PROPYLENE OXIDE

Method no.:	88
Matrix:	Air
Target concentration:	1 ppm (2.4 mg/m^3) and 20 ppm (47 mg/m^3)
Procedure:	Samples are collected by drawing air through standard size Anasorb 747 adsorbent tubes. The Anasorb 747 is desorbed with carbon disulfide and the desorbate is analyzed by gas chromatography using a flame ionization detector.
Recommended air volume and sampling rate:	5 L at 0.1 L/min
Reliable quantitation limit:	35 ppb (83 µg/m^3)
Standard error of estimate at target concentration: (Section 4.7)	5.7% (1 ppm) 6.5% (20 ppm)
Special requirement:	Stored samples should be kept at 0°C or colder to reduce migration. Reduced temperature shipment of samples to the laboratory is not necessary.
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

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1. General Discussion

1.1 Background

1.1.1 History

OSHA has set a time weighted average final rule limit of 20 ppm for workplace exposure to propylene oxide. (Ref. 5.1) Based on evidence that propylene oxide is an animal carcinogen, NIOSH recommends that occupational exposure to propylene oxide be reduced to the lowest feasible level. (Ref. 5.2) Because the PEL for ethylene oxide, which is chemically similar to propylene oxide and is also an animal carcinogen, has recently been reduced to 1 ppm (Ref. 5.3), evaluation data was collected at 1 ppm (TC-1) as well as 20 ppm (TC-20) to assure that an adequate monitoring procedure would be available if the PEL for propylene oxide were significantly lowered in the future.

The current methodology used by OSHA to determine propylene oxide in air is based on the coconut shell charcoal tube procedure evaluated by NIOSH. (Ref. 5.4) The concentration range studied was 50 to 200 ppm (5-L air samples) because the OSHA PEL was 100 ppm at that time. The method specifies that samples be shipped at reduced temperatures, although no sample stability tests were reported. Typically, air samples collected on adsorbent tubes are shipped refrigerated to minimize migration of analyte from the front section to the back section of adsorbent.

Preliminary tests at the 20-ppm level (5-L air samples) confirmed that migration was indeed a problem. Test samples were prepared by spiking the front sections of SKC, Inc. Lot 120 coconut shell charcoal tubes and storing them at room temperature for six days. Approximately 5 L of air at 80% relative humidity had been drawn through the charcoal tubes before they were spiked. Upon analysis, about 20% of the original amount of propylene oxide was found on the back sections. When added to the amount found on the front sections, the total amount found was only about 80% of the original amount spiked. This not only indicated that there is a migration problem but there may also be a stability problem for propylene oxide collected on charcoal.

Commercially available glass sampling tubes containing a new carbon-based adsorbent called Anasorb 747 were evaluated and found to be a superior alternative to charcoal sampling tubes. Storage samples generated from humid atmospheres exhibited excellent storage stability over the 15-day perious studied. Some minor migration of propylene oxide to the back section of the sampling tubes for the TC-20 anbient storage samples was observed, but was not considered significant enough to require shipment of samples to the laboratory at reduced temperatures.

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

Propylene oxide is an irritant and a mild depressant of the central nervous system. Excessive exposure may cause irritation of the eyes, nose, throat, and lungs. Contact with the liquid may cause skin or eye irritation or burns. (Ref. 5.5)

NIOSH has recently issued a Current Intelligence Bulletin on the carcinogenic effects of exposure to propylene oxide. Numerous studies are cited that show propylene oxide exposure produces cancer and benign tumors in both rats and mice. They conclude that propylene oxide should be considered a potential occupational carcinogen and worker exposure should be reduced to the lowest feasible levels. (Ref. 5.2)

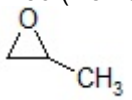
Currently OSHA has a transitional limit of 100 ppm and a final rule limit of 20 ppm for 8-hour time weighted average exposures to airborne propylene oxide. (Ref. 5.1)

1.1.3 Workplace exposure

Propylene oxide is produced by the chlorohydrin process, where propylene is reacted with chlorine, or by the hydroperoxide process, where an organic hydroperoxide is used to epoxidize propylene. The estimated U.S. production of propylene oxide in 1980 was 1,767 million pounds. It is used primarily as an intermediate for the manufacture of polyether polyols in the production of polyurethane foams, and for the manufacture of propylene glycol in the production of unsaturated polyester resins. Small quantities are also used for

sterilizing medical equipment and for fumigating foodstuffs. In 1983, NIOSH estimated that 209,000 U.S. workers were potentially exposed to propylene oxide. (Ref. 5.2)

