

2-BUTOXYETHANOL (BUTYL CELLOSOLVE)
2-BUTOXYETHYL ACETATE (BUTYL CELLOSOLVE ACETATE)



Method no.: 83

Matrix: Air

Procedure: Samples are collected by drawing air through standard size coconut shell charcoal tubes. The charcoal is desorbed with a 95/5 (v/v) methylene chloride/methanol solution and the desorbate is analyzed by gas chromatography using a flame ionization detector.

Recommended air volume and sampling rate: 48 L at 0.1 L/min

	<u>2-butoxyethanol</u>	<u>2-butoxyethyl acetate</u>
Target concentration:	5 ppm (24 mg/m ³)	5 ppm (33 mg/m ³)
Reliable quantitation limit:	31 ppb (150 µg/m ³)	24 ppb (157 µg/m ³)
Standard error of estimate at target concentration: (Section 4.7)	5.2%	5.5%

Special requirement: Samples for 2-butoxyethyl acetate should be stored at 0°C or colder to reduce hydrolysis. Reduced temperature shipment of samples to the laboratory is not necessary.

Status of method: Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

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1. General Discussion

1.1 Background

1.1.1 History

Methodologies to determine airborne concentrations of 2-methoxyethanol (methyl Cellosolve), 2-methoxyethyl acetate (methyl Cellosolve acetate), 2-ethoxyethanol (Cellosolve), and 2-ethoxyethyl acetate (Cellosolve acetate) have previously been evaluated by the OSHA Laboratory at two different target concentrations. (OSHA Method 53, Ref. 5.1 and OSHA Method 79, Ref. 5.2) These two methods were based on work done by NIOSH where samples are collected by drawing air through coconut shell charcoal and are analyzed by GC after desorption of the charcoal with 95/5 (v/v) methylene chloride/methanol. (Ref. 5.3) The NIOSH method also included an evaluation of 2-butoxyethanol at a range of 124 to 490 mg/m³ for 10-L air samples. NIOSH has no evaluated method for 2-butoxyethyl acetate.

OSHA has adopted a PEL of 25 ppm for 2-butoxyethanol (Ref. 5.4) and currently has no PEL for 2-butoxyethyl acetate. NIOSH is considering issuing recommendations to lower the PEL for 2-butoxyethanol and to establish a PEL at about the same recommended concentration for 2-butoxyethyl acetate, thus a target concentration of 5 ppm was chosen for both analytes in this evaluation. A number of modifications were made to OSHA Method 79 for this evaluation. Although an RTx-Volatiles (Restek Corp.) capillary column is acceptable for analysis of 2-butoxyethanol, there was less peak-tailing when a Nukol (Supelco, Inc.) capillary column was used. There is no significant peak-tailing for 2-butoxyethyl acetate on either of these columns. In OSHA Method 79, solid anhydrous magnesium sulfate was added to the desorption vials for 2-methoxyethanol and 2-ethoxyethanol samples to improve desorption efficiency. This was found to be unnecessary for 2-butoxyethanol samples.

To ascertain the validity of this method at higher concentrations, the collection efficiency of charcoal sampling tubes was confirmed at 50 ppm for each analyte. The stability and desorption efficiency of the analytes should not be affected at these higher loadings.

1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)

The effects of overexposure to 2-butoxyethanol and 2-butoxyethyl acetate are similar. Inhalation of vapors may be irritating to the respiratory tract and may cause nausea, headaches, vomiting, dizziness, drowsiness, and unconsciousness. The liquid is readily absorbed through the skin and may cause irritation to the skin and eyes. Ingestion may cause nausea, vomiting, headaches, dizziness, and gastrointestinal irritation. Chronic overexposure may damage the kidneys, liver and blood. (Ref. 5.5)

1.1.3 Workplace exposure

2-Butoxyethanol is used as a solvent for nitrocellulose, natural and synthetic resins, soluble oils, lacquers, varnishes and enamels. It is also used in textile dyeing and printing, in the treatment of leather, in the production of plasticizers, as a stabilizer in metal cleaners and household cleaners, and in hydraulic fluids, insecticides, herbicides and rust removers. (Ref. 5.6)

2-Butoxyethyl acetate is used as a high-boiling solvent for nitrocellulose lacquers, epoxy resins, and multicolor lacquers. It is also used as a film coalescing aid for polyvinyl acetate latex. (Ref. 5.7)

1.1.4 Physical properties (Ref. 5.6 unless otherwise noted)

chemical formula:

2-butoxyethanol: $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OH}$

2-butoxyethyl acetate: $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OOCCH}_3$

	<u>2-butoxyethanol</u>	<u>2-butoxyethyl acetate</u>
CAS no.:	111-76-2	112-07-2
mol wt:	118.17	160.21
bp at 101.3 kPa, °C:	171.2	192
appearance:	colorless liquid	colorless liquid
sp gr at 20/20°C:	0.9022 (Ref. 5.8)	0.9422
vp at 20°C, Pa:	101	33-40
vapor density, air=1:	4.1	5.5
flash point, °C		
open cup: (Ref. 5.8)	69.4	87.8
closed cup: (Ref. 5.8)	60.0	73.9
autoignition temp., °C	244	340
odor:	mild	pleasant, sweet, fruity
odor threshold, ppm:	approx. 0.4	0.1 (absolute perception limit) 0.35-0.48 (recognition)
explosive limits, %		
lower:	1.1	0.88
upper:	10.1	8.54
solubility:	soluble in water, alcohol, ether	moderately soluble (1 g per 100 g at 20°C) in water, soluble in hydrocarbons and other organic solvents
synonyms and trade names:		
2-butoxyethanol:	ethylene glycol monobutyl ether; ethylene glycol n-butyl ether; butyl Cellosolve	
2-butoxyethyl acetate:	ethylene glycol monobutyl ether acetate; 2-butoxyethanol acetate; acetic acid, 2-butoxyethyl ester; ethylene glycol butyl ether acetate; Ektasolve EB Acetate; butyl Cellosolve acetate	

The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations listed in ppm and ppb are referenced to 25°C and 101.3 kPa (760 mmHg).

