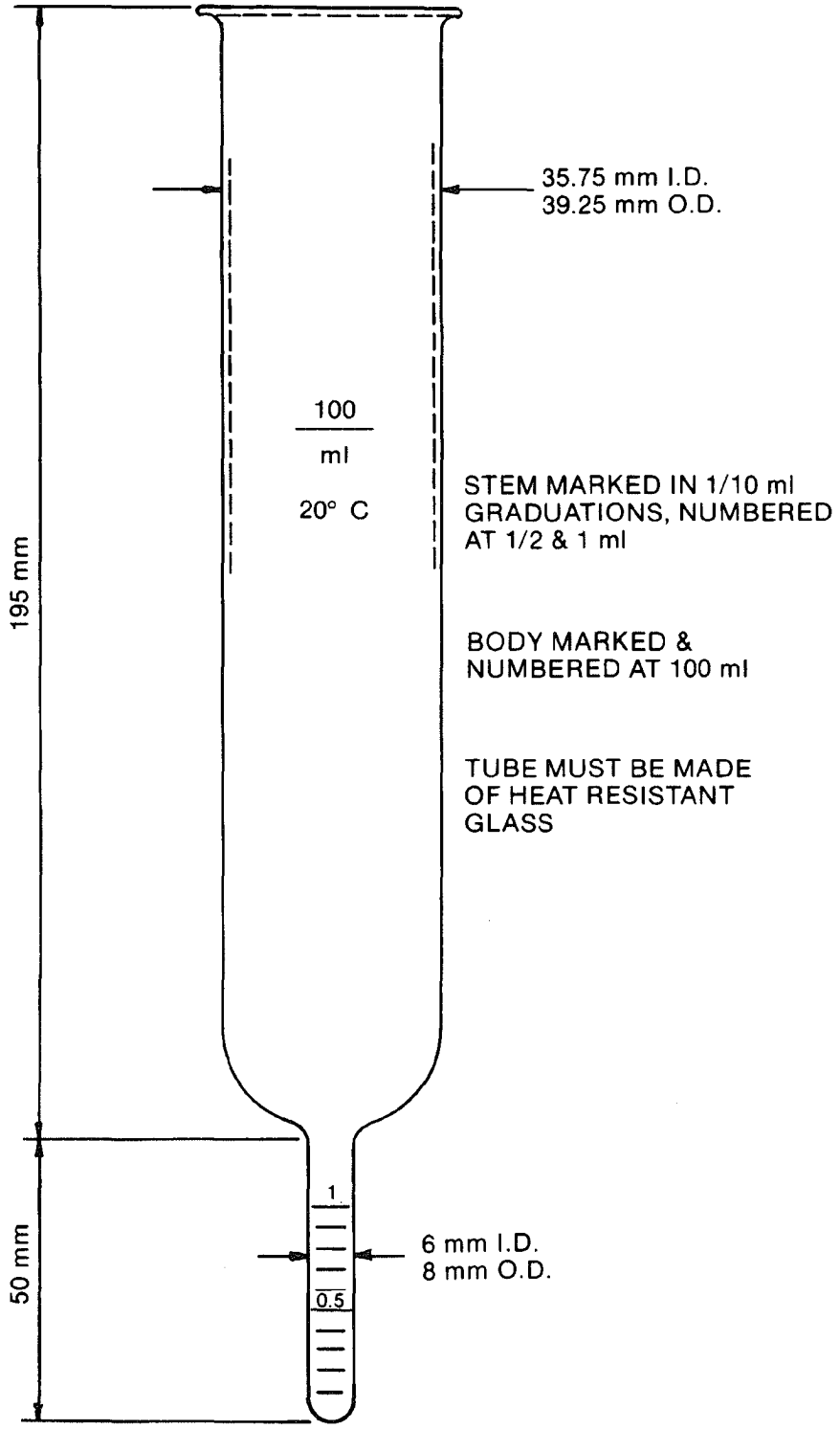


Figure 1—Glass residue tube

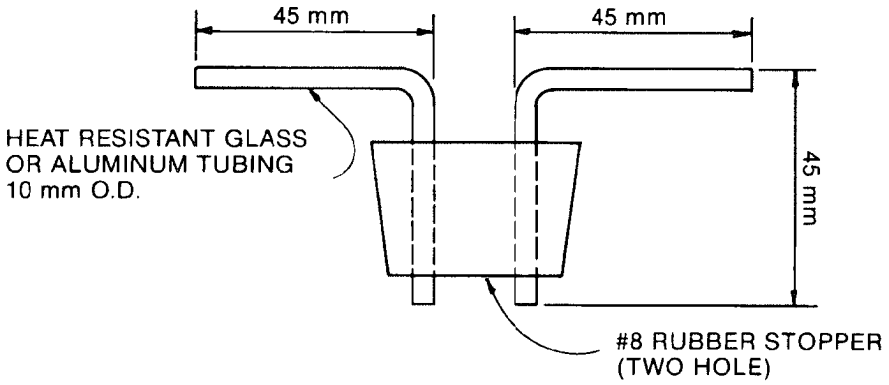


Figure 2—Ammonia sampling device

