

Method: ACRN-21 Revision: 5 Final Revision Date: 03/26/03	Acrylonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

The pH of a 5% solution of acrylonitrile in water is determined. The solution is then titrated with 0.1N H₂SO₄ until a pH of 4 is reached. The volume of acid required to obtain a pH 5.0 is determined from a titration curve and is known as the "titration value". Values below 0.2 mL are reported as <0.2 mL.

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-9 (SOHIO Test Method), "Titration Value" of Acrylonitrile"

INTERFERENCES

There are no known interferences to this method.

APPARATUS AND REAGENTS

1. **pH meter.**
2. **pH and Reference electrodes.**
3. **Stirrer**, magnetic, variable speed.
4. **Stirring bar**, Teflon magnetic.
5. **Graduated cylinder**, 50 mL.

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6. **Beaker**, 1000 mL.
7. **Buret**, 10 mL.
8. **Dropper bottles**, containing 0.01N NaOH and 0.01 N H₂SO₄.
9. **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.
10. **Reference buffer solutions**, pH 7, pH 4.
11. **Sulfuric acid 0.1N**, ACS grade.

CALIBRATION

The pH meter should be calibrated using procedure CAL-3.

PROCEDURE

1. Use a calibrated pH meter.
2. Use the solution from the ACRN-19 pH procedure, or prepare a pH 7 solution as follows:
 - a. Add 760 mL water into a 1000 mL beaker and place on the magnetic stir plate.
 - b. Add stirring bar and bring to pH 7.00±0.1 with dropwise addition of 0.01N NaOH or 0.01N H₂SO₄, only if necessary.
3. With a graduated cylinder, measure 50.0 mL of sample; add this to the stirring water. Stir briskly but avoid entrainment of air bubbles.
4. Take initial pH reading when acrylonitrile is dissolved. Solution will turn from cloudy to clear.
5. Add 0.1N H₂SO₄ in increments using a buret.
 - When values are determined from a titration curve, titrate until the pH is less than 4. Record pH readings for each incremental addition of acid.
 - Alternately, the titration value may be read directly when the pH reaches 5.0.

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

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CALCULATIONS

1. For the titration curve approach, plot pH readings vs. volume acid titrant used and draw best curve.
2. Record pH of 5% aqueous solution as being the initial pH (this is the same as method ACRN-19, pH).
3. From the titration curve, read the volume of titrant used to bring the aqueous solution to 5.0 pH. This volume is the "titration value".

REPORT

Report titration value to nearest 0.1 mL. If titration value is less than 0.2 mL then report as <0.2 mL.

For example: Titration Value = 0.4 mL.