1.2 Limit defining parameters

1.2.1 Detection limit of the analytical procedure

The detection limits of the analytical procedure are 0.12 and 0.13 ng per injection (1.0- μ L injection with a 58:1 split) for 2-butoxyethanol and 2-butoxyethyl acetate respectively. These are the amounts of each analyte that will give peaks with heights approximately 5 times the height of baseline noise. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limits of the overall procedure are 7.22 and 7.54 μ g per sample for 2-butoxyethanol and 2-butoxyethyl acetate respectively. These are the amounts of each analyte spiked on the sampling device that allow recovery of amounts of each analyte equivalent to the detection limits of the analytical procedure. These detection limits correspond to air concentrations of 31 ppb (150 μ g/m³) and 24 ppb (157 μ g/m³) for 2-butoxyethanol and 2-butoxyethyl acetate respectively. (Section 4.2)

1.2.3 Reliable quantitation limit

The reliable quantitation limits are the same as the detection limits of the overall procedure because the desorption efficiencies are essentially 100% at these levels. These are the smallest amounts of each analyte that can be quantitated within the requirements of recoveries of at least 75% and precisions (± 1.96 SD) of $\pm 25\%$ or better. (Section 4.3)

The reliable quantitation limits and detection limits reported in the method are based upon optimization of the GC for the smallest possible amounts of each analyte. When the target concentration of an analyte is exceptionally higher than these limits, they may not be attainable at the routine operating parameters.

1.2.4 Instrument response to the analyte

The instrument response over the concentration ranges of 0.5 to 2 times the target concentrations is linear for both analytes. (Section 4.4)

1.2.5 Recovery

The recovery of 2-butoxyethanol and 2-butoxyethyl acetate from samples used in a 15-day storage test remained above 98 and 86% respectively when the samples were stored at ambient temperatures. (Section 4.5, from regression lines shown in Figures 4.5.1.2 and 4.5.2.2)

1.2.6 Precision (analytical procedure)

The pooled coefficients of variation obtained from replicate injections of analytical standards at 0.5, 1 and 2 times the target concentrations are 0.004 and 0.002 for 2-butoxyethanol and 2-butoxyethyl acetate respectively. (Section 4.6)

1.2.7 Precision (overall procedure)

The precisions at the 95% confidence level for the ambient temperature 15-day storage tests are ± 10.1 and $\pm 10.8\%$ for 2-butoxyethanol and 2-butoxyethyl acetate respectively. These include an additional $\pm 5\%$ for pump error. The overall procedure must provide results at the target concentration that are $\pm 25\%$ or better at the 95% confidence level. (Section 4.7)

1.2.8 Reproducibility

Six samples for each analyte collected from controlled test atmospheres and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after 8 days of refrigerated storage. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.8)

1.3 Advantages

1.3.1 Charcoal tubes provide a convenient method for sampling.

1.3.2 The analysis is rapid, sensitive, and precise.

1.4 Disadvantage

It may not be possible to quantitate certain co-collected solvent vapors using this method because most other common solvents which are collected on charcoal are normally analyzed after desorption with carbon disulfide and may exhibit unacceptably low desorption efficiencies when 95/5 (v/v) methylene chloride/methanol is used.

2. Sampling Procedure

2.1 Apparatus

2.1.1 Samples are collected using a personal sampling pump calibrated to within $\pm 5\%$ of the recommended flow rate with a sampling tube in line.

2.1.2 Samples are collected with solid sorbent sampling tubes containing coconut shell charcoal. Each tube consists of two sections of charcoal separated by a urethane foam plug. The front section contains 100 mg of charcoal and the back section, 50 mg. The sections are held in place with glass wool plugs in a glass tube 4-mm i.d. \times 70-mm length. For this evaluation, SKC Inc. charcoal tubes (Catalog Number 226-01, Lot 120) were used.

2.2 Reagents

None required

2.3 Technique

- 2.3.1 Immediately before sampling, break off the ends of the charcoal tube. All tubes should be from the same lot.
- 2.3.2 Connect the sampling tube to the sampling pump with flexible tubing. It is desirable to utilize sampling tube holders that have protective covers to shield the employee from the sharp, jagged end of the sampling tube. Position the tube so that sampled air passes through the 100-mg section first.
- 2.3.3 Air being sampled should not pass through any hose or tubing before entering the sampling tube.
- 2.3.4 To avoid channeling, place the sampling tube vertically in the employee's breathing zone.
- 2.3.5 After sampling, seal the tubes immediately with plastic caps and wrap lengthwise with OSHA Form 21.
- 2.3.6 Submit at least one blank sampling tube with each sample set. Blanks should be handled in the same manner as samples, except no air is drawn through them.
- 2.3.7 Record sample volumes (in liters of air) for each sample, along with a list of any other solvents being used in the sampling area.
- 2.3.8 Ship any bulk sample(s) in a container separate from the air samples.

2.4 Sampler capacity

Sampler capacity is determined by measuring how much air can be sampled before breakthrough of analyte occurs, i.e., the sampler capacity is exceeded. Individual breakthrough studies were performed on each of the analytes by monitoring the effluent from sampling tubes containing only the 100-mg section of charcoal while sampling at 0.1 L/min from atmospheres containing 50 ppm analyte. The atmospheres were at approximately 80% relative humidity and 20-25°C. No breakthrough was detected in any of the studies after sampling for more than 8 h (>48 L).

2.5 Desorption efficiency

- 2.5.1 The average desorption efficiencies of 2-butoxyethanol and 2-butoxyethyl acetate from SKC Inc. Lot 120 charcoal are 99.0 and 101.5% respectively over the range of 0.5 to 2 times the target concentrations. (Section 4.9)
- 2.5.2 Desorbed samples remain stable for at least 24 h. (Section 4.10)
- 2.5.3 Desorption efficiencies should be periodically confirmed because they may change slightly due to variations in charcoal and operator technique.

