

# AZELAIC ACID

5019

HOOC(CH<sub>2</sub>)<sub>7</sub>COOH

MW: 188.25

CAS: 123-99-9

RTECS: CM1980000

**METHOD:** 5019, Issue 2

**EVALUATION:** PARTIAL

**Issue 1:** 15 May 1985

**Issue 2:** 15 August 1994

**OSHA :** no PEL  
**NIOSH:** no REL  
**ACGIH:** no TLV

**PROPERTIES:** solid; d 1.225 g/mL @ 25 °C;  
 MP 106.5 °C; BP >360 °C (decomposes)

**SYNONYMS:** nonanedioic acid; 1,7-heptanedicarboxylic acid; lepargylic acid

**APPLICABILITY:** The working range is 2 to 20 mg/m<sup>3</sup> for a 500-L air sample. The measurement range can be lowered if adequate recoveries are found. The method may be applicable to other straight-chain aliphatic dicarboxylic acids.

**INTERFERENCES:** Moisture decomposes both the trimethylsilyl reagents and their corresponding derivatives.

**OTHER METHODS:** This is P&CAM 256 [2] in a revised format.

**REAGENTS:**

1. N,O-bis(trimethylsilyl)trifluoro-acetamide (BSTFA) containing 1% trimethylchlorosilane (TMCS) obtainable from chromatography products suppliers.  
NOTE: Store in desiccator.
2. Pyridine, spectroquality grade.\*
3. Ethanol, spectroquality grade.
4. Azelaic acid, highest purity available.
5. Helium, purified.
6. Hydrogen, purified.
7. Air, "zero grade."

\* See Special Precautions

**EQUIPMENT:**

1. Sampler: PVC membrane filter, 5- $\mu$ m pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator and column (page 5019-1).
4. Vacuum oven or hotplate.
5. Analytical balance, 0.01 mg.
6. Waterbath, 70 °C.
7. Vials, glass, 20-mL, PTFE-lined screw caps.
8. Pipets, 1- and 5-mL.
9. Syringes, 10- $\mu$ L.
10. Tweezers, microspatula, weighing paper and an applicator stick.

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**SPECIAL PRECAUTIONS:** Pyridine may cause central nervous system depression and irritation of the skin and respiratory tract [3]. Work must be performed in a fume hood.

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**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 3 L/min for a sample size of 200 to 1000 L.
3. Cap the samplers with plastic plugs and pack securely for shipment.

**SAMPLE PREPARATION:**

4. Remove filter from cassette holder with tweezers and insert in a glass vial. Push it gently to the bottom of the vial.
5. Pipet 5.0 mL ethanol into the vial. Cap the vial.
6. Place the vial in a 70 °C waterbath for 20 min. Shake the vial every 5 min.  
NOTE: Use of an ultrasonic bath is not recommended because it breaks up the PVC filter.
7. Lift the filter with tweezers so it is above the ethanol level in the vial. Rinse the filter with ten 1-mL aliquots of ethanol. Discard the filter.
8. Place the vial in a vacuum oven or on a hotplate at 45 to 50 °C. Evaporate to dryness.
9. Derivatize.
  - a. Add 1 mL pyridine to the vial to dissolve the residue.
  - b. Add 1 mL BSTFA mixture to the vial. Shake the vial for 1 min.  
NOTE: Avoid working in a humid atmosphere because water decomposes the trimethylsilyl reagent and derivative.
  - c. Cap the vial. Place the vial in a waterbath at 70 °C for 20 min. Shake the contents of the vial every 5 min.

**CALIBRATION AND QUALITY CONTROL:**

10. Calibrate daily with at least six working standards over the range 0.001 to 10 mg azelaic acid per sample.
  - a. Add weighed amounts of azelaic acid to marked vials containing 5 mL ethanol.
  - b. Evaporate and derivatize (steps 8 and 9).

- c. Analyze together with samples and blanks (steps 12 and 13).
  - d. Prepare calibration graph (peak area vs. mg azelaic acid).  
NOTE: If azelaic acid is not 100% pure, correct the mass of azelaic acid standards before preparation of calibration graph.
11. Determine the recovery of azelaic acid from PVC filters at least once for each lot of filters used.
    - a. Weigh various amounts of azelaic acid to cover the range of interest onto PVC filters in marked vials.
    - b. Treat as in steps 5 through 9 and 12 and 13.

**MEASUREMENT:**

12. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5019-1. Inject a 1- $\mu$ L sample.
13. Measure peak area after each injection. Calculate the mean area.  
NOTE: Remove daily the white flakes which accumulate on the FID.

**CALCULATIONS:**

14. Determine the mass, mg, of azelaic acid (W) per filter and the average media blank (B) from the calibration graph.
15. Calculate the concentration, C, of azelaic acid in the air volume sampled, V (L):

$$C = \frac{W - B}{V} \cdot 10^3, \text{ mg/m}^3.$$

**EVALUATION OF METHOD:**

The measurement precision,  $\bar{S}_r$ , of this method was determined to be 0.02 using standards in the range 1 to 10 mg per sample. The average recovery of azelaic acid from spiked PVC filters in the range 2 to 10 mg per filter was  $94.9 \pm 4.3\%$  [1].

**REFERENCES:**

- [1] Palassis, J. The Sampling and Determination of Azelaic Acid in Air, Am. Ind. Hyg. Assoc. J., **39**, 731-736 (1978).
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 256, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, USDHHS Publ. (NIOSH) 81-123 (1981).

**METHOD REVISED BY:**

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