

2,4,7-TRINITROFLUOREN-9-ONE

5018



MW: 315.20

CAS: 129-79-3

RTECS: LL9100000

METHOD: 5018, 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 yellow needles; MP 176 °C

SYNONYMS: TNF

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.5-µm, PTFE membrane)	TECHNIQUE:	HPLC, UV DETECTION
FLOW RATE:	1 to 3 L/min	ANALYTE:	2,4,7-trinitrofluoren-9-one
VOL-MIN:	100 L @ 2 µg/m ³	PREPARATION:	extract with 2 mL toluene; centrifuge
-MAX:	500 L	INJECTION VOLUME:	100 µL
SHIPMENT:	routine	MOBILE PHASE:	20% isooctane/80% methylene chloride; 2 mL/min; ambient temperature
SAMPLE STABILITY:	at least 2 weeks @ 25 °C	COLUMN:	Waters Radial PAK B (packed with 10-µm silica) with Radial Compression Module, or equivalent
BLANKS:	2 to 10 field blanks per set	DETECTOR WAVELENGTH:	280 nm
ACCURACY		CALIBRATION:	standard solutions of TNF in toluene
RANGE STUDIED:	not studied	RANGE:	0.2 to 4 µg per sample
BIAS:	none identified	ESTIMATED LOD:	0.04 µg per sample [1]
OVERALL PRECISION (Ŝ_{r,T}):	not evaluated	PRECISION (Ŝ_r):	0.056 [1]
ACCURACY:	not determined		

APPLICABILITY: The working range is 0.4 to 100 µg/m³ for a 500-L air sample. This method was developed for the determination of TNF in spent toner from business copy machines.

INTERFERENCES: None known.

OTHER METHODS: This revises P&CAM 348 [1].

REAGENTS:

1. Isooctane, HPLC grade.
2. Methylene chloride, HPLC grade.
3. Toluene.
4. Calibration stock solution, 0.05 mg/mL. Dissolve 0.5 mg 2,4,7-trinitrofluoren-9-one* (TNF)* in 10 mL toluene.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: PTFE filter, 0.5- μ m pore size, 37-mm diameter, with backup pad and in polystyrene cassette.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tube.
3. Liquid chromatograph with UV detector, recorder, integrator, autosampler or syringe, and column (page 5018-1)
4. Ultrasonic waterbath.
5. Centrifuge.
6. Test tubes, glass, 10-mL, screw-cap.
7. Pasteur pipets.
8. Volumetric flasks, 10-mL
9. Syringes or micropipets, 5- to 100- μ L
10. Pipets, 2-mL, glass, delivery, with pipet bulb.
11. Tweezers

SPECIAL PRECAUTIONS: Trinitrofluorenone is a suspect carcinogen and mutagen.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Remove cassette plugs and attach cassette 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 100 to 500 L.
4. Plug the inlet and outlet of the cassette. Pack securely for shipment.

SAMPLE PREPARATION:

5. Remove filter from cassette. Fold or roll the filter and slide it to the bottom of a test tube with tweezers.
6. Add 2.0 mL toluene to each test tube and seal.
7. Suspend test tube with filter in an ultrasonic bath. Agitate for 5 min.
8. Centrifuge sample at ca. 2500 rpm for 30 min.
9. Transfer an aliquot of clear sample liquid to a clean test tube or autosampler vial.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards over the range 0.04 to 4 μ g TNF per sample.
 - a. Add known amounts of calibration stock solution to toluene in 10-mL volumetric flasks and dilute to the mark to produce concentrations of 0.02 to 2 μ g/mL.
 - b. Analyze together with samples and blanks (steps 13 through 15).
 - c. Prepare calibration graph (peak area vs. μ g TNF).

11. Determine recovery (R) over the calibration range (step 10) at least once for each lot of filters used for sampling. Prepare three filters at each of five levels plus three blanks.
 - a. Add a known amount of TNF in toluene to media blank filters. Allow to dry.
 - b. Place filters in test tubes and allow to stand overnight.
 - c. Extract filters (steps 5 through 9) and analyze together with working standards (steps 13 through 15).
 - d. Prepare a graph of R (mass recovered/mass added) vs. μg TNF recovered.
12. Analyze three quality control blind spikes and three analyst spikes to ensure that calibration graph and recovery graph are in control.

MEASUREMENT:

13. Set up the liquid chromatograph according to manufacturer's recommendations and to the conditions given on page 5018-1.
14. Inject sample aliquot using syringe, fixed volume sample loop or autosampler.
15. Measure peak area.
NOTE: If peak area is above the range of the standards, dilute with toluene, reanalyze, and apply the appropriate dilution factor in calculations.

CALCULATIONS:

16. Determine the mass, μg (corrected for R) of TNF found in the sample (W) and in average media blank (B).
17. Calculate concentration, C, of TNF in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Lab-tested with 2,4,7-trinitrofluoren-9-one spiked filters. The average recovery was 95% over the range 0.2 to 4.0 μg per sample and had a pooled precision (\bar{S}_r) of 0.056. PTFE filters with a loading of 0.4 μg were stored for two weeks at room temperature with no loss of analyte [1,2].

REFERENCES:

- [1] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 7, P&CAM 348, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
- [2] Seymour, M. J. *J. Chrom.*, 236, 530-534 (1982).

METHOD WRITTEN BY:

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