



Isooctyl alcohol

Method number: PV2033

Target concentration: 50 ppm (270 mg/m³) OSHA TWA PEL

Procedure: Samples are collected by drawing a known volume of air through a charcoal tube. Samples are desorbed with 1 mL of 1:99 dimethyl formamide: carbon disulfide 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.

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1 General Discussion

1.1 Background

1.1.1 History of procedure

OSHA promulgated an exposure standard for isooctyl alcohol in January 1989, at a level of 50 ppm. This study was undertaken to gather the data necessary to verify the procedure we had been recommending. The procedure for collection (charcoal tubes) and analysis (GC/FID) of isooctyl alcohol described in this evaluation is basically that used in OSHA method 07. (Ref. 5.1) Isooctyl alcohol contains several peaks that elute over a time range of 1 minute. A solution of 1 mL of 1:99 dimethyl formamide:carbon disulfide gives a constant desorption of 92.3% over the range of 0.25 mg to 2.49 mg. Isooctyl alcohol had good retention and storage efficiencies on charcoal tubes.

1.1.2 Potential workplace exposure (Ref. 5.2)

Isooctyl alcohol is used to form esters (such as phthalates) for plasticizing vinyl and other resins.

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

Toxicological data for isooctyl alcohol was unavailable. Documentation of Threshold Limit Values and Biological Exposure Indices contained data for 2-ethyl-1-hexanol (CAS# 104-76-7), not for isooctyl alcohol (CAS# 26952-21-6). (Ref. 5.3) 2-ethyl-1-hexanol is not a synonym for isooctyl alcohol. Isooctyl alcohol is a mixture of 3-, 4-, and 5-methyl heptanols. (Ref. 5.4) The name was chosen by the manufacturers as a simple, semi descriptive name for marketing purposes. (Ref. 5.2)

1.1.4 Physical properties (Ref. 5.3):

CAS:	26952-21-6
IMIS:	A635
RTECS:	NS7700000; 44590
DOT:	8604
Compound:	R-CH ₂ OH, R = heptyl radicals
Synonyms:	Isooctanol
Molecular weight:	130.23
Density:	0.834
Freezing point:	- 76 °C
Boiling point:	185 °C
Flash point:	84 °C (185 °F) (open cup)
Color:	Colorless liquid
Molecular formula:	C ₈ H ₁₈ O

1.2 Limit defining parameters

1.2.1 The detection limit of the analytical procedure is 41.5 ug, with a 1-uL injection volume. This is the smallest amount which could be detected under normal operating conditions.

1.2.2 The overall detection limit is 0.78 ppm. (All ppm values 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 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.

2 Sampling procedure

2.1 Apparatus

- 2.1.1 A calibrated personal sampling pump, the flow of which can be determined within $\pm 5\%$ at the recommended flow with the sample tube 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. x 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 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 Bults submitted for analysis must be shipped in a separate mailing container from other samples.

2.3 Desorption efficiency

Six tubes were liquid spiked at loadings of 0.25 mg (4.67 ppm), 0.83 mg (15.6 ppm), and 2.49 mg (46.7 ppm) isooctyl 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 1:99 dimethyl formamide:carbon disulfide for 30 minutes with occasional shaking, and

analyzed by GC-FID. The overall average was 92.3% recovered. (Table 1) Values for the retention efficiency and the storage study were corrected for the desorption efficiency.

Table 1
Desorption Efficiency

tube #	% recovered		
	0.25 µg	0.83 µg	2.49 µg
1	93.0	92.3	93.3
2	90.0	90.9	95.6
3	91.5	91.2	94.5
4	92.0	90.8	95.6
5	92.6	90.0	92.2
6	92.0	lost	92.5
average	91.9	91.0	94.0

average = 93.2%
standard deviation = ±1.68

2.4 Retention efficiency

Six tubes were liquid spiked with 2.49 mg (46.7 ppm) isooctyl alcohol, allowed to equilibrate overnight, and then had 10 liters humid air (85% RH) pulled through them. They were then opened, desorbed, and analyzed by GC-FID. The retention efficiency averaged 92.7%. There was no isooctyl alcohol found on the backup portions of the tubes. (Table 2)

Table 2
Retention Efficiency

tube #	% recovered		
	'A'	'B'	total
1	93.3	0.0	93.3
2	92.2	0.0	92.2
3	91.3	0.0	91.3
4	93.1	0.0	93.1
5	93.5	0.0	93.5
6	93.3	0.0	93.3

average = 92.7%

2.5 Storage

Tubes were spiked with 2.49 mg (46.7 ppm) isooctyl alcohol and stored at room temperature until opened and analyzed. The recoveries averaged 102% for the 14 days stored. (Table 3)

Table 3
Storage Study

day	% recovered
8	103
8	104
8	104
14	100
14	101
14	100
average	102%

2.6 Precision

The precision was calculated using the area counts from six injections of each standard at concentrations of 0.25, 0.83, 2.49 mg/mL isooctyl alcohol in 1:99 dimethyl formamide:carbon disulfide. The pooled coefficient of variation was 0.0115. (Table 4)

