1- and 2-BROMOPROPANE 1025

$CH_3CH_2CH_2Br$	MW: 123.00	CAS: 106-94-5	RTECS:TX4110000
$(CH_3)_2CHBr$	123.00	75-26-3	TX4111000

METHOD: 2552, ISSUE 1 **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

OSHA : None
NIOSH: None
ACGIH: None

PROPERTIES: **1-BP**: liquid; d= 1.354 g/mL @ 20 °C; BP= 71 °C; MP = -110 °C; FP= 25 °C.
2-BP: liquid; d= 1.310 g/mL @ 20 °C; BP= 59 °C; MP= -89 °C; FP= 19 °C.

NAMES & SYNONYMS: 1-Bromopropane: Propyl bromide, 1-BP.
2-Bromopropane: Isopropyl bromide, 2-BP.

SAMPLING

SAMPLER: Solid Sorbent Tube [1]
(Anasorb CSC, 100/50 mg)
Alternative sampler
(Anasorb CMS, 150 mg/75 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 0.1L
-MAX: 12 L

SHIPMENT: Routine

SAMPLE STABILITY: 30 days at 5°C

BLANKS: 10% of field samples

ACCURACY

RANGE STUDIED: Not determined.

BIAS: Not determined.

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

ACCURACY: Not determined.

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: 1-Bromopropane and 2-Bromopropane

DESORPTION: 1-mL CS_2 for 30 minutes with agitation.

INJECTION VOLUME: 1-µL

TEMPERATURE:
-INJECTION: 200°C
-DETECTOR: 250°C
-COLUMN: 35°C (3 min) to 150°C (8°C/min)

CARRIER GAS: Helium

COLUMN: Capillary, fused silica,30-m x 0.32-mm ID; 1.8-µm film phenyl/methyl polysiloxane, Rtx-502.2 or equivalent

CALIBRATION: Standard solutions of analytes in CS_2.

RANGE: 1-BP: 3.0 to 406.0 µg per sample [1]
2-BP: 4.5 to 393.0 µg per sample [1]

ESTIMATED LOD: 1-BP: 1.0 µg per sample [1]
2-BP: 1.0 µg per sample [1]

PRECISION ($\bar{S}_r$): 1-BP: 0.015 [1]
2-BP: 0.022 [1]

APPLICABILITY: Method can be applied to any process where bromopropanes are volatilized. The method was field tested in an industrial setting where 1-bromopropane was used in the application of adhesive to foam strips [2].

INTERFERENCES: Any compounds with similar retention times.

OTHER METHODS: None.

REAGENTS:

1. 1-Bromopropane, GC grade.
2. 2-Bromopropane, GC grade.
3. Carbon disulfide, GC grade.
4. Helium, prepurified and filtered.
5. Hydrogen, prepurified and filtered.
6. Air, compressed, purified, filtered.
7. Calibration stock solution: Add known amounts of analytes to carbon disulfide in 10-mL volumetric flask.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of Anasorb® CSC or equivalent (100/50 mg) separated by a 2-mm urethane plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section.
2. Alternative sampler: glass tube, 7 cm long, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of Anasorb® CMS or equivalent (150/75 mg) separated by a 2-mm urethane plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section.
3. Personal sampling pump, 0.01 to 0.2 L/min, connected with flexible tubing.
4. Gas chromatograph equipped with FID, integrator and capillary column (see page 2552-1).
5. Autosampler vials, 2-mL, glass, with PTFE-lined crimp caps.
6. Syringes, 10-µL, 25-µL, and 1-mL.
7. Pipettes, 3-mL and 5-mL.
8. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic, explosive, and a fire hazard (FP= -30°C). Work with carbon disulfide in a well ventilated hood.

SAMPLING:

1. Calibrate each sampling pump with a representative sampler in line.
2. Break the ends of sampling tube immediately before sampling. Attach sampling tube to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 12 L.
4. Cap the samplers with plastic caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Place the glass wool preceding the front section into the vial containing the front sorbent section. Discard the urethane foam plugs.
6. Add 1.0 mL of carbon disulfide into each vial. Attach crimp caps to each vial.
7. Allow to stand for 30 minutes with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards from below the LOD to 10 times the LOQ. If necessary, additional standards may be added to extend the calibration curve.
   a. Add known amounts of analytes to carbon disulfide solvent in a 10-mL volumetric flask and dilute to the mark. Prepare additional standards by serial dilution in 10-mL volumetric flasks.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs µg analyte).

9. Determine desorption efficiency (DE) at least once for each lot of Anasorb CSC or Anasorb CMS used for sampling in the calibration ranges (step 8).
   a. Prepare three tubes at each of five levels plus three media blanks.
   b. Inject a known amount of DE stock solution (5 to 25 µL) directly onto the front sorbent section of each tube with a microliter syringe.
   c. Allow the tubes to air equilibrate for several minutes, then cap the ends of each tube and allow to stand overnight.
   d. Desorb (steps 5-7) and analyze together with standards and blanks (steps 11 and 12).
   e. Prepare a graph of DE vs µg analyte recovered.
10. Analyze a minimum of three quality control blind spikes and three analyst spikes to ensure 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 1025-1. Inject a 1-µL sample aliquot manually using the solvent flush technique or with an autosampler.
    NOTE: If peak area is above the linear range of the working standards, dilute with solvent, reanalyze, and apply the appropriate dilution factor in the calculations.
12. Measure peak areas.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE), of analyte 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 analyte in the air volume sampled, V(L):

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

EVALUATION OF METHOD:

Desorption efficiency was checked for 1- and 2-bromopropane by spiking known amounts (in CS_2) on 2 different sorbents, Anasorb CSC and Anasorb CMS. The effect of volatility on sample recovery was also determined for each analyte spiked on Anasorb CSC and Anasorb CMS sorbent tubes using GelAir portable pumps to pull air through each tube at 0.2 L/min for 60 minutes (total volume was 12 L). Storage stability was determined for each analyte after 7, 14, and 30 days.

The average DE determined for 1-bromopropane from Anasorb CSC was 96.8% (RSD = 0.015) and for 2-bromopropane was 101.0% (RSD = 0.020). When air was pulled through spiked sorbent tubes to determine the effects of volatility on sample recovery, the average DE determined for 1-bromopropane was 103.7% (RSD = 0.013) and for 2-bromopropane was 99.7% (RSD = 0.026).

The average 30-day storage stability recovery for 1-bromopropane on Anasorb CSC was 106.9% (RSD = 0.009) and for 2-bromopropane was 98.2% (RSD = 0.013). The 30 day storage recovery using Anasorb CMS was 106% (RSD = 0.014) for 1-Bromopropane and 100.6% for 2-Bromopropane.

REFERENCES:

[1] Pendergrass, SM [1998]. 1- & 2-Bromopropane Backup Data Report for method development, National Institute for Occupational Safety and Health, DART, Cincinnati, OH. Unpublished report.
[2] Pendergrass, SM [1998]. Analytical Report for Sequence 9015 - 1-Bromopropane, National Institue for Occupational Safety and Health, DART, Cincinnati, Ohio, September.

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