2.6 Recommended air volume and sampling rate

- 2.6.1 For TWA samples, the recommended air volume is 48 L collected at 0.1 L/min (8-h samples).
- 2.6.2 For short-term samples, the recommended air volume is 15 L collected at 1.0 L/min (15-min samples).
- 2.6.3 When short-term samples are required, the reliable quantitation limits become larger. For example, the reliable quantitation limit is 99 ppb ($478 \mu\text{g}/\text{m}^3$) for 2-butoxyethanol when 15 L is sampled.

2.7 Interferences (sampling)

- 2.7.1 It is not known if any compound(s) will severely interfere with the collection of the two analytes on charcoal. In general, the presence of other solvent vapors in the air will reduce the capacity of charcoal to collect the analytes.
- 2.7.2 Other solvents used in the sampling area should be reported to the laboratory as potential interferences.

2.8 Safety precautions (sampling)

- 2.8.1 Attach the sampling equipment to the employee so that it will not interfere with work performance or safety. Use sampling tube holders with protective covers if possible.
- 2.8.2 Wear eye protection when breaking the ends of the charcoal tubes.
- 2.8.3 Follow all safety procedures that apply to the work area being sampled.

3. Analytical Procedure

3.1 Apparatus

- 3.1.1 A GC equipped with a flame ionization detector. For this evaluation, a Hewlett-Packard 5890 Series II Gas Chromatograph equipped with a 7673A Automatic Sampler was used.
- 3.1.2 A GC column capable of separating the analyte of interest from the desorption solvent, internal standard and any interferences. A 30-m × 0.25-mm i.d. (0.25- μ m film), fused silica Nukol column (Catalog Number 2-4107M, Supelco, Inc., Bellefonte, PA) was used in this evaluation.
- 3.1.3 An electronic integrator or some other suitable means of measuring peak areas or heights. A Waters 860 Networking Computer System was used in this evaluation.
- 3.1.4 Two-milliliter vials with Teflon-lined caps.
- 3.1.5 A dispenser capable of delivering 1.0 mL to prepare standards and samples. If a dispenser is not available, a 1.0-mL volumetric pipet may be used.
- 3.1.6 Syringes of various sizes for preparation of standards.
- 3.1.7 Volumetric flasks and pipets to dilute the pure analytes in preparation of standards.

3.2 Reagents

- 3.2.1 2-Butoxyethanol and 2-butoxyethyl acetate, reagent grade. Aldrich Lot 01604KT 2-butoxyethanol and Lot 01106KP 2-butoxyethyl acetate were used in this evaluation.
- 3.2.2 Methylene chloride, chromatographic grade. American Burdick and Jackson Lot AQ553 was used in this evaluation.
- 3.2.3 Methanol, chromatographic grade. American Burdick and Jackson Lot AW106 was used in this evaluation.
- 3.2.5 A suitable internal standard, reagent grade. Aldrich Lot 01601HT 2-ethyl-1-hexanol was used in this evaluation.
- 3.2.6 The desorption solvent consists of methylene chloride/methanol, 95/5 (v/v) containing an internal standard at a concentration of 1.5 mL/L.
- 3.2.7 GC grade nitrogen, air, and hydrogen.

3.3 Standard preparation

- 3.3.1 Prepare concentrated stock standards by diluting the pure analytes with methylene chloride. Prepare working standards by injecting microliter amounts of concentrated stock standards into vials containing 1.0 mL of desorption solvent delivered from the same dispenser used to desorb samples. For example, to prepare a stock standard of 2-butoxyethanol, dilute 3.0 mL of pure 2-butoxyethanol (sp gr = 0.9022) to 10.0 mL with methylene chloride. This stock solution would contain 270.7 μ g/ μ L. A working standard of 1137 μ g/sample is prepared by injecting 4.2 μ L of this stock into a vial containing 1.0 mL of desorption solvent.
- 3.3.2 Bracket sample concentrations with working standard concentrations. If samples fall outside of the concentration range of prepared standards, prepare and analyze additional standards to ascertain the linearity of response.

3.4 Sample preparation

- 3.4.1 Transfer each section of the samples to separate vials. Discard the glass tubes and plugs.
- 3.4.2 Add 1.0 mL of desorption solvent to each vial using the same dispenser as used for preparation of standards.
- 3.4.3 Immediately cap the vials and shake them periodically for about 30 min before analysis.

3.5 Analysis

3.5.1 GC conditions

column:	30-m × 0.25-mm i.d. fused silica, Nukol, 0.25-µm film
injection volume:	1.0 µL (with a 58:1 split)
zone temperatures:	column - 90°C injector - 150°C detector - 200°C
gas flows: head pressure)	hydrogen (carrier)-1.7 mL/min (83 kPa nitrogen (makeup) - 20 mL/min hydrogen (flame) - 65 mL/min air - 315 mL/min
retention times:	2-butoxyethanol - 7.55 min 2-butoxyethyl acetate - 9.75 min (2-ethyl-1-hexanol - 11.5 min)
chromatogram:	Section 4.11

- 3.5.2 Peak areas (or heights) are measured by an integrator or other suitable means.
- 3.5.3 An internal standard (ISTD) calibration method is used. Calibration curves are prepared by plotting micrograms of analyte per sample versus ISTD-corrected response of standard injections. Sample concentrations must be bracketed by standards.

3.6 Interferences (analytical)

- 3.6.1 Any compound that produces a flame ionization detector response and has a similar retention time as the analyte or internal standard is a potential interference. Any potential interferences reported to the laboratory by the industrial hygienist should be considered before samples are desorbed.
- 3.6.2 GC parameters (i.e., column and column temperature) may be changed to possibly circumvent interferences.
- 3.6.3 Retention time on a single column is not considered proof of chemical identity. Analyte identity should be confirmed by GC/mass spectrometer if possible.

