2-BUTANONE

Method no.:	84
Matrix:	Air
Target concentration:	200 ppm (590 mg/m^3)
Procedure:	Samples are collected by drawing air through glass sampling tubes containing Carbosieve S-III (carbon based molecular sieve) adsorbent. Samples are desorbed with a mixture of 99/1 (v/v) carbon disulfide (CS_2)/dimethylformamide (DMF) in the presence of anhydrous sodium sulfate and are analyzed by GC using a flame ionization detector.
Recommended air volume and sampling rate:	3 L at 0.05 L/min
Reliable quantitation limit:	79.9 ppb (236 µg/m^3)
Standard error of estimate at the target concentration: (Section 4.7)	8.4%
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

Date: July 1990 Chemist: Donald Burright

Organic Methods Evaluation Branch
OSHA Analytical Laboratory
Salt Lake City, Utah

1. General Discussion

1.1 Background

1.1.1 History

2-Butanone is one of the industrial solvents that exhibit storage stability problems when collected with charcoal sampling tubes. This problem was overcome in OSHA Method 16, where samples were collected with silica gel sampling tubes and analyzed on a GC equipped with a flame ionization detector (FID) after desorption with dimethyl sulfoxide (DMSO). (Ref. 5.1) In order to attain adequate sampler capacity, the sampling procedure of OSHA Method 16 requires two standard size (6-mm o.d. × 4-mm i.d. x 7 cm) silica gel tubes connected in series.

The purpose of this evaluation was to develop a sampling and analytical procedure for 2-butanone that would avoid the inconvenience of using two sampling tubes in series, as recommended in OSHA Method 16, and preserve the storage stability not achievable with charcoal sampling tubes. These goals were accomplished by using sampling tubes containing carbon molecular sieve adsorbent, Carbosieve S-III. Carbosieve S-III has been used earlier in OSHA Method 69 (Ref. 5.2) to overcome a capacity problem with acetone, a ketone similar to 2-butanone. The use of Carbosieve S-III also permitted to use of a 99/1 (v/v) carbon disulfide/dimethylformamide desorbing solution, which is less detrimental to analytical GC columns than the dimethyl sulfoxide required in OSHA Method 16.

This procedure does not invalidate OSHA Method 16, but provides an alternative to the inconvenience of using sampling tubes connected in series.

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

Inhalation of vapors may cause headache, nausea, vomiting, dizziness, drowsiness, irritation of the respiratory tract, and loss of consciousness. Contact with skin or eyes may cause irritation. Prolonged exposure may cause dermatitis. Liquid may cause permanent eye damage. Ingestion may cause nausea, vomiting, headaches, dizziness, gastrointestinal irritation. (Ref. 5.3) The TLV for 2-butanone (200 ppm) and the STEL (300 ppm) was established to prevent any injurious effects and to minimize complaints about odor and irritation. (Ref. 5.4) The OSHA PEL has been established equal to the TLV.

1.1.3 Workplace exposure

In 1985, 244 million kilograms were produced in the United States. The major uses of 2-butanone in 1981 were as follows: solvent for vinyl coatings, 30%; solvent for adhesives, 18%; solvent for acrylic coatings, 11%; solvent for other coatings, 7%; solvent for magnetic tapes, 7%; extraction solvent for lube oil dewaxing, 5%; solvent for printing inks, 5%. (Ref. 5.5) In 1978, NIOSH estimated that over 3 million workers are potentially exposed to 2-butanone in the United States (Ref. 5.6).

1.1.4 Physical properties and other descriptive information (Ref. 5.7, unless otherwise stated)

CAS no.:	78-93-3
molecular weight:	72.11
chemical formula:	$CH_3COCH_2CH_3$
melting point:	-86.4°C
boiling point:	79.6°C
vapor pressure:	10.33 kPa (77.5 mmHg) at 20°C
vapor density:	2.41 (air=1)
specific gravity:	0.805 (water=1)
explosive limits:	1.8% (lower) (Ref. 5.8) 11.5% (upper) (Ref. 5.8)

self-ignition temperature:	516°C (Ref. 5.8)
flash point:	-6°C (Ref. 5.9)
odor:	sharp, fragrant, acetone-like
odor threshold:	2 ppm
appearance:	colorless liquid
solubility:	27 g/100 mL of water; soluble in most common organic liquids
synonyms:	MEK; methyl ethyl ketone; ethyl methyl ketone; methyl acetone

The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations listed in ppm and ppb are referenced to 25°C and 101.3 kPa (760 mmHg).

