CYANURIC ACID

$C_3N_3(OH_3)$ MW: 129.08 CAS: 108-80-5 RTECS: XZ1800000

METHOD: 5030, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA : no standard
NIOSH: no recommended exposure limit
ACGIH: no TLV

PROPERTIES: solid; d 2.50 g/mL @ 20 °C; MP >330 °C; (decomposes to cyanic acid); VP not significant

SYNONYMS: 2,4,6-trihydroxy-1,3,5-triazine; isocyanuric acid; 1,3,5-triazine-2,4,6(1 H,3H,5H)trione.

SAMPLING

SAMPLER: Filter (5-µm PVC membrane)

FLOW RATE: 1 to 3 L/min

VOL-MIN: 10 L @ 0.1 mg/m^3
-MAX: 1000 L

SHIPMENT: routine

SAMPLE STABILITY: ≥ 69 days at 25 °C [1]

FIELD BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: not studied

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): not determined

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: HPLC, UV DETECTION

ANALYTE: cyanuric acid

EXTRACTION: 3 mL 0.005 M Na_2HPO_4, 5:95 (v:v) methanol:water (pH = 7.0), 10 min ultrasonic

INJECTION VOLUME: 15 µL

MOBILE PHASE: 0.005 M Na_2HPO_4, 5:95 (v:v) methanol:water (pH = 7.0)

COLUMN: µ-Bondapak C_{18}, 10-µm particle, size; Radial PAK cartridge, 11 cm x 8-mm ID

DETECTOR: UV, 225 nm

CALIBRATION: standard solutions of cyanuric acid in mobile phase

RANGE: 1 to 750 µg per sample

ESTIMATED LOD: 0.3 µg per sample

PRECISION ($\hat{S}_r$): 0.020 @ 12 to 412 µg per sample [1]

APPLICABILITY: The working range is 0.01 to 10 mg/m^3 for a 100-L air sample.

INTERFERENCES: Trichloroisocyanuric acid interferes because it reacts with water (which is present in the eluent used for recovery) to form cyanuric acid.

OTHER METHODS: None identified for air analysis. HPLC method for measurement in solution is a variation of the method of Briggle et al. [2].

REAGENTS:

1. Cyanuric acid, ≥98% pure.*
2. Water, distilled.
3. Methanol, chromatographic quality.*
4. $Na_2HPO_4 \cdot 7H_2O$, reagent grade.
5. HCl, 1 M (aqueous).
6. Eluent and mobile phase: 0.005 M Na_2HPO_4, 5:95 (v:v) methanol:water at pH = 7.0. Dissolve 5.36 g of $Na_2HPO_4 \cdot 7H_2O$ in 2000 mL water. Add 200 mL methanol. Add water until total volume is 3990 mL. Add 1 M HCl until pH is 7.0.
7. Calibration stock solution, 1 mg/mL. Dissolve 25 mg cyanuric acid in eluent to make 25 mL solution. Prepare fresh monthly.
   NOTE: Ultrasonic bath will facilitate dissolution of cyanuric acid.
8. Recovery stock solution, 1.7 µg/µL. Dissolve 42.5 mg cyanuric acid in water to make 25 mL solution.
   NOTE: Ultrasonic bath will facilitate dissolution of cyanuric acid.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm, 5-µm polyvinyl chloride (PVC) membrane filter in 2-piece cassette filter holder.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. High performance liquid chromatograph (HPLC), UV absorption detector, 225 nm, recorder, intergrator and column (page 5030-1).
4. Vials, 4-mL, with PTFE-lined caps.
5. Volumetric flasks, 25-mL.
6. Carboy, 4-L.
7. Graduated cylinders, 1-L, readable to 10-mL; 250-mL, readable to 2 mL.
8. Syringes, 500-µL, readable to 10 µL; 100-µL, readable to 1 µL; 10-µL, readable to 0.1 µL.
9. Beakers, 50-mL, internal diameter ≥37 mm.
10. Ultrasonic water bath.
11. Film, plastic, flexible, water-resistant.
12. Filter units, 25-mm PTFE membrane, 0.45-µm pore size, in polypropylene housing.
13. Vacuum oven.
14. Tweezers.
15. pH meter.
16 Adhesive tape.

SPECIAL PRECAUTIONS: Cyanuric acid is a possible tumorigenic agent and a slight eye irritant [3,4]. Methanol is toxic. Ingestion of methanol may cause blindness or death. Methanol is a fire hazard (flash point 12 °C).

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 1 and 3 L/min for a total sample size of 10 to 1000 L. Limit the maximum loading of particulate matter on the filter to about 1 mg.
4. Seal the ends of the sampler with plugs. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the PVC filter face down into a 50-mL beaker (the filter should lie flat on the bottom of the beaker). Retain the cassette filter holder (step 9).
6. Add 3 mL eluent to the beaker. Seal the mouth of the beaker with plastic film.
   NOTE: The volume of 3 mL is sufficient when the quantity of cyanuric acid on the filter is 1 mg or less. The limit of solubility of cyanuric acid in eluent is about 2 mg/mL at room temperature.
7. Place the beaker into an ultrasonic bath for 10 min.