3.7 Calculations

The analyte concentration for samples is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for desorption efficiency. The air concentration is calculated using the following formulae. The back (50-mg) section is analyzed primarily to determine if there was any breakthrough from the front (100-mg) section during sampling. If a significant amount of analyte is found on the back section (e.g., greater than 25% of the amount found on the front section), this fact should be reported with sample results. If any analyte is found on the back section, it is added to the amount found on the front section. This total amount is then corrected by subtracting the total amount (if any) found on the blank.

$$\text{mg/m}^3 = \frac{\text{micrograms of analyte per sample, blank corrected}}{(\text{liters of air sampled})(\text{desorption efficiency})}$$

$$\text{ppm} = \frac{(\text{mg/m}^3)(24.46)}{\text{molecular weight of analyte}}$$

where 24.46 = molar volume (L) at 25°C and 101.3 kPa (760 mmHg)

molecular weight = 118.17 for 2-butoxyethanol
 160.21 for 2-butoxyethyl acetate

3.8 Safety precautions (analytical)

- 3.8.1 Avoid skin contact and inhalation of all chemicals.
- 3.8.2 Restrict the use of all chemicals to a fume hood when possible.
- 3.8.3 Wear safety glasses and a lab coat at all times while in the lab area.

4. Backup Data

4.1 Detection limit of the analytical procedure

The injection size listed in the analytical procedure (1.0 µL with a 58:1 split) was used in the determination of the detection limits of the analytical procedure. The detection limits of 0.12 and 0.13 ng were determined by making injections of 7.22 and 7.54 ng/µL standards for 2-butoxyethanol and 2-butoxyethyl acetate respectively. These amounts were judged to produce peaks with heights approximately 5 times the baseline noise. A chromatogram of such an injection is shown in Figure 4.1.

4.2 Detection limit of the overall procedure

Six samples for each analyte were prepared by injecting 7.22 µg of 2-butoxyethanol and 7.54 µg of 2-butoxyethyl acetate into the 100-mg section of charcoal tubes. The detection limits of the overall procedure correspond to air concentrations of 31 ppb (150 µg/m³) and 24 ppb (157 µg/m³) for 2-butoxyethanol and 2-butoxyethyl acetate respectively. The results are given in Tables 4.2.1 and 4.2.2.

Table 4.2.1
 Detection Limit of the Overall Procedure for 2-Butoxyethanol

sample no.	µg spiked	µg recovered
1	7.22	6.87
2	7.22	7.03
3	7.22	7.49
4	7.22	7.36
5	7.22	6.98
6	7.22	7.16

Table 4.2.2
 Detection Limit of the Overall Procedure for 2-Butoxyethyl Acetate

sample no.	µg spiked	µg recovered
1	7.54	8.57
2	7.54	8.51
3	7.54	7.76
4	7.54	7.58
5	7.54	7.72
6	7.54	7.34

4.3 Reliable quantitation limit

The reliable quantitation limits were determined by analyzing charcoal tubes spiked with loadings equivalent to the detection limits of the analytical procedure. Samples were prepared by injecting 7.22 µg of 2-butoxyethanol and 7.54 µg of 2-butoxyethyl acetate into the 100-mg section of charcoal tubes. These amounts correspond to air concentrations of 31 ppb (150 µg/m³) and 24 ppb (157 µg/m³) for 2-butoxyethanol and 2-butoxyethyl acetate respectively. The results are given in Tables 4.3.1 and 4.3.2.

Table 4.3.1
 Reliable Quantitation Limit for 2-Butoxyethanol
 (Based on samples and data of Table 4.2.1)

percent recovered	statistics
95.2	$\bar{X} = 99.0$
97.4	SD = 3.25
103.7	Precision = (1.96)(±3.25)
101.9	= ±6.37
96.7	
99.2	

Table 4.3.2
 Reliable Quantitation Limit for 2-Butoxyethyl Acetate
 (Based on samples and data of Table 4.2.2)

percent recovered	statistics
113.7	$\bar{X} = 105.0$
112.9	SD = 6.76
102.9	Precision = (1.96)(±6.76)
100.5	= ±13.2
102.4	
97.3	

4.4 Instrument response to the analyte

The instrument response to the analytes over the range of 0.5 to 2 times the target concentrations was determined from multiple injections of analytical standards. These data are given in Tables

4.4.1 and 4.4.2 and Figures 4.4.1 and 4.4.2. The response is linear for both analytes with slopes (in ISTD-corrected area counts per micrograms of analyte per sample) of 226.3 and 219.6 for 2-butoxyethanol and 2-butoxyethyl acetate respectively.

Table 4.4.1
Instrument Response to 2-Butoxyethanol

× target conc	0.5×	1×	2×
µg/sample	568.9	1138	2276
ppm	2.45	4.91	9.81
area	130013	265338	511143
counts	130843	264851	511543
	130236	266530	510113
	130759	264206	507834
	130483	266271	508070
	130198	265353	514955
X	130422	265425	510610

Table 4.4.2
Instrument Response to 2-Butoxyethyl Acetate

× target conc	0.5×	1×	2×
µg/sample	791.3	1583	3165
ppm	2.52	5.03	10.07
area	174117	355187	690704
counts	174715	355740	688825
	173817	355966	690567
	173654	355907	690419
	173498	355731	693114
	173516	354933	692883
X	173886	355577	691085

4.5 Storage test

Storage samples are normally generated by sampling the recommended air volume at the recommended sampling rate from test atmospheres at 80% relative humidity containing the analyte at the target concentration. Because this would require generation of 8-h samples, in the interest of time, samples were generated by sampling from atmospheres containing the analytes at about 4 times the target concentrations for 60 min at 0.2 L/min (12-L samples). For each set of 36 samples for each analyte, six samples were analyzed immediately after generation, fifteen were stored in a refrigerator at 0°C and fifteen were stored in a closed drawer at ambient temperatures of 20-28°C. Six samples, three from refrigerated and three from ambient storage, were analyzed at intervals over a period of fifteen days. The results are given in Tables 4.5.1 and 4.5.2 and shown graphically in Figures 4.5.1.1, 4.5.1.2, 4.5.2.1 and 4.5.2.2. The loss of analyte on the 2-butoxyethyl acetate samples was due to hydrolysis of the ester to 2-butoxyethanol and acetic acid. This was supported by the fact that amounts of 2-butoxyethanol were found on these samples which corresponded to the loss of 2-butoxyethyl acetate. The loss of analyte in this study after 15 days was about 3% for refrigerated storage and about 10% for ambient storage. If possible, stored 2-butoxyethyl acetate samples should be refrigerated to reduce hydrolysis.