1.1.4 Physical properties (Ref. 5.5 unless otherwise noted)

CAS number:	75-56-9
molecular weight:	58.08
specific gravity (water=1):	0.830
boiling point at 101.3 kPa (760 mmHg)	34°C
melting point:	-112 °C
vapor density (air = 1 at bp of propylene oxide):	2
vapor pressure at 20°C:	58.9 kPa (442 mmHg)
appearance:	colorless liquid
odor:	etherlike
evaporation rate (butyl acetate = 1):	33.7
flash point (closed cup):	-37 °C
autoignition temperature:	748°C
flammable limits in air, % by volume:	2.1 to 37.0
odor threshold:	200 ppm
solubility:	59% by weight in water at 25°C; miscible with acetone, benzene, carbon tetrachloride, ether, and methanol (Ref. 5.2)
synonyms:	1,2-propylene oxide; epoxypropane; 1,2-epoxy-propane; 2,3-epoxypropane; ethylene oxide, methyl-; methyl ethylene oxide; methyl oxirane; oxirane, methyl-; NCI-C50099; oxyde de propylene (French); propene oxide; propylene epoxide; propyleneoxide; propane, 1,2-epoxy-; propane, epoxy-; UN 1280 (Ref. 5.6)
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 24 pg per injection. This is the amount of propylene oxide that will produce a peak with a height approximately 5 times the height of baseline noise. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 0.415 µg per sample (35 ppb or 83 µg/m^3). This is the amount of propylene oxide spiked on an Anasorb 747 adsorbent tube that, upon analysis, produces a peak similar in size to that of the detection limit of the analytical procedure. (Section 4.2)

1.2.3. Reliable quantitation limit

The reliable quantitation limit is 0.415 µg per sample (35 This is the smallest amount of propylene oxide that can be quantitated within the requirements of a recovery of at least 75% and 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 GC for the smallest possible amount of analyte. When the target concentration of an 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 ranges of 0,5 to 2 times the TC-1 and TC-20 target concentrations is linear. (Section 4.4)

1.2.5 Recovery

The recovery of propylene oxide from samples used in 15-day storage tests remained above 86% and 91% at the TC-1 and TC-20 levels respectively when the samples were stored at ambient temperatures. (Section 4.5, from regression lines shown in Figures 4.5.1.2 and 4.5.2.2)

1.2.6 Precision (analytical procedure)

The pooled coefficients of variation obtained from replicate injections of analytical standards at 0.5, 1 and 2 times the target concentrations are 0.013 and 0.012 at the TC-1 and TC-20 levels respectively. (Section 4.6)

1.2.7 Precision (overall procedure)

The precisions at the 95% confidence level for the refrigerated 15-day storage tests are +11.2% and +12.7% at the TC-1 and TC-20 levels respectively. These include 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. (Section 4.7)

1.2.8 Reproducibility

Six samples for each target concentration collected from controlled test atmospheres and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The TC-1 and TC-20 samples were analyzed after 37 and 44 days of refrigerated storage respectively. One of the TC-1 results was rejected as being an outlier by the Q test. (Ref. 5.7) None of the other sample results deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.8)

1.3 Advantage

Reduced temperature shipment of samples to the laboratory is not necessary.

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 a sampling tube in line.

2.1.2 Samples are collected with solid sorbent sampling tubes containing Anasorb 747. Each tube consists of two sections of Anasorb 747 separated by a urethane foam plug. The front section contains 140 mg and the back section, 70 mg. The sections are held in place with glass wool plugs in a glass tube 4-mm i.d. × 70-mm length. For this evaluation, SKC Inc. Lot 645 tubes (Catalog Number 226-81) were used.

2.2 Reagents

None required

2.3 Technique

2.3.1 Immediately before sampling, break off the ends of the Anasorb 747 tube. All tubes should be from the same lot.

2.3.2 Connect the sampling tube to the sampling pump with flexible tubing. It is desirable to utilize sampling tube holders that have protective covers to shield the employee from the sharp, jagged end of the sampling tube. position the tube so that sampled air passes through the 140-mg section first.

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

2.3.4 To avoid channeling, place the sampling tube vertically in the employee's breathing zone.

2.3.5 After sampling, seal the tubes immediately with plastic caps and wrap lengthwise with OSHA Form 21.

2.3.6 Submit at least one blank sampling tube with each sample set. Blanks should be handled in the same manner as samples, except no air is drawn through them.

2.3.7. Record sample volumes (in liters of air) for each sample.

2.3.8. List any compounds that could be considered potential interferences, especially solvents, that are being used in the sampling area.

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

2.4 Sampler capacity

Sampler capacity is determined by measuring how much air can be sampled before breakthrough of analyte occurs, i.e., the sampler capacity is exceeded. Breakthrough studies were performed by monitoring the effluent from sampling tubes containing only the 140-mg section of Anasorb 747 while sampling at 0.1 L/min from an atmosphere containing 40 ppm of propylene oxide. Breakthrough was considered to occur when the propylene oxide concentration in the effluent was 5% of the upstream concentration. The atmosphere was at approximately 80% relative humidity and 20-25 °C. The average 5% breakthrough volume from three determinations was 11.1 L.

2.5 Desorption efficiency

2.5.1 The average desorption efficiency is 98.5% and 98.8% over the range of 0.5 to 2 times the TC-1 and TC-20 levels respectively. (Section 4.9)

2.5.2 Desorbed samples remain stable for at least 24 h. (Section 4.10)

2.5.3 Desorption efficiencies should be periodically confirmed because differences may occur due to variations in Anasorb 747, desorption solvent, and operator technique.

2.6 Recommended air volume and sampling rate

2.6.1 For TWA samples, the recommended air volume is 5 L collected at 0.1 L/min (50-min samples). Although the break through volume was experimentally determined to exceed 11 L (Section 2.4), a recommended air volume of 5 L was chosen to assure that the method will be valid over a wide range of sampling conditions.

2.6.2 For short-term samples, the recommended air volume is 5 L collected at 1.0 L/min (5-min samples).

2.7 Interferences (sampling)

2.7.1 It is not known if any compound(s) will severely interfere with the collection of propylene oxide on Anasorb 747. In general, the presence of other solvent vapors in the sampled air wall reduce the capacity of Anasorb 747 to collect propylene oxide.

