

Research Report

Validation of Ethyl tert-Butyl Ether (tert-Butyl Ethyl Ether, TBEE) using SKC Passive Sampler 575-001

Abstract

A sampling method for ethyl tert-butyl ether (tert-butyl ethyl ether, TBEE) in air has been validated for concentration levels from 0.4 to 140 ppm and for exposure times from 30 minutes to 8 hours. The 575-001 passive sampler used has a sample medium of Coconut Charcoal. Desorption was with carbon disulfide and analysis by gas chromatography with flame ionization detection (GC-FID).

The analytical recovery over the range of 0.4 to 140 ppm (11.3 to 3750 µg) was 101%.

The sampling rate is 13.1 ml/min. Samples can be taken from 10° C to 40° C.

Minimum recommended sampling time is 15 minutes. Maximum recommended sampling time is 8 hours.

A full validation of methyl tert-butyl ether was done according to NIOSH protocol¹ and a partial NIOSH validation by the bi-level procedure was conducted for TBEE and other higher members of this homologous series.

Authors

Christine O'Lear
Martin Harper
Lloyd V. Guild

Compiled by Martin Harper

SKC Inc.
863 Valley View Road
Eighty Four, PA 15330 USA

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Ethyl tert-Butyl Ether (tert-Butyl Ethyl Ether, TBEE)*

Importance of Passive Sampler Validation

There are distinct differences between a passive sampler and a sample tube.

The most important difference is that a passive sampler does not have a foolproof back up section that guarantees that all of the chemical hazard has been collected and that there is a true and total measure of worker exposure.

Secondly, the sorbent medium is exposed to the external environment and this poses problems not associated with a sample tube where the air sample passes into the sample tube directly contacting the sorbent medium. That is why it is critical to use a strong sorbent medium in passive samplers to assure complete capture and retention.

Therefore, for compliance purposes a passive sampler must be laboratory tested and validated under worst case field conditions for all factors that affect sampling accuracy as well as interaction between affects.

NIOSH has laid out a rigorous and complete validation protocol to assure that the sample collected is a complete and true measure of worker exposure. The following are the factors that the NIOSH protocol addresses:

Factors That Affect Complete Sample Uptake & Retention

Chemical hazard concentration	Temperature
Time of exposure	Humidity
Sorbent capacity	Interfering chemicals
Sorbent strength	Reverse diffusion from sorbent surface
Wind velocity	Sampler orientation
Interaction of any of the above factors	

Validation by NIOSH protocol assures that the sample results are a true and total measure of worker exposure.

**SKC validation follows the NIOSH Validation Protocol.
Certain experiments may have been modified for practical
reasons, or to provide more rigorous tests.**

User Responsibility

The sampler manager should be a professional trained in air sampling and aware of the limitations and advantages of the method being used. It is also very helpful if he or she has a working relationship with the analytical techniques being used and the requirements of record-keeping.

In accordance with ASTM D6346-98 and ANSI 104-1998 standards, use of samplers outside the range of conditions used in these validation tests does not assure accurate results and is not recommended. It is the user's responsibility to determine whether the conditions of the sampling site fall within the range tested. For bi-level validations it can be assumed that the applicable range is that used for testing the lower member of the homologous series.

Workers should be trained in the use of the equipment. In collecting the sample, care should be taken in the location of the sampler on the worker. It is to be openly exposed near the breathing zone. Exact times of exposure must be recorded. No moisture condensation should occur on the sampler. Workers should not be allowed to touch the sampler as they may transfer contamination. Particular attention must be paid to environments where liquid aerosols may be present, since droplets of liquid solvent on the sampler face will invalidate the sample. Any other field conditions outside of the limits used in the NIOSH protocol, such as extreme temperatures or stagnant air conditions that might affect the sampler operation, should be recorded.

Good laboratory practice must be followed. Follow the operating instructions for the desorption time needed for complete desorption. Use only the correct desorption instrument. If gas chromatography is used as the analysis method, base line separation should occur with the chemical hazard of interest and proper instrument calibration procedures used.

NIOSH or OSHA analytical methods should be used.