Table 4.5.1
Storage Test of 2-Butoxyethanol

storage time (days)	% recovery (ambient)			% recovery (refrigerated)		
	0	98.5	96.9	98.0	98.5	96.9
		97.7	98.2	98.4	97.7	98.2
2		99.5	98.9	99.7	99.7	99.2
6		97.5	98.2	99.0	97.9	98.3
8		95.2	96.4	96.3	95.5	96.0
13		98.4	99.4	100.0	100.3	100.2
15		98.2	99.6	98.2	99.1	99.0

Table 4.5.2
Storage Test of 2-Butoxyethyl Acetate

storage time (days)	% recovery (ambient)			% recovery (refrigerated)		
	0	99.2	98.6	98.9	99.2	98.6
		97.7	94.0	99.4	97.7	94.0
2		93.2	96.5	95.8	98.6	96.4
6		91.6	86.7	92.4	97.5	97.1
8		89.3	90.1	90.9	96.0	96.5
13		88.2	85.2	90.0	97.4	95.1
15		87.0	89.7	89.1	97.2	93.1

4.6 Precision (analytical procedure)

The precision of the analytical procedure for each analyte is the pooled coefficient of variation determined from replicate injections of standards. The precision of the analytical procedure for each analyte is given in Tables 4.6.1 and 4.6.2. These tables are based on the data presented in Section 4.4.

Table 4.6.1
Precision of the Analytical Method
for 2-Butoxyethanol
(Based on Table 4.4.1)

× target conc	0.5×	1×	2×
µg/sample	568.9	1138	2276
ppm	2.45	4.91	9.81
SD ¹	330.6	867.7	2624
CV	0.0025	0.0033	0.0051
CV	0.004		

¹standard deviation is in area counts

Table 4.6.2
Precision of the Analytical Method
for 2-Butoxyethyl Acetate
(Based on Table 4.4.2)

× target conc	0.5×	1×	2×
µg/sample	791.3	1583	3165
ppm	2.52	5.03	10.07
SD ¹	466.2	418.9	1632
CV	0.0027	0.0012	0.0024
CV	0.002		

¹standard deviation is in area counts

4.7. Precision (overall procedure)

The precision of the overall procedure is determined from the storage data. The determination of the standard error of estimate (SEE) for a regression line plotted through the graphed storage data allows the inclusion of storage time as one of the factors affecting overall precision. The SEE is similar to the standard deviation, except it is a measure of dispersion of data about a regression line instead of about a mean. It is determined with the following equation:

$$SEE = \sqrt{\frac{\sum(Y_{obs} - Y_{est})^2}{n - k}}$$

where

n = total number of data points

k = 2 for linear regression

k = 3 for quadratic regression

Y_{obs} = observed % recovery at a given time

Y_{est} = estimated % recovery from the regression line at the same given time

An additional 5% for pump error is added to the SEE by the addition of variances. The SEEs are 5.2% and 5.5% for 2-butoxyethanol and 2-butoxyethyl acetate respectively. The precision of the overall procedure is the precision at the 95% confidence level, which is obtained by multiplying the SEE (with pump error included) by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). The 95% confidence intervals are drawn about their respective regression lines in the storage graphs. The precisions of the overall procedure are ±10.1% and ±10.8% for 2-butoxyethanol and 2-butoxyethyl acetate respectively. The SEE and precision of the overall procedure for each analyte were obtained from Figures 4.5.1.2 and 4.5.2.2 for 2-butoxyethanol and 2-butoxyethyl acetate respectively.

4.8 Reproducibility

Six samples for each analyte, collected from controlled test atmospheres (at about 80% R.H., 24-28°C, 86-88 kPa) containing the analytes at about 4 times the target concentrations, were analyzed by a chemist unassociated with this evaluation. The samples were generated by drawing the test atmospheres through sampling tubes for 60 min at approximately 0.2 L/min. The samples were stored in a refrigerator for 8 days before being analyzed. The results are presented in Tables 4.8.1 and 4.8.2.

Table 4.8.1
Reproducibility for 2-Butoxyethanol

µg found	µg expected	% found	% deviation
1008	1090	92.5	-7.5
992.6	1073	92.5	-7.5
994.5	1073	92.7	-7.3
993.2	1063	93.4	-6.6
1007	1091	92.3	-7.7
1036	1104	93.8	-6.2

Table 4.8.2
Reproducibility for 2-Butoxyethyl Acetate

µg found	µg expected	% found	% deviation
1347	1396	96.5	-3.5
1337	1372	97.4	-2.6
1315	1371	95.9	-4.1
1318	1361	96.8	-3.2
1364	1373	99.3	-0.7
1380	1414	97.6	-2.4

4.9 Desorption efficiency

The desorption efficiency for each analyte was determined by injecting microliter amounts of stock standards onto the front section of charcoal tubes. Eighteen samples were prepared, six samples for each concentration level listed in the following table.

