

<b>Method:</b> ACRN-12 Revision: 5 Final Revision Date: 05/23/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review: 08/15/07	<b>Hydrogen Cyanide</b>	Page 1 of 4
Next Review: 08/15/11		Reviewed by: Karen Runge

## METHOD SUMMARY

Hydrogen cyanide is extracted from the sample as a water soluble salt by means of a solution containing sodium hydroxide, ammonium hydroxide, and potassium iodide. The aqueous extract is then titrated with standard silver nitrate until all the cyanide is complexed as the soluble  $\text{Ag}(\text{CN})_2$ . The first excess of silver, the end point, is indicated visually with the formation of insoluble silver iodide (turbidity). The hydrogen cyanide content is calculated from the quantity of silver nitrate consumed.

## 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 C-10 (SOHIO Test Methods) "Hydrogen Cyanide in Acrylonitrile and Acetonitrile"

ASTM E1178-97(2002), "Standard Test Methods for Analysis of Acrylonitrile"<http://www.astm.org/>

## INTERFERENCES

There are no known interferences to this method.

Technical information contained herein is furnished without charge or obligation, and is given and accepted at recipient's sole risk. Because conditions of use may vary and are beyond our control, INEOS USA LLC makes no representation about, and is not responsible or liable for the accuracy or reliability of data, nor for toxicological effects or Industrial Hygiene requirements associated with particular uses of any product described herein. Nothing contained in this document shall be considered a recommendation for any use that may infringe patent rights, or an endorsement of any particular material, equipment, service, or other item not supplied by INEOS USA LLC. The "Properties" and "Applications" listed in this document are not specifications. They are provided as information only and in no way modify, amend, enlarge, or create any specification or warranty, and ALL WARRANTIES, EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION THE WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE, ARE EXCLUDED.

October 2006

©2006 INEOS USA LLC

This document is UNCONTROLLED. For the latest revision of this test method, visit <http://techservice.innovene.com> and select Acrylonitrile in "Browse by Product."

<b>Method:</b> ACRN-12	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Revision: 5 Final Revision Date: 05/23/03		
Last Review: 08/15/07	<b>Hydrogen Cyanide</b>	Page 2 of 4
Next Review: 08/15/11		Reviewed by: Karen Runge

## APPARATUS AND REAGENTS

1. **Separatory funnel**, 250 mL.
2. **Buret**, micro, 10 mL.
3. **Flasks, Erlenmeyer**, 250 mL.
4. **Cylinder, graduated**, 100 mL.
5. **Flasks, volumetric**, 1000 mL.
6. **Bottle**, plastic, 1000 mL.
7. **Balance**, top loading.
8. **Water**, ASTM, Type II, or equivalent. Minimum electrical resistivity 1.0 MΩ•cm at 298 K; maximum total organic carbon 50 µg/L; maximum sodium 5 µg/L; maximum chlorides 5 µg/L; maximum total silica 3 µg/L. Detailed specifications can be obtained from ASTM: [www.astm.org](http://www.astm.org).
9. **Silver Nitrate, 0.01N** - The solution may be prepared in the following manner. Dilute 100 mL of 0.1N AgNO<sub>3</sub> (standardized, Fisher SS72 or equivalent) to one liter with water.
10. **Potassium Iodide**, reagent grade, Fisher P 410 or equivalent.
11. **Sodium Hydroxide**, reagent grade, Fisher S318 or equivalent.
12. **Ammonium Hydroxide**, ACS reagent, Fisher A669 or equivalent.
13. **Caustic - Iodide Solution** - The solution may be prepared in the following manner: Dissolve 3.6g ± 1g of potassium iodide and 44.1g of sodium hydroxide in 700 mL of water. Add 180 mL of concentrated ammonium hydroxide and dilute to one liter with water. Solutions are stable for at least six months. When larger quantities are needed, it may be prepared in one of the following ways.
  - a. To make 45 gallons of solution: Dissolve 612g ± 1.0g of potassium iodide and 7500g ± 5g of sodium hydroxide in 31.5 ± 0.5 gallons of water. Add 8.1 ± 0.1 gallons of concentrated ammonium hydroxide. Dilute to 45 ± .5 gallons with water.
  - b. To make 40L of solution: Dissolve 144g of potassium iodide and 1764g of sodium hydroxide in 27L of water. Add 7200 mL of concentrated ammonium hydroxide. Dilute to 40L with water.

<b>Method:</b> <b>ACRN-12</b> Revision:        5    Final Revision Date: 05/23/03	<b>Acrylonitrile Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review:   08/15/07 Next Review:   08/15/11	<b>Hydrogen Cyanide</b>	Page 3 of 4 Reviewed by: Karen Runge

## CALIBRATION

The silver nitrate titrant is prepared from a purchased volumetric standard, which is standardized against NIST reference material. Under normal circumstances it is not necessary to re-standardize this titrant.

## PROCEDURE

1. Add 100 mL of caustic iodide to a 250 mL Erlenmeyer flask using the graduated cylinder.
2. Blank the caustic iodide by slowly titrating with 0.01N AgNO<sub>3</sub> to a slight opalescence that persists.
3. Transfer the contents of the Erlenmeyer flask to 250 mL separatory funnel. Add 100 mL of sample with a graduated cylinder. CAUTION: Avoid breathing acrylonitrile vapors or ammonia vapors from the caustic iodide reagent. Use a well ventilated hood.
4. Stopper and shake the separatory funnel for 1 minute ± 10 seconds: Be sure to vent the funnel to relieve pressure. Then let the funnel stand in a holder until the layers separate.
5. Draw off the bottom (aqueous) layer into a 250 mL Erlenmeyer flask.
6. Slowly titrate contents of the Erlenmeyer flask with standard 0.01N AgNO<sub>3</sub> until an opalescence is obtained. Record this volume of AgNO<sub>3</sub>.

## CALCULATIONS

Calculate the ppm HCN in the sample as follows:

$$\text{HCN, ppm} = \frac{(\text{mL AgNO}_3)(\text{N AgNO}_3)(0.054) \times 10^6}{(\text{mL sample})(0.807 \text{ gms AN/mL})}$$

<b>Method:</b> <b>ACRN-12</b> Revision:        5    Final Revision Date: 05/23/03	<b>Acrylonitrile  Specification Tests</b>	<b>INEOS Nitriles</b>
Last Review:   08/15/07	<b>Hydrogen Cyanide</b>	Page 4 of 4
Next Review:   08/15/11		Reviewed by: Karen Runge

Where:    0.054 = milliequivalent wt of HCN

$$\text{Or: HCN, ppm} = \frac{(\text{mL AgNO}_3)(N \text{ AgNO}_3)(66914)}{(\text{mL sample})}$$

## REPORT

Report ppm HCN to the nearest 0.1 ppm.  
Example: HCN, ppm = 2.1