ETHYLENE GLYCOL DINITRATE
See NITROGLYCERINE (Method 2507) for Procedure

$O_2NOCH_2CH_2ONO_2$ MW: 152.06   CAS: 628-96-6   RTECS: KW5600000

METHOD: 2507, Issue 2   **EVALUATION: FULL**   **Issue 1: 15 February 1984**
                                                                 Issue 2: 15 August 1994

OSHA : C 0.2 mg/m^3 (skin)
NIOSH: STEL 1 mg/m^3 (skin)
ACGIH: 0.31 mg/m^3 (skin)
           (1 ppm = 6.22 mg/m^3 @ NTP)

PROPERTIES: liquid; MP -22 °C; explodes 114 °C;
d 1.49 g/mL @ 25 °C; VP 6.8 Pa
(0.05 mm Hg; 68 ppm) @ 20 °C

SYNONYMS: EGDN; ethylene dinitrate.

SAMPLING	MEASUREMENT
SAMPLER: SOLID SAMPLER TUBE (Tenax-GC, 100 mg/50 mg)	**TECHNIQUE:** GAS CHROMATOGRAPHY, ECD
	ANALYTE: EGDN
FLOW RATE: 0.2 to 1.0 L/min	**DESORPTION:** 2 mL ethanol; stand 30 min
VOL-MIN: 3 L @ 1 mg/m^3 **-MAX:** 100 L	**INJECTION VOLUME:** 2 µL
SHIPMENT: routine	**TEMPERATURE-INJECTION:** 160 °C **-DETECTOR:** 280 °C **-COLUMN:** 125 °C
SAMPLE STABILITY: at least 25 days @ 25 °C [5]	
BLANKS: 2 to 10 field blanks per set	**CARRIER GAS:** 95% argon/methane, 75 mL/min
	COLUMN: glass, 1 m x 4-mm, 2-mm ID 10% OV-17 on 60/80 mesh Gas Chrom Q

ACCURACY	MEASUREMENT (cont.)
	CALIBRATION: solutions of EGDN in ethanol
RANGE STUDIED: 0.5 to 1.8 mg/m^3 [3]	**RANGE:** 3 to 45 µg per sample
BIAS: -0.02%	
OVERALL PRECISION ($\hat{S}_{rT}$): 0.089 [2]	**ESTIMATED LOD:** 0.6 µg per sample
ACCURACY: ±20.3%	**PRECISION ($\bar{S}_r$):** 0.063 [2]

APPLICABILITY: The working range is 0.03 to 0.5 ppm (0.2 to 3 mg/m^3) for a 15-L air sample, provided that the linear range of the ECD is not exceeded.

INTERFERENCES: A high concentration of 2-hydroxyethyl nitrate (ethylene glycol mononitrate) tails into the EGDN peak but does not prevent a satisfactory determination of EGDN.

OTHER METHODS: This method combines and replaces Methods S216 [2,5] and P&CAM 203 [4].

REAGENTS:

1. Ethanol, absolute.
2. Nitroglycerin (NG)* - one of the following:
   a. pure material.
   b. standard solution in ethanol, available from dynamite manufacturer.
   c. tablets, sublingual, USP (analyzed; 80 to 112% NG).
      NOTE: to prepare solutions, weigh, crush and extract with ethanol. Filter and dilute to volume.
   d. nitroglycerin lactose trituration, available from NG tablet manufacturer.
3. Ethylene glycol dinitrate (EGDN)* - one of the following:
   a. pure material.
   b. dynamite, analyzed, available.
      NOTE: Dynamite is heterogeneous. Use care to obtain representative sample.
4. Argon/methane, 95:5 (v/v), purified.
5. DE stock solution, 3 µg/µL. Dissolve 30.0 mg NG and/or 30.0 mg EGDN in ethanol to make 10 mL solution.
6. Calibration stock solution, 0.3 µg/µL. Dilute 1 mL DE stock solution to 10 mL with ethanol.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 10 cm long, 8-mm OD, 6-mm ID, flame-sealed ends and plastic caps, containing two sections of 35/60 mesh Tenax-GC resin (front = 100 mg; back = 50 mg), separated by a urethane foam plug. Silylated glass wool plugs precede the front section and follow the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.2 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, ECD, integrator and column (page 2507-1).
4. Vials, 5-mL, with PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Pipets, 2-mL, delivery, glass, with pipet bulb.
7. Volumetric flasks, 1- and 10-mL.

