Carbitol
Carbitol acetate

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|---|---|
| Method number: | PV2013 |
| Target concentration: | 25 ppm (140 mg/m$^3$) carbitol<br>25 ppm (180 mg/m$^3$) carbitol acetate |
| Procedure: | Samples are collected by drawing a known volume of air through a charcoal tube. Samples are desorbed with 95:5 methylene chloride:methanol and analyzed by gas chromatography with a flame ionization detector (GC-FID). |
| Air volume and sampling rate: | 50 minutes at 0.2 L/m (10 liters) |
| Status of method: | Partially Validated method. This method has been only partially evaluated and is presented for information and trial use. |

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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 carbitol and carbitol acetate. They have been directed to follow OSHA Method 79 for 2-ethoxyethanol and 2-ethoxyethyl acetate, since carbitol and carbitol acetate are related to this compound. (Ref. 5.1) In this method, the samples are collected on charcoal tubes and desorbed with 95:5 methylene chloride:methanol. This study was undertaken to gather the data necessary to verify that this is the proper procedure. Desorption of carbitol acetate was attempted using carbon disulfide but the recovery was nonlinear ranging from 44.9% to 74.9% for loadings 1.011 to 4.046-mg. These recoveries were low and a better desorbing solvent was needed. The 95:5 methylene chloride:methanol was tried and found to give desorption of 96.1% for carbitol and 100% for carbitol acetate. Retention and storage studies showed good recoveries for charcoal tubes.

### 1.1.2 Potential workplace exposure (Ref. 5.2)

Carbitol is used in finger nail polish remover, for setting the twist and conditioning yarns and cloth, in lecithin manufacturing, in textile printing and soaps, in lacquers, in organic synthesis, as a brake fluid diluent, and as a solvent for dyes, nitrocellulose, resins, mineral oil soap, mineral oil-sulfonated oil mixtures, and nonaqueous stains for wood. Carbitol acetate is used in lacquers, printing inks, coatings, and as a solvent for cellulose esters, gums, and resins.

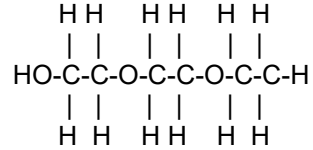
### 1.1.3 Toxic Effects (This section is for information purposes and should not be taken as the basis for OSHA policy.) (Ref. 5.3)

Carbitol and carbitol acetate are considered to be of low toxicity by the FDA and have been approved for use in cosmetics. Rabbits exposed on the skin to 500 mg/kg of carbitol and carbitol acetate showed mild reactions. The $LD_{50}$ orally for guinea pigs was 3.67 g/kg for carbitol and 3.93 g/kg for carbitol acetate.

### 1.1.4 Physical properties (Ref. 5.4.):

**Carbitol**

Compound:

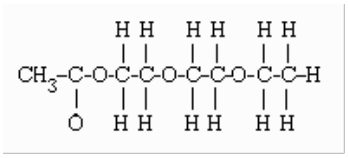


| | |
|---|---|
| CAS: | 111-90-0 |
| IMIS: | D615 |
| Synonyms: | APV; Carbitol cellosolve; Carbitol solvent; Diethylene glycol ethyl ether; Diglycol monoethyl ether; Dioxitol; Dowanol; Dowanol DE; Ethoxy diglycol; 2-(2-ethoxyethoxy) ethanol; Ethyl carbitol; Ethyl digol; Ethyl diethylene glycol; Ethylene diglycol monoethyl ether; Poly-solv; Losungsmittel APV; Monoethyl ether of diethylene glycol; Solvolsol |

| | |
|---|---|
| Molecular weight: | 134.2 |
| Density: | 0.9855 |
| Boiling point: | 196 °C |
| Flash point: | 96 °C (205 °F) (open cup) |
| Odor: | mildly sweet |
| Color: | clear liquid |
| Molecular formula: | $C_6H_{14}O_3$ |
| RTECS: | 34453; RR8750000 |