1.2. Limit defining parameters

1.2.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 0.141 ng per injection (1.0-µL injection with a 5:1 split). This is the amount of analyte that will give a peak whose height is approximately 5 times the height of the baseline noise. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 0.707 µg per sample (79.9 ppb or 236 µg/m^3). This is the amount of analyte spiked on the sampling device that allows recovery of an amount of analyte equivalent to the detection limit of the analytical procedure. (Section 4.2)

1.2.3 Reliable quantitation limit

The reliable quantitation limit is 0.707 µg per sample (79.9 ppb or 236 µg/m^3). This is the smallest amount of analyte spiked on the sampling device that can be quantitated within the requirements of a recovery of at least 75% and a precision (±1.96 SD) of ±25% or better. (Section 4.3)

The reliable quantitation limit and detection limits reported in the method are based upon optimization of the instrument for the smallest possible amount of analyte. When the target concentration of analyte is exceptionally higher than these limits, they may not be attainable at the routine operating parameters.

1.2.4 Instrument response to the analyte

The instrument response over the concentration range of 0.5 to 2 times the target concentration is linear. (Section 4.4)

1.2.5 Recovery

The recovery of 2-butanone from samples used in 17-day storage test remained above 80.1% when the samples were stored at about 22°C. (Section 4.5, regression line of Figure 4.5.1)

1.2.6 Precision (analytical procedure only)

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1 and 2 times the target concentration is 0.0172. (Section 4.6)

1.2.7 Precision (overall procedure)

The precision at the 95% confidence level for the 17-day ambient temperature storage test is ±16.4%. (Section 4.7) This includes an additional ±5% for pump error. The overall procedure must provide results at the target concentration that are ±25% or better at the 95% confidence level.

1.2.8 Reproducibility

Six samples collected from a controlled test atmosphere and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after 7 days of refrigerated storage. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.8)

1.3 Advantages

1.3.1 This procedure allows the use of a single standard size (6-mm o.d. x 4-mm i.d. x 7 cm) adsorbent tube for the collection of 2-butanone.

1.3.2 The desorbing solvent is no longer DMSO, which has a detrimental effect on GC columns.

1.4 Disadvantages

1.4.1 The fine mesh size of Carbosieve S-III (60/80) results in a greater pressure drop across the sample tube than occurs with the conventional silica gel sampling tube. This results in the need for the 0.05 L/min sampling rate.

1.4.2 The recommended sample size is 3 L as opposed to the 10 L sample size of previous methods.

2. Sampling Procedure

2.1 Apparatus

2.1.1 Samples are collected using a personal sampling pump calibrated to within ±5% of the recommended flow rate with the sampling device attached.

2.1.2 Samples are collected with 4-mm i.d. x 6-mm o.d. x 7.0 cm glass sampling tubes packed with two sections of 60/80 mesh Carbosieve S-III. The front section contains 130 mg and the back section contains 65 mg of adsorbent. The sections are held in place with glass wool plugs. For this evaluation, Supelco's ORBO-91 Carbosieve S-III tubes (catalog no. 2-0360) were used.

2.2 Reagents

No sampling reagents are required.

2.3 Technique

2.3.1 Immediately before sampling, break off the ends of the Carbosieve S-III tube. All tubes should be from the same lot.

2.3.2 Attach the sampling tube to the sampling pump with flexible tubing. It is desirable to utilize sampling tube holders which have a protective cover that shield the employee from the sharp, jagged end of the sampling tube. Position the tube so that sampled air first passes through the 130-mg section.

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

2.3.4 Attach the sampler vertically in the worker's breathing zone in such a manner that it does not impede work performance or safety.

2.3.5 After sampling for the appropriate time, remove the sampler and seal the tube with plastic end caps. Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.6 Submit at least one blank sampler with each set of samples. Handle the blank sampler in the same manner as the other samples except draw no air through it.

2.3.7 Record sample volumes (in liters of air) for each sample, along with any potential interferences.

2.3.8 Ship any bulk samples in a container separate from the air samples.

2.4 Sampler capacity

The sampling capacity of the front section of a Carbosieve S-III sampling tube was determined by sampling a controlled test atmosphere containing 400 ppm (1080 mg/m^3, 77% relative humidity) of 2-butanone at ambient temperature. The sampling rate was 0.0516 L/min. The 5% breakthrough air volume was 11.1 L. (Section 4.9)

2.5 Desorption efficiency

2.5.1 The average desorption efficiency for 2-butanone from Carbosieve S-III adsorbent was 98.6% over the range of 0.5 to 2 times the target concentration. (Section 4.10.1)

2.5.2 Desorbed samples remain relativity stable for at least 24 h.(Section 4.10.2)

2.6 Recommended air volume and sampling rate

2.6.1. For time-weighted average samples, the recommended air volume is 3 L collected at 0.05 L/min (1-h samples). The 3-L air volume was selected so that the sampling time would be consistent with other sampling procedures using Carbosieve S-III.

