Benzyl alcohol

Method number:	PV2009
Target concentration:	5 ppm (22 mg/m^3)
Procedure:	Samples are collected by drawing a known volume of air through an XAD-7 tube. Samples are desorbed with methanol and analyzed by gas chromatography with a flame ionization detector (GC-FID).
Air volume and sampling rate studied:	120 minutes at 0.2 Lpm (24 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

OSHA method 32 recommends collection of phenol and cresol on XAD-7 tubes and desorption with methanol (Ref. 5.1). Since benzyl alcohol is similar in structure to phenol and cresol, this method of collection and analysis was tried and found to give good recoveries for retention, desorption, and storage.

1.1.2 Potential workplace exposure (Ref. 5.2 and 5.3)

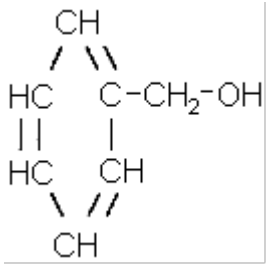
Benzyl alcohol is a component of many naturally occurring perfume oils, including jasmine, hyacinth, and ylang-ylang. Benzyl alcohol and its esters are used in making perfumes and flavorings. Benzyl alcohol is used in the manufacture of other benzyl compounds, in cosmetics, in ointments, as a bacteriostat, as a photographic developer for color film, and as an embedding material in microscopy. It is used as a solvent for gelatin, casein, cellulose acetate, shellac, dyestuffs, and waxes.

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)

Benzyl alcohol is a moderate skin and mucous membrane irritant, and a severe eye irritant. In a human skin exposure, over a 48-hour period, 16 mg caused mild contact dermatitis.

1.1.4 Physical properties (Ref. 5.2 and 5.3):

Structure:



CAS:	100-51-6
IMIS:	0337
RTECS:	DN3150000; 19460
Synonyms:	Benzenecarbinol; Benzenemethanol; Benzoyl alcohol; Hydroxytoluene; Phenylcarbinol; Phenylmethanol; α-Toluenol
Molecular weight:	108.13
Density:	1.04156
Freezing point:	- 15.19 °C
Boiling point:	204.7 °C
Flash point:	10l °C (213 °F) (closed cup); 105 °C (220 °F) (open cup)
Odor:	faintly aromatic
Color:	clear liquid
Molecular formula:	C_7H_8O

1.2 Limit defining parameters

1.2.1 The detection limit of the analytical procedure is 1-µg benzyl alcohol. This is the smallest amount that could be detected under normal operating conditions.

1.2.2 The overall detection limit is 0.009 ppm. (All ppm amounts in this study are based on a 24 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 Reanalysis 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

None known

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 XAD-7 tubes containing 15/50 mesh XAD-7 with a 100-mg adsorbing section with a 50-mg backup section separated by a silanized glass wool plug, with a silanized glass wool plug before and after the adsorbing sections. The ends are flame sealed and the glass tube containing the adsorbent is 8-cm x 8-mm o.d. and 6-mm i.d., SKC tubes or equivalent.

2.2 Sampling technique

2.2.1 Open the ends of the XAD-7 tube immediately before sampling.

2.2.2 Connect the XAD-7 tube to the sampling pump with flexible tubing.

2.2.3 Place the tubes 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 XAD-7 tube.

2.2.5 Seal the XAD-7 tube with plastic caps immediately after sampling. Seal each sample lengthwise with 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 at each loading of 52.1 µg (0.49 ppm), 260 µg (2.45 ppm), 521 µg (4.91 ppm), and 1042 µg (9.82 ppm) benzyl alcohol. They were allowed to equilibrate overnight at room temperature. They were opened; each section placed into a separate 2-mL vial, desorbed with 1 mL of the desorbing solution of methanol with 0.25 µL/mL DMF internal standard, for 30 minutes with occasional shaking, and then analyzed by GC-FID. The overall average was 97.1%. (Table 1)

Table 1
Desorption Efficiency

tube #	% recovered			
	52.1 µg	260 µg	521 µg	1042 µg
1	97.4	97.3	95.6	98.5
2	97.8	97.1	96.8	98.5
3	05.9	96.3	97.0	98.1
4	97.4	95.9	98.0	97.6
5	98.3	95.9	98.0	97.5
6	95.9	96.5	95.9	98.3
average	97.1	96.5	96.9	98.1

overall average = 97.1%
standard deviation = ±0.96

2.4 Retention efficiency

Six tubes were spiked with 1042 µg (9.8 ppm) benzyl alcohol, allowed to equilibrate 4 hours at room temperature, and had 24 liters humid air (81% RH) pulled through them at 0.2 Lpm. They were then opened, desorbed, and analyzed by GC-FID. The recoveries in table 2 are corrected for desorption efficiency. The retention efficiency averaged 99.1%. There was little benzyl alcohol found on the backup ('B') portions of the tubes. (Table 2)

