

Method: ACEN-22 Revision: 5 Final Revision Date: 11/19/03	Acetonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

A sample is evaporated to dryness in a tared evaporating dish on a hot plate. The dish is cooled and reweighed. The ppm by weight of residue is calculated and reported. Limit of quantitation is 10 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 C7-75, "Non Volatile Matter in Acrylonitrile," SOHIO Test Method, 1975.

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/>

INTERFERENCES

There is no known interference to this method.

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APPARATUS AND REAGENTS

1. **Hot plate**, 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. **Balance**, analytical, sensitive to 0.1 mg (CAL-2)
3. **Evaporating dish** – Glass, porcelain or one of the noble metals, deep form, with a capacity of 150 to 200 ml.
4. **Tongs**, for handling evaporating dishes.
5. **Desiccator** - with indicating desiccant.
6. **Graduated cylinder**, 250 mL.
7. **Oven**, constant temperature, capable of maintaining a temperature of $105^\circ \pm 2^\circ\text{C}$.
8. **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.
9. **Water**, ASTM type II or equivalent.

CALIBRATION

The balance used for this method should be certified and/or calibrated in accordance with Method CAL-2.

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 $105^\circ\text{C} \pm 2^\circ\text{C}$ for 30 minutes.

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3. Allow the dish to cool for 30 minutes in the dessicator near the balance.

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. Weigh the evaporating dish and record the weight to the nearest 0.1 mg. Handle the evaporating dish with clean tongs.

PROCEDURE

1. Weigh a clean, dry evaporating dish on an analytical balance to 0.1 mg.
2. Measure 125 mL of sample with a graduate and transfer to the dish.
3. Place the dish on a hot plate set for a surface temperature of approximately 80°C in a fume hood and slowly evaporate to dryness.
4. Place the dish in an oven at 105° C ± 2 °C for 30 minutes.
5. Cool in a desiccator. Reweigh and determine weight of residue.

CALCULATIONS

Calculate the concentration 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:

$$W_1 = \text{wt dish, g}$$

$$W_2 = \text{wt dish + residue, g}$$

$$V_1 = \text{sample volume, mL}$$

$$0.78 = \text{specific gravity of acetonitrile}$$

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$$10^6 = \frac{10^3 \text{ mg}}{\text{g}} \times \frac{10^3 \text{ mL}}{\text{L}}$$

REPORT

Report ppm non-volatile matter in the sample to the nearest 10 ppm. If the result is less than 10 ppm, report as <10 ppm.

Ex: Non-volatile matter, ppm = 50