Desflurane

Method number:	106
Matrix:	Air
Target concentrations:	1 ppm (6.9 mg/m3) and 75 ppm (515 mg/m3)
OSHA PEL:	None
ACGIH TLV:	None
Procedure:	Samples are collected by drawing a known volume of air through standard size (6-mm o.d., 140/70) Anasorb 747 tubes. Samples are desorbed with toluene and analyzed by GC using a flame ionization detector (FID).
Recommended air volume and sampling rate:	3 L at 0.05 L/min
Reliable quantitation limit:	33.1 ppb (228 µg/m3)
Standard error of estimate at the target concentrations:	5.3% at 1 ppm 5.6% at 75 ppm
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

Date: April 1995 Chemist: Donald Burright

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Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

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1. General Discussion

1.1 Background

1.1.1 History

This method is an extension of the work that was done earlier to produce OSHA Method 103. (Ref. 5.1) Desflurane was identified as a new anesthetic gas undergoing testing for FDA approval during the earlier work. FDA approved desflurane for use in the general population in September 1992. Desflurane was not included in Method 103 because it required different analytical conditions. Initial studies were conducted using both Anasorb CMS and Anasorb 747 as adsorbents for collection of desflurane to parallel the method used for isoflurane, halothane and enflurane. Anasorb 747 was selected as the adsorbent for collection because a higher desorption efficiency was obtained. The desorbing solvent was changed from carbon disulfide to toluene. This reduced the loss of desflurane from the sample which was caused by the heat generated when carbon disulfide was added to the adsorbent. The evaluation was performed at two target concentrations, 1 and 75 ppm, to parallel the earlier work. There is no OSHA PEL or ACGIH TWA for desflurane, but NIOSH has a recommended exposure limit (REL) for halogenated anesthetic gases of 2 ppm as a 60-min ceiling value (Refs. 5.2 and 5.3). Because OSHA sometimes sets the TWA concentration at about one-half of the ceiling value, the REL is the basis for the lower target concentration. The higher target concentration was chosen because of the close structural similarity between desflurane and enflurane, which has an ACGIH TLV of 75 ppm.

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

Acute exposure to desflurane may cause irritation and redness to the eyes, and dryness and irritation to the skin. Overexposure by inhalation can lead to headaches, dizziness, drowsiness, unconsciousness or death. Irritation of the mouth and throat can occur with an acute exposure by inhalation. Acute exposure by ingestion may lead to unconsciousness or death. (Ref. 5.4)

1.1.3 Workplace exposure

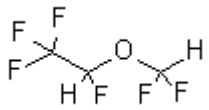
Desflurane is a new organic anesthetic gas and may be found in operating rooms, teaching hospitals, dental offices, and veterinary hospitals. The number of people potentially exposed is not known but will rise as desflurane is accepted into general use.

1.1.4 Physical properties and other descriptive information (Ref. 5.4)

CAS number:	57041-67-5
molecular weight:	168.04
boiling point, °C:	22.8
color:	clear
specific gravity:	1.47 @ 15°C
molecular formula:	C3H2OF6
vapor pressure,	

kPa (mmHg):	89.2 (669.2) @ 20°C
odor:	mild, ethereal
flash point:	>93°C (CC)
synonyms:	Suprane™; 1,2,2,2-tetrafluoroethyl difluoromethyl ether; I-653

structural formula:



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 13.6 pg. This is the amount of analyte that will give a response that is significantly different from the background response of a reagent blank. (Sections 4.1 and 4.2)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 0.205 µg per sample (9.93 ppb or 68.4 µg/m3). This is the amount of analyte spiked on the sampler that will give a response that is significantly different from the background response of a sampler blank. (Sections 4.1 and 4.3)

1.2.3 Reliable quantitation limit

The reliable quantitation limit is 0.683 µg per sample (33.1 ppb or 228 µg/m3). This is the amount of analyte spiked on a sampler that will give a signal that is considered the lower limit for precise quantitative measurements. (Section 4.4)

1.2.4 Precision (analytical procedure)

The precisions of the analytical procedure, measured as the pooled relative standard deviation over a concentration range equivalent to the range of 0.5 to 2 times the target concentration are 0.58% and 0.46% for the lower and higher target concentrations, respectively. (Section 4.5)

1.2.5 Precision (overall procedure)

The precisions of the overall procedure at the 95% confidence level for the ambient temperature 18- and 16-day storage tests (at the target concentration) are 10.4% and 11.0% for the lower and higher target concentrations, respectively. This includes an additional 5% for sampling error. (Section 4.6)

1.2.6 Recovery

The recoveries of desflurane from samples used in the 18- and 16-day storage tests remained above 99.7% and 100.2% for the lower and higher target concentrations, respectively when the samples were stored at 22°C. (Section 4.7)

1.2.7 Reproducibility

Twelve samples spiked by liquid injection were submitted for analysis by one of the OSHA Salt Lake Technical Center's service branch laboratories. The samples were analyzed according to the instructions in a draft copy of this procedure after 10 days of storage at 4°C. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.5. (Section 4.8)

2. Sampling Procedure

2.1 Apparatus

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

2.1.2 Samples are collected with 7-cm × 4-mm i.d. × 6-mm o.d. glass sampling tubes packed with two sections of Anasorb 747 (140/70 mg). The sections are held in place with a glass wool plug and two urethane foam plugs. For this evaluation, commercially prepared sampling tubes were purchased from SKC, Inc. (catalog no. 226-81A).

2.2 Reagents

None required.

2.3 Technique

2.3.1 Only properly trained personnel can sample in an operating room or dental office, this is necessary to be in compliance with OSHA's Exposure Control Plan for blood born pathogens. (Ref. 5.5)

2.3.2 Break off the ends of the sampling tube immediately before sampling. All tubes should be from the same lot.

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

2.3.4 Sampled air should not pass through any hose or tubing before entering the sampling tube.

2.3.5 Attach the sampler vertically with the larger section pointing downward in the worker's breathing zone to avoid channeling. Position the sampler so it does not impede work performance or safety.

2.3.6 Remove the sampling tube and seal it with plastic end caps immediately after sampling for the appropriate time.

2.3.7 In order t prevent occupational exposure to SLTC personnel, sampling tubes that may become contaminated with blood or other potentially infectious materials are to be examined prior to shipping and decontaminated (e.g., wiped off with bleach or other disinfectant) as necessary. Contaminated items are not to placed or stored in areas where food is kept, and decontamination should be accomplished as soon as possible following the inspection where contamination occurred. Decontamination is not to take place in any area where food or drink is consumed. (Ref. 5.5)

2.3.8 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.9 Submit at least one blank sample with each set of samples. Handle the blank sampling tube in the same manner as the other samples, except draw no air through it.