Table 4.9
Desorption Efficiency Data

analyte	2-butoxyethanol			2-butoxyethyl acetate		
	0.5×	1×	2×	0.5×	1×	2×
× target concn µg/sample	568.9	1138	2276	791.3	1583	3165
ppm	2.45	4.91	9.81	2.52	5.03	10.07
desorption efficiency, %	99.0	99.4	99.9	101.6	100.6	102.2
	98.7	99.1	99.1	101.6	101.7	101.8
	100.4	98.5	98.4	101.9	101.4	101.3
	99.2	98.9	99.0	101.5	101.2	102.2
	98.5	98.0	99.2	101.4	100.9	101.7
	99.1	98.2	99.3	101.1	100.9	102.4
\bar{X} mean	99.2	98.7	99.2	101.5	101.1	101.9
		99.0			101.5	

4.10 Stability of desorbed samples

The stability of desorbed samples was checked by reanalyzing the target concentration samples from Section 4.9 one day later using fresh standards. The sample vials were resealed with new septa after the original analyses and were allowed to stand at room temperature until reanalyzed. The results are given in Table 4.10.

Table 4.10
Stability of Desorbed Samples
at the Target Concentration

sample number	% desorption after 24 h	
	2-butoxyethanol	2-butoxyethyl acetate
1	101.8	103.4
2	102.7	103.8
3	102.7	103.8
4	101.7	103.8
5	101.0	103.6
6	100.7	103.4
\bar{X}	101.8	103.6

4.11 Chromatograms

A chromatogram of the two analytes is shown in Figure 4.11. The chromatogram is from an injection of a standard equivalent to a 48-L air sample at the target concentrations.

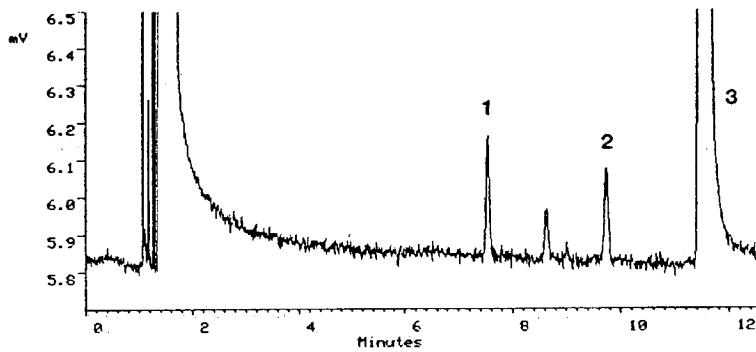


Figure 4.1. Detection limit chromatogram. Key: (1) 2-butoxyethanol, (2) 2-butoxyethyl acetate, (3) 2-ethyl-1-hexanol.

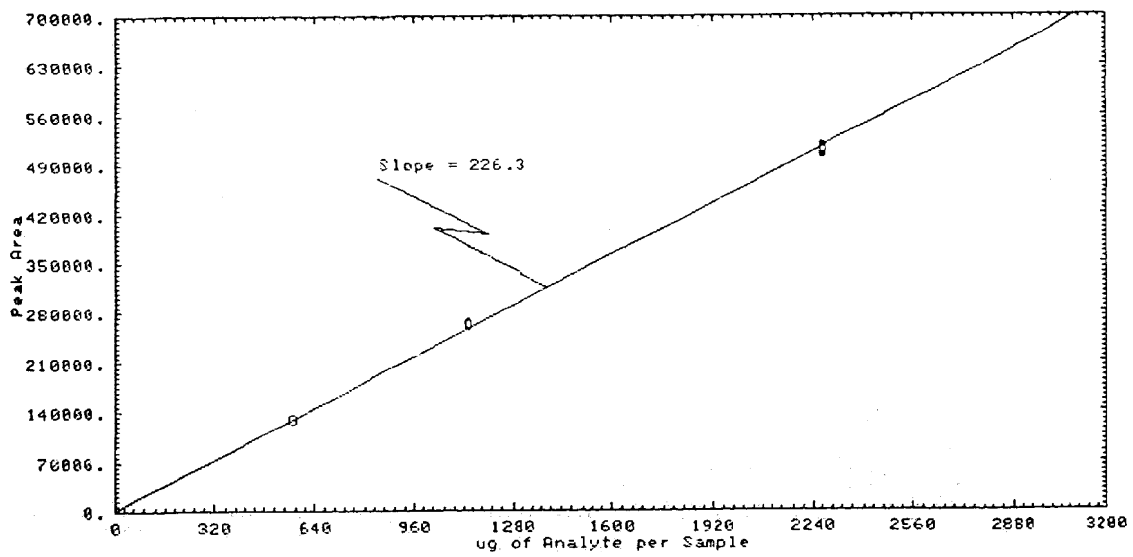


Figure 4.4.1. Instrument response to 2-butoxyethanol.

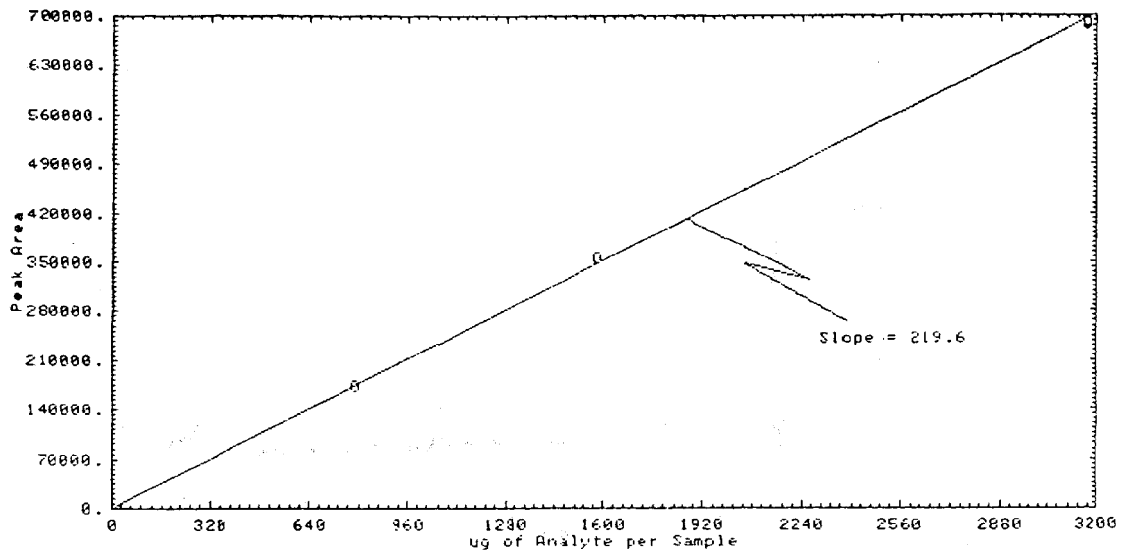


Figure 4.4.2. Instrument response to 2-butoxyethyl acetate.