2.7.2 Potential interferences used in the sampling area should be reported to the laboratory with each sample set.

2.8 Safety precautions (sampling)

2.8.1 Attach the sampling equipment to the employee so that it will not interfere with work performance or safety. Use sample tube holders with protective covers whenever possible.

2.8.2 Wear eye protection when breaking the ends of the Anasorb 747 tubes.

2.8.3 Follow all safety procedures 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. For this evaluation, a Hewlett-Packard 5890 Series II Gas Chromatograph equipped with a 7673A Automatic Sampler was used.

3.1.2 A GC column capable of separating propylene oxide from the desorption solvent, internal standard and any interferences. A thick film, 60-m × 0.32-mm i.d., fused silica RTx-Volatiles column (Cat. no. 10904, Restek Corp., Bellefonte, PA) was used in this evaluation.

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

3.1.4 Two-milliliter vials with Teflon-lined caps.

3.1.5 A dispenser capable of delivering 1.0 mL of desorption 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 Propylene oxide, reagent grade. Fisher scientific Lot 713390 propylene oxide was used in this evaluation.

3.2.2 Carbon disulfide, chromatographic grade. Omnisolv, glass distilled carbon disulfide from EM Science was used in this evaluation.

3.2.3 A suitable internal standard, reagent grade. Benzene was used in this evaluation.

3.2.4 The desorption solvent consists of carbon disulfide containing an internal standard at a concentration of 25 µL/L.

3.2.5 GC grade nitrogen, air, and hydrogen.

3.3 Standard preparation

3.3.1 Prepare concentrated stock standards by diluting the reagent grade propylene oxide with carbon disulfide. Prepare working standards by injecting microliter amounts of concentrated stock standards into vials containing 1.0 mL of desorption solvent delivered from the same dispenser used to desorb samples. For example, prepare a stock standard by diluting 3.00 mL of propylene oxide (sp gr = 0.830) to 50.0 mL with carbon disulfide. This stock solution concentration would equal 49.8 g/µL. A working standard of 239.0 µg/sample is prepared by injecting 4.8 µL of this stock into a vial containing 1.0 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 Transfer each Anasorb 747 section of the samples to separate vials. Discard the glass tubes and plugs.

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 cap the vials and shake them periodically for about 10 min before analysis.

3.5 Analysis

3.5.1 GC conditions

column: 60-m × O.32-mm i.d. fused silica, RTx-Volatiles, thick film
injection volume: 1.0 µL (with a 17:1 split) zone temperatures:

column:`	70 °C
injector:	200 °C
detector	250 °C
gas flows:	
hydrogen (carrier):	3.7 mL/min (110 kPa head pressure)
nitrogen (makeup):	22 mL/min
hydrogen (flame);	43 mL/min
air	400 mL/min
retention times:	
propylene oxide:	3.0 min
benzene:	6.0 min
chromatograms:	Section 4.11.

3.5.2 Peak areas (or heights) are measured by an integrator or other suitable means.

3.5.3 An internal standard (ISTD) calibration method is used. Calibration curves are prepared by plotting micrograms of propylene oxide per sample versus ISTD-corrected response of standard injections. Sample concentrations must be bracketed by standards.

3.6 Interferences (analytical)

3.6.1 Any compound that produces a flame ionization detector response and has a similar retention time as propylene oxide or the internal standard is a potential interference. Any potential interferences reported to the laboratory by the industrial hygienist should be considered before samples are desorbed.

3.6.2 GC parameters (i.e., column and column temperature) may be changed to possibly circumvent interferences.

3.6.3 Retention time on a single column is not considered proof of chemical identity. Confirmation should be performed by GC/mass spectrometry or another suitable technique.

3.7 Calculations

The propylene oxide concentration for samples is obtained from the calibration curve in terms of micrograms per sample, uncorrected for desorption efficiency. The air concentration is calculated using the following formulae. 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 sample results. If any analyte is found on the back section, it is added to the amount found on the front section. This total amount is then corrected by subtracting the total amount (if any) found in the corresponding blank sampling tube.

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

$$ppm = \frac{(mg/m^3)(24.46)}{58.08} = (mg/m^3)(0.421)$$

where 24.46 = molar volume (L) at 25 °C and 101.3 kPa (760 mmHg)
58.08 = molecular weight of propylene oxide

3.8 Safety precautions (analytical)

3.8.1 Avoid skin contact and inhalation of all chemicals.

3.8.2 Restrict the use of all chemicals to a fume hood then possible.

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

4. Backup Data

4.1 Detection limit of the analytical procedure

The injection size listed in the analytical procedure (1.0 µL with a 17:1 split) was used in the determination of the detection limit of the analytical procedure. The detection limit of 24 pg was determined by making injections of a 415 pg/L standard. This amount was judged to produce a peak with a height approximately 5 times the baseline noise. A chromatogram of such an injection is shown in Figure 4.1.

4.2 Detection limit of the overall procedure

Six samples were prepared by injecting 0.415 µg (5.0 µL of a 0.083 µg/µL standard) of propylene oxide into the 140-mg section of Anasorb 747 tubes. The detection limit of the overall procedure corresponds to an air concentration of 35 ppb (83 µg/m^3).

