



Butyl Acrylate

Method number: PV2011

Target Concentration:
ACGIH TLV-TWA: 2 ppm
NIOSH: 10 ppm (55 mg/m³)

Procedure: Samples are collected by drawing known volumes of air through glass sampling tubes containing charcoal adsorbent which has been coated with 4-*tert*-butylcatechol (TBC). Samples are desorbed with carbon disulfide and analyzed by gas chromatography (GC) using a flame ionization detector (FID).

Recommended air volume and sampling rate: 240 min at 0.05 L/min (12 L)

Detection limit of the overall procedure: 0.25 ppm (1.4 mg/m³) based on the recommended air volume and the analytical detection limit.

Status of method: Partially validated method. This method has been partially evaluated and is presented for information and trial use only.

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1 General Discussion

1.1 Background

1.1.1 History of procedure

This evaluation was undertaken to determine the effectiveness of the TBC-coated charcoal tube as a sampling device for butyl acrylate. TBC-coated charcoal tubes were initially used for 1,3-butadiene in OSHA Method 56 (Ref. 5.1).

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

Butyl acrylate has an oral LD₅₀ of 3.73 g/kg in rats (Ref. 5.2). The dermal LD₅₀ for rabbits was about 1.80 g/kg. Butyl acrylate was found to be moderately irritating to the skin, and as an eye irritant, produced corneal necrosis in the unwashed rabbit eye, similar to that produced by ethyl alcohol (Ref. 5.3).

1.1.3 Potential workplace exposure

The monomer of butyl acrylate is used in the manufacture of acrylate polymers and resins for textile and leather finishes. It is also used in paint formulations (Ref. 5.2).

1.1.4 Physical properties (Ref. 5.2 and 5.3)

CAS number:	141-32-2
IMIS number:	0450
Molecular weight:	128.17
Molecular formula:	C ₇ H ₁₂ O ₂
Density:	0.8986
Boiling point:	145 °C at 101.3 kPa (760 mmHg)
Solubility:	in water at 20 °C = 0.14 g/100 mL, at 40 °C = 0.12 g/100 mL
Chemical name:	Acrylic acid n-butyl ester; Butyl 2-propenoate
Appearance:	monomer: colorless, flammable liquid polymer: Elastic, tacky substance
Structure:	CH ₂ =CHCOOC ₄ H ₉

1.2 Limit defining parameters

The detection limit of the analytical procedure, including a 7:1 split ratio, is 2.4 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise.

2 Sampling Procedure

2.1 Apparatus

2.1.1 Samples are collected by using a personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device in line.

2.1.2 Samples are collected with 7.0 cm glass sampling tubes, 6-mm o.d. × 4-mm i.d., packed with two sections of charcoal that has been coated with 10% by weight of 4-*tertiary*-butylcatechol (TBC). The front section contains 100-mg and the back section contains 50-mg of TBC-coated charcoal. The sections are held in place with glass wool plugs. Tubes were purchased from SKC, Inc. (catalog no. 226-73).

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

- 2.3.1 Immediately before sampling, break off the ends of the TBC-coated charcoal tube. All tubes should be from the same lot.
- 2.3.2 Attach the sampling tube to the sampling pump with flexible tubing. Position the tube so that sampled air first passes through the 100-mg section.
- 2.3.3 Attach the tube vertically in the employee's breathing zone in such a manner that it does not impede work performance.
- 2.3.4 After sampling for the appropriate time, remove the sample and seal the tube with plastic caps.
- 2.3.5 Wrap each sample end-to-end with a Form OSHA-21 seal.
- 2.3.6 Record the air volume for each sample, and list any possible interference.
- 2.3.7 Submit at least one blank for each set of samples. Handle the blank in the same manner as the samples, except no air is drawn through it.
- 2.3.8 Submit bulk samples for analysis in a separate container. Do not ship them with air samples.

2.4 Desorption efficiency

Sixteen vials, each containing 100-mg portions of TBC-coated charcoal, were divided into four groups of four vials each. They were liquid spiked with 67.4, 337, 674, and 1348 μg of butyl acrylate, respectively. These represent 0.1 \times , 0.5 \times , 1.0 \times , and 2.0 \times the target concentration. The vials were stored overnight in a freezer (-5 °C), desorbed with 1.0 mL of the desorbing solution, shaken for 30 min, and analyzed as in Section 3. The average desorption efficiency was 100.1%. The results are listed in Table 2.4.