**Carbitol acetate**

Compound:



| | |
|---|---|
| CAS: | 112-15-2 |
| IMIS: | C128 |
| Synonyms: | 2-(2-Ethoxyethoxy) ethanol acetate; Diethylene glycol monoethyl ether acetate; Diglycolmonoethyl ether acetate; Ektasolve DE acetate; Glycol ether DE acetate |
| Molecular weight: | 176.24 |
| Density: | 1.0114 |
| Boiling point: | 219 °C |
| Freezing point: | - 25 °C |
| Flash point: | 110 °C (230 °F) (open cup) |
| Odor: | mildly sweet |
| Color: | clear liquid |
| Molecular formula: | $C_8H_{16}O_4$ |
| RTECS: | 34454; KK8925000 |

1.2 Limit defining parameters

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

1.2.2 The overall detection limit is 0.09 ppm carbitol and 0.07 ppm 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 and GC 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 with the sampler attached.

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 silanized 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. Wrap 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 were spiked with 0.138 mg (2.51 ppm), 0.69 mg (12.6 ppm), 1.38 mg (25.1 ppm), and 2.76 mg (50.3 ppm) carbitol, and 0.182 mg (2.52 ppm), 0.910 mg (12.6 ppm), 1.82 mg (25.2 ppm), and 3.64 mg (50.5 ppm) carbitol acetate. They were allowed to equilibrate overnight at room temperature. The samples 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 carbitol was 96.1% recovered (Table 1). The overall average for carbitol acetate was 100% recovered (Table 2).

Table 1
Carbitol Desorption Efficiency

| tube # | % recovered (mg) | | | |
|---|---|---|---|---|
| | 0.138x | 0.690x | 1.38x | 2.76x |
| 1 | 93.7 | 97.4 | 97.4 | 97.3 |
| 2 | 94.0 | 96.4 | 98.0 | 97.1 |
| 3 | 94.1 | 95.6 | 97.4 | 96.5 |
| 4 | 93.8 | 95.8 | 97.4 | 96.9 |
| 5 | 94.2 | 94.6 | 97.9 | 97.0 |
| 6 | 93.7 | 95.0 | 97.5 | 96.8 |
| average | 93.9 | 95.8 | 97.6 | 96.9 |

overall average = 96.1%
standard deviation = ±1.52

Table 2
Carbitol Acetate Desorption Efficiency

| tube # | % recovered (mg) | | | |
|---|---|---|---|---|
| | 0.182x | 0.910x | 1.82x | 3.64x |
| 1 | 99.5 | 100 | 101 | 101 |
| 2 | 99.8 | 100 | 100 | 101 |
| 3 | 98.5 | 100 | 100 | 100 |
| 4 | 99.2 | 100 | 101 | 101 |
| 5 | 98.2 | 101 | 101 | 100 |
| 6 | 98.1 | 100 | 101 | 100 |
| Average | 98.9 | 100 | 101 | 101 |

overall average = 100%
standard deviation = ±0.885

## 2.4 Retention efficiency

Six tubes were spiked with 2.76 mg (50.3 ppm) carbitol and 3.64 mg (50.5 ppm) carbitol acetate, allowed to equilibrate overnight, and had 10 liters of humid air (91% RH) pulled through them at 0.2 L/min. They were opened, desorbed, and analyzed by GC-FID. There was no carbitol or carbitol acetate found on the backup portions of the tubes. The values for carbitol were corrected for desorption efficiency. The retention efficiency averaged 98.4% for carbitol and 101% for carbitol acetate (Table 3).

Table 3
Retention Efficiency

| Tube # | % recovered | | | | | |
|---|---|---|---|---|---|---|
| | Carbitol | | | carbitol acetate | | |
| | 'A' | 'B' | total | 'A' | 'B' | total |
| 1 | 98.2 | 0.0 | 98.2 | 102 | 0.0 | 102 |
| 2 | 98.3 | 0.0 | 98.3 | 100 | 0.0 | 100 |
| 3 | 99.2 | 0.0 | 99.2 | 100 | 0.0 | 100 |
| 4 | 98.4 | 0.0 | 98.4 | 100 | 0.0 | 100 |
| 5 | 97.5 | 0.0 | 97.5 | 101 | 0.0 | 101 |
| 6 | 98.8 | 0.0 | 98.8 | 101 | 0.0 | 101 |
| | average = 98.4% | | | average = 101% | | |