2.6.2 For short-term exposure limit samples, the recommended air volume is 0.75 L collected at 0.05 L/min (15-min samples).

2.6.3 When short-term exposure limit samples are required, the reliable quantitation limit becomes larger. For example, the reliable quantitation limit is 0.320 ppm (0.943 mg/m^3) for 2-butanone when 0.75 L is collected.

2.7 Interferences (sampling)

2.7.1 It is not known if any compounds will severely interfere with the collection of 2-butanone on Carbosieve S-III. In general, the presence of other contaminant vapors in the air will reduce the capacity of Carbosieve S-III to collect 2-butanone.

2.7.2 Suspected interferences should be reported to the laboratory with submitted samples.

2.8 Safety precautions (sampling)

2.8.1 The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

2.8.2 All safety practices that apply to the work area being sampled should be followed.

2.8.3 Protective eyewear should be worn when breaking the ends of the glass Carbosieve S-III tubes.

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 an FID was used in this evaluation.

3.1.2 A GC column capable of separating 2-butanone and the internal standard from the desorbing solvent and any potential interferences. A 30-m x 0.32-mm i.d. SUPELCOWAX 10 (0.25-µm film thickness) capillary column (Supelco Inc.) was used in this evaluation.

3.1.3 An electronic integrator or some other suitable means of measuring detector response. A Hewlett-Packard 5895A GC ChemStation was used in this evaluation.

3.1.4. Two-milliliter vials with polytetrafluoroethylene-lined caps.

3.1.5. A dispenser capable of delivering 1.0 mL of desorbing solution is used to prepare standards and samples. If a dispenser is not available, a 1.0-mL volumetric pipet may be used.

3.2 Reagents

3.2.1 2-Butanone. Reagent grade or better should be used. The 2-butanone (b&j brand HIGH PURITY SOLVENT) used in this evaluation was purchased from American Burdick & Jackson (Muskegon, MI).

3.2.2 Carbon disulfide, CS_2. Reagent grade or better CS_2 should be used. The CS_2 (REAGENT ACS) was purchased from Fisher Scientific (Fair Lawn, NJ). In this evaluation, benzene-free CS_2 was used. The CS_2 had been passed through Molecular Sieve 13X (45/60 mesh) to remove the benzene contamination. Fifty grams of molecular sieve should remove the benzene from 1 L of carbon disulfide.

3.2.3 Dimethylformamide, DMF. Reagent grade or better should be used. The DMF (b&j brand HIGH PURITY SOLVENT) used in this evaluation was purchased from American Burdick & Jackson (Muskegon, MI).

3.2.4 Sodium sulfate, anhydrous. Sodium sulfate is used as a drying agent. The sodium sulfate (AR grade) used in this evaluation was purchased from Mallinckrodt (Paris, KY).

3.2.5 Desorbing solution. This consists of a solution of 99:1 (v/v) benzene-free CS_2/DMF. An internal standard such as ethyl benzene can be used.

3.2.6 Ethyl benzene. This was used as the internal standard in the desorbing solution. The solution is prepared by adding 250 µL of ethyl benzene to 1 L of desorbing solution. The ethyl benzene (reagent grade) used in this evaluation was purchased from Eastman Kodak (Rochester, NY).

3.3 Standard preparation

3.3.1 Prepare concentrated stock standards by diluting the 2-butanone with DMF. Prepare working analytical standards by injecting microliter amounts of concentrated stock standards into 2-mL vials containing 1 mL of desorbing solution delivered from the same dispenser used to desorb samples. For example, to prepare a target level standard, inject 10 µL of a stock solution containing 177 mg/mL 2-butanone in DMF into 1 mL of desorbing solution.

3.3.2 Prepare a sufficient number of analytical standards to generate a calibration curve. Ensure that the amount of 2-butanone found in the samples is bracketed by the range of the standards. Prepare additional standards if necessary.

3.4 Sample preparation

3.4.1 Remove the plastic caps from the sample tube and carefully transfer each section of the adsorbent to separate vials. Discard the glass tube and glass wool plugs.

3.4.2 Add approximately 150 mg of anhydrous sodium sulfate to each sample.

3.4.3 Add 1.0 mL of desorbing solution to each vial and immediately seal the vials with polytetrafluoroethylene-lined caps.