Summary of NIOSH Validation Protocol¹

Characteristic	Experimental Design	Interpretation of Results														
1. Analytical recovery	Spike 16 samplers, 4 at each of 4 concentration levels (0.1, 0.5, 1.0 & 2.0 x STD). Equilibrate about 12 h and analyze.	The higher 3 levels require $\geq 75\%$ recoveries with $S_r \leq 0.1$.														
2. Sampling rate and capacity	Expose samplers (4 per time period) for 1/8, 1/4, 1/2, 1, 2, 4, 6, 8, 10 & 12 h to 2 x STD, 80% RH, and 20 cm/s face velocity. Plot concentration vs. time exposed. Determine MRST and SRST.	Verify sampling rate. State useful range at 80% RH & 2 x STD. Capacity - sample loading corresponding to the downward break in conc. vs. time curve from constant concentration. SRST - time linear uptake rate achieved. MRST-0.67 x capacity (1 analyte). MRST-0.33 x capacity (multi-analyte).														
3. Reverse diffusion	Expose 20 samplers to 2 x STD, 80% RH for 0.5 x MRST. Remove and analyze 10 samplers. Expose others to 80% RH and no analyte for remainder of MRST.	Require $\leq 10\%$ difference between means of the two sampler sets at the 95% CL.														
4. Storage stability	Expose 3 sets of samplers (10 per set) at 80% RH, 1 x STD, and 0.5 x MRST. Analyze first set within 1 day, second set after 2 weeks storage at room temperature, and third set after 2 weeks storage at about 5° C.	Require $\leq 10\%$ difference at the 95% CL between means of stored sampler sets and set analyzed within 1 day.														
5. Factor effects	<div>Test the following factors at the levels shown. Use a 16-run fractional factorial design (4 samplers per exposure) to determine significant factors.</div> <table><tr><th>Factor</th><th>Test Levels</th></tr><tr><td>Analyte concentration</td><td>0.1 & 2 x STD</td></tr><tr><td>Exposure time</td><td>SRST & MRST</td></tr><tr><td>Face velocity</td><td>10 & 150 cm/s</td></tr><tr><td>Relative humidity</td><td>10 & 80% RH</td></tr><tr><td>Interferant</td><td>0 & 1 x STD</td></tr><tr><td>Sampler orientation</td><td>parallel & perpendicular (to airflow)</td></tr></table>	Factor	Test Levels	Analyte concentration	0.1 & 2 x STD	Exposure time	SRST & MRST	Face velocity	10 & 150 cm/s	Relative humidity	10 & 80% RH	Interferant	0 & 1 x STD	Sampler orientation	parallel & perpendicular (to airflow)	Indicate any factor that causes a statistically significant difference in recovery at the 95% CL. Investigate further to characterize its effect.
Factor	Test Levels															
Analyte concentration	0.1 & 2 x STD															
Exposure time	SRST & MRST															
Face velocity	10 & 150 cm/s															
Relative humidity	10 & 80% RH															
Interferant	0 & 1 x STD															
Sampler orientation	parallel & perpendicular (to airflow)															
6. Temperature effects	Expose samplers (10 per temp) to 0.5 x STD at 10, 25, & 40° C for 0.5 x MRST.	Define temperature effect and verify correction factor, if provided.														
7. Accuracy and precision	Calculate precision and bias for samplers (10 per conc. level) exposed to 0.1, 0.5, 1 & 2 x STD at 80% RH for \geq MRST. Use data from previous experiments.	Require bias within $\pm 25\%$ of true value at 95% CL with precision $S_r \leq 10.5\%$ for 0.5, 1, & 2 x STD levels.														

Summary of NIOSH Validation Protocol (cont.)

Characteristic	Experimental Design	Interpretation of Results
8. Shelf-life	Observe samplers throughout evaluation for changes in blank values, physical appearance, etc. Test samplers from more than one lot, if possible.	Note shelf storage time at which changes begin to occur. Indicate whether correctable or not.
9. Behavior in the field	Consider problems not predictable from laboratory experiments.	Record temperature, humidity, air velocity, other contaminants, etc.
<i>Area sampling:</i>	Expose passive samplers and independent method samplers (13 each) to the same environment.	Calculate precision and bias. Compare with laboratory results.
<i>Personal sampling:</i>	Conduct personal sampling with ≥ 25 sampler pairs. Place pairs of passive samplers and independent samplers on the same lapel of each worker.	Calculate bias. Compare with area sampling and laboratory results.

Bi-level Validation (previously designated by SKC as 5B)

Passive sampler validation is essential to ensure accurate determination of airborne chemical levels. To assist manufacturers and users, the National Institute for Occupational Safety and Health (NIOSH), the Health and Safety Executive (HSE)², and the Comité Européen de Normalisation (CEN)^{3,4} have developed comprehensive protocols for the validation of passive samplers.

Bi-level validation can also be used to assure that a sample gives the total and complete exposure to a chemical hazard.

Bi-level validation is only for a series of chemically-related compounds, i.e., members of a homologous series. Bi-level validation includes a full protocol validation on key compounds followed by a partial validation on other members of the series.

