Butyl carbitol
Butyl Carbitol acetate

Method number:	PV2095
Target concentration:	25-ppm (166 mg/m^3) butyl carbitol 25-ppm (205 mg/m^3) butyl carbitol acetate
Procedure:	Samples are collected by drawing a known volume of air through a charcoal tube. Samples are desorbed with 5:95 methanol:methylene chloride and analyzed by gas chromatography with a flame ionization detector (GC-FID).
Air volume and sampling rate studied:	50 minutes at 0.2 Lpm (10 L)
Status of method:	Partially validated method. This method has been only partially evaluated and is presented for information and trial use.

February 1993

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1 General Discussion

1.1 Background

1.1.1 History of procedure

There have been many requests for sampling and analytical procedures for butylcarbitol and butyl carbitol acetate. They have been directed to follow OSHA method 83 for 2-butoxyethanol, since butyl carbitol is related to this compound (Ref. 5.1). This study was undertaken to gather the data necessary to verify that this is the proper procedure. In OSHA method 83, collection of 2-butoxyethanol is on charcoal tubes. Desorption of butyl carbitol from charcoal tubes was previously attempted using carbon disulfide, but the recovery was 71%, while butyl carbitol acetate had an 83.4% recovery. These recoveries were low and a better desorbing solvent was needed. A solution of 5:95 methanol:methylene chloride is used in OSHA method 83 to desorb 2-butoxyethanol from charcoal. This solvent was tried and found to give desorption of 99.2% for butyl carbitol and 100% for butyl carbitol acetate. Retention and storage studies showed good recoveries from charcoal tubes.

1.1.2 Potential workplace exposure (Ref. 5.2)

Butyl carbitol and butyl carbitol acetate are used as a solvent for nitrocellulose, oils, dyes, gums, soaps, and polymers, and as a plasticizer in lacquers and coatings.

1.1.3 Toxic Effects (This section is for information purposes only and should not be taken as the basis for OSHA policy.) (Ref. 5.3)

Butyl carbitol and butyl carbitol acetate are skin, eye, and mucous membrane irritants. In rabbits, 5 mg butyl carbitol applied to the eye resulted in severe irritation. For butyl carbitol, the LD_{50} orally in guinea pigs is 2000 mg/kg, and through the skin in rabbits is 4120 mg/kg. For butyl carbitol acetate, the LD_{50} orally in guinea pigs was 2340 mg/kg, and through the skin in rabbits was 14500 mg/kg. The eye irritation in rabbits of butyl carbitol acetate was mild for a 500 mg exposure.

1.1.4 Physical properties (Ref. 5.2):

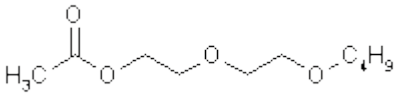
<u>Butyl carbitol</u>

CAS:	112-34-5
IMIS:	0471
RTECS:	34278 (KJ9100000)
Synonyms:	2-(2-butoxyethoxy)ethanol, diethylene glycol n-butyl ether, diglycol monobutyl ether, butyl diethoxol, butyl dioxitol, butoxydiglycol, butyl diicinol, Dowanol DB, Polysolv DB, Ektasolve DB
Molecular weight:	162.22
Density:	0.9536
Boiling point:	230.4 °C
Freezing point:	- 68 °C
Flash point:	78 °C (172 °F) (closed cup)
Autoignition temperature:	228 °C (442 °F)
Odor:	odorless
Color:	clear liquid
Molecular formula:	$C_8H_{18}O_3$
Compound:	

Butyl carbitol acetate

CAS:	124-17-4
IMIS:	M316
RTECS:	34278 (KJ9100000
Synonyms:	ethanol-2-(2-Butoxyethoxy)-acetate; Diethylene glycol butyl ether acetate; 2-(2-Butoxyethoxy)ethanol acetate; Diethylene glycol monobutyl ether acetate; Diglycol monobutyl ether acetate; Ektasolve DB acetate; Glycol ether DB acetate
Molecular weight:	204.30
Density:	0.981
Boiling point:	247 °C
Freezing point:	- 32 °C
Flash point:	115 °C (240 °F) (open cup)
Autoignition temperature:	299 °C (570 °F)
Odor:	odorless
Color:	clear liquid
Molecular formula:	$C_{10}H_{20}O_4$

Compound:



1.2 Limit defining parameters

1.2.1 The detection limit of the analytical procedure is 5 ng butyl carbitol and butyl carbitol acetate, with a 1-µL injection volume. This is a 5 pg/mL analytical standard. This is the smallest amount which could be detected under normal operating conditions.