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

2.3.11 Ship any bulk sample(s) in a container separate from the air samples.

2.3.12 Submit the samples to the laboratory for analysis as soon as possible after sampling. If delay is unavoidable, store the samples at reduced temperature.

2.4 Sampler capacity

Sampler capacity is determined by measuring how much air can be sampled before the analyte breaks through the sampler, i.e., the sampler capacity is exceeded. Breakthrough is considered to occur when the effluent from the sampler contains a concentration of analyte that is 5% of the upstream concentration (5% breakthrough). Testing for breakthrough was performed by using an FID to monitor the effluent from sampling tubes containing only the 140-mg section of Anasorb 747. Dynamically generated test atmospheres, which were about two times the higher target concentration, were used for the capacity tests. The samples were collected at 0.05 L/min and the relative humidity was about 80% at 25°C. The 5% breakthrough air volume was calculated from the data of duplicate determinations and is 3.83 L. (Section 4.9)

2.5 Desorption efficiency

2.5.1 The average desorption efficiencies for desflurane from Anasorb 747 over the range of 0.5 to 2.0 times the target concentrations were 101.1% and 102.9% for the lower and higher target concentration respectively. (Section 4.10)

2.5.2 The desorption efficiencies at 0.05, 0.1 and 0.2 times the target concentrations (TC) were found to be very high and are listed below. (Section 4.10)

Table 2.5.2
Desorption Efficiencies at 0.05 to 0.2 times TC, %

TC	0.05×TC	0.1×TC	0.2×TC
low	98.0	103.2	100.2
high	100.0	100.2	101.0

2.5.3 Desorbed samples remain stable for at least 20 and 33 h for the lower and higher target concentration respectively.

2.6 Recommended air volume and sampling rate

2.6.1 For long-term samples, collect 3 L at 0.05 L/min.

2.6.2 For short-term samples, collect 0.75 L at 0.05 L/min.

2.6.3 When short-term samples are collected, the air concentration equivalent to the reliable quantitation limit becomes larger. For example, the reliable quantitation limit is 133 ppb (911 µg/m3) when 0.75 L is sampled.

2.7 Interferences (sampling)

2.7.1 There are no known compounds that will severely interfere with the collection of desflurane on Anasorb 747. In general, the presence of other contaminant vapors in the air will reduce the capacity of Anasorb 747 to collect desflurane.

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 sampling tubes.

3. Analytical Procedure

3.1 Apparatus

3.1.1 Gas chromatograph equipped with an FID. For this evaluation, a Hewlett-Packard 5890A Gas Chromatograph equipped with a 7673A Automatic Sampler was used. A Forma Scientific Model 2006 refrigerated circulator was used to cool the sample tray of the HP 7673A to 10°C.

3.1.2 A GC column capable of separating the analyte of interest from the desorption solvent, internal standard and any interferences. A 60-m × 0.32-mm i.d. fused silica SPB-1® column with a 4-µm df (Supelco, Inc, Bellefonte, PA) was used in the evaluation.

3.1.3 An electronic integrator or some other suitable means of measuring peak areas. A Waters 860 Networking Computer System was used in this evaluation.

3.1.4 Two-milliliter vials with poly(tetrafluoroethylene)-lined caps.

3.1.5 A dispenser capable of delivering 1.0 mL of desorbing solvent 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 Desflurane, USP. The desflurane used in this evaluation was manufactured by Anaquest (Liberty Corner, NJ).

3.2.2 Toluene, chromatographic grade or better. The toluene used in this evaluation was Optima Grade and was purchased from Fisher Scientific (Fair Lawn, NJ).

3.2.3 Desorption solvent. The desorption solvent was toluene and the benzene contaminant was used as the internal standard.

3.2.4 GC grade nitrogen, air, and hydrogen.

3.3 Standard preparation

3.3.1 Prepare two concentrated stock standards of desflurane in toluene. Prepare working analytical standards by injecting microliter amounts of concentrated stock standards into 2-mL vials containing 1 mL of desorption solvent delivered from the same dispenser used to desorb samples. For example, to prepare a target level standard of desflurane, inject 20 µL of a stock solution containing 77.37 mg/mL of desflurane in toluene into 1 mL of desorption solvent.

3.3.2 Bracket sample concentrations with working standard concentrations. If samples fall outside of the concentration range of prepared standards, prepare and analyze additional standards to ascertain the linearity of response.

3.4 Sample preparation

3.4.1 Remove the plastic end caps from the sample tube and carefully transfer each section of the adsorbent to separate 2-mL vials. Discard the glass tube, urethane foam plugs and glass wool plug.

3.4.2 Add 1.0 mL of desorption solvent to each vial using the same dispenser as used for preparation of standards.

3.4.3 Immediately seal the vials with poly(tetrafluoroethylene)-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	
zone	
temperatures:	60°C (column), hold 4 min, ramp at 5°C/min to 90°C, hold 0 min, ramp at 20°C/min to 150°C, hold 5 min 250°C (injector) 300°C (detector)
run time:	18 min
column gas flow:	2.7 mL/min (hydrogen)
septum purge:	1.9 mL/min (hydrogen)
injection size:	1.0 µL (15.5 : 1 split)
column:	60-m × 0.32-mm i.d. capillary SPB-1 (4.0-µm df)
retention times:	3.05 min (desflurane) 10.7 min (benzene)
FID conditions	
hydrogen flow:	38 mL/min
air flow:	450 mL/min
makeup flow:	30 mL/min (nitrogen)

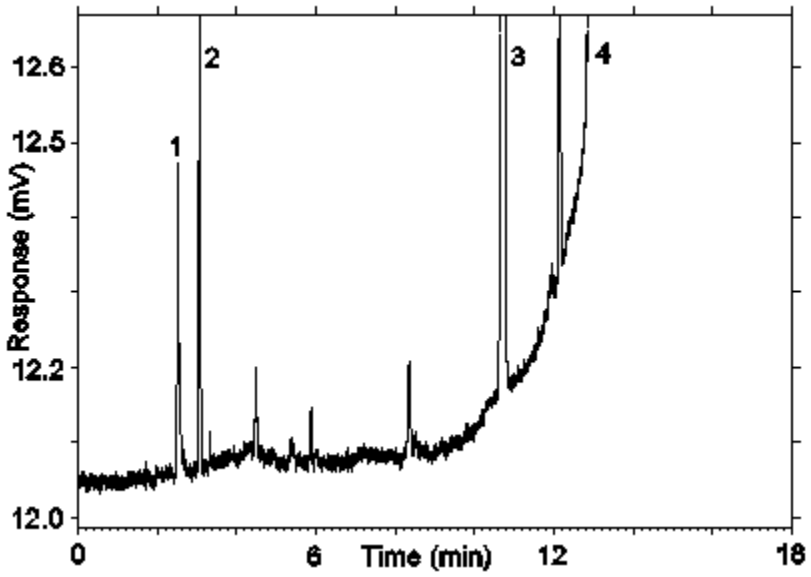


Figure 3.5.1.1. Chromatogram obtained at the low TC with the recommended conditions. Peak identification: (1) air peak, (2) desflurane, (3) benzene, (4) toluene.