Table 4.2
Detection Limit of the Overall Procedure

sample no.	µg spiked	µg recovered
1	0.415	0.379
2	0.415	0.386
3	0.415	0.401
4	0.415	0.388
5	0.415	0.395
6	0.415	0.392

4.3 Reliable quantitation limit

The reliable quantitation limit was determined by analyzing Anasorb 747 tubes that had been spiked with a loading equivalent to the detection limit of the overall procedure. Samples were prepared by injecting 0.415 µg (5.0 L of a 0.083 µg/µL standard) of propylene oxide into the 140-mg section of Anasorb 747 tubes. This amount corresponds to an air concentration of 35 ppb (83 µg/m^3).

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

percent recovered	statistics
91.3	
93.0	mean = 94.0
96.6	SD = 1.84
93.5	Precision = (1.96)(±1.84)
95.2	= ±3.61
94.5	

4.4 Instrument response to the analyte

The instrument response to the analyte over the °range of 0.5 to 2 times each target concentration was determined from multiple injections of analytical standards. These data are given in Tables 4.4.1 and 4.4.2 and Figures 4.4.1 and 4.4.2. The response is linear with slopes (in ISTD-corrected area counts per microgram of analyte per sample) of 278.9 and 280.2 for the TC-l and TC-20 levels respectively.

Table 4.4.1
Instrument Response

× TC-1	0.5×	1.0×	2.0×
µg/sample	5.976	11.95	23.90
ppm	0.503	1.01	2.01
area counts	1716	3426	6742
	1703	3383	6717
	1714	3467	6813
	1651	3368	6684
	1729	3376	6656
	1692	3389	6633
mean	1701	3402	6708

Table 4.4.2
Instrument Response

× TC-20	0.5×	1.0×	2.0×
µg/sample	119.5	239.0	478.1
ppm	10.1	20.1	40.3
area counts	34508	67259	132472
	34113	66119	133972
	34482	67717	132993
	34083	67721	136494
	34174	68350	136590
	33508	66971	134849
mean	34145	67356	134562

4.5 Storage test

Thirty-six samples were generated at each target concentration by sampling from atmospheres that were at ambient temperature and approximately 80% relative humidity. Samples were collected for 50 min at 0.1 L/min (5-L samples). For each set of 36 samples for each target concentration, six samples were analyzed immediately after generation, fifteen were stored in a refrigerator at 0 °C and fifteen were stored in a closed drawer at ambient temperatures of 21-25 °C. Six samples, three from refrigerated and three from ambient storage, were analyzed at intervals over a period of fifteen days. The results are given in Tables 4.5.1 and 4.5.2 and shown graphically in Figures 4.5.1.1,

4.5.1.2, 4.5.2.1 and 4.5.2.2. After nine days of ambient temperature storage of the TC-20 samples, approximately 1% of the collected propylene oxide was found on the back sections. Similarly, approximately 2% and 4% was found on the back sections of the twelve- and fifteen-day ambient storage samples respectively There was no observed migration of propylene oxide for the refrigerated TC-20 samples or any of the TC-1 samples.

Table 4.5.1
Storage Test at TC-1

time (days)	percent recovery (ambient)			percent recovery (refrigerated)		
0	89.2	87.6	91.2	89.2	87.6	91.2
	90.5	87.4	90.4	90.5	87.4	90.4
3	92.6	92.6	93.5	92.8	90.8	92.7
6	87.9	86.1	86.2	85.8	84.7	85.0
9	89.2	87.1	86.7	86.8	88.2	90.0
12	85.6	84.2	82.6	87.4	85.8	87.4
15	87.7	87.3	88.2	94.2	92.0	89.5

Table 4.5.2
Storage Test at TC-20

time (days)	percent recovery (ambient)			percent recovery (refrigerated)		
0	100.1	99.8	103.6	100.1	99.8	103.6
	98.8	98.6	99.7	98.8	98.6	99.7
3	91.1	92.1	91.6	93.6	94.7	91.5
6	98.2	95.7	96.5	102.0	96.7	99.8
9	91.3	90.1	87.4	90.9	87.5	87.8
12	100.6	96.8	94.8	95.8	96.2	100.8
15	93.4	88.8	90.2	91.5	90.9	92.8

4.6 Precision (analytical Procedure)

The precision of tide analytical procedure is the pooled coefficient of variation determined from replicate injections of standards. The precision of the analytical Procedure for each target concentration is given in Tables 4.6.1 and 4.6.2. These tables are based on the data Presented in Section 4.4.

Table 4.6.1
Precision of the Analytical Procedure
at 0.5 to times TC-1
(Based on Table 4.4.1)

× TC-1	0.5×	1.0×	2.0×
µg/sample	5.976	11.95	23.90
ppm	0.503	1.01	2.01
SD[1]	27.4	37.8	65.1
CV	0.0161	0.0111	0.0097
$\overline{CV}$	0.013		

1 - in area counts

Table 4.6.2
Precision of the Analytical Procedure
at 0.5 to 2 times TC-20
(Based on Table 4.4.2)

× TC-20	0.5×	1.0×	2.0×
µg/sample	119.5	239.0	478.1
ppm	10.1	20.1	40.3
SD[1]	362.4	766.7	1738.1
CV	0.0106	0.0114	0.0129
$\overline{CV}$	0.012		

1 - 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 percent recovery at a given time
Y_{est} = estimated percent 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 SEEs are 5.7% and 6.5% at the TC-1 and TC-20 levels respectively. The precision of the overall procedure is the precision at the 95% confidence level, which 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 lines in the storage graphs The precisions of the overall procedure are +11.2% and +12.7% at the TC-1 and TC-20 levels respectively. The SEE and precision of the overall procedure for each level were obtained from Figures 4.5.1.1 and 4.5.2.1.

