

<b>Method:</b> ACEN-7A Revision: 5 Final Revision Date: 11/17/03	<b>HPLC Acetonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
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## METHOD SUMMARY

This method is based on the coulometric Karl Fischer titration that utilizes a methanolic solution of iodine, sulphur dioxide and a base as buffer to electrochemically generate iodine when a sample containing water is introduced into the reaction vessel. As iodine is produced, the voltage difference across a pair of indicator electrodes decreases. A voltametric method is used to detect the endpoint. This method is best for very low levels of water, and can be used for samples with up to 0.5% water.

## 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 E1064-00, "Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration"

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

MSDS of acetonitrile, available on <http://techservice.innovene.com>  
ACEN-7-5, "Water in Acetonitrile"

Metrohm Model 756 KF Coulometer Instruction Manual

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October 2006

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## INTERFERENCES

There are no known interferences to this method.

## APPARATUS AND REAGENTS

- Balance**, analytical, sensitive to 0.1 mg (CAL-2)
- Coulometer**, Metrohm Model 756.
- Syringe**, 3 or 5 cc. For sample introduction.
- Hydranal-Coulomat AG reagent**.
- Stand**, Metrohm Ti 703.
- Keyboard**, Metrohm Model 756.
- Hydranal Water Standard 1.00** (Sigma 34828), 1 mg water per gram
- Hydranal water standard 0.10** (Sigma 34847), 0.1 mg water per gram

## CALIBRATION

The manual for the Coulometric Karl Fischer titration apparatus states that this is an absolute method and does not require calibration. A validation of the instrument should be performed and documented on a quarterly basis, using a Hydranal water standard in accordance with manufacturer's recommendations. The result of this validation test should not exceed  $\pm 5\%$  of the specified value.

## PROCEDURE

- Use the following table to determine the appropriate sample size. The minimum amount of H<sub>2</sub>O to be determined should at no time be less than 50  $\mu\text{g}$ .

<b>Content of sample</b>	<b>Sample weight</b>	<b>H<sub>2</sub>O to be determined</b>
100000 ppm	50 mg	5000 $\mu\text{g}$
10000 ppm	10 mg – 100mg	100 $\mu\text{g}$ – 1000 $\mu\text{g}$
1000 ppm	100 mg – 1g	100 $\mu\text{g}$ – 1000 $\mu\text{g}$
100 ppm	1 g	100 $\mu\text{g}$
10 ppm	5 g	50 $\mu\text{g}$

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2. Tare a 3 cc or 5 cc syringe with the appropriate amount of sample.
3. Press the "START" button on the 756 KF Coulometer keyboard.
4. Inject the sample into the reaction vessel.
5. Reweigh the syringe.
6. Enter the sample weight on the 756 keyboard and press "ENTER".
7. Titrator will print ppm water.

## CALCULATIONS

The titrator performs the necessary calculations automatically. The titrator will report  $\mu\text{g}$  water found, and using the sample weight entered in step 6 above will calculate the ppm water.

## REPORT

Report water to the nearest ppm or wt% to four decimal places.

Ex: ppm water = 35 ppm

Wt % water = 0.0035 %