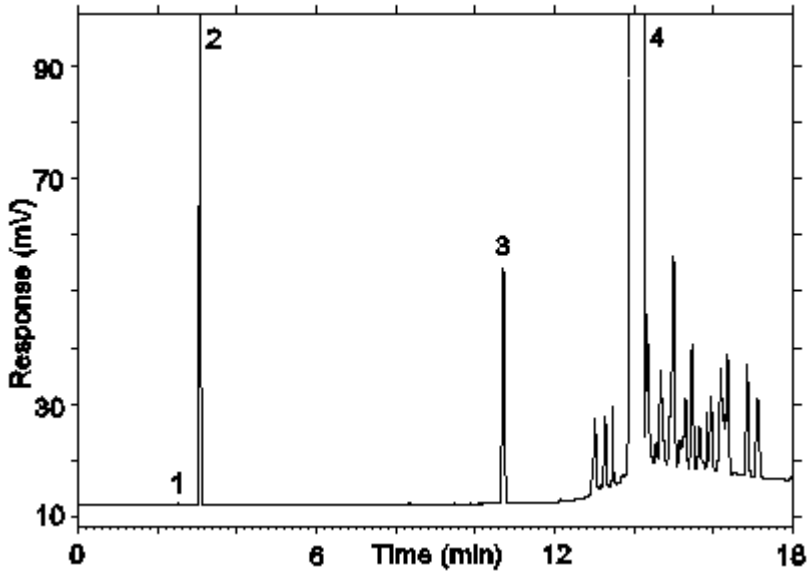


Figure 3.5.1.2. Chromatogram obtained at the height TC with the recommended conditions. Peak identification: (1) air peak, (2) desflurane, (3) benzene, (4) toluene.

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

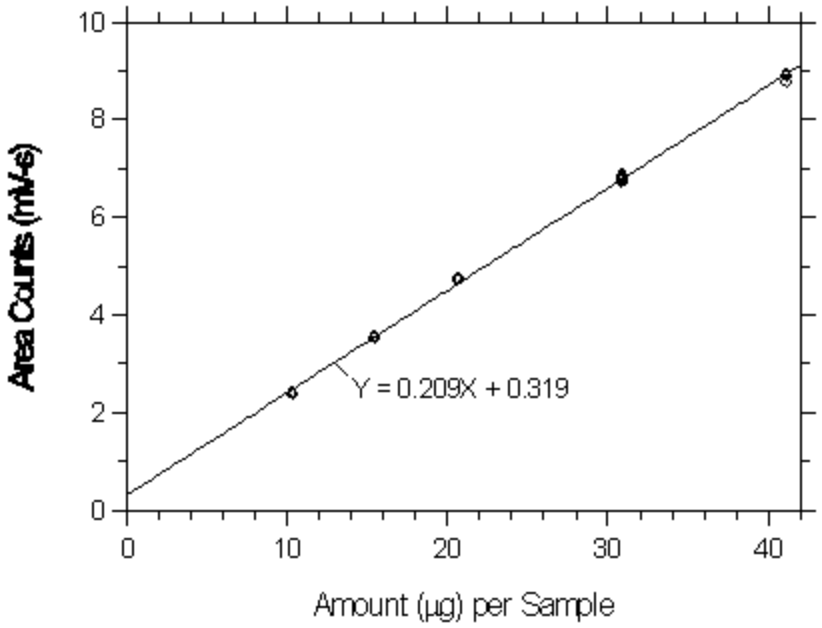


Figure 3.5.2.1. Calibration curve at the low TC made from data of Table 4.5.1.

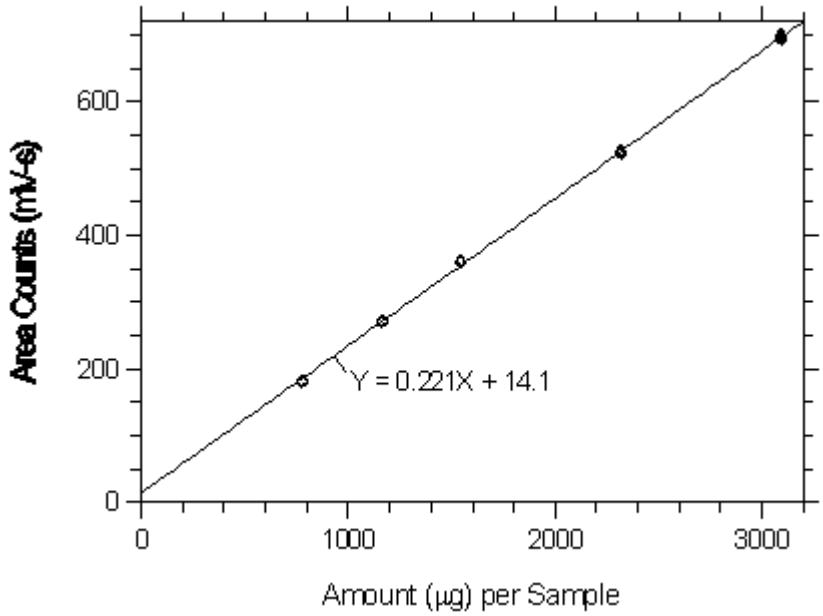


Figure 3.5.2.2. Calibration curve at the high TC made from data of Table 4.5.2.

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 the samples are desorbed. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

3.6.2 When necessary, the identity or purity of an analyte peak may be confirmed with additional analytical data (Section 4.11).