1.2.2 The overall detection limit is 0.08-ppm butyl carbitol and 0.06 ppm butyl carbitol acetate based on a 10-liter air volume. (All ppm amounts in this study are based on a 10 L air volume.)

1.3 Advantages

1.3.1 The sampling procedure is convenient.

1.3.2 The analytical method is reproducible and sensitive.

1.3.3 Re-analysis of samples is possible.

1.3.4 It may be possible to analyze other compounds at the same time.

1.3.5 Interferences may be avoided by proper selection of column parameters.

1.4 Disadvantages

Methylene chloride is very volatile. A fan blowing on the instrument may be advisable to obtain replicate injections, when using an autosampler.

2 Sampling procedure

2.1 Apparatus

2.1.1 A calibrated personal sampling pump, the flow of which can be determined within ±5% at the recommended flow.

2.1.2 Charcoal tubes, lot 120, containing 100-mg adsorbing section with a 50-mg backup section separated by a 2-mm portion of urethane foam, with a silane treated glass wool plug before the adsorbing section and a 3-mm plug of urethane foam at the back of the backup section. The ends are flame sealed and the glass tube containing the adsorbent is 7-cm x 6-mm o.d. and 4-mm i.d., SKC tubes or equivalent.

2.2 Sampling technique

2.2.1 The ends of the charcoal tube are opened immediately before sampling.

2.2.2 Connect the charcoal tube to the sampling pump with flexible tubing.

2.2.3 Tubes should be placed in a vertical position to minimize channeling, with the smaller section towards the pump.

2.2.4 Air being sampled should not pass through any hose or tubing before entering the charcoal tube.

2.2.5 Seal the charcoal tube with plastic caps immediately after sampling. Seal each sample lengthwise with a Form OSHA-21 seal.

2.2.6 With each batch of samples, submit at least one blank tube from the same lot used for samples. This tube should be subjected to exactly the same handling as the samples (break ends, seal, & transport) except that no air is drawn through it.

2.2.7 Transport the samples (and corresponding paperwork) to the lab for analysis.

2.2.8 Bulks submitted for analysis must be shipped in a separate mailing container from other samples.

2.3 Desorption efficiency

Six tubes each were spiked with 0.167 mg (2.52 ppm), O.834 mg (12.6 ppm), 1.67 mg (25.2 ppm), and 3.34 mg (50.3 ppm) butyl carbitol, and 0.206 mg (2.47 ppm), 1.03 mg (12.3 ppm), 2.06 mg (24.7 ppm), and 4.12 mg (49.3 ppm) butyl carbitol acetate. They were allowed to equilibrate overnight at room temperature. They were then opened, each section placed into a separate 2-mL vial, desorbed with 1 mL of the desorbing solution for 30 minutes with occasional shaking, and analyzed by gas chromatography with a flame ionization detector. The overall average for butyl carbitol was 99.2%. (Table 2.3.1) The overall average for butyl carbitol acetate was 100%. (Table 2.3.2)

Table 2.3.1
Butyl Carbitol Desorption Efficiency

tube #	% recovery			
	0.167 mg	0.834 mg	1.67 mg	3.34 mg
1	99.3	97.4	99.9	98.9
2	97.9	98.1	101	100
3	97.8	98.8	102	99.4
4	98.5	97.7	101	99.6
5	97.6	98.9	101	99.9
6	96.7	97.5	99.7	101
average	98.0	98.1	101	99.8
overall average = 99.2% standard deviation = ±1.41				

Table 2.3.2
Butyl Carbitol Acetate Desorption Efficiency

tube #	% recovery			
	0.206 mg	1.03 mg	2.06 mg	4.12 mg
1	99.2	103	99.9	99.8
2	100	101	99.5	100
3	100	100	99.8	101
4	99.7	99.8	101	100
5	101	100	100	101
6	99.4	99.2	99.3	100
average	99.9	101	99.9	100

overall average = 100%
standard deviation = ±0.85

2.4 Retention efficiency

Six tubes each were spiked with 3.34 mg (50.3 ppm) butyl carbitol and 4.12 mg (49.3 ppm) butyl carbitol acetate, allowed to equilibrate overnight, and then had 10 liters humid air (90% RH) pulled through them at 0.2 Lpm. They were analyzed by GC-FID immediately. There was no butyl carbitol or butyl carbitol acetate found on the backup portions of the tubes. The retention efficiency averaged 99.3% for butyl carbitol and 99.4% for butyl carbitol acetate. (Table 2.4) Recoveries were desorption corrected.