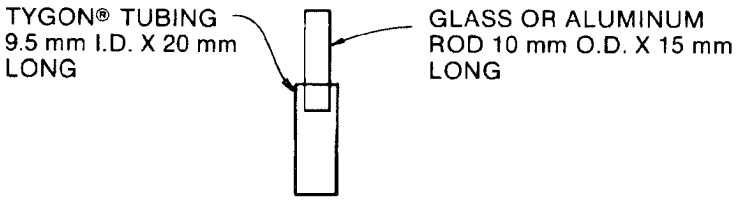


Figure 3—Cap device

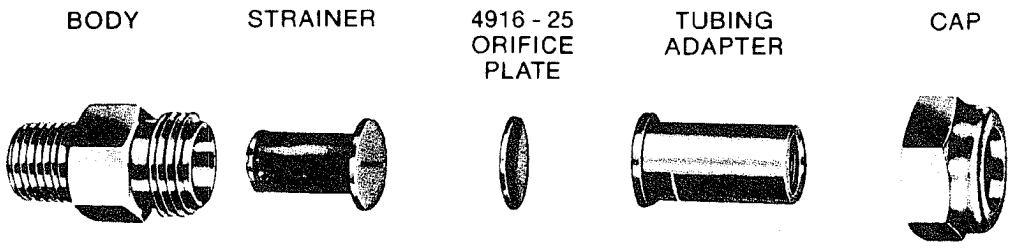
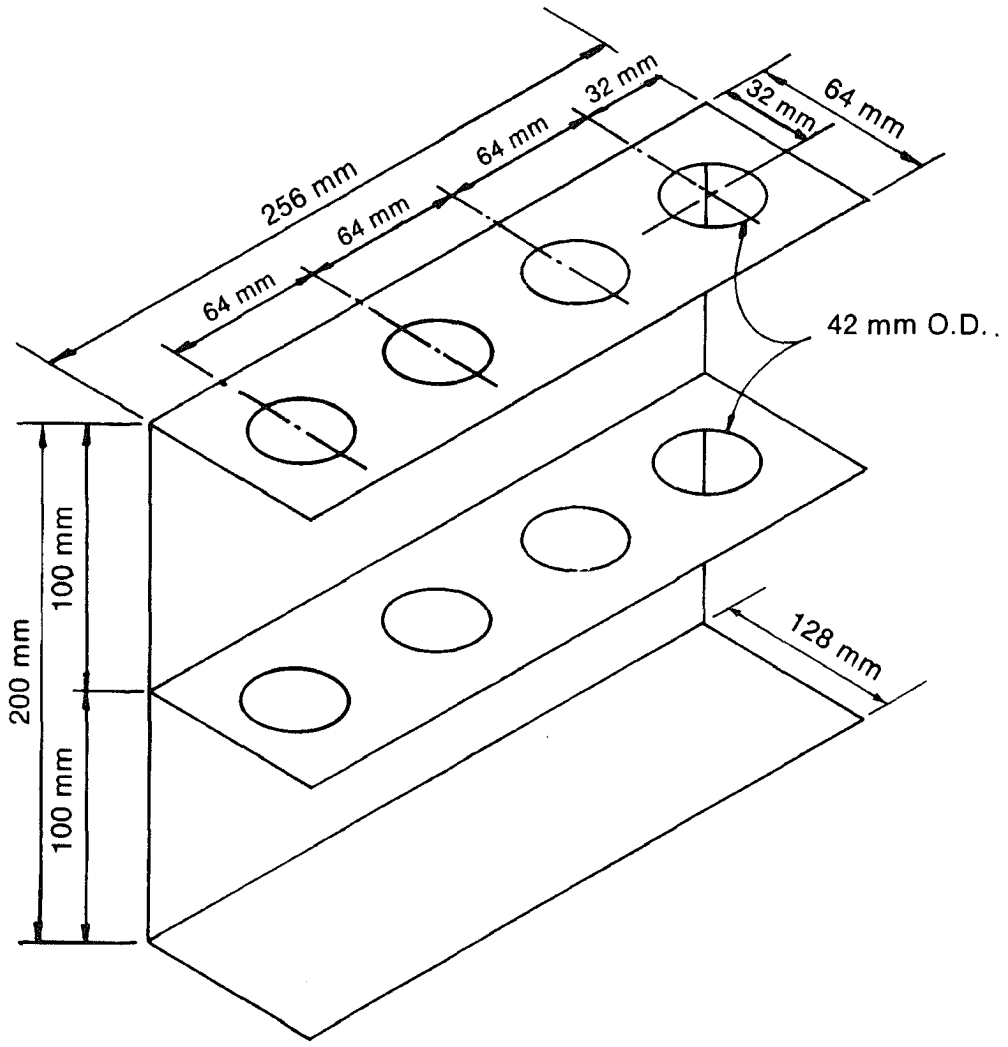


