

Method: ACEN-25 Revision: 5 Final Revision Date: 11/19/03	Acetonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

A sample is titrated directly with 0.01N HCl, in a closed system, using a 1:3 mixture of bromocresol green and methyl red indicators. The acidity is calculated from the amount of hydrochloric acid required. Titration may be done with a burette to a visual colorimetric end-point.

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.

REFERENCES

ACS Reagent Chemicals, Ninth Edition

INTERFERENCES

There are no known interferences to this method.

APPARATUS AND REAGENTS

1. **Hydrochloric Acid Solution (0.01N).** [CAS 7647-01-0] Fisher SA62-1, or the solution may be prepared in the following manner: Dilute 100 mL of 0.1N Hydrochloric acid (Fisher SA54-1) to one liter with ASTM type II water.
2. **Ethyl Alcohol (Ethanol)** [CAS 64-17-5].
3. **Bromocresol Green Indicator: 0.05% solution.** Dissolve 0.05 gm of bromocresol green [CAS 62625-32-5] in 100 mL of ethyl alcohol. Mix well.

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4. **Methyl Red Indicator:** Dissolve 0.1 gm of methyl red [CAS 493-52-7] in 100 mL of ethyl alcohol. Mix well.
5. **Magnetic stirrer and stirring bar.**
6. **Buret:** 10 mL, Class A.
7. **Flask, Erlenmeyer,** 125 mL.
8. **Cylinder, graduated,** TD100 mL Class A
9. **1:3 Indicator Mixture:** Mix 15 mL of the bromocresol green indicator with 45 mL. of the methyl red indicator.
10. **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.

CALIBRATION

The hydrochloric acid 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.

PROCEDURE

1. With a Class A graduated cylinder, add 100 mL acetonitrile to a 250 mL Erlenmeyer flask.
2. Add 0.4 mL of the 1:3 indicator mixture.
3. Titrate with 0.01N HCl to a light orange-pink end point.

CALCULATIONS

$$\text{Titration Base} = \frac{(\text{mL HCl})(0.01\text{N})}{(\text{mL sample})(\text{S.G. sample})} \times \frac{1000 \text{ ueq}}{1 \text{ meq}}$$

Where S.G. sample = Specific Gravity of Acetonitrile (0.78 g/mL) and mL sample = 100

REPORT

Report titration base to the nearest <0.01 ueq/gm.