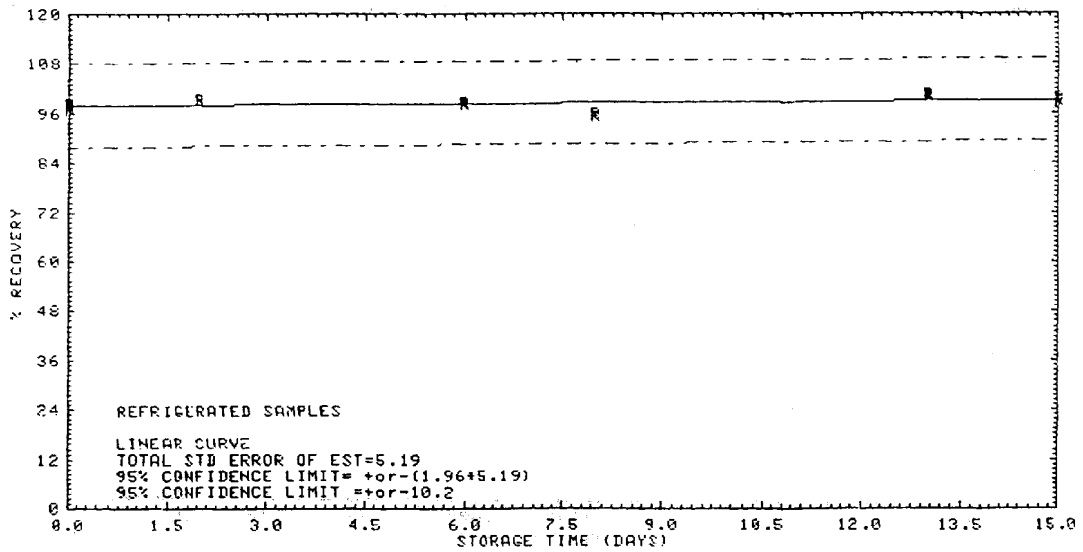


Figure 4.5.1.1. 2-Butoxyethanol refrigerated storage samples.

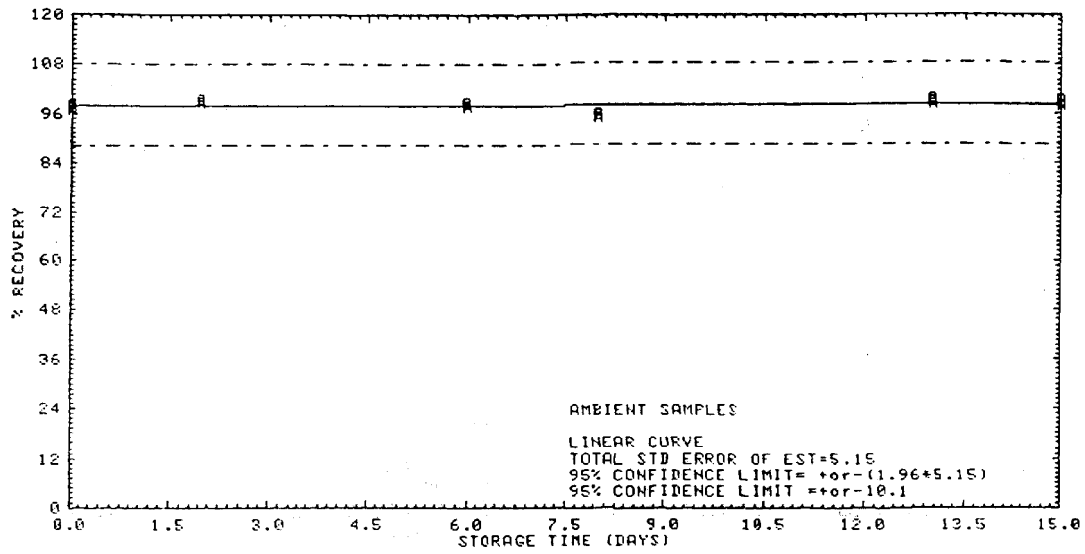


Figure 4.5.1.2. 2-Butoxyethanol ambient storage samples.