3.7 Calculations

The amount of desflurane per sample is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for desorption efficiency. The back (70-mg) section is analyzed primarily to determine if there was any breakthrough from the front (140-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 fact should be reported with the sample results. If any analyte is found on the back section, it is added to the amount on the front section. This amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulae.

$$\text{mg/m3} = \frac{\text{micrograms of analyte per sample, blank corrected}}{\text{liters of air sampled} \times \text{desorption efficiency}}$$

$$\text{ppm} = \frac{\text{mg/m3} \times 24.46}{\text{molecular weight of analyte}}$$

where 24.46 is the molar volume at 25°C and 101.3 kPa (760 mmHg)
168.04 = molecular weight of desflurane

3.8 Safety precautions (analytical)

3.8.1 Adhere to the rules set down in your Chemical Hygiene Plan.

3.8.2 Avoid skin contact and inhalation of all chemicals.

3.8.3 Wear safety glasses, gloves and a lab coat while in the laboratory areas and working with chemicals.

4. Backup Data

4.1 Determination of detection limits

Detection limits, in general, are defined as the amount (or concentration) of analyte that gives a response (YDL) that is significantly different (three standard deviations (SDBR)) from the background response (YBR).

$$YDL - YBR = 3(SDBR)$$

The measurement of YBR and SDBR in chromatographic methods is typically inconvenient and difficult because YBR is usually extremely low. Estimates of these parameters can be made with data obtained from the analysis of a series of analytical standards or samples whose responses are in the vicinity of the background response. The regression curve obtained for a plot of instrument response versus concentration of analyte will usually be linear. Assuming SDBR and the precision of the data about curve

are similar, the standard error of estimate (SEE) for the regression curve can be substituted for SDBR in the above equation. The following calculations derive a formula for DL:

$$SEE = \frac{\sqrt{\sum(Yobs - Yest)^2}}{n - k}$$

Yobs = observed response

Yest = estimated response from regression curve

n = total number of data points

k = 2 for linear regression curve

At point YDL on the regression curve

YDL = A(DL) + YBR A = analytical sensitivity (slope)

therefore

$$DL = \frac{(YDL - YBR)}{A}$$

Substituting 3(SEE) + YBR for YDL gives

$$DL = \frac{3(SEE)}{A}$$

4.2 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte actually introduced into the chromatographic column. Ten analytical standards were prepared in equal descending increments with the highest standard containing 2.573 µg/mL of desflurane. This is the concentration that would produce a peak approximately 10 times the background noise of a reagent blank near the elution time of the analyte. These standards, and the reagent blank, were analyzed with the recommended analytical parameters (1-µL injection with a 15.5 : 1 split), and the data obtained were used to determine the required parameters (A and SEE) for the calculation of the DLAP. Values of 3.071 and 13.965 were obtained for A and SEE respectively. DLAP was calculated to be 13.6 pg.

Table 4.2
Detection Limit of the Analytical Procedure

concentration (µg/mL)	mass on column (pg)	area counts (µV-s)
0	0	0
0.257	16.58	48.2
0.515	33.23	120.7
0.772	49.81	154.9
1.029	66.39	196.2
1.286	82.97	285.9
1.544	99.61	291.3
1.801	116.2	365.8
2.058	132.8	424.7
2.315	149.4	460.4
2.573	166.0	505.8

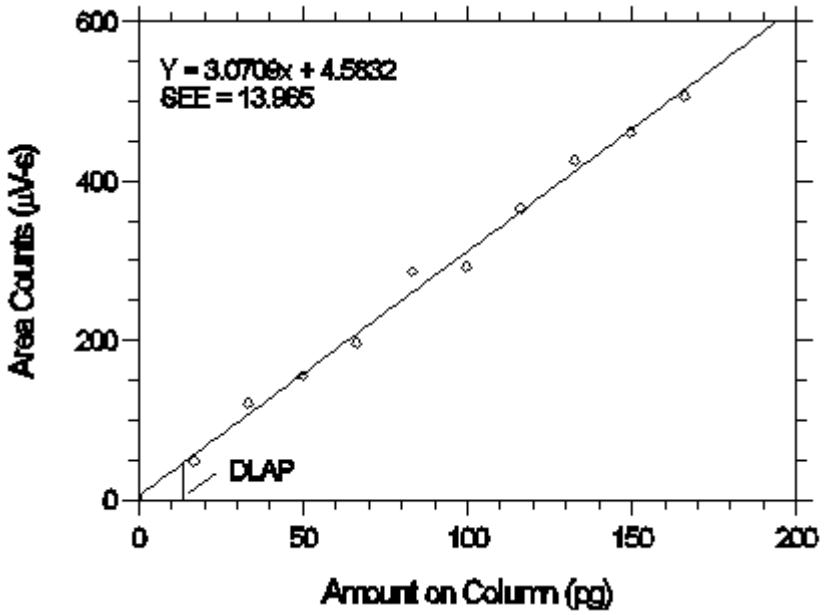


Figure 4.2. Plot of the data from Table 4.2 to determine the DLAP of desflurane.

4.3 Detection limit of the overall procedure (DLOP)

The DLOP is measured as mass per sample and expressed as equivalent air concentration, based on the recommended sampling parameters. Ten samplers were spiked with equal descending increments of analyte, such that the highest sampler loading was 2.573 µg/sample. This is the amount spiked on a sampler that would produce a peak approximately 10 times the background response for a sample blank. These spiked samplers, and a sample blank, were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (A and SEE) for the calculation of the DLOP. Values of 200.32 and 13.69 were obtained for A and SEE, respectively. The DLOP was calculated to be 0.205 µg/sample (9.93 ppb or 68.4 µg/m3).

Table 4.3
Detection Limit of the Overall procedure

mass per sample (µg)	area counts (µV-s)
0	0
0.257	61.8
0.515	113.7
0.772	144.1
1.029	192.2
1.286	265.5
1.544	305.5
1.801	342.7
2.058	396.8
2.315	485.3
2.573	523.0

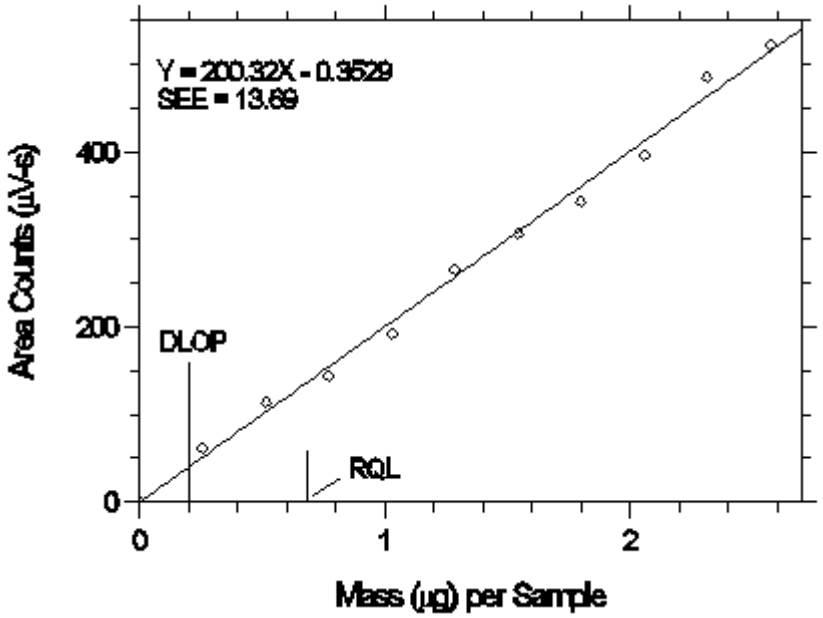


Figure 4.3. Plot of the data to determine the DLOP.

