

Method: ACEN-7 Revision: 5 Final Revision Date: 11/17/03	Acetonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

A known weight of sample is titrated in methanol with standardized Karl Fischer or Hydranal reagent using a dual platinum electrode "dead-stop" electrometric titration apparatus. The end-point is indicated by a characteristic change in current, measured in microamperes. The % water is calculated from the volume of Karl Fischer or Hydranal reagent consumed. This method should only be used to analyze samples with higher (greater than 0.05%) water levels.

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

ASTM E1178-97(2002), "Standard Test Methods for Analysis of Acrylonitrile"

ASTM E203-01, "Standard Test Method for Water, Using Karl Fischer Titration"
<http://www.astm.org/>

INTERFERENCES

There are no known interferences to this method.

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APPARATUS AND REAGENTS

1. **Karl Fischer titrator**, the Brinkman Metrohm Auto Titrator or equivalent with 665 Dosimat, includes a 10-20 mL burette, dual platinum electrode, programming keyboard, reaction vessel with cover, magnetic stirrer with variable setting, stir bar, drying tubes, desiccant and connectors.
2. **Pipettes**, assorted sizes.
3. **Methanol**, [CAS 67-56-1] absolute low moisture.
4. **Karl Fisher reagent**, Fisher Scientific #SO-K-3 or Hydranal-Composite/5, #CC34805-212, Fisher Scientific.
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. **Balance**, analytical, sensitive to ±0.1 mg.
7. **Syringe**, 50 µL.

CALIBRATION

Standardize according to the appropriate calibration document or do the following:

1. Refer to individual instrument operating manual for specific operating procedures.
2. Add enough methanol to cover platinum leads on electrode. Set magnetic stirrer at moderate speed and maintain at this setting. Assure no entrainment of air.
3. Accurately weigh and transfer 1 or 2 drops (20-100 mg) of water to the reaction vessel. Fill the 50 µL syringe with water and weigh the full syringe. Inject the water through the septum in the Karl Fisher apparatus and re-weigh the empty syringe. The difference in weights is the mg water added.
4. Titrate and calculate the reagent factor:

$$RF = \frac{\text{mg water added}}{\text{mL reagent added}}$$

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PROCEDURE

1. The methanol in the reaction vessel should have all moisture neutralized.
2. Weigh the sample, add to the reaction vessel and titrate. When using Karl Fischer reagent, add 10.0 mL of sample. When using Hydranal, add 1.0 mL of sample. Most titrators will calculate % water.

CALCULATIONS

If the titrator does not calculate weight percent water:

$$\text{Wt \% H}_2\text{O} = \frac{\text{mL reagent used}}{\text{mL sample}} \times \frac{(\text{RF}) \times 100\%}{(0.78 \text{ g/mL})(1000 \text{ mg/g})}$$

0.78 g/mL - specific gravity of acetonitrile
RF – reagent factor as defined in step 4 above.

REPORT

Report wt % water to two decimal places:

$$\text{wt \% water} = 0.42$$