

<b>Method:</b> ACEN-22A Revision: 1 Revision Date: 08/15/07	<b>HPLC Acetonitrile  Specification Tests</b>	<b>INEOS Nitriles</b>
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## METHOD SUMMARY

A sample is evaporated to dryness in a tared evaporating dish on a hot plate and oven. The dish is cooled and reweighed. The ppm by weight of residue is calculated and reported. This procedure is used for detecting values below 10 ppm residue. The theoretical detection limit is 0.25 ppm.

## 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 (SOHIO Test Method) C7-75, "Non-Volatile Matter in Acrylonitrile"

ACEN-22, "Non-Volatile Matter"

ASTM D2109-96(2000)e1, "Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures" <http://www.astm.org/>

CAL-2, "Laboratory Instrument Calibration Method – Balances"

ASTM D 2021-65(1995), "Specifications for Neutral Detergent 40 Percent Alkylbenzene Sulfonate Type," <http://www.astm.org/>

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October 2006

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## APPARATUS AND REAGENTS

1. **Hot plate** (or steam bath) capable of maintaining a temperature of  $80 \pm 2^\circ\text{C}$ . The heating element should be enclosed so that the acetonitrile fumes cannot be ignited. The hot plate must be located in a fume hood.
2. **Rotary Evaporator with Steam Bath** (and associated glassware)
3. **Oven**, thermostatically controlled at  $105 \pm 2^\circ\text{C}$ .
4. **Balance**, analytical, sensitive to 0.1 mg. (CAL-2)
5. **Evaporating dish**, Porcelain or noble metal, tall form, with capacity of 100 to 200 mL.
6. **Desiccator**, with indicating desiccant.
7. **Graduated cylinder**, 500 mL. Class A
8. **Oven**, constant temperature, capable of maintaining a temperature of  $105^\circ \pm 2^\circ\text{C}$ .
9. **Cleaning Agent**. A liquid surface-active agent conforming to ASTM D 2021 (2.2). Fisher Sparkleen 1 (04-320-4). Approved equivalents may be used, provided they can be completely rinsed from the surface and leave neither solid nor oily residue.
10. **Water**, ASTM Type II or equivalent.
11. **Tongs**, for handling evaporating dishes.

## CALIBRATION

Insure that the balance is calibrated per Method CAL-2.

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## PREPARATION OF THE APPARATUS

1. Wash the evaporating dish with a solution of the cleaning agent in hot water and rinse thoroughly with water.
2. Place the clean evaporating dish in the oven and heat it to  $105^{\circ} \pm 2^{\circ} \text{C}$  for 30 minutes.
3. Allow the dish to cool for 30 minutes in the dessicator near the balance. Handle the evaporating dish with clean tongs.

The cover should be kept securely on the dessicator except when articles are being inserted or removed. The dessicant must be replaced when it changes from blue to pink.

4. Store the dish in the dessicator.

## PROCEDURE

1. Rinse the 500 mL graduated cylinder and 1000 mL rotary evaporating flask with acetonitrile sample to be tested.
2. Measure 500 mL of the sample into a clean 500 mL graduate and transfer 470 mL to a clean 1000 mL rotary evaporating flask. Evaporate the sample to about 40 to 50 mL on the rotary evaporation device.
3. Dry a cleaned 150 mL capacity evaporating dish in an oven at  $105^{\circ} \pm 2^{\circ} \text{C}$  for 15 minutes and cool in a desiccator for 15 minutes. Repeat until the weight is constant or within 0.1 mg of the last weighing.
4. Transfer the residue to the evaporating dish. Rinse the flask twice with a 15 mL portion of the sample retained in the graduate (All of the retained sample is used to rinse the flask and must be included in the sample volume.). Add the rinsate to the evaporating dish.
5. Allow the sample to evaporate by gently heating on a warm hotplate in a hood. Protect the evaporating dish from external contamination and do not exceed  $77^{\circ} \text{C}$ . When the evaporation is complete, dry the evaporating dish in the oven at  $105^{\circ} \text{C}$  for 15 minutes, cool in a desiccator for 15

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minutes, and reweigh. Repeat oven drying, cooling and weighing steps until the weight is within 0.1 mg of the previous weighing.

## CALCULATIONS

Calculate the concentrations of non-volatile matter in ppm, with the following equation:

$$\text{NVM, ppm} = \frac{(W_2 - W_1) \times 10^6}{(V_1)(0.78)}$$

Where:

- $W_1$  = wt. dish, g
- $W_2$  = wt. dish + residue, g
- $V_1$  = sample volume, mL
- 0.78 = specific gravity of acetonitrile
- $10^6 = \frac{10^3 \text{ mg}}{\text{g}} \times \frac{10^3 \text{ mL}}{\text{L}}$

## REPORT

Report ppm non-volatile matter in the sample to the nearest 1ppm. If the result is less than 1 ppm, report as < 1 ppm.

Example: Non-volatile matter, ppm = 2