Table 2.4
Desorption Efficiency

sample #	µg spiked	µg found	% recovered
D6	67.4	69.4	103.0
D7	67.4	67.8	100.6
D8	67.4	69.3	102.8
D9	67.4	68.9	102.2
average of 0.1 x PEL = 102.2%			
D10	337	334	99.1
D11	337	346	102.7
D12	337	344	102.1
D13	337	351	104.2
average of 0.5 x PEL = 102.0%			
D1	674	617	91.5
D2	674	655	97.2
D3	674	668	99.1
D4	674	681	101.0
D5	0	0	blank
average of 1 x PEL = 97.2%			
DD1	1348	1327	98.4
DD2	1348	1310	97.2
DD3	1348	1334	99.0
DD4	1348	1354	100.4
average of 2 x PEL = 98.8%			

2.5 Retention efficiency

Five TBC-coated charcoal tubes were each liquid spiked with 3 µL (1× PEL) of a 225 µg/µL solution of butyl acrylate in carbon disulfide. These were allowed to dry for 2 hours and then 12 L of humid air (~80% relative humidity) were drawn through each tube at 0.05 L/min. The tubes were stored overnight in a freezer (-5 °C), desorbed with 1.0 mL of desorbing solution, shaken for 30 min and then analyzed as in Section 3. The results are listed in Table 2.5.

Table 2.5
Retention Efficiency

sample #	µg spiked	µg found	% recovered
1	674	638	94.7
2	674	635	94.2
3	674	654	97.0
4	674	637	94.5
5	674	649	96.3
average = 95.3%			

2.6 Sample storage

Ten TBC-coated charcoal tubes were each liquid spiked with 3 µL (1× PEL) of 225 µg/µL solution of butyl acrylate in carbon disulfide. These were allowed to dry for 2 hours and then 12 L of humid air (~80% relative humidity) were drawn through each tube at 0.05 L/min. Half of the tubes were stored in a drawer at ambient temperature, and the other half were stored in a

freezer (- 5 °C). After ten days, they were extracted and analyzed as in Section 3. The results are given in Tables 2.6.1 and 2.6.2.

Table 2.6.1
Ambient Storage

days stored	µg spiked	µg found	% recovered
10	674	629	93.3
10	674	629	93.3
10	674	635	94.2
10	674	635	94.2
10	674	634	94.1

average = 93.8%

Table 2.6.2
Freezer Storage

days stored	µg spiked	µg found	% recovered
10	674	633	93.9
10	674	652	96.7
10	674	649	96.3
10	674	654	97.0
10	674	649	96.3

average = 96.0%

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 12 L.

2.7.2 The recommended flow rate is 0.05 L/min.

2.8 Interferences (sampling)

It is not known if any compounds will interfere with the collection of butyl acrylate. Any suspected interferences should be reported to the laboratory with submitted samples.

2.9 Safety precautions (sampling)

2.9.1 Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2 Follow all safety practices 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 (FID). A Hewlett-Packard 5890 Gas Chromatograph equipped with a 7673A Autosampler and FID was used in this evaluation.

- 3.1.2 A GC column capable of separating butyl acrylate and the internal standard from any interference. A 60-m × 0.32 mm i.d. (0.25 µm d_f DX-4) capillary column was used in this evaluation.
- 3.1.3 An electronic integrator or some other suitable means to measure detector response. A Waters 860 Networking Computer System was used in this evaluation.
- 3.1.4 Volumetric flasks, pipettes, and syringes for preparing standards, making dilutions and performing injections.
- 3.1.5 Vials, 2-mL, and 4-mL, with PTFE-lined caps.

3.2 Reagents

- 3.2.1 Butyl acrylate. Reagent grade or better. Eastman Chemical Co.
- 3.2.2 Carbon disulfide - Desorbing solvent. Reagent grade or better.
- 3.2.3 *p*-Cymene – Internal standard. Reagent grade or better. (0.25 µL *p*-Cymene/mL Carbon disulfide). Aldrich Chemical Company, Inc.

3.3 Standard preparation

Prepare standards by diluting known volumes of butyl acrylate with desorbing solution. Standards must be used the day they are prepared.

3.4 Sample preparation

- 3.4.1 Transfer the 100-mg section of the sampling tube to a 2-mL vial. Place the 50-mg backup section in a separate 2-mL vial.
- 3.4.2 Add 1.0 mL of desorbing solution to each vial and seal with a PTFE-lined cap.
- 3.4.3 Shake vials for 30 minutes on a mechanical shaker then analyze samples.