4.4 Reliable quantitation limit (RQL)

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculations of the DLOP (Section 4.3), providing at least 75% of the analyte is recovered. The RQL is defined as the amount of analyte that gives a response (YRQL) such that

$$YRQL - YBR = 10(SDBR)$$

therefore

$$RQL = \frac{10(SEE)}{A}$$

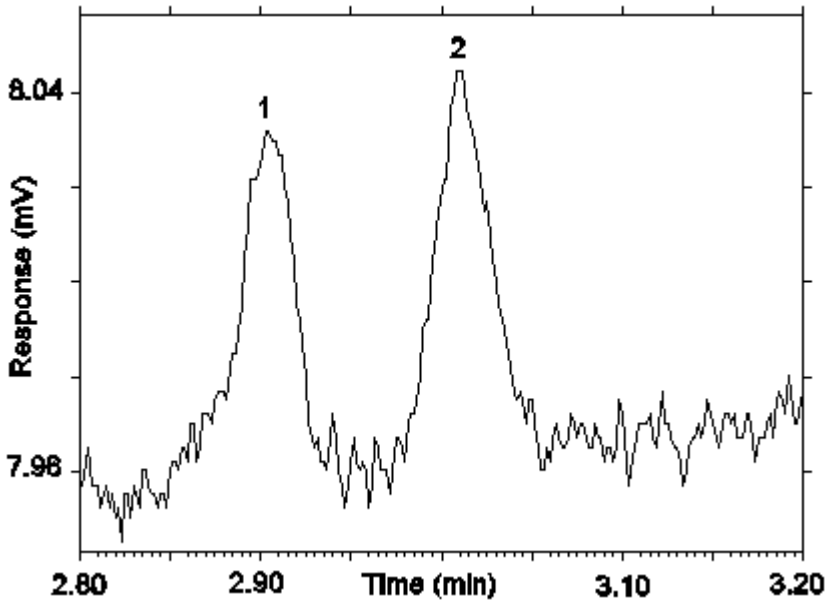


Figure 4.4. Chromatogram of the RQL for desflurane on Anasorb 747.
Peak identification: (1) contaminant form Anasob 747, (2) desflurane.

The RQL for desflurane was calculated to be 0.683 µg/sample (33.1 ppb or 228 µg/m3). The recovery at this concentration is 91.2%.

4.5 Precision (analytical method)

The precision of the analytical procedure is measured as the pooled relative standard deviation (RSDP). Relative standard deviations are determined from six replicate injections of desflurane standards at 0.5, 0.75, 1, 1.5 and 2 times the target concentrations. After assuring that the RSDs satisfy the Cochran test for homogeneity at the 95% confidence level, RSDP was calculated to be 0.58% and 0.46% for the lower and higher target concentration, respectively.

Table 4.5.1
Instrument Response to Desflurane at Low TC

× target concn (µg/mL)	0.5× 10.29	0.75× 15.44	1× 20.58	1.5× 30.87	2× 41.16
area counts (mV-s)	2.391	3.554	4.743	6.747	8.930
	2.394	3.559	4.746	6.777	8.847
	2.381	3.549	4.703	6.827	8.793
	2.416	3.548	4.729	6.763	8.917
	2.417	3.550	4.716	6.863	8.906
	2.388	3.530	4.763	6.880	8.886
$\bar{x}$	2.398	3.548	4.733	6.810	8.880
SD	0.015	0.010	0.022	0.055	0.051
RSD (%)	0.63	0.28	0.46	0.81	0.57

Table 4.5.2
Instrument Response to Desflurane at High TC

× target concn (µg/mL)	0.5× 774	0.75× 1161	1× 1547	1.5× 2321	2× 3095
area counts (mV-s)	182.784	271.647	361.916	520.504	697.847
	182.971	273.385	360.981	523.876	699.750
	181.180	271.537	360.914	527.304	693.565
	180.916	270.495	358.954	523.414	696.571
	182.850	270.950	359.275	524.745	700.723
	180.309	270.262	358.821	524.994	694.286
$\bar{x}$	181.835	271.379	360.144	524.140	697.124
SD	1.168	1.126	1.293	2.233	2.877
RSD (%)	0.64	0.41	0.36	0.43	0.41

The Cochran test for homogeneity:

$$g = \frac{\text{largest RSD2}}{\text{RSD0.5x2 + RSD0.75x2 + RSD1x2 + RSD1.5x2 + RSD2x2}}$$

The critical value of the g-statistic, at the 95% confidence level, for five variances, each associated with six observations is 0.5065. The g-statistics are 0.3915 and 0.3773 for the low and high target concentrations respectively. Because the g-statistic does not exceed this value, the RSDs can be considered equal and they can be pooled (RSDP) to give an estimated RSD for the concentration range studied.

$$RSD = \frac{\sqrt{5(RSD0.5 \times 2 + RSD\ 0.75 \times 2 + RSD\ 1.5 \times 2 + RSD\ 2 \times 2)}}{5+5+5+5+5}$$

The (RSDP)s are 0.58% and 0.46% for the low and high target concentration respectively.

4.6 Precision (overall procedure)

The precision of the overall procedure is determined from the storage data in Section 4.7. The determination of the standard error of estimate (SEER) 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 SEER is similar to the standard deviation, except it is a measure of the dispersion of data about a regression line instead of about a mean. It is determined with the following equation:

$$SEER = \frac{\sqrt{\sum(Yobs - Yest)^2}}{n-k}$$

Yobs = observed % recovery at a given time

Yest = estimated % recovery from the regression line at the same given time

n = total number of data points

k = 2 for linear regression

k = 3 for quadratic regression

An additional 5% for pump error (SP) is added to the SEER by the addition of variances to obtain the total standard error of the estimate.

$$SEE = \sqrt{(SEER)2 + (SP)2}$$

The precision at the 95% confidence level is obtained by multiplying the standard error of estimate (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 lines in the storage graphs, as shown in Figures 4.7.1.1 through 4.7.2.2. The precisions of the overall procedure are 10.4% and 11.0% for the low and high target concentration, respectively.

4.7 Storage test

4.7.1 Storage test at the low target concentration

Storage samples were generated by spiking Anasorb 747 tubes with a toluene solution containing desflurane while pulling air through the tubes at 0.05 L/min. The relative humidity was approximately 80% at 22°C. Humid air was then pulled through the tubes for 30 min. Thirty-six storage samples were prepared. Six samples were analyzed immediately after generation, fifteen tubes were stored at reduced temperature (4°C) and the other fifteen were stored in the dark at ambient temperature (about 22°C). At 2-5 day intervals, three samples were selected from each of the two sets and analyzed.