8. Filter the solution.
9. If cyanuric acid is found on the PVC filter (steps 13 and 14), prepare additional sample solution by treatment of the interior surface of the front piece of the cassette filter holder with eluent.
   NOTE: The quantity of cyanuric acid collected on the interior surface of the front piece of the cassette filter holder may be significant (see EVALUATION OF METHOD).
   a. Use adhesive tape to secure in place the plug in the inlet of the front piece of the cassette filter holder.
   b. Add eluent (ca. 6 mL) to the front piece until about 95% of the interior surface is in contact with eluent.
   c. Place the front piece into an ultrasonic bath for 5 min.
   d. Transfer the solution from the front piece to a beaker, seal the mouth of the beaker with plastic film, and place the beaker into an ultrasonic water bath for 10 min.
      NOTE: Solid particles may be present in the solution after the first 5 min of ultrasonic agitation. Transfer of the solution to a beaker helps protect the solution from contamination.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards over the range 0.1 to 250 mg cyanuric acid per mL of solution.
    a. Prepare a series of working standards in vials from calibration stock solution and eluent by serial dilution.
    NOTE: Working standards may be stored at -3 °C for at least 18 days without deterioration.
    b. Analyze together with samples and blanks (steps 13 and 14).
    c. Prepare calibration graph (peak area or height vs. µg of cyanuric acid).
11. Determine recovery (R) at least once for each lot of PVC membrane filters used for sampling in the calibration range (step 10). Prepare three filters at each of five levels plus three media blanks.
    a. Place an aliquot of recovery stock solution onto a PVC filter which is situated in the back piece of a cassette filter holder. If the volume of the aliquot is greater than 15 µL, transfer the aliquot to the filter in portions which are about 15 µL in size.
    NOTE: Portions of stock solution will appear as beads on the filter. A maximum of about sixteen 15-µL portions of solution can be distributed on the filter. Thus, a maximum of about 400 mg of cyanuric acid can be applied to the filter with the recovery stock solution. A recovery stock solution which has a much higher concentration of cyanuric acid in water at room temperature can not be prepared because the limit of solubility of cyanuric acid in water at room temperature is about 2.5 mg/mL. If higher loadings are needed, allow the first portions to dry and repeat the process.
    b. Join the front piece of the cassette filter holder with the back piece. Remove the plug from the inlet.
    c. Carefully transfer the filter holder to a vacuum oven.
    d. Dry the filter at about 65 °C and 40 kPa (300 mm Hg). Filter will be dry in ca. 0.3 to 4.5 h.
    e. Prepare sample (steps 5 through 8) and analyze with working standards (steps 13 and 14).
    f. Prepare a graph of R vs. µg cyanuric acid recovered.
12. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph is in control.

MEASUREMENT:

13. Set liquid chromatograph to manufacturer's recommendations and to conditions given on page 5030-1. Inject sample aliquot manually or with autosampler.
    NOTE: If peak area is above the range of the working standards, dilute with eluent, reanalyze, and apply the appropriate dilution factor in calculations.
14. Measure peak area or peak height.

CALCULATIONS:

15. Determine the mass, µg (corrected for R), of cyanuric acid found on the filter (W_f) and on the average media blank (B_f).
16. Determine the mass, µg, of cyanuric acid found on the interior surface of the cassette filter holder (W_c) and on the interior surface of a blank cassette filter holder (B_c).
17. Calculate concentration, C, of cyanuric acid in the air volume sampled, V (L):

$$C = \frac{(W_f + W_c - B_f - B_c)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Average recoveries after fortification of 37-mm PVC membrane filters with 12-, 36-, and 412-µg quantities of cyanuric acid were 0.98, 1.00, and 1.00, respectively; precision ($\bar{S}_r$) was 0.020 (16 samples, pooled). The average recovery of 36-µg quantities of cyanuric acid from PVC filters after 22 days of storage at room temperature was 1.08; $\bar{S}_r$ was 0.065 (5 samples). Recovery of 1.08 was not significantly different from 1.00 at the 95% confidence level. Recoveries of 10-, 80-, 200-, and 400-µg quantities of cyanuric acid were 0.88, 1.04, 0.98 and 0.98, respectively, after storage of fortified PVC filters for 69 days at room temperature (one sample at each level). This method was not evaluated with controlled atmospheres in a laboratory. However, the method was employed for measurement of cyanuric acid in air at a plant in which trichloroisocyanuric acid was manufactured from cyanuric acid [1,5]. Significant quantities (ca. 40% of the totals) of cyanuric acid were found on interior surfaces of the front pieces of cassette filter holders.

Working standards of cyanuric acid at concentrations near 1 µg/mL in eluent deteriorated in about 3 weeks during storage at room temperature; standards were stable for at least 18 days during storage at - 3 °C. Deterioration of a C_{18} analytical column took place and caused the LOD of cyanuric acid to increase from 0.1 to 0.25 µg/mL during 6 weeks.

Trichloroisocyanuric acid is an interference because it reacts with water (present in the eluent) to form cyanuric acid. Average yields of cyanuric acid after treatment of 8.4-, 64-, and 424-µg quantities of trichloroisocyanuric acid with eluent in glass vials were 74%, 89%, and 93%, respectively.

REFERENCES:

[1] Tucker, S.P., and L.M. Blade. Anal. Lett. 25 (12), 2265-2277 (1992).
[2] Briggle, T. V., L. M. Allen, R. C. Duncan, and C. D. Pfaffenberger. J. Assoc. Off. Anal. Chem., 64 (5), 1222-1226 (1981).
[3] Canelli, E. Amer. J. Pub. Health. 64 (2), 155-162 (1974).
[4] Hammond, B. G., S. J. Barbee, T. Inoue, N. Ishida, G. J. Levinskas, M. W. Stevens, A. G. Wheeler and T. Cascieri. Environ. Health Perspect., 69, 287-292 (1986).
[5] Tucker, S. Analytical Report for DPSE/MRSB Analytical Sequence #6426, NIOSH, Unpubl. (1989).

METHOD WRITTEN BY:

Samuel P. Tucker, Ph.D., NIOSH/DPSE.