TRIMELLITIC ANHYDRIDE 5036

$C_9H_4O_5$ MW: 192.13 CAS: 552-30-7 RTECS: DC2050000

METHOD: 5036, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 August 1994**

OSHA : No PEL
NIOSH: 0.005 ppm/10 h
ACGIH: 0.005 ppm
(1 ppm = 7.86 mg/m^3 @ NTP)

PROPERTIES: solid; MP 161 to 163 °C;
VP 5.3 x 10^{-4} Pa (4 x 10^{-6} mm Hg)
@ 20 °C

SYNONYMS: anhydrotrimellitic acid; 1,3-dioxo-phthalancarboxylic acid; 1,3-dihydro-1,3-dioxo-5-isobenzofurancarboxylic acid; 1,2,4-benzenetricarboxylic acid anhydride; 1,2,4-benzenetricarboxylic acid cyclic-1,2-anhydride.

SAMPLING

SAMPLER: FILTER (0.8-µm PVC-copolymer)

FLOW RATE: 1.5 to 2.0 L/min

VOL-MIN: 400 L
-MAX: not specified

SHIPMENT: routine

SAMPLE STABILITY: unknown

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.048 to 0.24 mg/m^3 [1] (400-L samples)

BIAS: not significant

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

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

EXTRACTION: methanol, 3 mL

DERIVATIZATION: boron trifluoride/methanol

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 315 °C
-DETECTOR: 325 °C
-COLUMN: 250 °C, 2 min; 16 °C/min; 310 °C, 4 min

COLUMN: stainless steel, tenax 60/80 mesh, (9 ft x 2-mm)

CARRIER GAS: Helium, 35 mL/min

CALIBRATION: standard solutions of trimethyl trimellitate (derivative) in a mixture of pyridine, diethyl ether, and pentane

RANGE: 19 to 96 µg per sample

ESTIMATED LOD: 2 µg per sample

PRECISION ($\hat{S}_r$): 0.087 @ 19.2 to 96 µg per sample

APPLICABILITY: The working range is 0.048 to 0.24 mg/m^3 for a 400-L air sample. The method was used for analysis of bulk samples and air (field samples) [2,3]. This method will not differentiate between trimellitic anhydride and trimellitic acid [1].

INTERFERENCES: Any compound with the same retention time is an interference. The interference may be eliminated by changing the temperature program or carrier gas flow rate.

OTHER METHODS: This revises Method P&CAM 322 [1], which was based on a procedure by Biondi and Cagnosso [4].

REAGENTS:

1. Methanol; ACS reagent grade.
2. Pentane, ACS reagent grade.*
3. Diethyl ether, ACS reagent grade.*
4. Boron trifluoride/methanol, 14° (w/v) mixture* (commercially available from GC suppliers).
5. Pyridine, ACS reagent grade.
6. Pyridine mixture,* prepare a 7:3 pyridine/pentane mixture in a screw-cap tube.
7. Trimellitic anhydride.
8. Calibration stock solution, 1.92 µg/µL. Dissolve 0.4803 g of trimellitic anhydride in methanol and dilute to 250 mL.
9. Hydrogen, prepurified.
10. Air, compressed, filtered.
11. Helium, purified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm, PVC-copolymer filter (0.8-µm pore size) supported by a cellulose backup pad in a three-piece plastic filter holder.
2. Personal sampling pump, 1.5 to 2 L/min with flexible polyethylene or PTFE tubing.
3. Gas Chromatograph, FID, integrator and column.
4. Test tubes, glass, (13 x 100-mm) with teflon-lined screw caps.
5. Syringe, 10-µL.
6. Pipettes, 10-µL, 25-µL, and 0.25 - 5-mL.
7. Volumetric flask, 250-mL.
8. Beakers, 50-mL and 250-mL.
9. Hotplate, variable heat settings.
10. Watch Glasses, for 50-mL beakers.
11. Ultrasonic bath.
12. Septa, high temperature (350 °C), GC injection.
13. Analytical balance, to ± 0.01 mg.
14. Tweezers
15. Applicator stick.
16. Weighing paper.