2.5 Storage

Tubes were spiked with 1.38 mg (25.1 ppm) carbitol and 1.82 mg (25.2 ppm) carbitol acetate, and stored at room temperature until opened and analyzed. The recoveries for carbitol were corrected for desorption efficiency. The recoveries averaged 98.8% for carbitol and 98.0% for carbitol acetate over the 14 days stored. (Table 4)

Table 4
Storage Study

| day | % recovered | |
|---|---|---|
| | carbitol | carbitol acetate |
| 7 | 100 | 98.0 |
| 7 | 95.7 | 99.9 |
| 7 | 99.3 | 98.6 |
| 14 | 98.5 | 95.6 |
| 14 | 98.2 | 96.3 |
| 14 | 101 | 99.5 |
| average | 98.8% | 98.0% |

2.6 Precision

The precision was calculated using the area counts from six injections of each standard at concentrations of 0.138, 0.690, 1.38, and 2.76 mg/mL carbitol; and 0.182, 0.910, 1.82, and 3.64 mg/mL carbitol acetate in the desorbing solution. The pooled coefficient of variation was 0.00349 for carbitol and 0.00369 for carbitol acetate. (Tables 5 and 6)

Table 5
Carbitol Precision Study

| injection number | 0.138 mg/mL | 0.690 mg/mL | 1.38 mg/mL | 2.76 mg/mL |
|---|---|---|---|---|
| 1 | 26886 | 122733 | 249185 | 493394 |
| 2 | 26869 | 123008 | 248652 | 498230 |
| 3 | 26832 | 122859 | 245787 | 494524 |
| 4 | 26847 | 123003 | 246254 | 497276 |
| 5 | 26859 | 122850 | 246099 | 496050 |
| 6 | 26884 | 122959 | 246506 | 495515 |
| average | 26863 | 122902 | 247081 | 495832 |
| standard deviation | ±21.2 | ±108 | ±1452 | ±1769 |
| CV | 0.000789 | 0.000879 | 0.00588 | 0.00357 |

pooled CV = 0.00349

Table 6
Carbitol Acetate Precision Study

| injection number | 0.182 mg/mL | 0.910 mg/mL | 1.82 mg/mL | 3.64 mg/mL |
|---|---|---|---|---|
| 1 | 35941 | 173819 | 356698 | 705803 |
| 2 | 36111 | 173402 | 357304 | 707101 |
| 3 | 36454 | 174829 | 357788 | 706641 |
| 4 | 36301 | 174738 | 357078 | 707308 |
| 5 | 36141 | 174710 | 357560 | 706848 |
| 6 | 36521 | 174072 | 355886 | 709041 |
| average | 36245 | 174262 | 355886 | 707124 |
| standard deviation | ±221 | ±587 | ±685 | ±1074 |
| CV | 0.00610 | 0.00337 | 0.00192 | 0.00152 |

pooled CV = 0.00369

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

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

Where:

A1, A2, A3, A4 = number of injections at each level
CV1, CV2, CV3, CV4 = Coefficients 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 the 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. with a (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 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 Carbitol, Reagent grade

3.2.3 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 the internal standard

3.2.7 The desorbing solution is 95:5 methylene chloride:methanol 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 carbitol and 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 (0.986 mg/mL carbitol and 1.011 mg/mL carbitol acetate) and one standard at 4 µL/mL (3.942 mg/mL carbitol and 4.046 mg/mL carbitol acetate) were used.

3.5 Analysis

3.5.1 Gas chromatograph: 15-m x 0.32-mm i.d. (with 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): | 60 | Column: | 60 °C then 10 °C/min |
| Air: | 410 | | to 110 °C |

Injection size: 1 µL
Chromatogram: see Figure 1

3.5.2 Gas chromatograph: 60-m x 0.32-mm i.d. with (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): | 60 | Column: | 100 °C then 10 °C/min | |
| Air: | 410 | | to 180 °C | |

Injection size: 1 µL
Chromatogram: see Figure 2

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 is an 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 0.986 mg/mL (1 µL/mL) carbitol and 1.011 mg/mL carbitol acetate in the desorbing solution. The linearity of the calibration is checked with a standard of 3.942 µL/mL (4 µL/mL) carbitol and 4.046 mg/mL (4 µL/mL) carbitol acetate in the desorbing solution.