The concept of a bi-level validation of chemically-related compounds for a given sorbent and sampler design is based on the following premises and has been studied by Guild et al.⁵

1. Full validation by NIOSH, HSE, or CEN protocol of a lower member of the series is essential to assure accurate, routine sampling under all field conditions without the need for error-corrective measures.
2. Capacity and retentivity are directly related to the affinity of a sorbent for a specific chemical. For a series of chemically-related compounds, the affinity of a sorbent for a particular member compound will increase with the molecular weight and boiling point of the member. If a sorbent is suitable for collecting a low molecular weight member of the series, it will be suitable for the higher molecular weight members of the series as well.
3. For chemically-stable compounds, sample loss by reverse diffusion and loss during storage are inversely related to the affinity of the sorbent for the adsorbate. Therefore, compounds with higher molecular weights and boiling points will exhibit less loss by reverse diffusion and storage. Again, if a sorbent is suitable for a member with a lower molecular weight and boiling point, it will be suitable for the higher members.
4. The linearity of uptake with time is also a function of sorbent affinity and capacity. Uptake becomes increasingly linear as the molecular weight and boiling point increases and the sample load decreases. (Protocol validation requires study of concentrations ranging from 0.1 to 2.0 times the Permissible Exposure Limit [PEL].)

Bi-level Validation (cont.)

5. Temperature affects the accuracy of passive samplers in two different ways, the relation of temperature to adsorption affinity and the relation of the molecular diffusion of the sample to the sampler.
 - a. It is well known that the affinity of a sorbent for a chemical decreases with increasing temperature. If the sorbent has adequate affinity for a low molecular weight member of the series at 40° C (the maximum temperature tested under protocol), it will also be adequate at lower temperatures and for higher molecular weight members of the series.
 - b. The effects of temperature on sample uptake follow established mathematical relationships and are not significant compared to other random sampling errors.
6. The effects of humidity will be most pronounced for lower members of the series because of competition or modification of sorbent affinity.
7. Adsorption affinity decreases with the mass adsorbed. Therefore, the “key” member chosen for full validation should have a high Permissible Exposure Limit (PEL) relative to the other members of the series.
8. Air velocity and sampler orientation effects are functions of sampler design and will be similar for all compounds.
9. If all the factors affecting sampling accuracy improve with increasing molecular weight and boiling point and there are no interacting effects of these parameters with a lower member of the series, then there will be no interacting effects with higher members.
10. The accuracy of a sampler is determined by its bias and precision. For most passive samplers, the bias is the result of the deviation of the calculated sample rate from the actual rate. By determining the sample rate under known conditions at 1 PEL, the bias is reduced to zero. Therefore, measured sample rates should be determined for all compounds.
11. The precision of a sampler is a function of the consistency of sampler manufacture and the analytical procedures in the laboratory.
12. Analytical recovery tends to decrease with increased sorbent affinity and is a function of the chemical compound, the concentration, and the sorbent. Therefore, analytical recovery should be determined for every compound over the concentration range of 0.1 to 2.0 PEL, as recommended by protocol.

Conclusion: The above premises have been verified, peer-reviewed, and published.⁵ Therefore, Bi-level validation (5B) is an excellent way to assure accurate performance of a passive sampler for higher members of a homologous series.

Comments on the Relationship Between the NIOSH and CEN Diffusive Sampler Evaluation Protocols

The Comité Européen de Normalisation (CEN) is engaged in writing standards for air sampling equipment that include the limitations on precision and accuracy (EN 482) and the required performance tests. In the case of passive samplers, the relevant performance test standard is EN 838.

The precision and accuracy requirements in EN 482 are based on the use that will be made of the results, principally for either problem identification or compliance purposes. The standard for compliance purposes is a combined precision and accuracy of less than 30%, which is a looser standard than the 25% in the NIOSH protocol.

The performance tests are closely related to those in the NIOSH protocol, as might be expected, since they are trying to confirm the performance of the samplers over a similar range of environmental conditions. As in the NIOSH protocol, there are tests for desorption efficiency, uptake rate at different concentrations and for different time-periods, reverse diffusion, storage stability, wind velocity and orientation, humidity, and temperature. As in the NIOSH protocol, these factors are normally tested using a "high" and a "low" measure, either alone or in combination. Since there is little difference between workplace conditions in the U.S.A. and Europe, these "high" and "low" conditions in the two protocols are very similar. In general, the NIOSH test provides the more stringent conditions (e.g., 7.5 minutes up to 12 hours in the NIOSH uptake rate experiment versus 30 minutes and 8 hours in the CEN equivalent). In addition, for the majority of the experiments, the NIOSH protocol requires more samples to be taken for each data point (typically 10 rather than 6). The reverse diffusion test is one test that might be considered significantly different. A paper showing that the results of the tests are actually comparable has been submitted for publication.⁶

In addition, the CEN protocol requires tests for shelf-life and packaging integrity that have been carried out for one analyte (n-Hexane) only. The 575 Series passive sampler successfully passed these tests.

