

<b>Method:</b> ACEN-14 Revision: 5 Final Revision Date: 11/18/03	<b>Acetonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
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## METHOD SUMMARY

This procedure allows determination of iron in acetonitrile by heated graphite atomizer atomic absorption spectrophotometry. The detection limit is 0.02 ppm for iron. Precision is better than  $\pm 0.01$  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 C15-76, "Iron in Acrylonitrile and Acetonitrile," SOHIO Test Method, 1976.

ACEN-14A, "Iron by Spectrophotometry"

## APPARATUS AND REAGENTS

1. **1000 ppm iron reference solution** (Fisher SI124 or equivalent)
2. **Methanol**, [CAS 67-56-1], (Reagent grade)
3. **Acetonitrile**, [CAS 2206-26-0] containing less than 5 ppb of iron

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4. **Polyethylene bottles**, one-ounce, washed with 1:1 nitric acid, rinsed with deionized water and air dried.
5. **Disposable pipettes** or medicine droppers
6. **Micropipette and tips** for dispensing 20.0 microliter aliquots
7. **Atomic absorption spectrophotometer** equipped with heated graphite atomizer
8. **Hollow cathode lamp** for iron
9. **Top-loading balance** with sensitivity and accuracy better than 0.01 gram (CAL-2)

## CALIBRATION AND PROCEDURE

### Preparation of Standards

50 ppm stock solution -- Transfer 5.00 grams of the 1000 ppm iron solution to a dry four-ounce polyethylene bottle. Dilute to 100 grams with methanol.

2.5 ppm stock solution -- Transfer 1.00 grams of the 50 ppm stock solution to a dry one-ounce bottle. Dilute to 20.0 grams with metal-free acetonitrile.

100 ppb working standard -- Transfer 0.80 grams of the 2.5 ppm working solution to a dry four ounce bottle. Dilute to 20.0 grams with metal-free acetonitrile. This solution is also used to spike samples to verify absence of interferences.

50 ppb working standard -- Transfer 10.00 grams of the 100 ppb solution to a dry one-ounce bottle and dilute to 20.00 grams with metal-free acetonitrile.

25 ppb working standard -- Transfer 10.00 grams of the 50 ppb solution to a dry one-ounce polyethylene bottle and dilute to 20.00 grams with metal-free acetonitrile.

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1. Set up instrument according to manufacturer's recommendation or operator's previous experience. Install iron hollow cathode lamp and align. Set monochromator to select iron line at 248.3 nm.
2. Set temperatures on the furnace to 100 °C for drying, 900 °C for charring, 2100 °C for atomization and 3000 °C for cleaning. Drying time should be 20 seconds, ashing time 15 seconds, and atomization and cleaning times 5 seconds each.
3. Prepare a calibration curve by injecting 20 microliter aliquots of working standard solutions in ascending order of concentration (i.e. blank, 25, 50, and 100 ppb) to prepare a calibration curve. Each solution should be injected at least three times to assure accuracy; additional injections may be employed to improve precision.
4. After calibration, inject and analyze at least two blanks to assure that baseline is stable. Results should be between - 10 and + 10 ppb.
5. Analyze duplicate portions of the 25 ppb standard to check accuracy. Results should be between 15 and 35 ppb.
6. Analyze samples as received by injecting 20 microliter aliquots. At least two aliquots of each sample should be used to assure accuracy. Values should agree to within  $\pm 10$  ppb of mean; if not, repeat until standard deviation of three or less is obtained.
7. After every fifth sample, analyze a blank and a standard to assure that calibration is stable. Results should agree to within 10 ppb of nominal value.
8. Prepare spiked samples at a frequency of 10% by weighing 9.00 grams of sample into a dry one-ounce bottle and adding 1.00 gram of the 100 part per billion working standard. Analyze spikes by same procedure as samples. Spike recovery should be between 8 and 12 ppb.

## CALCULATIONS

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Calculations are very straightforward. Results are obtained directly from the calibration curve. If any sample is found to contain more than 100 ppb, dilute with metal free acetonitrile and repeat the analysis.

Spike recoveries are typically between 80 and 120%.

Duplicate injection should agree within 10 ppb or better.

Detection limit is 20 ppb for iron. It may be improved by increasing the atomizing temperature and/or increasing sample size.

## REPORT

Report iron concentration to the nearest 0.01 ppm.

Ex: iron, ppm = 0.03