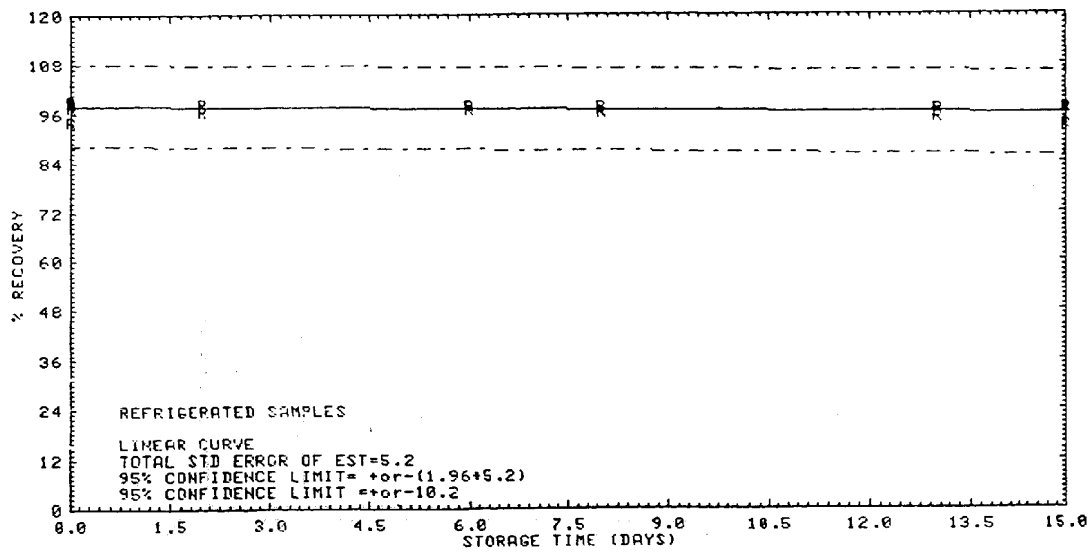


Figure 4.5.2.1. 2-Butoxyethyl acetate refrigerated storage samples.

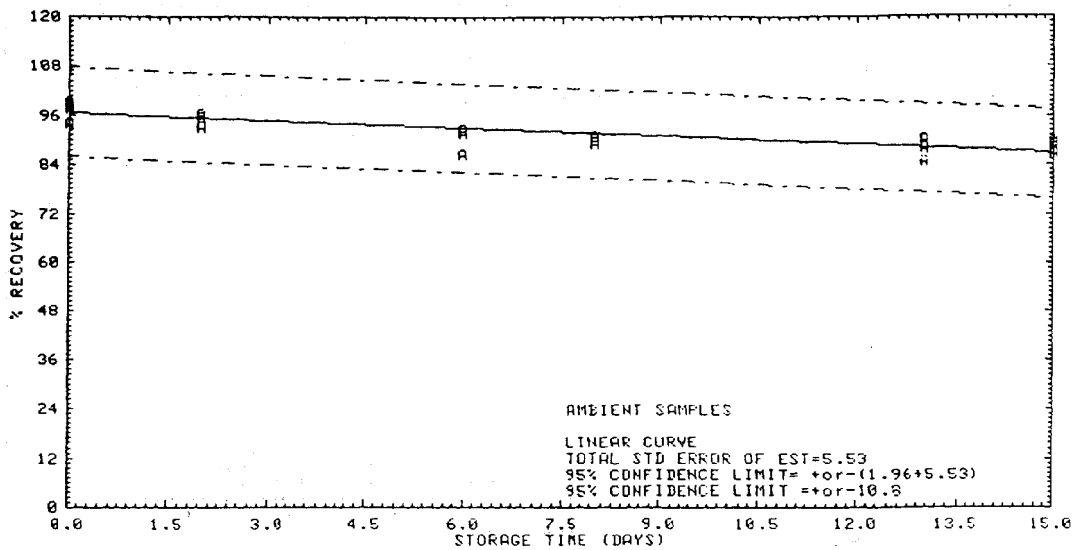


Figure 4.5.2.2. 2-Butoxyethyl acetate ambient storage samples.

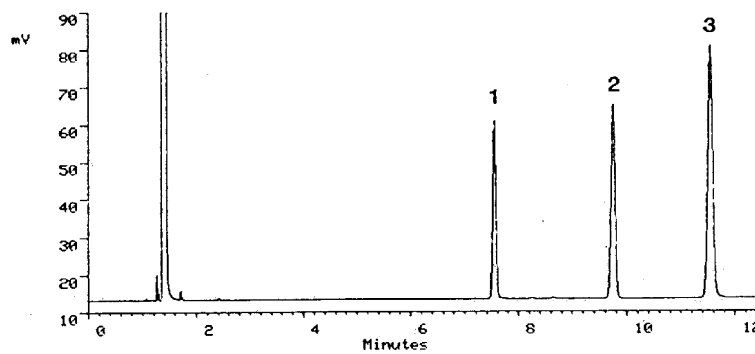


Figure 4.11. Chromatogram of a standard at the target concentrations. Key: (1) 2-butoxyethanol, (2) 2-butoxyethyl acetate, (3) 2-ethyl-1-hexanol.

5. References

- 5.1 "OSHA Analytical Methods Manual" U.S. Department of Labor, Occupational Safety and Health Administration; OSHA Analytical Laboratory: Salt Lake City, UT, 1985; Method 53; American Conference of Governmental Industrial Hygienists (ACGIH): Cincinnati, OH, ISBN: 0-936712-66-X.
- 5.2 Elskamp, C.J. "OSHA Method 79; 2-Methoxyethanol, 2-Methoxyethyl Acetate, 2-Ethoxyethanol, and 2-Ethoxyethyl Acetate", OSHA Analytical Laboratory, unpublished, Salt Lake City, UT 84165, January, 1990.
- 5.3 "NIOSH Manual of Analytical Methods", 3rd ed.; U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering; Cincinnati, OH, 1984, Method 1403, DHHS (NIOSH) Publ. No. 84-100.
- 5.4 "Air Contaminants - Permissible Exposure Limits", Code of Federal Regulations, Title 29; 1910.1000, U.S. Department of Labor, OSHA; Washington, DC, 1989, DOL (OSHA) Publ. No. OSHA 3112.
- 5.5 J.T. Baker Chemical Co.: Material Safety Data Sheets (MSDS) for 2-butoxyethanol and 2-butoxyethyl acetate, Jan. 1989.
- 5.6 ChemInfo Database on CCINFO CD-ROM disc 89-2, Canadian Centre for Occupational Health and Safety, Hamilton, Ontario.
- 5.7 "Hawley's Condensed Chemical Dictionary" 11th ed.; Sax, N.I., Lewis, R.J., Eds.; Van Nostrand Reinhold, New York, 1987.
- 5.8 Brown, E.S. et al. In "Kirk-Othmer Encyclopedia of Chemical Technology" 3rd ed.; Grayson, M., Ed.; John Wiley & Sons, New York, 1980, Vol. 11, pp 933-956.