3.4.4 Shake the vials vigorously several times during the next 30 min.

3.5 Analysis

3.5.1 Analytical conditions

GC conditions

temperatures:	40°C (column) 200°C (injector) 220°C (detector)
temp program:	hold initial temp 1.0 min, increase temp at 5°C/min to 65°C, then increase temp at 25°C/min to 190°C.
column gas flow:	1.2 mL/min (hydrogen)
septum purge:	1.5 mL/min (hydrogen)
injection size:	1.0 µL (5:1 split)
column:	30 m x 0.32-mm i.d. capillary SUPELCOWAX 10 (0.25-µm film thickness)
retention times:	2.75 min (2-butanone) 5.75 min (ethyl benzene)

FID conditions

hydrogen flow:	34 mL/min
air flow:	450 mL/min
nitrogen makeup flow:	33 mL/min
chromatogram:	Figure 3.5.1

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

3.5.3 An internal standard (ISTD) calibration method is used. A calibration curve can be constructed by plotting micrograms of 2-butanone 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 compound that produces an FID response and has a similar retention time as the analyte or internal standard is a potential interference. If any potential interferences were reported, they should be considered before samples are desorbed. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

3.6.2 Retention time on a single column is not considered proof of chemical identity. Analysis by an alternate GC column or confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

The analyte concentration for samples is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for desorption efficiency. The air concentration is calculated using the following formulae. The back (65-mg) section is analyzed primarily to determine if there was any breakthrough from the front (130-mg) section during sampling. If a significant amount of analyte is found on the back section (e.g., greater than 25% of the amount found on the front section), this should be reported with sample results. If any analyte is found on the back section, it is added to the amount on the front section. This total amount is then corrected by subtracting the total amount (if any) found on the blank.

$$mg/m^3 = \frac{\text{microgram of analyte per sample, blank corrected}}{\text{liters of air sampled} \times \text{desorption efficiency}}$$

$$ppm = \frac{mg/m^3 \times 24.46}{\text{molecular weight of analyte}}$$

where 24.46 = molar volume (liters) at 101.3 kPa (760 mmHg) and 25°C
molecular weight = 72.11

3.8 Safety precautions (analytical)

3.8.1 Restrict the use of all chemicals to a fume hood.

3.8.2 Avoid skin contact and inhalation of all chemicals.

3.8.3 Wear safety glasses and a lab coat at all times while in the laboratory areas.

4. Backup Data

4.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 0.141 ng per injection, based on a 1.0-µL injection (with a 5:1 split) of a 0.707 µg/mL standard. This amount produced a 2-butanone peak whose height is about 5 times the height of the baseline noise. A chromatogram of the detection limit of the analytical procedure is shown in Figure 4.1.

4.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 0.707 µg per sample (79.9 ppb or 236 µg/m^3). The injection size listed in the analytical procedure (1.0 µL, 5:1 split) was used in the determination of the detection limit of the overall procedure. Eight vials containing 130 mg of Carbosieve S-III resin were spiked with 0.707 µg of 2-butanone. The samples were desorbed about 24 h after being spiked.

Table 4.2
Detection Limit of the Overall Procedure for 2-Butanone

sample no.	µg spiked	µg recovered
1	0.707	0.677
2	0.707	0.689
3	0.707	0.743
4	0.707	0.756
5	0.707	0.747
6	0.707	0.720
7	0.707	0.722
8	0.707	0.739

4.3 Reliable quantitation limit data

The reliable quantitation limit is 0.707 µg per sample (79.9 ppb or 236 µg/m^3). The injection size listed in the analytical procedure (1.0 µL, 5:1 split) was used in the determination of the reliable quantitation limit. Eight vials containing 130 mg of Carbosieve S-III resin were liquid-spiked with 0.707 µg of 2-butanone. Because the recovery of 2-butanone from the spiked samples was greater than 75% and had a precision of ±25% or better, the detection limit of the overall procedure and reliable quantitation limit are the same.

Table 4.3
Reliable Quantitation Limit
(Based on samples and data of Table 4.2)

percent recovered	statistics
95.8	$\overline{X}$ = 102.4
97.4	SD = 4.0
105.1	Precision = (1.96)(±4.0)
106.9	= ±7.8
105.6	
101.8	
102.1	
104.5	

4.4 Instrument response to 2-butanone

The instrument response to 2-butanone over the range of 0.5 to 2 times the target concentration is linear with a slope of 14.47 (in ISTD-corrected area counts per microgram/milliliter). The precision of the response to 2-butanone was determined by multiple injections of 2-butanone standards. The data below is presented graphically in Figure 4.4.