4.8 Reproducibility

Six samples collected for each target concentration from controlled test atmospheres (at about 80% R.H., 23-24C, 86-88 kPa) were analyzed by a chemist unassociated with this evaluation. The samples were generated by drawing the test atmospheres through sampling tubes for 50 min at approximately 0.1 L/min. The TC-1 and TC-20 samples were stored in a refrigerator for 37 and 44 days respectively before being analyzed. About 2-3% of the total amount of anlyte found was on the back sections of the TC-20 samples. Sample number 4 at the TC-1 level was rejected as being an outlier by the Q test. (Ref. 5.7)

Table 4.8.1
Reproducibility Data at the TC-1 Level

µg spiked	µg recovered	percent recovered	percent deviation
11.18	10.87	97.2	-2.8
11.06	11.02	99.6	-0.4
11.27	11.50	102.0	+2.0
10.88	9.47	87.0	-13.0
10.73	10.79	100.6	+0.6
10.95	11.00	100.5	+0.5

Table 4.8.2
Reproducibility Data at the TC-20 Level

µg spiked	µg recovered	percent recovered	percent deviation
227.1	216.2	95.2	-4.8
224.4	215.0	95.8	-4.2
227.1	218.4	96.2	-3.8
224.1	208.6	93.1	-6.9
224.3	212.0	94.5	-5.5
228.7	218.8	95.7	-4.3

4.9 Desorption efficiency

The desorption efficiency over the range of 0.5 to 2 times each target concentration was determined by injecting microliter amounts of stock standards into the front section of Anasorb 747 tubes.

Table 4.9
Desorption Efficiency Data

level	TC-1			TC-20		
× target concn	0.5×	1×	2×	0.5×	1×	2×
µg/sample	5.976	11.95	23.90	119.5	239.0	478.1
ppm	0.503	1.01	2.01	10.1	20.1	40.3
desorption	97.7	97.8	98.5	98.7	97.1	100.0
efficiency, %	98.5	98.6	97.8	97.4	99.0	101.8
	102.3	98.6	97.5	97.0	98.7	102.5
	101.1	97.8	97.9	96.1	96.7	102.3
	99.8	98.0	97.7	95.3	99.0	103.3
	97.0	97.7	98.4	98.2	99.4	95.1
$\bar{x}$	99.4	98.1	98.0	97.1	98.3	100.8
average $\bar{x}$		98.5			98.8	

4.10 Stability of desorbed samples

The stability of desorbed samples was checked by reanalyzing the target concentration samples from Section 4.9 one day later using fresh standards. The sample vials were resealed with new septa after the original analyses and were allowed to stand at room temperature until reanalyzed.

Table 4.10
Stability of Desorbed Samples at the Target Concentration

	% desorption after 24 h	
sample #	TC-1	TC-20
1	92.9	97.8
2	95.0	98.6
3	94.3	96.9
4	94.3	96.4
5	94.8	95.1
6	94.5	98.9
$\bar{x}$	94.3	97.3

4.11 Chromatograms

Chromatograms at each target concentration are shown in Figures 4.11.1 and 4.11. 2. The chromatograms are from injections of standards equivalent to 5-L air samples at the target concentrations.

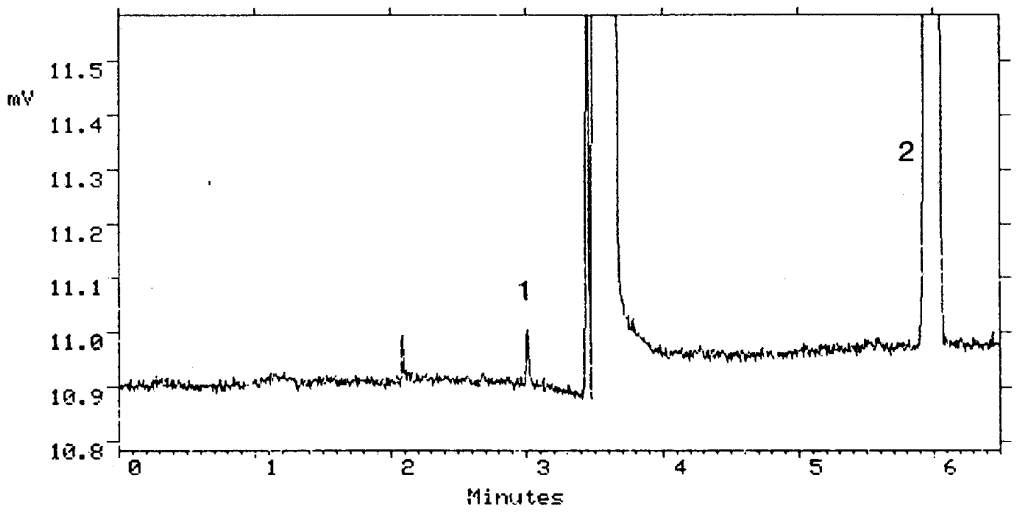


Figure 4.1. Detection limit chromatogram. Key: (1) propylene oxide (2) benzene.