3.5 Analysis

3.5.1 Instrument conditions

Column: 60-m × 0.32 mm i.d. (0.25 µm d_f DX-4)

GC Temperatures

Injector: 200 °C
 Detector: 220 °C
 Column: 70 °C (initial temp)

Temperature program hold initial temp 3 min, increase temp at 10 °C/min to 220 °C, hold temp 5 min

Gas flow rates

	<u>mL/min</u>
column:	1.5 (hydrogen)
septum purge:	1.2 (hydrogen)
FID:	19 (hydrogen)
FID:	10 (nitrogen)
FID:	480 (air)

Injection volume: 1 μ L
Split ratio: 7:1
Retention time: 1.97 min (butyl acrylate)
4.38 min (*p*-cymene)

3.5.2 An extended chromatographic run time was used to remove the late eluting TBC from the analytical column.

3.5.3 Chromatogram

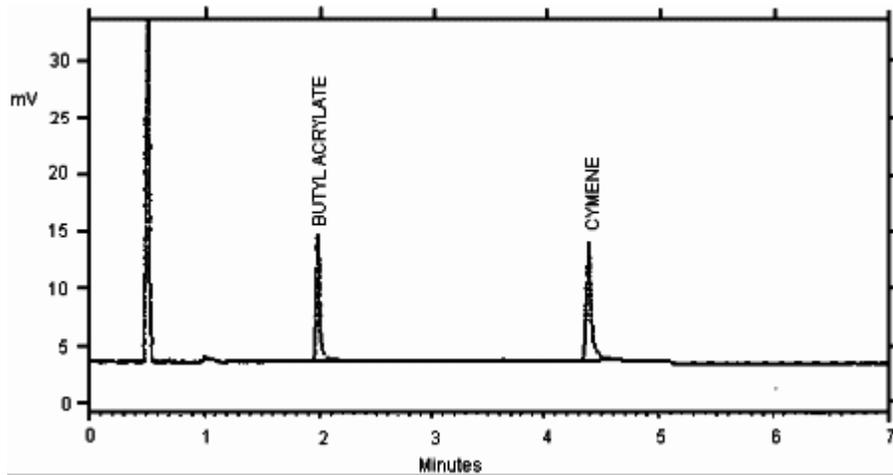


Figure 1. Chromatogram of butyl acrylate

3.5.4 Measure detector response using a suitable method such as electronic integration.

3.5.5 An internal standard (ISTD) calibration method is used. A calibration curve may be constructed by plotting concentration of analyte per sample versus ISTD-corrected response of standard injections. Bracket the samples with freshly prepared analytical standards over a range of concentrations.

3.6 Interferences (analytical)

3.6.1 Any collected compound which produces an FID response and has a similar retention time as butyl acrylate or the internal standard is a potential interference.

3.6.2 GC conditions may generally be varied to circumvent interferences.

3.6.3 Retention time on a single column is not proof of chemical identity. Analysis by an alternate GC column, high performance liquid chromatography (HPLC) and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 A calibration curve is constructed by plotting detector response versus ISTD-corrected response of standard concentration (μ g/mL) of butyl acrylate.

3.7.2 Determine the μ g/mL of butyl acrylate in both sections of each sample and blank from the calibration curve. If butyl acrylate is found on the backup section, it is added to the amount found on the front section. Blank corrections should be performed before adding the results together.

3.7.3 Determine the air concentration by using the following formula.

$$mg / m^3 = \frac{(\mu g / mL, \text{ blank corrected})(\text{desorption volume, mL})}{(\text{air volume, L})(\text{desorption efficiency, decimal})}$$

$$ppm = \frac{(mg / m^3)(24.46)}{(128.17)}$$

Where: 24.46 = molar volume (liters/mole) at 101.3 kPa, 760 mmHg and 25 °C
128.17 = molecular weight (g/mole) of butyl acrylate

3.8 Safety precautions (analytical)

3.8.1 Avoid skin contact and air exposure to butyl acrylate.

3.8.2 Avoid skin contact with all solvents.

3.8.3 Wear safety glasses at all times.

4 Recommendation for Further Study

This method should be fully validated.

5 References

5.1 "OSHA Analytical Methods Manual", 2nd ed., U.S. Department of Labor, Occupational Safety and Health Administration; OSHA Analytical Laboratory; Salt Lake City, UT, 1990; Method 56 - 1,3-Butadiene; American Conference of Government Industrial Hygienists (ACGIH); Cincinnati, OH, Publ. No. 4542.

5.2 Merck Index, 10th ed.; Windholz, Martha ed.; Merck: Rahway, N.J., 1983; p 1512.

5.3 Documentation of the Threshold Limit Values and Biological Exposure Indices, American Conference of Governmental Industrial Hygienist INC., fifth edition, 1986; p 75.