For the reasons given above, SKC considers the validations presented in these research reports to be at least sufficient to meet the requirements of the European Standards EN 838 and EN 482 for compliance monitoring. This conclusion is supported by a detailed comparison, which has been published.⁷

Full validation is equivalent to level 1A of EN 838. Partial validation, according to the Bi-level Theory of Validation, is equivalent to level 1B of EN 838.

Shelf-life Study on 575 Series Passive Samplers

Protocol: Four expired and two unexpired 575-001 samplers were exposed to an atmosphere of 100 ppm n-Hexane (2 x PEL) at 80% relative humidity (25° C) for 30 minutes, and then analyzed. This study was conducted in August 1995.

Results:

Calculated atmosphere concentration:	106 ppm
Gas sample analysis concentration:	102 ppm (RSD = 7.0%)
Sorbent tube analysis concentration:	115 ppm (RSD = 3.2%)
Sampler analysis concentration: [◇]	
Sampler expired 12/92:	106 ppm
Sampler expired 4/94:	106 ppm
Sampler expired 10/94:	108 ppm
Sampler expired 10/94:	110 ppm
Sampler unexpired (7/96):	100 ppm
Sampler unexpired (7/96):	100 ppm

[◇] Based on 111.6% desorption efficiency

Conclusion: Samplers will perform as expected up to their expiration date.

Packaging Integrity Study on 575 Series Samplers

Protocol: Six 575-001 samplers in unopened Tedlar® pouches were exposed to an atmosphere of 100 ppm n-Hexane (2 x PEL) at 80% relative humidity (25° C) for four hours, and then opened and analyzed.

Results:

Calculated atmosphere concentration:	103 ppm
Gas sample analysis concentration:	104 ppm (RSD = 8.7%)
Sorbent tube analysis concentration:	103 ppm (RSD = 2.7%)

Sampler analysis: No detectable n-Hexane in any sampler.

Estimated LOD = 1.5 micrograms, equivalent to 0.125 ppm.

Conclusion: Packaging will prevent contamination of stored samplers.

Scope of the Method

Analyte:	Ethyl tert-Butyl Ether (tert-Butyl Ethyl Ether, TBEE)
Matrix:	Air
Procedure:	Adsorption on a 575-001 SKC passive sampler, desorption with 2 ml of carbon disulfide, and analysis by GC-FID.
Exposure guidelines:	ACGIH-TLV® (1994/95) None OSHA (1995) None NIOSH (1995) None

Validation range & recovery:

<u>Compound</u>	<u>Validation Range-ppm in air</u>	<u>Mean % Recovery</u>
TBEE	0.4 to 140	101

Detection limits: The limit of detection is less than 10 µg/sample.

Humidity effects: High humidity conditions (the presence of 200 µl/water) did not significantly affect the recovery of TBEE, nor did high humidity conditions (80% RH at 25° C) affect the uptake of TBEE on the 575-001 passive sampler.

Storage effects: None observed with MTBE, none expected with TBEE.

Interferences: Any compound that has the same retention time as TBEE will interfere with analysis.

Validation completion date: February 1996

Physical properties:

<u>Mol. Weight (g/mole)</u>	<u>Boiling Pt. at 760 mm Hg</u>	<u>Density (g/ml)</u>
102.17	71° C	0.750

Background

History of Methodology

None

Research Purpose

The present work was to evaluate and validate the SKC 575 Series passive sampler containing coconut charcoal as a method for sampling TBEE. The passive sampler was validated over a concentration range of 0.4 to 140 ppm. Critical parameters include analytical recovery, concentration, and relative humidity.

Experimental

Ninety-seven percent pure TBEE (Acros Chemicals) was used. The HPLC-grade carbon disulfide (99.9%) was obtained from Aldrich Chemical Company. The 575 Series passive sampler containing coconut charcoal (SKC Cat. No. 575-001) and the coconut charcoal tubes used for atmosphere calibrations (SKC Cat. No. 226-01) are available from SKC, Inc.