Table 4.7.1
Storage Test at the Low TC

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	90.2	102.0	101.2	90.2	102.0	101.2
	103.9	102.3	102.1	103.9	102.3	102.1
5	97.0	101.9	99.6	101.9	102.3	101.8
8	98.3	102.6	104.2	105.3	106.0	107.0
11	95.8	102.1	102.5	100.1	101.9	102.5
13	101.9	98.5	100.2	103.0	102.1	103.2
18	96.7	99.0	100.8	102.3	100.2	100.6

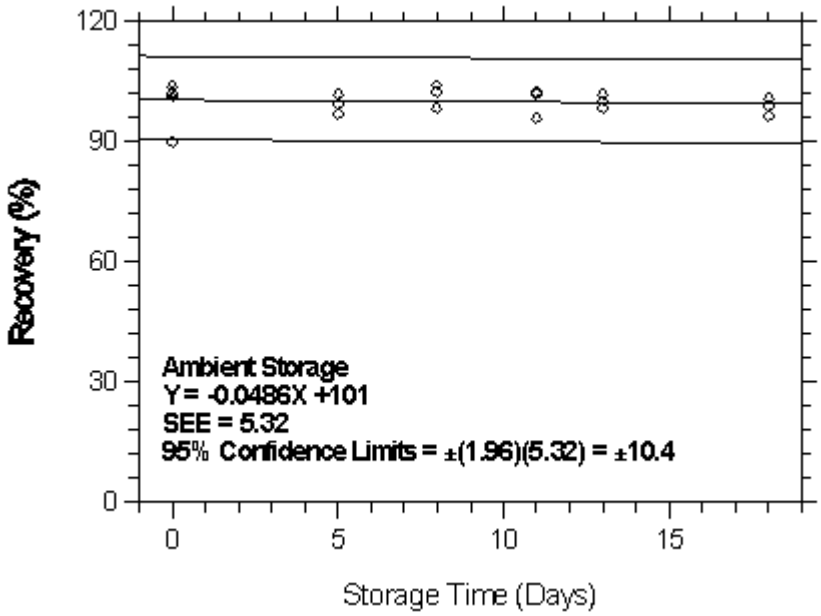


Figure 4.7.1.1. Ambient storage test at 1 ppm.

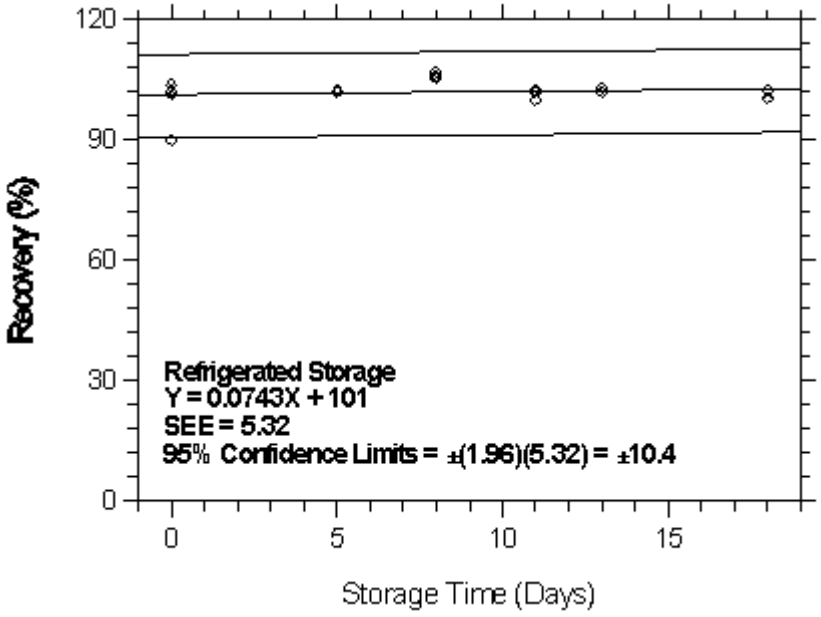


Figure 4.7.1.2. Refrigerated storage test at 1 ppm.

4.7.2 Storage test at the high target concentration

Storage samples were generated by sampling from a controlled test atmosphere containing 1151.2 mg/m3 of desflurane, about 2 times the target concentration. Anasorb 747 tubes were used to sample for 30 min at 0.05 L/min. The relative humidity was approximately 80% at 22°C. Thirty-six storage samples were prepared. Six samples were analyzed immediately after generation, fifteen tubes were

stored at reduced temperature (4°C) and the other fifteen were stored in the dark at ambient temperature (about 22°C). At 2-5 day intervals, three samples were selected from each of the two sets and analyzed.

Table 4.7.2
Storage Test at the High TC

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	87.7	102.6	96.7	87.1	102.6	96.7
	102.3	105.3	106.0	102.3	105.3	106.0
3	94.8	103.4	97.2	105.4	110.0	100.8
8	106.1	108.2	101.5	90.2	107.0	94.0
11	94.0	107.5	94.9	110.9	104.8	105.5
14	111.4	105.0	104.6	94.4	101.4	96.9
16	94.5	104.0	95.2	104.7	107.5	113.0

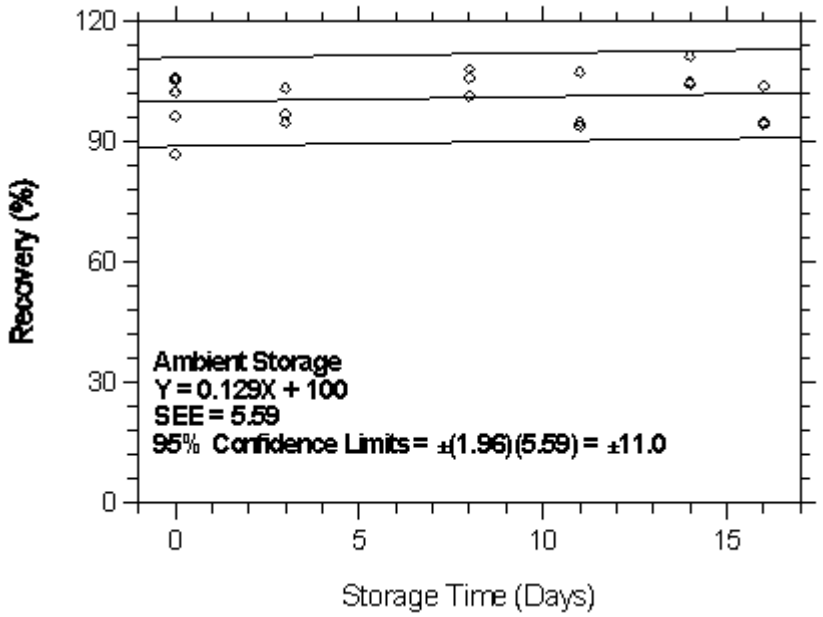


Figure 4.7.2.1. Ambient storage test at 75 ppm.