Table 2.4
Retention Efficiency

tube #	butyl carbitol			butyl carbitol acetate		
	'A'	'B'	total	'A'	'B'	total
1	97.3	0.0	97.3	99.9	0.0	99.9
2	98.7	0.0	98.7	98.2	0.0	98.2
3	101	0.0	101	99.7	0.0	99.7
4	98.5	0.0	98.5	100	0.0	100
5	101	0.0	101	99.1	0.0	99.1
6	99.1	0.0	99.1	99.5	0.0	99.5
	average = 99.3%			average = 99.4%		

2.5 Storage

Six tubes each were spiked with 1.67 mg (25.2 ppm) butyl carbitol and 2.06 mg (24.7 ppm) butyl carbitol acetate, and stored at room temperature until opened and analyzed. The recoveries averaged 98.8% for butyl carbitol and 98.7% for butyl carbitol acetate over the 14 days stored. Recoveries are desorption corrected. (Table 2.5)

Table 2.5
Storage Study

day	% recovered	
	butyl carbitol	butyl carbitol acetate
7	96.8	101
7	98.4	97.5
7	98.3	101
14	102	97.4
14	99.6	97.0
14	97.7	98.2
average	98.8	98.7

2.6 Precision

The precision was calculated using the area counts from six injections of each standard at concentrations of 0.167, 0.834, 1.67, and 3.34 mg/mL for butyl carbitol, and 0.206, 1.03, 2.06, and 4.12 mg/mL for butyl carbitol acetate in the desorbing solution. The pooled coefficient of variation was 0.00392 for butyl carbitol and 0.00155 for butyl carbitol acetate. (Tables 2.6.1 and 2.6.2)

Table 2.6.1
Butyl Carbitol Precision Study

injection #	0.167 mg/mL	0.834 mg/mL	1.67 mg/mL	3.34 mg/mL
1	68887	153640	326647	634611
2	68884	152347	326851	634061
3	69226	154281	323728	635744
4	69149	152240	327083	639198
5	68920	153501	324953	637233
6	69105	152466	326786	637233
average	69029	153079	326008	636367
standard deviation –	±150	±842	±1356	±1924
CV -	0.00217	0.0055	0.00416	0.00302

pooled CV = 0.00392

Table 2.6.2
Butyl Carbitol Acetate Precision Study

injection #	0.206 mg/mL	1.03 mg/mL	2.06 mg/mL	4.12 mg/mL
1	66260	258265	518901	1037664
2	66352	258402	518456	1036916
3	66152	258504	517160	1038185
4	66521	257457	518661	1036115
5	66174	258713	517187	1039708
6	66527	257765	518581	1039627
average	66331	258184	518158	1038036
standard deviation –	±165	±487	±776	±1444
CV -	0.00249	0.00185	0.00150	0.00139

pooled CV = 0.00155

Where:

$$CV\ \left(Coefficient\ of\ Variation\right) = \frac{\left(standard\ deviation\right)}{\left(average\right)}$$

$$Pooled\ CV = \sqrt{\frac{A1\left(CV1\right)^2 + A2\left(CV2\right)^2 + A3\left(CV3\right)^2 + A4\left(CV4\right)^2}{A1 + A2 + A3 + A4}}$$

A1, A2, A3, A4 = number of injections at each level
CV1, CV2, CV3, CV4 = Coefficient of variation at each level

2.7 Air volume and sampling rate studied

2.7.1 The air volume studied is 10 liters.

2.7.2 The sampling rate studied is 0.2 liters per minute.

2.8 Interferences

Suspected interferences should be listed on sample data sheets.

2.9 Safety precautions

2.9.1 Sampling equipment should be placed on an employee in a manner that does not interfere with work performance or safety.

2.9.2 Safety glasses should be worn at all times.

2.9.3 Follow all safety practices that apply to the workplace being sampled.

3 Analytical method

3.1 Apparatus

3.1.1 Gas chromatograph equipped with a flame ionization detector. A Hewlett Packard 5890 gas chromatograph was used in this study.

3.1.2 An electronic integrator or some other suitable method of measuring peak areas.

3.1.3 GC column capable of separating the analyte and an internal standard from any interference. The column used in this study was a 15-m x 0.32-mm i.d., (0.25 µm d_f DB-WAX) capillary column. An alternate column is a 60-m x 0.32-mm i.d., (1.0 µm d_f DB-WAX) capillary column.

