

<b>Method:</b> <b>ACRN-13</b> Revision:        5    Final Revision Date: 07/24/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review:    04/01/08	<b>Inhibitor</b>	Page 1 of 6
Next Review:    04/01/12		Reviewed by: Jennifer Young

## METHOD SUMMARY

The polymerization inhibitor p-methoxyphenol, MEHQ, absorbs ultraviolet energy near the 295 nm region of the spectrum. No other compounds absorbing in this region are usually present in acrylonitrile. The magnitude of the MEHQ absorbance is directly proportional to the MEHQ concentration. The absorbance is measured with a UV spectrophotometer and is converted directly to ppm MEHQ by weight by means of a standard calibration curve. Quantitation range is 12-50 ppm.

## SAFETY

Acrylonitrile is hazardous to the health and dangerous to handle. Use acrylonitrile 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 C11-75 (SOHIO Test Method), "Inhibitor in Acrylonitrile"

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

## INTERFERENCES

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October 2006

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<b>Method:</b> ACRN-13 Revision: 5 Final Revision Date: 07/24/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review: 04/010/08 Next Review: 04/01/12	<b>Inhibitor</b>	Page 2 of 6 Reviewed by: Jennifer Young

Any impurity that absorbs at 295 nm will interfere. Hydroquinone interferes with this test causing high biased results. Experience has shown that some impurities produced in aged acrylonitrile absorb in this region. The test method should, therefore, be applied only to acrylonitrile that is within the color specification.

## APPARATUS AND REAGENTS

- Spectrophotometer**, UV-Visible, Hitachi U-2000 or equivalent (CAL-1)
- Absorption cells**, 1 cm silica.
- Pipettes**: 1 mL, 2 mL, 3 mL, Class A
- Flasks**, volumetric, glass stoppered, 100 mL capacity, Class A
- MEHQ (p-methoxyphenol)**. Eastman inhibitor free #350 or equivalent.
- Acrylonitrile**.
- Balance**, analytical, sensitive to  $\pm 0.1$  mg (CAL-2).
- Sodium Hydroxide, 1N** – ACS Certified, or the solution may be prepared in the following manner. Add 40g of reagent grade sodium hydroxide to ~ 600 mL of water. Dissolve and dilute to 1 liter.
- Separatory funnel**, 500 ml.

## CALIBRATION

- Prepare about 650 ml of inhibitor-free acrylonitrile as follows: shake 200 ml of 1N NaOH with 100 ml of acrylonitrile in a 500 ml separatory funnel. Repeat the extraction with NaOH three or four times. Shake with 200 ml of water. The absorbance near 295 nm should be less than 0.01. Alternately, distill 750 ml of acrylonitrile retaining the 650 ml heart-cut.
- Accurately weigh  $0.10 \pm 0.05$  g for Stock I and  $0.40 \pm 0.05$  g for Stock II of MEHQ into separate 100 ml volumetric flasks. Dilute to volume with inhibitor-free acrylonitrile (~1250 and 5000 ppm w/w).

$$\frac{\text{g MEHQ (1000 mg/g)}}{(100 \text{ ml}) (0.807 \text{ g/ml}) (1 \text{ kg/1000 g})} = \text{ppm (w/w) MEHQ}$$

where: 0.807 g/ml is the density of acrylonitrile.

- Carefully pipette 1.0, 2.0 and 3.0 ml of Stock I and 1.0 ml of Stock II into separate 100 ml volumetric flasks and dilute to volume with inhibitor-free

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Last Review:    04/010/08 Next Review:    04/01/12	<b>Inhibitor</b>	Page 3 of 6 Reviewed by: Jennifer Young

acrylonitrile. This should give standards of about 12, 25, 37, and 50 ppm MEHQ.

4. Scan from 245 to 345 nm to find the maximum absorbance at about 295 nm. Measure the absorbance of each standard at the maximum using 1 cm cell against an empty 1 cm cell.
5. Prepare a calibration curve(see Attachment 1). Determine the slope of the calibration points by calculating the linear regression with the absorbance as the ordinate and ppm as the abscissa.
6. Enter the date, slope, and y-intercept in the appropriate log book.
7. The calibration curve for inhibitor will be re-determined yearly or anytime the instrument is found to be out of calibration.

## PROCEDURE

Fill the sample cell with sample and measure the absorbance on the spectrophotometer in exactly the same manner as the standards. Record the absorbance.

## CALCULATIONS

Calculate the ppm MEHQ present by calculating the value from the slope and y-intercept.

$$\text{ppmw} = \frac{1}{\text{slope}} \times \text{sample absorbance} - \text{y-intercept}$$

## REPORT

Report MEHQ to the nearest 1 ppmw.

Inhibitor - 39 ppmw.

## ATTACHMENTS

Attachment 1      Inhibitor Calibration Curve

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<b>Method:</b> ACRN-13 Revision: 5 Final Revision Date: 07/24/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review: 04/010/08	<b>Inhibitor</b>	Page 5 of 6
Next Review: 04/01/12		Reviewed by: Jennifer Young

Correlation \_\_\_\_\_

Date: \_\_\_\_\_ CALIBRATOR: \_\_\_\_\_  
COMMENTS:

**Attachment 2**  
**CALIBRATION REVIEW**

**Waste Water Spec:** Tolerances =  $\pm$  10%

	Old Calibration	New Calibration	Percent Difference
Slope			
Y- intercept			
Results @ 0.5 Absorbance			

Comments:

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**Acrylo Spec:** Tolerances =  $\pm$  10%

	Old Calibration	New Calibration	Percent Difference
Slope			
Y- intercept			
Results @ 0.5 Absorbance			

Comments:

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<b>Method:</b> <b>ACRN-13</b> Revision:        5    Final Revision Date: 07/24/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review:   04/010/08	<b>Inhibitor</b>	Page 6 of 6
Next Review:   04/01/12		Reviewed by: Jennifer Young

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