Table 4.4
Instrument Response to 2-Butanone
Injection Split = 5:1

× target conc µg/mL	0.5× 885	1× 1770	2× 3540
area counts	12714	25768	50082
	13154	25721	51115
	12992	26147	51561
	13173	26068	50283
	12749	25967	50497
	12629	25817	52899
	12676	25528	50759
	13062	25527	53058
$\bar{X}$	12894	25818	51282

4.5 Storage data

Storage samples are generated by sampling the recommended air volume at the recommended sampling rate from a test atmosphere at 80% relative humidity containing 2-butanone at the target concentration. Samples were generated by sampling from an atmosphere containing 2-butanone at 2 times the target concentration. Thirty-six storage samples were collected by sampling a dynamically generated atmosphere containing 1080 mg/m^3 or 400 ppm of 2-butanone and 77% relative humidity for 30 min at 0.05 L/min. One-half of the tubes was stored in a freezer (-20°C) and the other half was stored in a closed drawer at ambient temperature (about 22°C). At 3-4 day intervals, three samples were selected from each of the two storage sets and analyzed. The results are listed below and shown graphically in Figures 4.5.1 and 4.5.2.

Table 4.5
Storage Test of 2-Butanone

storage time (days)	% recovery (ambient)			% recovery (refrigerated)		
0	116.2	103.1	98.0	116.2	103.1	98.0
	97.0	87.9	97.7	97.0	87.9	97.7
4	96.8	94.1	99.9	99.9	93.9	89.0
7	84.1	87.2	88.6	96.6	86.3	87.7
11	83.0	87.9	80.1	91.7	89.0	89.3
14	77.8	77.0	81.9	91.1	89.3	93.6
17	87.9	82.4	90.5	82.3	92.7	91.2

4.6 Precision (analytical method)

The precision of the analytical procedure is defined as the pooled coefficient of variation determined from replicate injections of 2-butanone standards at 0.5, 1 and 2 times the target concentration. Based on the data of Table 4.4, the coefficients of variation (CV) for the three levels and the pooled coefficient of variation ($\overline{CV}$) were calculated and are listed below.

Table 4.6
Precision of the Analytical Method
(Based on the Data of Table 4.4)

× target conc µg/mL	0.5× 885	1× 1770	2× 3540
SD[1]	225	231	1147
CV	0.01746	0.00895	0.02236
$\overline{CV}$	0.0172		

[1]standard deviation is in area counts

4.7 Precision (overall procedure)

The precision of the overall procedure is determined from the storage data. The determination of the standard error of estimate (SEE) for a regression line plotted through the graphed storage data allows the inclusion of storage time as one of the factors affecting overall precision. The SEE is similar to the standard deviation, except it is a measure of dispersion of data about a regression line instead of about a mean. It is determined with the following equation:

$$SEE = \sqrt{\frac{\sum(Y_{obs} - Y_{est})^2}{n - k}}$$

where

n = total number of data points
k = 2 for linear regression
k = 3 for quadratic regression
Y_{obs} = observed % recovery at a given time
Y_{est} = estimated % recovery from the regression line at the same given time

An additional 5% for pump error is added to the SEE by the addition of variances. The precision at the 95% confidence level is obtained by multiplying the SEE (with pump error included) by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). The 95% confidence intervals are drawn about their respective regression line in the storage graph as shown in Figure 4.5.1. The data for Figure 4.5.1 was used to determine the SEE of ±8.4% and the precision of the overall procedure of ±16.4%.

4.8 Reproducibility data

Six samples, collected from a dynamically generated atmosphere containing 2-butanone, were given to a chemist unassociated with this study. The samples were generated by drawing a 423-ppm test atmosphere through sampling tubes for 20-60 min at approximately 0.050 L/min. The samples were analyzed after being stored for 7 days at 5°C. No sample result had a deviation greater than the precision of the overall procedure, which is ±16.4%.

Table 4.8
Reproducibility Data

µg spiked	µg recovered	% recovered	% deviation
3490	2978	85.3	-14.7
3680	3275	89.0	-11.0
1763	1608	91.2	-8.8
1774	1633	92.1	-7.9
1215	1082	89.1	-10.9
1173	1113	94.9	-5.1

4.9 Sampler capacity

Sampler capacity was determined by sampling from a dynamically generated atmosphere of 400 ppm (1080 mg/m^3) 2-butanone with a Carbosieve S-III sampling tube that contained only the front section. The tube was followed by a whole Carbosieve S-III sampling tube. The backup tube was periodically changed over a 4 h time. The relative humidity of the test atmosphere was 77%. The sampling rate was 0.0516 L/min. The air volumes listed below are the midpoints of each sampling interval plus the total of all preceding sampling intervals. The data is graphically shown in Figure 4.9.

