

<b>Method:</b> ACEN-4 Revision: 5 Final Revision Date: 11/17/03	<b>Acetonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review: 08/15/07 Next Review: 08/15/11		<b>Acidity</b>

## METHOD SUMMARY

A sample is titrated directly, in a closed system, with alcoholic 0.01N NaOH using bromothymol blue indicator. The acidity is calculated from the amount of sodium hydroxide required and is reported as ppm acetic acid. Titration may be done with a burette to a visual colorimetric end-point or with an automatic titrator using a color probe to detect the color change. Values less than 0.001 wt.% are reported as <0.001 wt.%.

## 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

STM C-9-75, "Acidity in Acrylonitrile and Acetonitrile"

## INTERFERENCES

There are no known interferences to this method.

## APPARATUS AND REAGENTS

- Sodium Hydroxide Standard Solution, (0.01N)** – [CAS 1310-73-2] Fisher LC24520-2(in methanol), or the solution may be prepared in the following

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manner: Dilute 100 mL of 0.1N sodium hydroxide (volumetric standard, Aldrich 31,948-1 or equivalent) to one liter with isopropanol or methanol.

2. **Bromothymol Blue Indicator Solution, (1 g/L)** - This solution may be prepared in the following manner: Weight 100 mg of bromothymol blue [CAS 76-59-5] ACS reagent grade, Aldrich 11,441-3 or equivalent) into a 150 mL beaker, add 1.5 mL of 0.1N NaOH to aid in dissolving and then gradually add 100 mL of methanol. Neutralize to a deep green by dropwise addition of 0.01N NaOH.
3. **Methanol**, [ CAS 67-56-1] ACS grade.
4. **Magnetic stirrer and stirring bar.**
5. **Microburet**, 10 mL fitted w/rubber stopper.
6. **Flask**, Erlenmeyer, 250 mL.
7. **Flasks**, volumetric, 100 mL.
8. **Cylinder**, graduated, 100 mL.
9. **Isopropanol**, [CAS 67-63-0] ACS grade.

Addition Reagents for Autotitration Method:

1. **Automatic titrator** such as Metrohm EA536.
2. **Automatic dispensing** burette (10 mL) and magnetic stirrer.
3. **Color probe** such as Brinkman PC701 colorimeter -- equipped with a 420 nm filter.
4. **Covered 250 mL titration beaker** with stoppers for all but two holes

## CALIBRATION

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 water and carbon dioxide and change normality.

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## PROCEDURE

1. Measure 100 mL of sample with a graduated cylinder and pour into a 250 mL Erlenmeyer flask or titration beaker of autotitrator.
2. Add 4 to 6 drops of Bromothymol blue indicator. Insert a magnetic stirring bar and seal the flask or beaker with a lid that allows the burette tip (and color probe) to extend into the vessel.
3. Stir slowly and titrate with 0.01N alcoholic sodium hydroxide until the blue visual end-point.
4. For the auto titrator, determine the end-point from the inflection point on the titration curve.

## CALCULATIONS

Acidity, calculated as acetic acid, wt.% =

$$\frac{(\text{mL NaOH}) (N \text{ NaOH}) (0.06005) \times 100\%}{(\text{mL sample}) (0.78)}$$

Where

0.06005 = meq. wt of acetic acid, g/meq.

0.78 = specific gravity of acetonitrile, g/mL

N = Normality NaOH, 0.01 meq./mL

## REPORT

Report acidity (as acetic acid) to the nearest .001%.

Ex: Acidity = < .001%