Figure 4—Flow regulator assembly



Construct from 16 gauge stainless steel sheet

Figure 5—Residue tube rack

3 Procedure

3.1

Clean all glassware with a suitable material to remove oil or other residue. Be certain that the glassware is dry before using. If necessary, a short length of 16 gauge stainless steel wire may be inserted into the glass residue tube to minimize "bumping" of the liquid anhydrous ammonia sample during evaporation. The wire must be removed before reading the residue volume or otherwise corrected for.

3.2

Install the steel bar stock valve at the appropriate connection point to permit withdrawal of a liquid ammonia sample from the bulk storage container, cargo tank, or loading line. See 49 CFR 173.315(1), (2) and (3) [1].

3.3

Connect the flow regulator assembly with 25 mil orifice to the steel bar stock valve on the downstream side to limit the liquid ammonia flow rate into the glass residue tube.

3.4

Connect a short length of Tygon tubing from the tubing adapter (see fig. 4) to the inlet tube of the ammonia sampling device.

3.5

Fit the rubber stopper portion of the ammonia sampling device into the mouth of the glass residue tube. The fit must be snug to prevent introduction of any extraneous moisture into the sample which could lead to an error in the test result. Provide a firm support for the glass residue tube to avoid overturn and loss of sample.

3.6

Connect a 30 inch (760 mm) minimum length of Tygon tubing to the outlet tube of the ammonia sampling device. Prepare the desiccant tube with desiccant and fit one end of the tube to the downstream outlet of the 30 inch (760 mm) length of Tygon tubing. The outlet of the desiccant tube should be supported and directed in a manner to permit safe discharge of ammonia vapor. If possible, chill the entire sampling train from the steel bar stock valve to the glass residue tube after complete assembly and before taking a sample of the liquid anhydrous ammonia.

3.7

Open the steel bar stock valve and carefully draw off liquid ammonia into the glass residue tube. Read and record the temperature of the liquid anhydrous ammonia in the container being sampled or in the line or pipe from which the sample is being taken.

3.8

Close the steel bar stock valve when the level of liquid anhydrous ammonia reaches the 100 ml graduation marked on the glass residue tube.

3.9

Remove the Tygon tubing from the inlet tube of the ammonia sampling device and quickly close the inlet tube with the cap device. Alternatively, use a screw clamp adjacent to the sampler on the Tygon tube and then disconnect tube at the tubing adapter.

3.10

If necessary to avoid vapor discharge or possible sample loss from the glass residue tube during transport of the assembly, which especially may occur due to the elevated ambient temperatures encountered during summer months, the glass residue tube may be chilled by placing it in loosely packed dry ice. The desiccant tube and desiccant must not be removed from the ammonia sampling device in order to prevent entry of moisture into the sample from the surrounding atmosphere. Once transported, the glass residue tube is removed from the dry ice and placed into a well supported residue tube rack and the ammonia is allowed to begin evaporating in a controlled manner such that the vapor is discharged into a well ventilated hood, scrubber, or duct so that safe environmental levels are not exceeded.

3.11

Allow the ammonia in the glass residue tube to evaporate in the ambient air to a volume of about 30 ml. Thereafter, place the glass residue tube and residue tube rack securely supported into a controlled temperature water bath with the water at about 70 °F (21.1 °C). As the sample continues to evaporate and the boiling diminishes, set the water bath temperature control to 120 °F ±2 degrees (48.9 °C) and maintain the bath at that tem-

perature for 10-15 minutes after the boiling has stopped to assure that the residue in the glass residue tube is at 120 °F ±2 degrees (48.9 °C).