Table 4
Precision Study

injection number	0.25 mg/mL	0.83 mg/mL	2.49 mg/mL
1	68295	211712	624546
2	69661	213739	632078
3	68623	214503	627399
4	70118	214381	620275
5	70579	214725	630681
6	69951	220254	622585
average	69538	214886	626261
standard deviation –	±893	±2851	±4625
CV -	0.0128	0.0133	0.00738

pooled CV = 0.0115

Where:

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

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

A1, A2, A3, A4 = number of injections at each level
CV1, CV2, CV3, CV4 = Coefficients 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 HP 5890 II 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 μ m d_f DB-WAX) 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- μ L 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 Isooctyl alcohol, Reagent grade
- 3.2.3 Carbon disulfide, Reagent grade
- 3.2.4 Dimethyl formamide, HPLC grade
- 3.2.5 p-Cymene, Reagent grade
- 3.2.6 The desorbing solution is 0.25 μ L/mL p-cymene in 1:99 dimethyl formamide:carbon disulfide.

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 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 isooctyl alcohol with the desorbing solution. Standards prepared at 1 μ L/mL isooctyl alcohol in the desorbing solution are equivalent to 0.830 mg/mL.
- 3.4.2 At least two separate standards should be made. These standards should be within \pm 5%, and then are averaged.

3.4.3 A third analytical standard should be prepared at a higher concentration to check for linearity of the calibration. For this study, two standards at 1 µL/mL (0.830 mg/mL) and one standard at 4 µL/mL (3.32 mg/mL) isooctyl alcohols were used.

3.5 Analysis

3.5.1 Gas chromatograph conditions.

Flow rates	(mL/min)	Temperature	(°C)
Nitrogen (make-up):	32	Injector:	180
Hydrogen (carrier):	2	Detector:	240
Hydrogen (detector):	60	Column:	65
Air:	410		
Injection size:	1 µL		

Elution time: 10.8 to 11.9 min. (Chromatogram - see Figure 1)

3.5.2 Peak areas are measured by an integrator or other suitable means. The peak areas were summed.

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 A curve with area counts versus concentration is prepared from the calibration standards.

3.7.2 The area counts for the samples are plotted with the calibration curve to obtain the concentration of isooctyl alcohol in solution.

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

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

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

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

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

* All units must cancel.

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

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

Where:

$\mu g/mL$ = 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 = 10 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 chemicals.

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

4 Recommendations for further study

Collection studies should be performed.

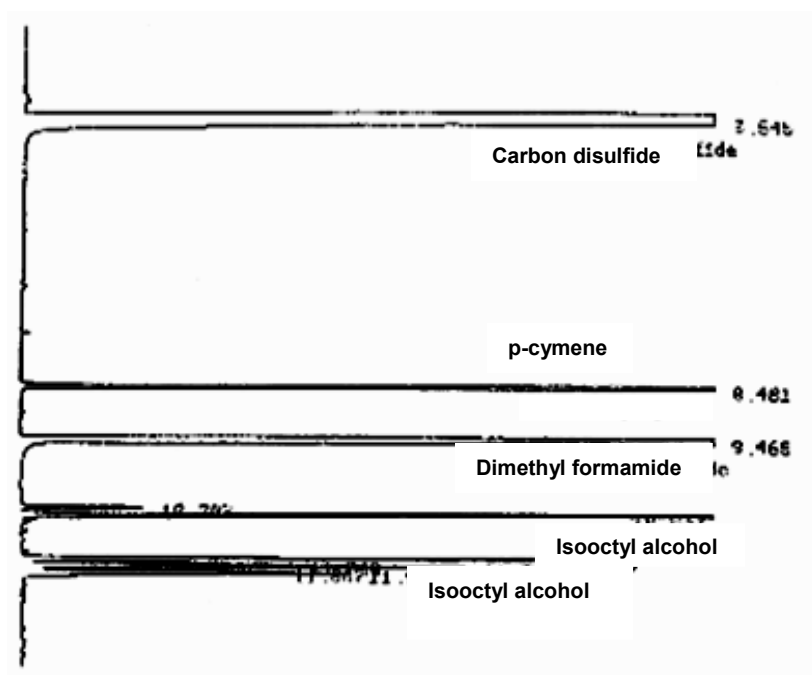


Figure 1. An analytical standard of 0.25 mg/mL isooctyl alcohol in 1:99 dimethyl formamide:carbon disulfide with 0.25 $\mu L/mL$ p-cymene internal standard. A chart speed of 1.0 cm/min was used.

5 References

- 5.1 Shulsky, M., Method 07, "Organic Vapors," Organic Methods Evaluation Branch, OSHA Analytical Laboratory, 1989.
- 5.2 "Kirk-Othmer Encyclopedia of Chemical Technology," Second Edition, John Wiley & Sons, Inc., New York, 1967, Vol. 14, p. 373-375.
- 5.3 "Documentation of the Threshold Limit Values and Biological Exposure Indices," Fifth Edition, American Conference of Governmental Industrial Hygienists, Inc., Cincinnati, 1986, p. 332.
- 5.4 Chem Service, Inc., Material Safety Data Sheet (MSDS) for Isooctyl Alcohol, July 1991.