Table 4.9
Breakthrough on Carbosieve S-III Tube

air vol (L)	sample time (min)	downstream (mg/m^3)	breakthrough (%)
2.322	45.0	0	0
5.031	97.5	0	0
5.805	112.5	0	0
6.631	128.5	0	0
7.405	143.5	0	0
8.179	158.5	0	0
8.953	173.5	0	0
9.727	188.5	0	0
10.50	203.5	18.5	1.71
11.27	218.5	75.4	6.99
12.05	233.5	206.6	19.1

4.10 Desorption efficiency and stability of desorbed samples

4.10.1 Desorption efficiency

The desorption efficiency (DE) of 2-butanone was determined by liquid-spiking 130-mg portions of Carbosieve S-III adsorbent with 2-butanone at 0.5 to 2 times the target concentrations. These samples were stored overnight and then desorbed with desorbing solution and analyzed. The average desorption efficiency over the studied range was 98.6%.

Table 4.10.1
Desorption Efficiency of 2-Butanone

× target conc µg/sample	0.5× 885	1× 1770	2× 3540
DE, %	100.7	101.1	95.9
	96.9	100.0	96.9
	96.4	101.6	96.9
	99.6	99.3	98.7
	99.7	95.9	98.7
	96.1	102.7	98.4
$\overline{X}$	98.2	100.1	97.6

4.10.2 Stability of desorbed samples

The stability of desorbed samples was investigated by reanalyzing the target concentration samples 24 h after initial analysis. The original analysis was performed and the vials were not recapped after injection. The samples were reanalyzed with fresh standards. The average recovery, compared to the average recovery of the original analysis, was 95.8 or a -4.3% change.

Table 4.10.2
Stability of Desorbed Samples

initial recovery (percent)	recovery after 24 h (percent)	percent change
101.1	97.5	-3.6
100.0	95.9	-4.1
101.6	88.8	-12.8
99.3	97.7	-1.6
95.9	94.3	-1.6
102.7	100.3	-2.4

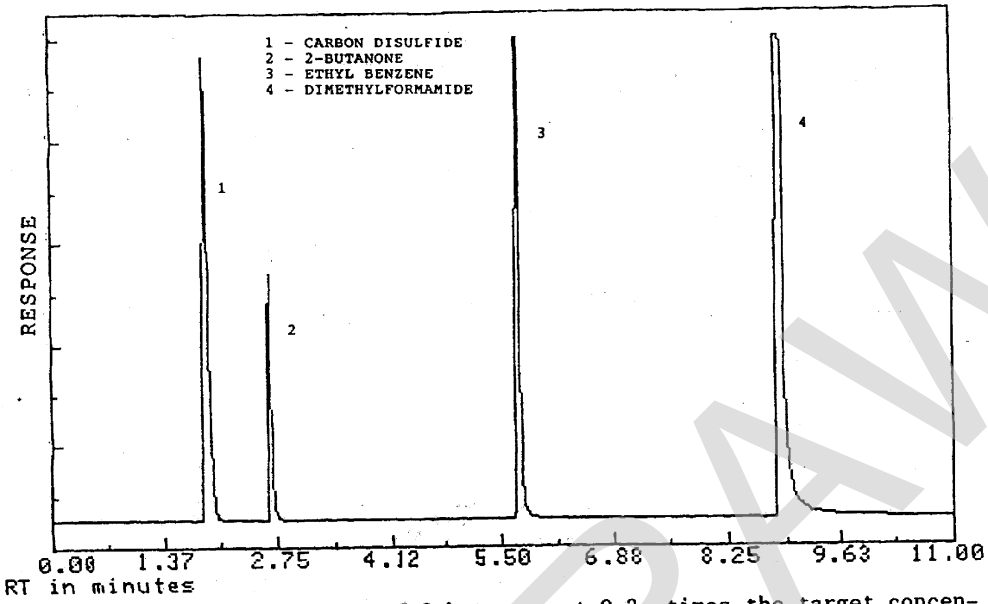

Figure 3.5.1. Chromatogram of 2-butanone at 0.2 times the target concentration.