CAUTION: *Should the, glass residue tube break or overturn, a violent reaction will occur when the anhydrous ammonia contacts the water in the water bath. Accordingly, a person handling the apparatus must wear the personal protective equipment described in 1.4.2 as a minimum requirement.*

3.12

Remove the glass residue tube from the water bath and allow the residue to attain room temperature.

3.13

Read and record the volume of the water residue in the calibrated stem of the glass residue tube estimated to the nearest 0.05 ml and accurately corrected to allow for the presence of any boiling aid device (if used).

NOTE—Oil should not interfere with the reading, but will float on the surface of the water residue.

4 Anhydrous ammonia evaporation factor

4.1

If the temperature of the anhydrous ammonia in the container or line sampled as recorded in 3.7 is equal to a temperature given in table 1, the anhydrous ammonia evaporation factor (EF) may be read directly. It should be noted that the evaporation factor required to correct for the vaporized ammonia flashed during sampling must be based upon the temperature of the liquid ammonia in the container or line from which the sample was taken. Most often, pressurized facilities are below saturation temperatures and a pressure reading of the subcooled ammonia does not permit an accurate determination of the evaporation factor.

4.2

The anhydrous ammonia evaporation factor (EF) may be calculated from the following equations:

- EF = 0.9491 - 0.00189F or
- EF = 0.8886 - 0.0034C

Where: F equals degrees Fahrenheit and C equals degrees Celsius.

4.3

Determine the anhydrous ammonia evaporation factor (EF) as read from table 1 or as calculated in 4.2 and record.

5 Calculation

5.1

Calculate the percent water by weight in the anhydrous ammonia sampled using the following formula:

Percent water by weight = (milliliters of water residue remaining in glass residue tube) x (1.076) x EF

5.2

The percent water by weight result calculated in 5.1 must equal or exceed 0.2% if the anhydrous ammonia sampled is to meet the requirements as set forth in 49 CFR 173.315(1) Note 14 [1].

Table 1—Anhydrous ammonia evaporation factors

Ammonia temperature in sampled container		Corresponding saturated ammonia pressure		Evaporation Factor (EF)
° F	(° C)	psig	(kPa)	
–28	(–33.3)	0.0	(0.0)	1.00
–20	(–28.9)	3.6	(24.8)	0.987
–10	(–23.3)	9.0	(62.1)	0.968
0	(–17.8)	15.7	(108)	0.949
5	(–15.0)	19.6	(135)	0.940
10	(–12.2)	23.8	(164)	0.930
15	(–9.4)	28.4	(196)	0.921
20	(–6.6)	33.5	(231)	0.911
25	(–3.9)	39.0	(269)	0.902
30	(–1.1)	45.0	(310)	0.892
35	(1.7)	51.6	(356)	0.883
40	(4.4)	58.6	(404)	0.874
45	(7.2)	66.3	(457)	0.864
50	(10.0)	74.5	(514)	0.855
55	(12.8)	83.4	(575)	0.845
60	(15.6)	92.9	(641)	0.836
65	(18.3)	103.1	(711)	0.826
70	(21.1)	114.1	(787)	0.817
75	(23.9)	125.8	(867)	0.807
80	(26.7)	138.3	(954)	0.798
85	(29.4)	151.7	(1046)	0.788
90	(32.2)	165.9	(1144)	0.779
95	(35.0)	181.1	(1249)	0.770
100	(37.8)	197.2	(1360)	0.760
105	(40.6)	214.2	(1477)	0.751
110	(43.3)	232.3	(1602)	0.741

SOURCE: Myers and Jessup, Refrigerating Engineering 11, 345.

6 References

[1] *Code of Federal Regulations,* Title 49 CFR Parts 100-179 (Transportation), U.S. Department of Transportation. Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[2] CGA G-2, *Anhydrous Ammonia,* Compressed Gas Association, Inc., 4221 Walney Rd., 5th Floor, Chantilly, VA 20151.

[3] ANSI K61.1 (CGA G-2.1), *American National Standard Safety Requirements for the Storage and Handling of Anhydrous Ammonia,* American National Standards Institute, Inc., 1430 Broadway, New York, NY 10018 or Compressed Gas Association, Inc., 4221 Walney Rd., 5th Floor, Chantilly, VA 20151.

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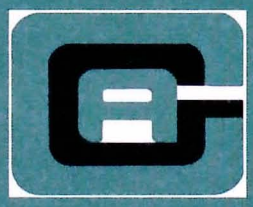

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