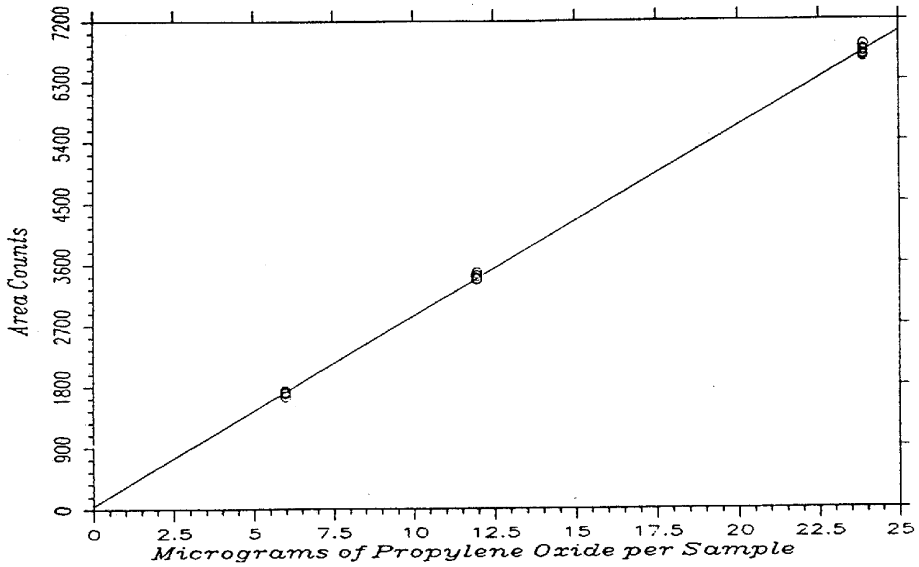


Figure 4.4.1. Instrument response to propylene oxide over the 0.5 to 2 times the TC-1 range.

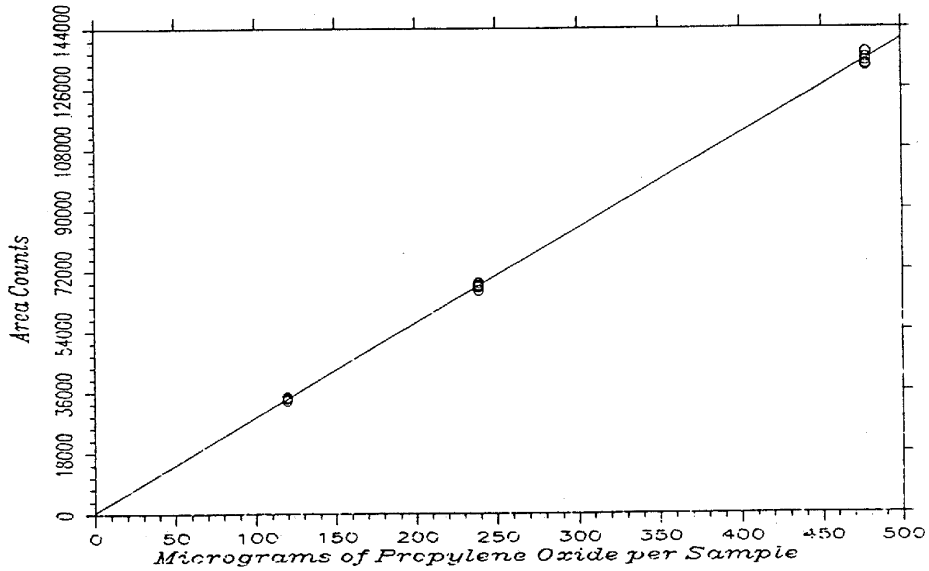


Figure 4.4.2. Instrument response to propylene oxide over the 0.5 to 2 times the TC-20 range.

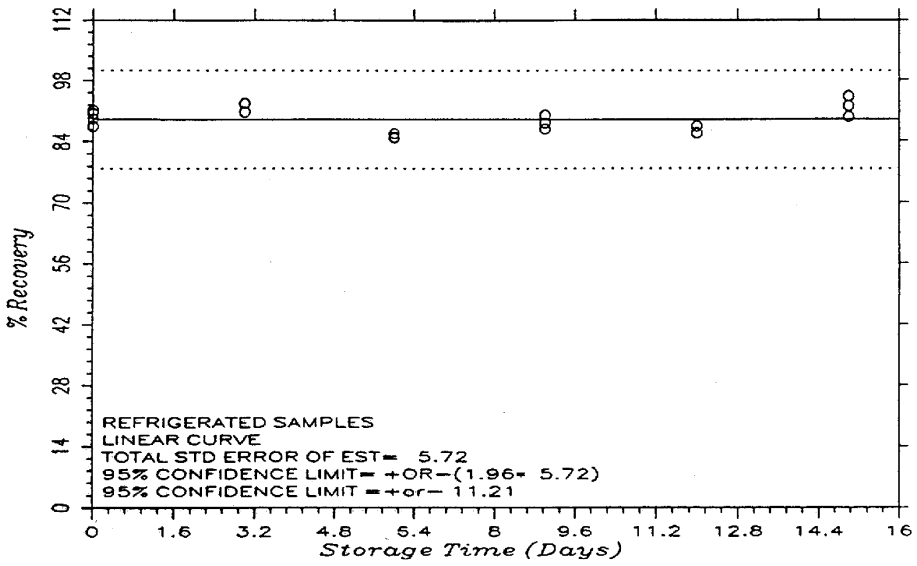


Figure 4.5.1.1. TC-1 refrigerated storage samples.