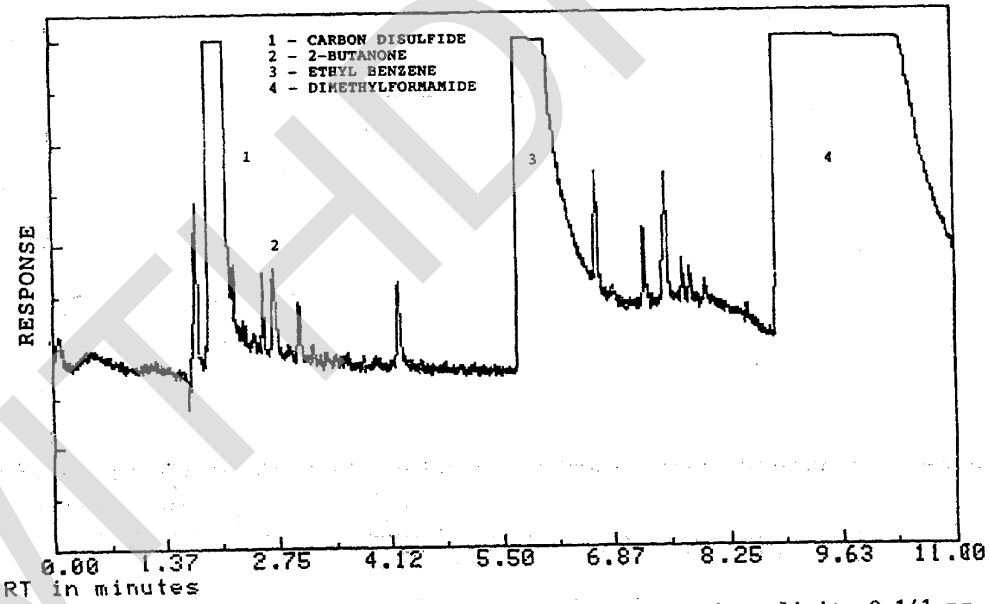

Figure 4.1. Chromatogram of 2-butanone at the detection limit, 0.141 ng per injection, injection split = 5:1.

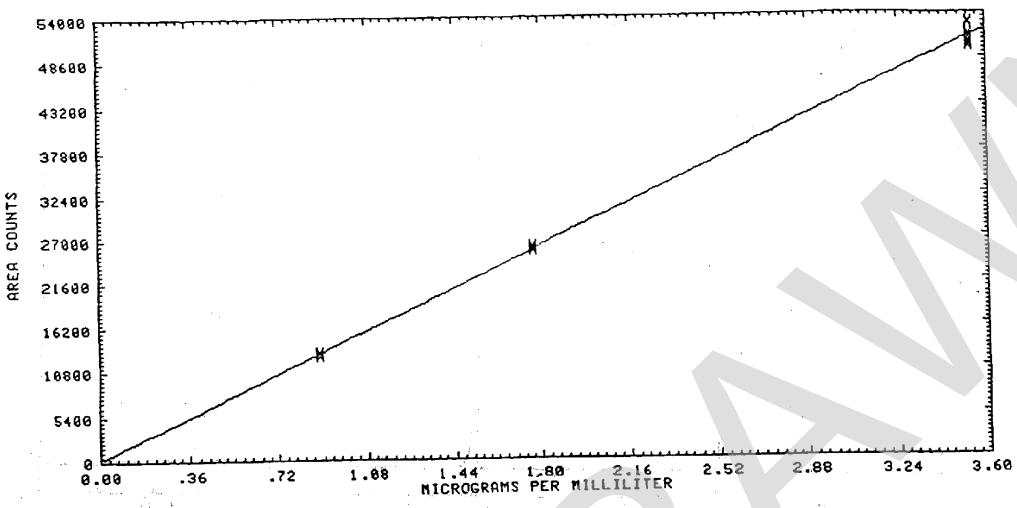

Figure 4.4. Instrument response curve for 2-butanone, slope = 14.47 area counts per micrograms per milliliter, injection split = 5:1.

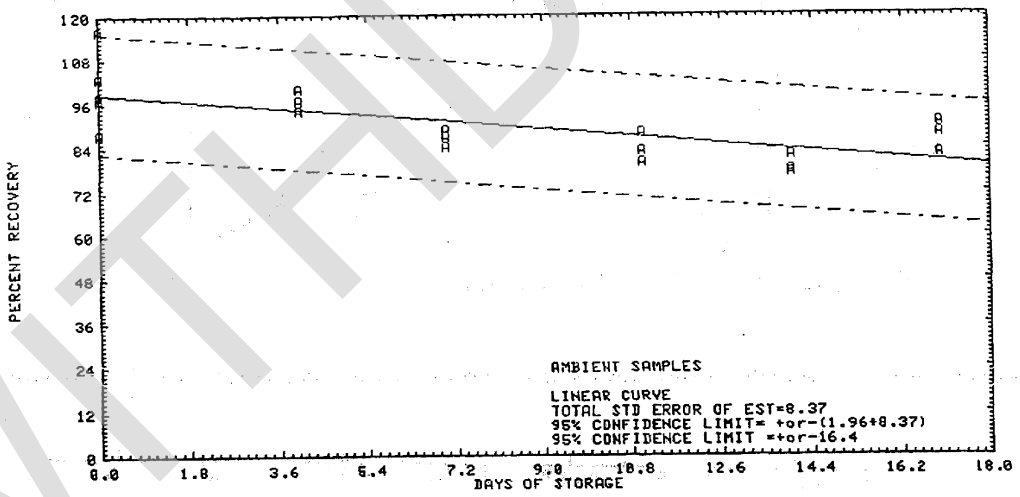

Figure 4.5.1. Ambient storage test for 2-butanone.