Table 2
Retention Efficiency

tube #	% recovered		
	'A'	'B'	total
1	100	0.0	100
2	99.1	0.2	99.3
3	98.3	0.0	98.3
4	97.8	0.0	97.8
5	99.8	0.1	99.9
6	99.2	0.1	99.3

average = 99.1%

2.5 Storage

Tubes were spiked with 521 µg (4.91 ppm) benzyl alcohol and stored at room temperature until opened and analyzed. The recoveries in table 3 are corrected for desorption efficiency. The recoveries averaged 99.0% for the 14 days stored. (Table 3)

Table 3
Storage Study

day	% recovered
8	97.9
8	99.3
8	101
14	100
14	97.7
14	97.9

average = 99.0%

2.6 Precision

The precision was calculated using the area counts from six injections of each standard at concentrations of 52.1-, 260-, 521-, and 1042 µg/mL benzyl alcohol in the desorbing solvent. The pooled coefficient of variation was 0.00974. (Table 4)

Table 4
Precision Study

injection number	µg/mL			
	52.1	260	521	1042
1	25772	129634	264566	531424
2	25910	128549	262945	527847
3	25757	129078	262405	528930
4	25260	130614	263425	531787
5	25202	129180	265395	528788
6	25396	128878	265619	528942
average	25550	129322	264059	529620
standard deviation	±300	±727	±1331	±1595
CV	0.0177	0.00562	0.00504	0.00301

pooled CV = 0.00974

Where

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

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

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 was 24 Liters.

2.7.2 The sampling rate studied was 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 in designated areas.

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 HP 5890 gas chromatograph was used in this study.

3.1.2 GC column capable of separating the analyte and an internal standard from any interference. The column used in this study was a 60-m x 0.32-mm i.d. (1.0-µm d_f DB-1) capillary column.

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

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 Benzyl alcohol, Reagent grade

3.2.3 Methanol, HPLC grade

3.2.4 Dimethyl formamide (DMF), Reagent grade, used as internal standard, concentration of 0.25 µL/mL.

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, methanol with 0.25 µL/mL dimethyl formamide used as internal standard.

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 At least two separate standards are prepared by diluting a known quantity of benzyl alcohol with the desorbing solution at a concentration of 521 µg/mL with a 0.5 µL/mL internal standard.

3.4.2 Additional analytical standards should be prepared at higher and lower concentrations, by diluting one of the two standards (521 µg/mL), to check the linearity of the detector. For this study one standard at 52.1 µg/mL, two standards at 521 µg/mL, and one standard at 2083 µg/mL were used.

3.5 Analysis

3.5.1 Gas chromatograph conditions.

Flow rates	**(mL/min.)**	**Temperature**	**(°C)**
Nitrogen (make-up):	30	Injector:	180
Hydrogen (carrier)	2	Detector:	220
Hydrogen (detector):	60	Column:	80 ^{0}C, increase l0°/min to
Air:	420		160 ^{0}C
Injection size:	1 µL		
Chromatogram:	See Figure 1		

3.5.2 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 521 µg/mL benzyl alcohol in the desorbing solution. The linearity of the calibration was checked with standards of 52.1-, and 2083 µg/mL benzyl alcohol in the desorbing solution.

3.7.2 If the calibration is non-linear, two additional 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:

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

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

$$Volume\ of\ analyte = (moles\ of\ analyte)(molar\ volume\ at\ 25\ ^0C\ and\ 760\ mmHg)$$

$$ppm = \frac{(Volume\ of\ analyte)(10^6)}{(Air\ volume)}$$

* All units must cancel.

3.7.4 The above equations can be consolidated to form the following formula. To calculate the ppm of analyte in the sample based on a 24-liter air sample:

$$ppm = \frac{(\mu g / mL)(DV)(24.46)}{(24\ L)(DE)(MW)}$$

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
24 L = 24 liter air sample
DE = Desorption efficiency

3.7.5 This calculation is done for each section of the sampling tube and the results are then blank corrected and 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 chemicals.

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

4 Recommendations for further study

Method needs to be validated.

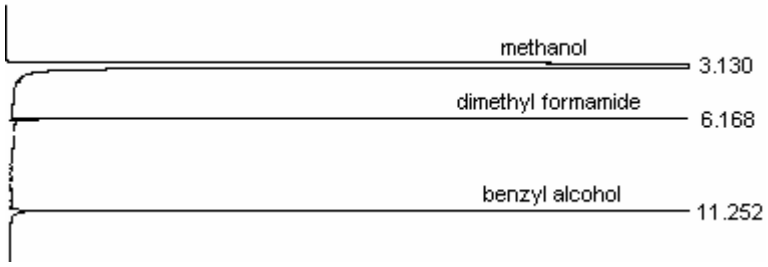


Figure 1 Analytical standard with 521 µg/mL benzyl alcohol and 0.25 µL/mL dimethyl formamide (internal standard) in methanol.

5 References

5.1 Cummins, K., Method 32, "Phenol and Cresol," Organic Methods Evaluation Branch, OSHA Salt Lake Technical Center, 1986.

5.2 Windholz, M., "The Merck Index," Eleventh Edition, Merck & Co., Rahway N.J., 1989, p. 176.

5.3 Lewis, R., "Hawley's Condensed Chemical Dictionary," Twelfth Edition, Van Nostrand Reinhold Co., New York, 1993, p. 134.