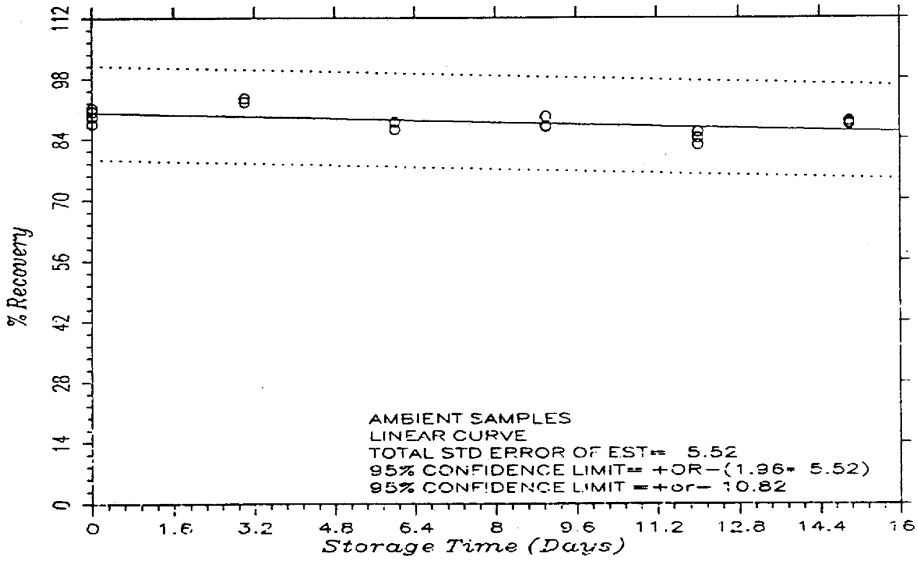


Figure 4.5.1.2. TC-1 ambient storage samples.

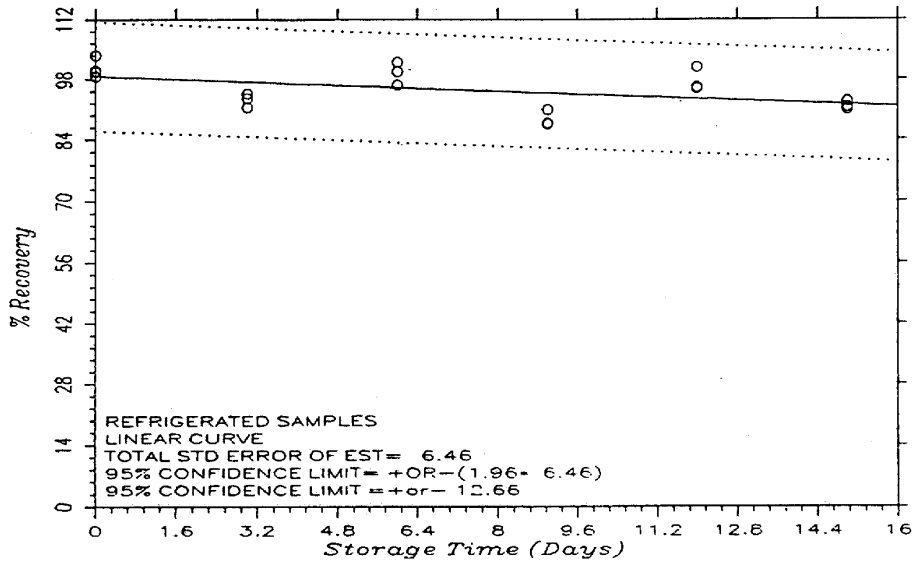


Figure 4.5.2.1. TC-20 refrigerated storage samples.

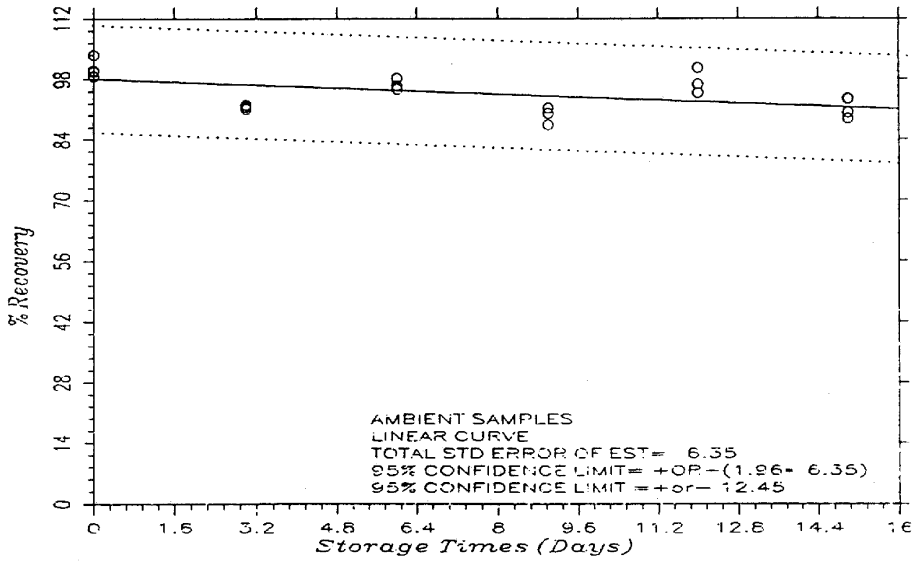


Figure 4.5.2.2. TC-20 ambient storage samples.

