

Method: ACEN-6 Revision: 5 Final Revision Date: 11/17/03	Acetonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

The concentration of ammonia or ammonium salts is determined by the amount of standard NaOH required to return the pH of a solution to neutral when the ammonia has been tied up as hexamethylene tetramine by the addition of neutral formaldehyde.

SAFETY

Acetonitrile is hazardous to the health and dangerous to handle. Use acetonitrile in a well ventilated hood. Review the MSDS for detailed information concerning toxicity, first aid procedures and safety precautions.

Refer to the appropriate safety section or site manual for the necessary protective equipment to use when handling any reagents or samples.

INTERFERENCE

There are no known interferences with this method.

APPARATUS AND REAGENTS

1. **Sodium hydroxide 0.01N.** The solution may be prepared in the following manner: Dilute 10 mL of 0.1N sodium hydroxide [CAS 1310-73-2] (volumetric standard, 0.1N, Aldrich 31, 948-1 or equivalent) to 100 mL with water.
2. **Neutral Formaldehyde Solution.** Neutralize formaldehyde [CAS 50-00-0] (37wt % ACS reagent, Aldrich 25, 254-9 or equivalent) with sodium hydroxide or sulfuric acid. Check pH each time before use and adjust pH as required.

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3. **pH meter**, Corning 125, Brinkman 632, or equivalent.
4. **Buret**, 10 mL.
5. **Water**, ASTM Type II, or equivalent. Minimum electrical resistivity 1.0 MΩ·cm at 298 K; maximum total organic carbon 50 µg/L; maximum sodium 5 µg/L; maximum chlorides 5 µg/L; maximum total silica 3 µg/L. Detailed specifications can be obtained from ASTM: www.astm.org.
6. **Reference buffer solutions**, pH7 and pH4.
7. **Dropper bottles**, containing various strengths of NaOH (0.01N - 1N).
8. **Dropper bottles**, containing various strengths of H₂SO₄ (0.01N - 1N).
9. **Beaker**, 250 mL.
10. **Graduated cylinder**, 100 mL, 25 mL, 50 mL.
11. **Magnetic Stirrer and Stirring bar**.

CALIBRATION

Refer to pH meter calibration procedure CAL-2. The sodium hydroxide titrant is prepared from a purchased volumetric standard, which is standardized against NIST reference material. Under normal circumstances it is not necessary to re-standardize this titrant. Care should be taken, however, to prevent prolonged exposure of this titrant to the atmosphere, from which it will absorb carbon dioxide and change normality.

PROCEDURE

1. Use a calibrated pH meter.
2. Use a graduated cylinder to add 100 mL of acetonitrile to a 250 mL beaker.
3. Add 50 mL of water.
4. Place on a magnetic stirrer, insert pH electrode and stir slowly.
5. Adjust the pH of the sample to 7.0±0.05 using the NaOH and H₂SO₄ dropper bottles.
6. Using a graduated cylinder, add 25 mL of neutral formaldehyde solution and continue to stir for 5 min.

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7. ~~Using Titrate to pH 7.0 with~~ 0.01N NaOH, titrate to a pH of 7.0 \pm 0.05.

CALCULATIONS

$$\text{NH}_3, \text{PPM} = \frac{V \times N \times 0.017 \times 1000000}{100 \times \text{Sp. Gr.}} = \frac{V \times N \times 170}{\text{Sp. Gr.}}$$

Where:

V	=	Volume of NaOH, mL
N	=	Normality of NaOH
0.017	=	meq. of NH ₃
1000000	=	factor for ppm
100	=	ml of sample
Sp.Gr	=	0.78

REPORT

Report ammonia content to the nearest 0.1 ppm.

Ex: NH₃, ppm = 2.3 ppm