

Method: ACEN-2 Revision: 5 Final Revision Date: 11/17/03	Acetonitrile Specification Tests	INEOS Nitriles
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METHOD SUMMARY

A sample is distilled in equipment meeting the specifications of ASTM D86-54. The temperatures (centigrade) of the first drop and 97 mL point are recorded and corrected to 760 mm pressure. These temperatures are the distillation range. They are reported to the nearest 0.1C.

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 C1-74 (SOHIO Test Methods), "Distillation Range of Acrylonitrile and Acetonitrile"

ASTM D86-02 (for Table 2 Approximate Corrected Thermometer Readings) "Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure" <http://www.astm.org/>

ASTM D850-03, "Standard Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials"

ASTM D1078-01, (for apparatus and equations), "Standard Test Method for Distillation Range of Volatile Organic Liquids"

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ASTM E-133-92(2000)E1, "Standard Specification for Distillation Equipment"

APPARATUS AND REAGENTS

1. **Distillation apparatus**, Precision Scientific Type 74731, for distillation or equivalent. Apparatus to include 100 mL Engler flask, seamless brass condenser, insulated cooling bath of at least 5.5 liters capacity, an SS shield, a variable transformer type heater control, an asbestos flask support, a 100 mL graduated receiver cylinder, and centigrade thermometer graduated in no more than 0.2°C increments and certified to NIST.
2. **Boil chips**, such as ceramic saddles.
3. **Barometer**, calibrated, traceable to NIST.

PROCEDURE

1. Measure 100 mL of sample and transfer to the 100 mL distillation flask and add 3 ceramic saddles or boiling chips.
2. Set up the apparatus according to ASTM D86-54.
3. Start heating and record the temperature of the first drop into the collection cylinder.
4. Maintain a distillation rate of 4 to 5 mL/minute and record the temperature at the 97 mL point in the collection cylinder.
5. Discontinue heating immediately when 97 mL of distillate has been recovered.

CALCULATIONS

Correct the temperatures for atmospheric pressure by adding 0.042°C for each mm. of pressure below 760 and subtracting 0.042°C for each mm above 760.

Distillation Range:

$$\text{Deg.C} = (\text{observed Temperature}) + (760 - P) 0.042$$

Where

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760 = Standard Barometric pressure, mm Hg
P = Barometric Pressure, mm of Hg
0.042 = Correction factor for liquids ASTM D-86, Table II.

REPORT

Report the following corrected thermometer readings to the nearest tenth of a deg C.

Example: Initial boiling point = 81.4°C
97% recovered temperature = 82.4°C