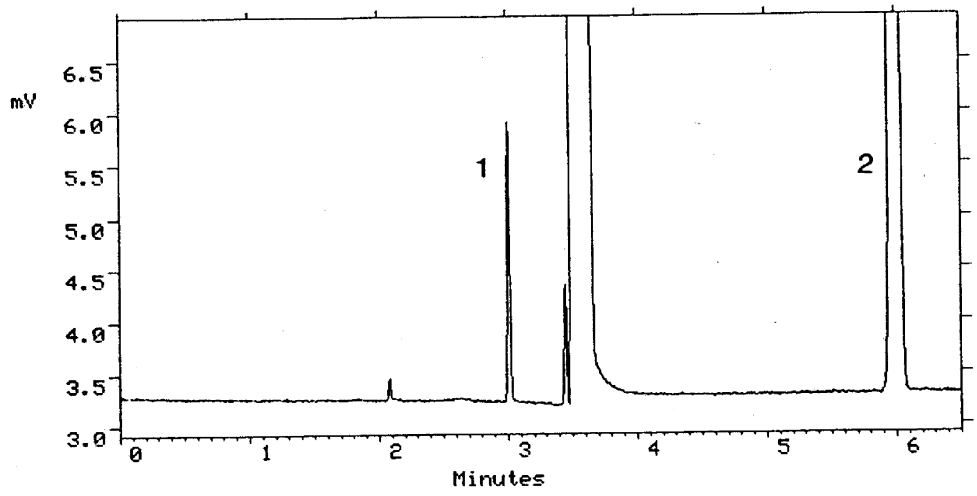


Figure 4.11.1. Chromatogram of a standard at the TC-1 target concentration. Key: (1) propylene oxide (2) benzene.

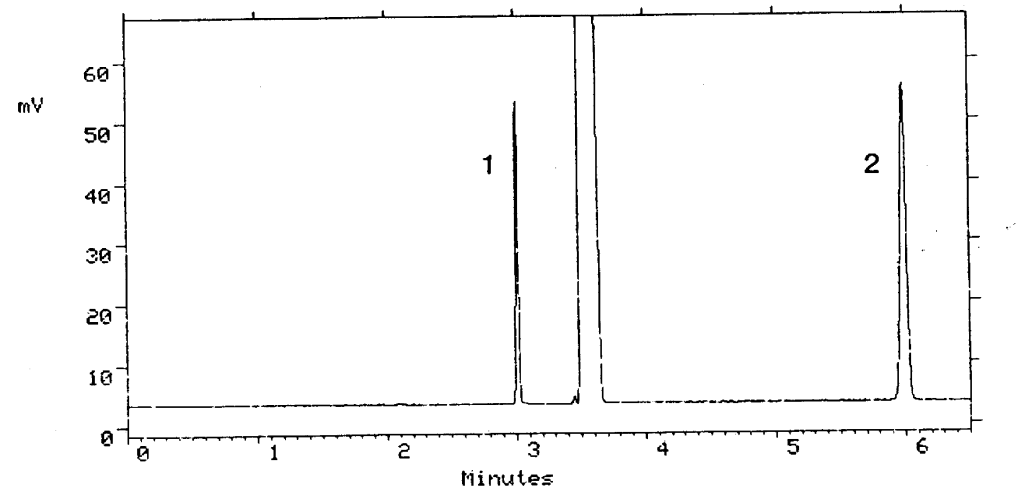


Figure 4.11.2. Chromatogram of a standard at the TC-20 target concentration. Key: (1) propylene oxide (2) benzene.

5. References

5.1 "Code of Federal Regulations", 29 CFR 1910. 1000, Table Z-1-A.-Limits for Air Contaminants, U.S.' Government Printing Office, Washington, DC, 1990.

5.2 "NIOSH Current Intelligence Bulletin 51: Carcinogenic Effects of Exposure to Propylene Oxide", U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Publications Dissemination, DSDTT; Cincinnati, OH, 1989, Publ. No. 89-111.

5.3 "Code of Federal Regulations", 29 CFR 1910.1047, Ethylene Oxide, U.S. Government Printing Office, Washington, DC, 1990.

5.4 "NIOSH Manual of Analytical Methods", 3rd ed. Vol. 2; U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering; Cincinnati, OH, 1985, Method 1612, DHHS (NIOSH).

5.5 "Occupational Health Guidelines for Chemical Hazards", NIOSH/OSHA, Jan. 1981, DHHS (NIOSH) Publ. No. 81-123.

5.6 "Registry of Toxic Effects of Chemical Substances", 1985-86 ed. Vol. 4; U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, U.S. Government Printing Office, Washington, DC, 1987.

5.7 Miller, J. C.; Miller, J. N. "Statistics for Analytical Chemistry", Ellis Horwood Limited: Chichester, England, 1984, p 59-62.