3.1.4 Two milliliter vials with PTFE-lined caps.

3.1.5 A 1-µL syringe or other convenient size for a sample injection.

3.1.6 Pipettes for dispensing the desorbing solution. The Glenco 1-mL dispenser was used in this method.

3.1.7 Volumetric flasks, 5-mL, and other convenient sizes for preparing standards.

3.2 Reagents

3.2.1 Purified GC grade nitrogen, hydrogen, and air.

3.2.2 Butyl carbitol, Reagent grade

3.2.3 Butyl carbitol acetate, Reagent grade

3.2.4 Methanol, HPLC grade

3.2.5 Methylene chloride, HPLC grade

3.2.6 n-Hexanol, Reagent grade, used as an internal standard

3.2.7 The desorbing solution is 5:95 methanol:methylene chloride with 0.25 µL/mL n-hexanol internal standard.

3.3 Sample preparation

3.3.1 Sample tubes are opened and the front and back section of each tube are placed in separate 2-mL vials.

3.3.2 Each section is desorbed with 1 mL of the desorbing solution.

3.3.3 The vials are sealed immediately and allowed to desorb for 30 minutes with occasional shaking.

3.4 Standard preparation

3.4.1 Standards are prepared by diluting a known quantity of butyl carbitol and butyl carbitol acetate with the desorbing solution.

3.4.2 At least two separate standards should be made.

3.4.3 A third analytical standard should be prepared at a higher concentration to check the linearity of the detection. For this study, two standards at 1 µL/mL (0.954 mg/mL butyl carbitol and 0.981 mg/mL butyl carbitol acetate) and one standard at 4 µL/mL (3.82 mg/mL butyl carbitol and 3.92 mg/mL butyl carbitol acetate) were used.

3.5 Analysis

3.5.1 Gas chromatograph

Column 15-m x 0.32-mm i.d., (0.25 µm d_f DB-WAX) capillary column

Flow rates	**(mL/min.)**	**Temperature**	**(°C)**
Nitrogen (make-up):	30	Injector:	180
Hydrogen (carrier):	2	Detector:	220
Hydrogen (detector):	30	Column:	60 °C for 2 min
Air:	350		10 °C/min to 130 °C

Injection size: 1 µL

Chromatogram:

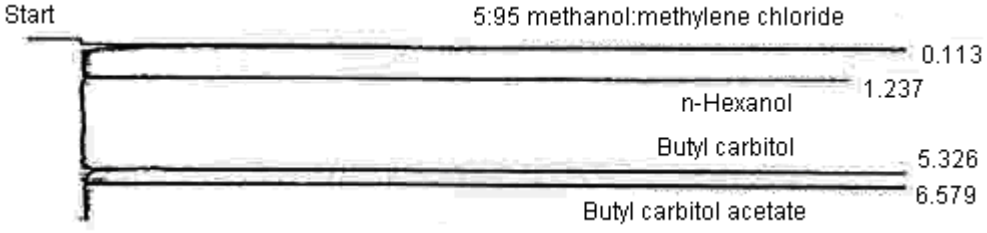


Figure 1. An analytical standard of 1 µL/mL butyl carbitol and butyl carbitol acetate in the desorbing solvent of 5:95 methanol:methylene chloride with 0.25 µL/mL n-hexanol internal standard, analyzed on a 15-meter DB-WAX capillary column.

3.5.2 Gas chromatograph

Column 60-m x 0.32-mm i.d., (1.0 µm d_f DB-WAX) capillary column

Flow rates	**(mL/min)**	**Temperature**	**(°C)**
Nitrogen (make-up):	30	Injector:	180
Hydrogen (carrier):	2	Detector:	220
Hydrogen (detector):	30	Column:	100 °C for 2 min
Air:	350		10 °C/min to 180 °C

Injection size: 1 µL

Chromatogram:

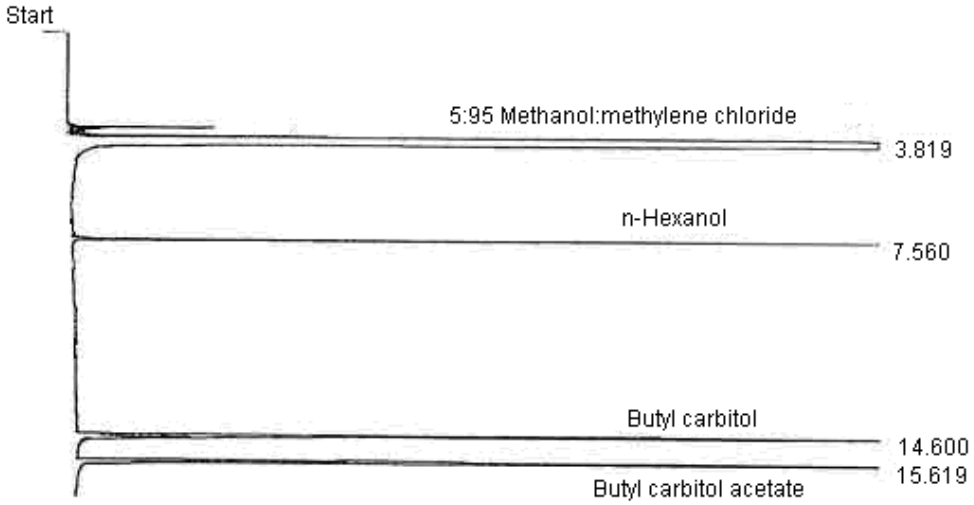


Figure 2. An analytical standard of 1 µL/mL butyl carbitol and butyl carbitol acetate in the desorbing solvent of 5:95 methanol:methylene chloride with 0.25 µL/mL n-hexanol internal standard, analyzed on a 60-meter DB-wax column.

3.5.3 Peak areas are measured by an integrator or other suitable means.

3.6 Interferences (analytical)

3.6.1 Any compound having the general retention time of the analyte or the internal standard used is interference. Possible interferences should be listed on the sample data sheet. GC parameters should be adjusted, if necessary, so these interferences will pose no problems.

3.6.2 Retention time data on a single column is not considered proof of chemical identity. Samples over the target concentration should be confirmed by GC/Mass Spec or other suitable means.

3.7 Calculations

3.7.1 The instrument is calibrated with a standard of 0.954 mg/mL butyl carbitol and 0.981 mg/mL butyl carbitol acetate in the desorbing solution. The linearity of the calibration is

checked with a standard of 3.82 mg/mL butyl carbitol and 3.92 mg/mL butyl carbitol acetate (4 µL/mL) in the desorbing solution.

3.7.2 If the calibration is non-linear, two more standards above and below the analyte peaks must be analyzed so a calibration curve can be plotted and sample values obtained.

3.7.3 To calculate the concentration of analyte in the air sample the following formulas are used:

$$\text{mass of analyte, } \mu g = \frac{(\mu g / mL)(\text{desorption volume, } mL)}{(\text{desorption efficiency, decimal})}$$

$$\text{moles of analyte} = \frac{(\text{mass of analyte, } \mu g)(1\,g)}{(\text{molecular weight})(10^6\,\mu g)}$$

$$\text{volume of analyte} = (\text{moles of analyte})(\text{molar volume})$$

$$ppm = \frac{(\text{volume of analyte})(10^6)^*}{(\text{air volume, } L)}$$

* All units must cancel.

3.7.4 The above equations can be consolidated to form the following formula, used to calculate the ppm of analyte in the sample based on a 10-liter air sample:

$$ppm = \frac{(\mu g / mL)(DV)(24.46)}{(L)(DE)(MW)}$$

Where:

µg/mL = concentration of analyte in sample
24.46 = Molar volume (liters/mole) at 25 °C and 760 mmHg
MW = Molecular weight (g/mole)
DV = Desorption volume, mL
10 L = Air volume, L
DE = Desorption efficiency, decimal

3.7.5 This calculation is done for each section of the sampling tube and the results added together.

3.8 Safety precautions

3.8.1 All handling of solvents should be done in a hood.

3.8.2 Avoid skin contact with all solvents.

3.8.3 Wear safety glasses, gloves, and a lab coat at all times in laboratory areas.

4 Recommendations for further study

Collection studies need to be performed. Method needs to be fully validated.

5 References

5.1 Elskamp, C., Method 83, "2-Butoxyethanol, and 2-butoxyethyl acetate," Organic Methods Evaluation Branch, OSHA Analytical Laboratory, 1990.

5.2 Sax, N., Lewis, R., "Hawley's Condensed Chemical Dictionary," Eleventh Edition, Van Nostrand Reinhold Co., New York, 1987, p. 390.

5.3 Sweet, D., "Registry of Toxic Effects of Chemical Substances," 1985-86 Edition, U.S. Department of Health and Human Services, Public Health Service, Center for Disease Control, NIOSH, 1987, Vol. 3, p. 2269.