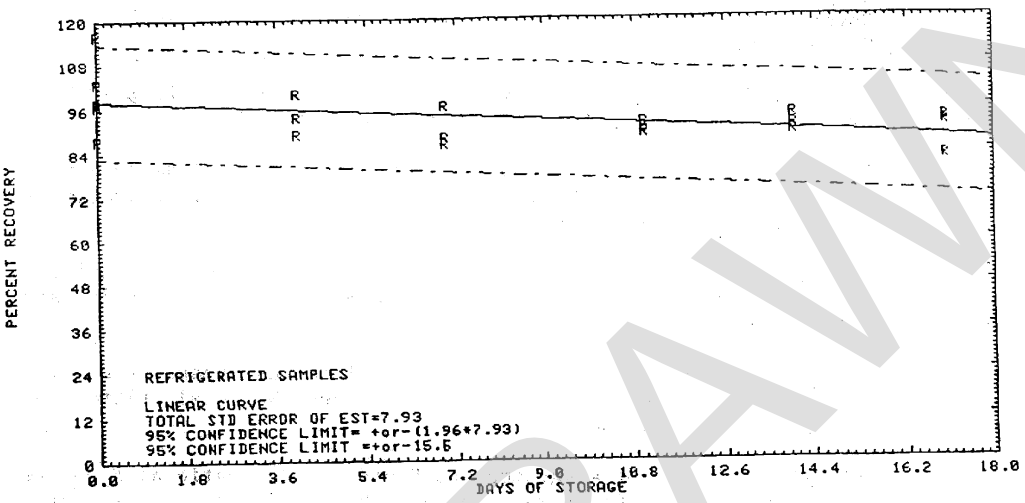

Figure 4.5.2. Refrigerated storage test for 2-butanone.

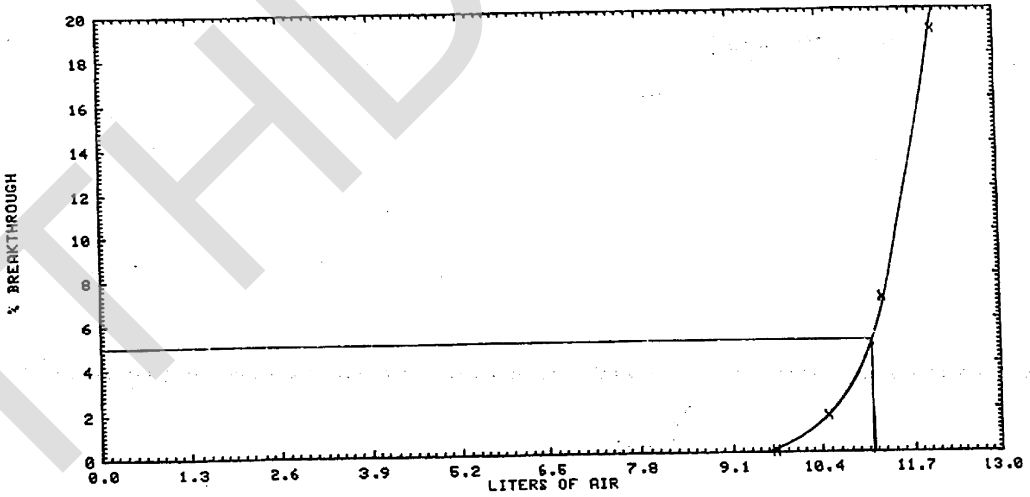

Figure 4.9. Determination of the 5% breakthrough air volume.

5. References

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5.6 "NIOSH Criteria for a Recommended Standard: Occupational Exposure to Ketones", U.S. Department of Health, Education, and Welfare, PHS/CDC/NIOSH, pp 23-24, June, 1978.

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5.9. Papa, Anthony J., Sherman, Paul D. in "Kirk-Othmer Encyclopedia of Chemical Technology"; 3rd ed.; Grayson, M., Ed.; John Wiley & Sons, New York, 1983, Vol. 13, p 899.