3.7.2 If the calibration is non-linear, two more standards 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:

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

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

$$volume\ of\ analyte = (moles\ of\ analyte)(molar\ volume)$$

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

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:

$$ppm = \frac{(\mu g\ /\ mL)(DV)(24.46)}{(10\ L)(DE)(MW)}$$

µg/mL = concentration of analyte in sample or standard
24.46 = Molar volume (liters/mole) at 25 °C and 760 mmHg.
MW = Molecular weight (g/mole)
DV = Desorption volume, mL
10 L = 10 liter air sample
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 at all times.

4 Recommendations for further study

Collection studies need to be performed. Complete validation of method.

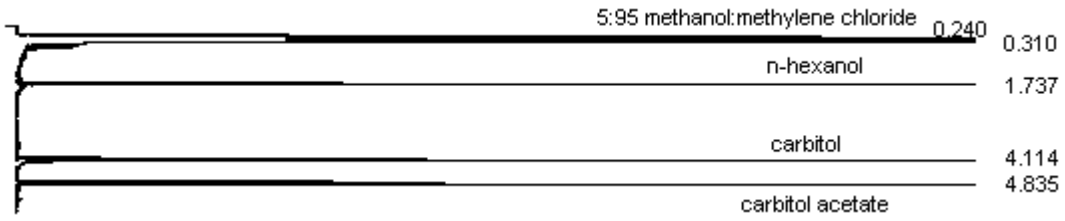



Figure 1
An analytical standard of 1 µL/mL carbitol and carbitol acetate in the desorbing solvent of 5:95 methanol:methylene chloride with 0.25 µL/mL n-hexanol internal standard, analyzed with a 15-m x 0.32-mm i.d. with (0.25 µm $d_f$ DB-WAX) capillary column.

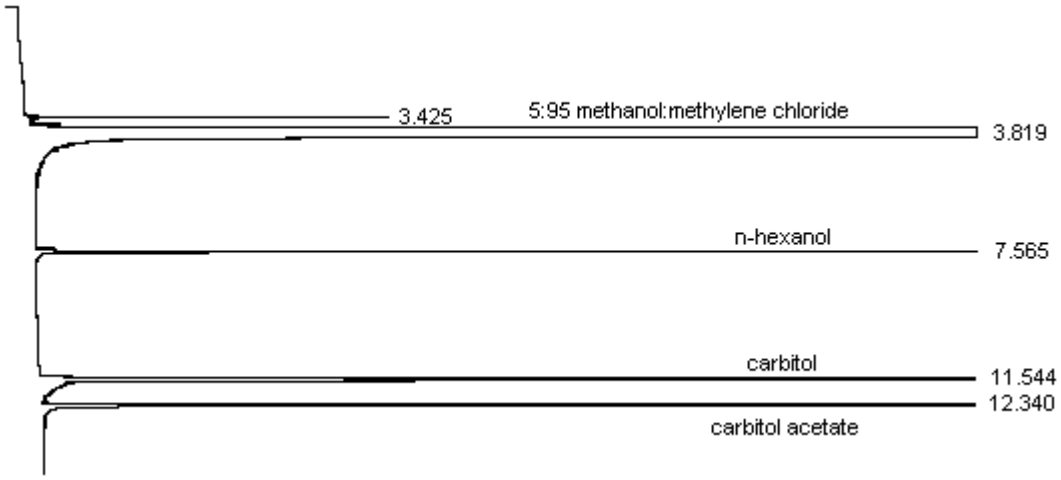



Figure 2
An analytical standard of 1 µL/mL carbitol and carbitol acetate in the desorbing solvent of 5:95 methanol:methylene chloride with 0.25 µL/mL n-hexanol internal standard, analyzed with a 60-m x 0.32-mm i.d. with (1.0 µm $d_f$ DB-WAX) capillary column,

## 5 References

5.1 Elskamp, C., Method 79, "2-Methoxyethanol, 2-methoxyethyl acetate, 2-ethoxyethanol, and 2-ethoxyethyl 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. 391.

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. 2280-l.

5.4 Windholz, M., "The Merck Index," Eleventh Edition, Merck & Co., Rahway N.J., 1989, p. 272.