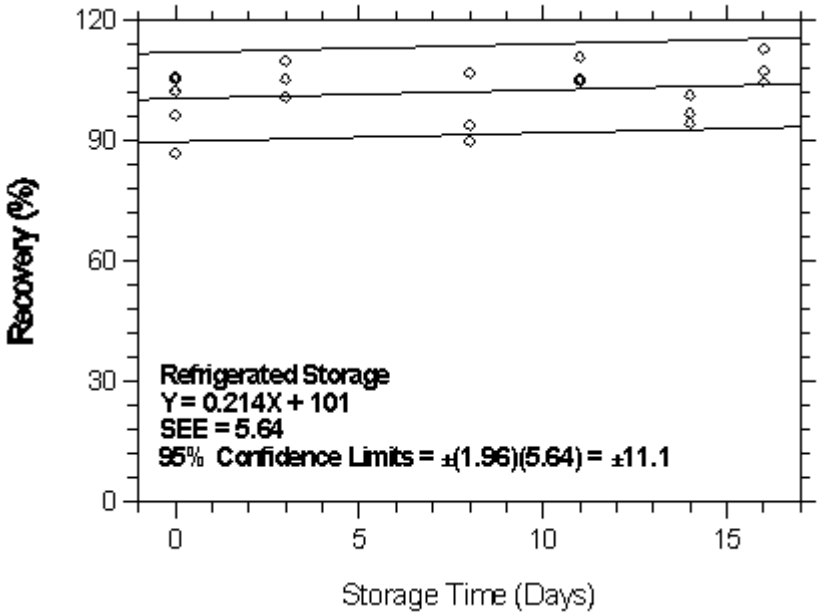


Figure 4.7.2.2. Refrigerated storage test at 75 ppm.

4.8 Reproducibility

4.8.1 Reproducibility at low target concentration

Six samples were prepared by injecting microliter quantities of a toluene solution containing desflurane into Anasorb 747 tubes while pulling air through the tubes at 0.05 L/min. The relative humidity was approximately 80% at 22°C. Humid air was then pulled through the tubes for 60 min. The samples were submitted to an OSHA Salt Lake Technical Center service branch. The samples were analyzed after being stored for 10 days at 4°C. Sample results were corrected for desorption efficiency. No sample result for desflurane had a deviation greater than the precision of the overall procedure determined in Section 4.6, which is ±10.4%.

Table 4.8.1
Reproducibility Data at Low TC

sample	expected (mg/m3)	reported (mg/m3)	recovery (%)	deviation (%)
1	6.18	5.77	93.4	-6.6
2	6.18	5.94	96.1	-3.9
3	6.18	5.87	95.0	-5.0
4	6.18	5.87	95.0	-5.0
5	6.18	6.00	97.1	-2.9
6	6.18	6.03	97.6	-2.4

4.8.2 Reproducibility at high target concentration

Six samples were prepared by injecting microliter quantities of a toluene solution containing desflurane into Anasorb 747 tubes while pulling air through the tubes at 0.05 L/min. The relative humidity was approximately 80% at 22°C. Humid air was then pulled through the tubes for 60 min. The samples were submitted to an OSHA Salt Lake Technical Center service branch. The samples were analyzed after being stored for 10 days at 4°C. Sample results were corrected for desorption efficiency. No sample result for desflurane had a deviation greater than the precision of the overall procedure determined in Section 4.6, which is ±11.0%.

Table 4.8.2
Reproducibility Data at High TC

sample	expected (mg/m3)	reported (mg/m3)	recovery (%)	deviation (%)
1	489.4	451.0	92.1	-7.9
2	489.4	457.6	93.5	-6.5
3	489.4	453.4	92.6	-7.4
4	489.4	448.4	91.6	-8.4
5	489.4	451.0	92.1	-7.9
6	489.4	453.3	92.6	-7.4

4.9 Sampler capacity

The sampling capacity of the front section of an Anasorb 747 sampling tube was tested by sampling from a dynamically generated test atmosphere of desflurane (1030 mg/m3 or 150 ppm). The samples were

collected at 0.05 L/min and the relative humidity was approximately 80% at 22°C. A GC with a gas sampling valve was placed in-line behind the 140-mg front test section. The valve was rotated to measure the amount of desflurane passing through the sampler at the time of rotation. The 5% breakthrough air volume was determined to be 3.83 L.

Table 4.9
Capacity of Desflurane on Anasorb 747

first test		second test	
air volume (L)	breakthrough (%)	air volume (L)	breakthrough (%)
1.60	0	1.78	0
2.58	0	2.72	0
3.35	0	3.46	0
3.87	1.26	3.95	1.56
4.39	37.3	4.56	34.2

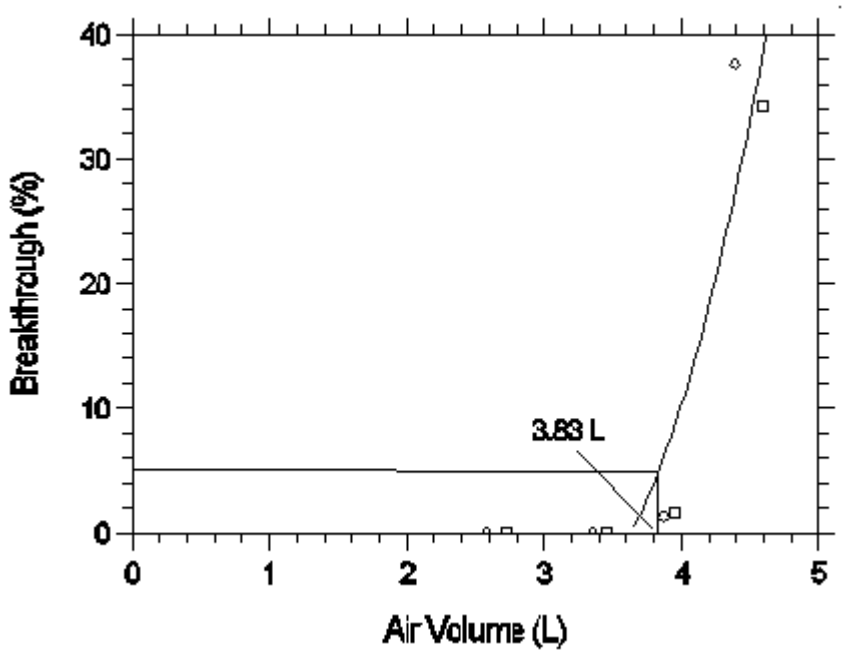


Figure 4.9. Five percent breakthrough air volume for desflurane on Anasorb 747.