SPECIAL PRECAUTIONS: Ethanol is flammable and all work should be done in an exhaust hood and away from any open flame.

Nitroglycerin and ethylene glycol dinitrate can be explosive in their pure forms and extreme caution should be used when handling the bulk material [5].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.2 and 1 L/min for a total sample size of 3 to 100 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.

6.   Add 2.0 mL ethanol to each vial. Attach crimp cap to each vial.
7.   Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8.   Calibrate daily with at least five working standards over the range 1 to 45 μg NG or EGDN per sample.
  a.   Add known amounts of calibration stock solution to ethanol in 10-mL volumetric flasks and dilute to the mark.
  b.   Analyze together with samples and blanks (steps 11 and 12).
  c.   Prepare calibration graph (peak area vs. μg NG or EGDN).
9.   Determine desorption efficiency (DE) at least once for each batch of Tenax-GC used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
  a.   Remove and discard back sorbent section of a media blank sampler.
  b.   Inject a known amount of DE stock solution directly onto front sorbent section with a microliter syringe.
  c.   Cap the tube. Allow to stand overnight.
  d.   Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
  e.   Prepare a graph of DE vs. μg NG or EGDN recovered.
10.   Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11.   Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2507-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
      NOTE: If peak area is above the linear range of the working standards, dilute with ethanol, reanalyze and apply the appropriate dilution factor in calculations.
12.   Measure peak area.

CALCULATIONS:

13.   Determine the mass, μg (corrected for DE) of NG or EGDN found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
      NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14.   Calculate concentration, C, of NG or EGDN in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S216 [3] was issued on November 11, 1975, and validated over the range of 0.56 to 3.2 mg/m^3 for nitroglycerin and 0.51 to 1.8 mg/m^3 for ethylene glycol dinitrate [2]. Eighteen 15-L samples for each analyte were collected from dynamically-generated test atmospheres. Eighteen additional samples for each analyte were spiked (six each at one-half, one and two times the OSHA standard) directly. The average recovery for all three levels for nitroglycerin was 97.6% and had a pooled precision ($\bar{S}_r$) of 0.051. The average recovery for the three levels for ethylene glycol dinitrate was 92.0% with a pooled

precision ($\bar{S}_r$) of 0.063. Since the collection efficiency of the Tenax-GC for both compounds was established as 100%, the corrected "found" value of the generated sample was taken as the measure of the generated air concentrations. Breakthrough for each compound was evaluated at a concentration twice the OSHA standard. Duplicate samples were collected at interim periods of 60, 120, and 240 min at 1.1 L/min. Breakthrough of EGDN occurred at some point between 130 and 257 L of air sampled. No breakthrough occurred with the NG samples up to an average sample volume of 261 L. Sample stability was evaluated with 2.6 µg EGDN per sample and 9.0 µg NG per sample for 25 days at ambient temperature. No losses were observed for either compound [1].

REFERENCES:

[1]   Sampling and Analysis of Four Organic Compounds Using Solid Sorbents, Final Report, NIOSH Contract No. HSM-99-73-63, Southern Research Institute, Birmingham, AL.

[2]   Documentation of the NIOSH Validation Tests, S216, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

[3]   NIOSH Manual of Analytical Methods, 2nd. ed., V. 3, S216, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

[4]   Ibid, V. 1, P&CAM 203, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

[5]   Criteria for a Recommended Standard...Occupational Exposure to Nitroglycerin and Ethylene Glycol Dinitrate, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-167 (1978).

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