SPECIAL PRECAUTIONS: Diethyl ether and pentane are extremely flammable. Boron trifluoride and pyridine are toxic. Work with these substances only in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Connect the filter holder to the pump with a piece of flexible tubing.
3. Sample at an accurately known flow rate between 1.5 and 2 L/min for a total sample size of 400 L or greater. Collect a larger sample if a qualitative analysis is to be performed.
4. Replace filter plugs, and pack securely for shipment.

SAMPLE PREPARATION:

5. Extraction:
   a. Place a field sample or spiked filter sample in a 50-mL beaker.
   b. Add 3 mL of methanol.
   c. Cover the beaker with a watch glass and place on a hot plate (65 °C) for one minute.
   d. Remove the beaker with the watch glass still in place and place in the ultrasonic bath for one minute.
   e. Rinse the bottom of the watch glass with 1 mL of methanol, collect the rinse in the beaker.
   f. Lift the filter with tweezers above the methanol level and rinse both sides of the filter slowly with two 3-mL aliquots of methanol, collecting the rinse in the beaker.
   g. Roll and squeeze the filter with tweezers against the inside wall of the beaker to remove methanol retained by the filter.
   h. Discard the filter.
   i. Place the beaker on the hot plate (55 °C) and evaporate the methanol to volume less than 5 mL.
   j. Transfer the methanol extract to a test tube.

k. Rinse the beaker two or three times with small amounts of methanol and combine the rinsings in the test tube.
l. Evaporate the methanol in the test tube to dryness.

6. Derivatization:
   a. Add 125 µL of BF_3/methanol to each sample or standard tube and screw the cap tightly.
   b. Place in a water bath (97 °C) for 20 min.
   c. Remove the tube and allow to cool to room temperature.
   d. Add 50 µL of diethyl ether to the tube. Screw the cap on and shake mildly.
   e. Add 50 µL of pentane, replace cap and shake mildly.
   f. Add 50 µL of pyridine, replace cap and shake mildly.
   g. Allow to stand for a few minutes until the white material precipitates.

CALIBRATION AND QUALITY CONTROL:

7. Prepare at least six working standards over the range of 19.2 to 96 µg (10 to 50 µL) of trimellitic anhydride.
   a. Add known amounts of calibration stock solution to test tubes.
   b. Derivatize according to step 6.
   c. Prepare a calibration graph (peak area vs. µg analyte).
8. Determine recovery (R) for each lot of filters used for sampling in the concentration range of interest (step 7). Prepare three filters at each of five levels plus three media blanks.
   a. Spike aliquots of calibration solution onto the center of each filter. Allow to dry overnight.
   b. Extract and derivatize (steps 5 and 6) and analyze (steps 10 through 12).
9. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration and recovery graphs are in control.

MEASUREMENT:

10. Set gas chromatographic conditions as given on page 5036-1.
11. Inject a 5 µL aliquot.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass of trimellitic anhydride, µg (corrected for recovery), for sample (W) and average media blank (B).
14. Calculate the concentration of trimellitic anhydride, C (mg/m^3) in the air volume sampled, V(L):

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

EVALUATION OF METHOD:

The extraction efficiency was evaluated over the range of 19.2 to 96 µg/sample levels with an average recovery of 103.3% and a measurement precision of 0.087.

REFERENCES:

[1] NIOSH Manual of Analytical Methods, V.6, P&CAM 322. U.S. Department of Health, Education and Welfare, (NIOSH) Publication No. 80-125 (1980).
[2] Sequence 1607, NIOSH (unpublished, 1979).
[3] Sequence 3632, Utah Biomedical Research Laboratory, Salt Lake City, Utah (unpublished, 1982).
[4] Biondi, P.A., and M. Cagnosso. Journal of Chromatography :109, 389 (1975).

METHOD REVISED BY:

Robert Kurimo, DPSE, NIOSH.