4.10 Desorption efficiency and stability of desorbed samples

4.10.1 Anasorb 747 at low target concentration (TC)

The desorption efficiencies (DE) of desflurane were determined by liquid-spiking 140-mg portions of Anasorb 747 with amounts equivalent to 0.05 to 2 times the target concentration. These samples were stored overnight at ambient temperature and then desorbed and analyzed. The average desorption efficiency over the working range of 0.5 to 2 times the target concentration is 101.1%.

Table 4.10.1.1
Desorption Efficiency of desflurane from Anasorb 747 at Low TC

× target concn (µg/sample)	0.05× 1.029	0.1× 2.058	0.2× 4.116	0.5× 10.29	1.0× 20.58	2.0× 41.16
DE (%)	96.6	100.7	100.9	101.4	102.3	99.8
	96.2	101.9	100.0	102.0	103.2	100.8
	98.2	104.3	99.1	101.4	102.3	99.0
	100.1	103.3	100.4	100.7	101.1	99.5
	95.9	104.8	100.7	99.8	102.8	99.2
	101.0	104.0	99.9	100.7	103.4	99.8
$\bar{x}$	98.0	103.2	100.2	101.0	102.5	99.7

The stability of desorbed samples was investigated by reanalyzing the target concentration samples 20 h after initial analysis. After the original analysis was performed, three vials were recapped with new septa while the remaining three retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was 2.4% for samples that were resealed with new septa and 0.9% for those that retained their punctured septa.

Table 4.10.1.2
Stability of Desorbed Samples from Anasorb 747

punctured septa replaced			punctured septa retained		
initial DE (%)	DE after one day (%)	difference	initial DE (%)	DE after one day (%)	difference
102.3	100.5	-1.8	101.1	101.7	+0.6
103.2	99.4	-3.8	102.8	101.3	-1.5
102.3	100.6	-1.7	103.4	101.6	-1.8
	averages			averages	
102.6	100.2	-2.4	102.4	101.5	-0.9

4.10.2 Anasorb 747 at high target concentration (TC)

The desorption efficiencies (DE) of desflurane were determined by liquid-spiking 140-mg portions of Anasorb 747 with amounts equivalent to 0.05 to 2 times the target concentration. These samples were stored overnight at ambient temperature and then desorbed and analyzed. The average desorption efficiency over the working range of 0.5 to 2 times the target concentration is 102.9%.

Table 4.10.2.1
Desorption Efficiency of Desflurane from Anasorb 747 at High TC

× target concn (µg/sample)	0.05× 77.37	0.1× 154.7	0.2× 309.5	0.5× 773.7	1.0× 1547	2.0× 3095
DE (%)	99.3	99.4	101.6	105.4	104.7	102.0
	100.4	100.0	101.2	102.9	102.6	101.5
	100.2	100.0	100.4	104.5	103.0	101.2
	99.7	100.6	97.4	103.8	102.6	101.4
	100.5	101.0	103.1	105.2	103.6	100.4
	100.1	99.9	102.0	103.7	103.4	100.3
$\bar{x}$	100.0	100.2	101.0	104.3	103.3	101.1

The stability of desorbed samples was investigated by reanalyzing the target concentration samples 33 h after initial analysis. After the original analysis was performed, three vials were recapped with new septa while the remaining three retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was 0% for samples that were resealed with new septa and +0.6% for those that retained their punctured septa.

Table 4.10.2.2
Stability of Desorbed Samples for Desflurane from Anasorb 747

punctured septa replaced			punctured septa retained		
initial DE (%)	DE after one day (%)	difference	initial DE (%)	DE after one day (%)	difference
104.7	103.5	-1.2	102.6	104.3	+1.7
102.6	103.4	+0.8	103.6	104.4	+0.8
103.0	103.2	+0.2	103.4	102.6	-0.8
	(averages)			(averages)	
103.4	103.4	0	103.2	103.8	+0.6

4.11 Qualitative analysis

The mass spectrum for desflurane was obtained from an HP5988A Mass Spec interfaced to a Hewlett-Packard 5890 Series II GC.

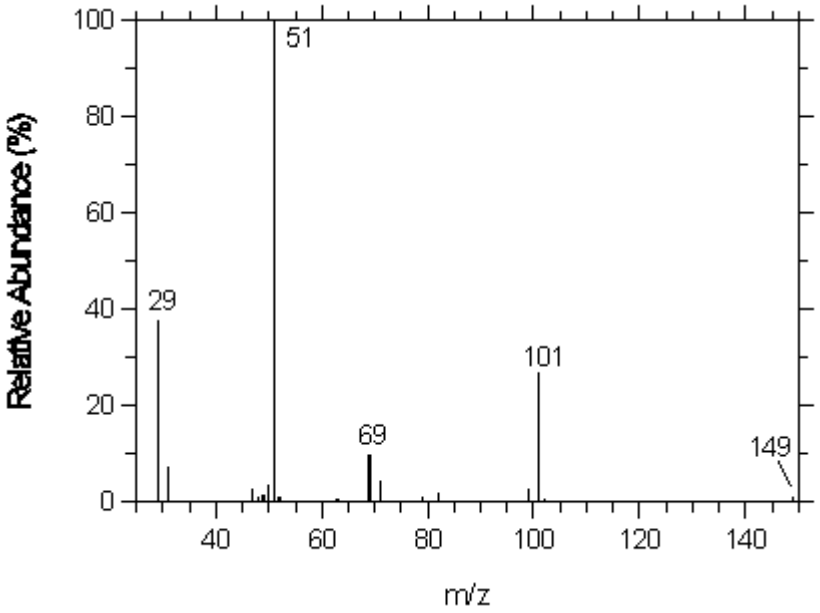


Figure 4.11. Mass spectrum of desflurane.

5. References

5.1 Burright, D.D. OSHA Method No. 103; Enflurane, Halothane and Isoflurane, OSHA Salt Lake Technical Center, unpublished, Salt Lake City, UT 84165, May 1994.

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5.4 Material Safety Data Sheet: Suprane™, Anaquest, Liberty Corner, NJ, March 1992.

5.5 OSHA Instruction CPL 2-2.60, Exposure Control Plan for Federal OSHA Personnel with Occupational Exposure to Blood born Pathogens, March 7, 1994; Occupational Safety and Health Administration, U.S. Department of Labor, Washington, D.C.