A dynamic atmosphere generation apparatus was used to generate precise concentrations of TBEE in air for exposure of the passive samplers. The system is described in Appendix A and Figure 1. The atmosphere was fed into an exposure test chamber. The passive samplers were exposed on a rotating bracket inside the test chamber to simulate wind velocity and orientation.

Analytical recoveries for the passive samplers were conducted by injecting a known amount of TBEE (as a CS₂ solution) into the back of each sampler. The passive samplers were capped, allowed to equilibrate overnight, and analyzed the next day to determine analytical recovery or desorption efficiency. The tests were conducted at mass loadings equivalent to an 8-hour Time-Weighted Average (TWA) sample (5.68 L based on a calculated sampling rate of 13.3 ml/min) at 0.4, 4, 20, 40, 80, and 140 ppm under dry conditions and at 20 ppm with 200 µl water added to mimic recovery from humid conditions.

The sampling rate experiment on the passive sampler was conducted under dynamic conditions in the test chamber described above.

The passive samplers were desorbed (in situ) with 2 ml of carbon disulfide and shaken on a flatbed shaker for 30 minutes. All extracts were transferred to autosampler vials and analyzed by flame ionization gas chromatography. A chromatogram with analytical conditions is shown in Appendix A, Figure 2.

Sampling Rate Determination

Sampling rates can be determined by one of several statistical methods from the experimental data and they differ by only a small amount. Any bias taken is toward the protection of the worker.

We use the Time-Weighted Average from one to eight hours where results fall within NIOSH criteria.

We constantly review our data and conduct experimental work to provide the most precise sampling rate. This rate may differ slightly from previously published sampling rates. Use the rate listed in this report.

Analytical Recovery

Ethyl tert-Butyl Ether (tert-Butyl Ethyl Ether, TBEE)

NIOSH Requirements

Experimental Design

Spike 16 samplers, 4 at each of 4 concentration levels (0.1, 0.5, 1.0 & 2.0 x STD). Equilibrate about 12 h and analyze. See Analytical Recoveries on page 11 for explanation.

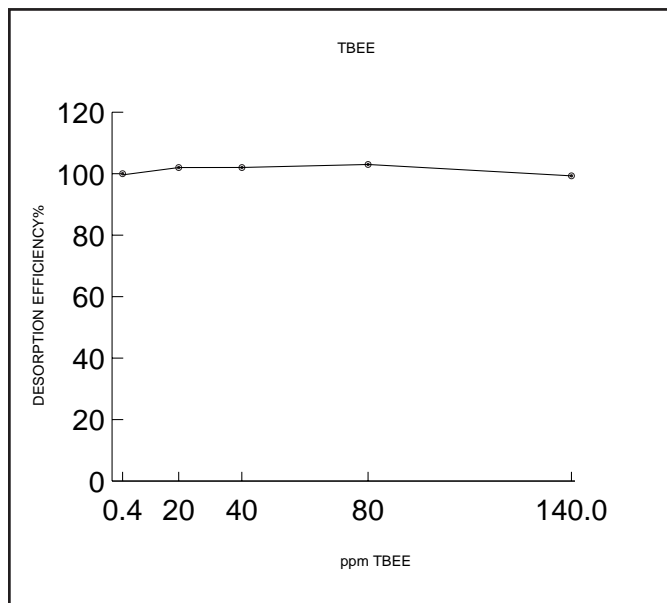
Interpretation of Results

The 3 higher levels require $\geq 75\%$ recoveries with $S_r \leq 0.1$.

Results:

8-hour TWA Concentrations	Spike (µg)	Recovery (µg)	Recovery %	Mean	RSD %
0.04	11.25	11.61	103		
		11.61	103		
		10.35	92.0		
		11.31	101	100	5.4
0.4	112.5	112.9	100		
		111.5	99.1		
		113.5	101	100	1.0
		114.1	101	103	
20		562.5	578.1		
		575.4	100		
		580.4	103		
		571.6	102	102	0.7
20*	562.5	532.8	94.7		
		550.4	97.8		
		544.8	96.9	96.5	1.7
40	1125.0	1147.0	102		
		1142.0	102		
		1137.0	101	102	0.4
		1140.4	102		
80	2250	2321.0	103		
		2319.0	103		
		2294.0	102		
		2365.0	105	103	1.3
140	3750	3665.0	97.7		
		3779.0	101		
		3703.0	98.7		
		3751.0	100	99.3	1.4
Overall Mean				101	

* 200 µl water added; results not used in overall mean.



Sampling Rate and Capacity

Ethyl tert-Butyl Ether (tert-Butyl Ethyl Ether, TBEE)

NIOSH Requirements

Experimental Design

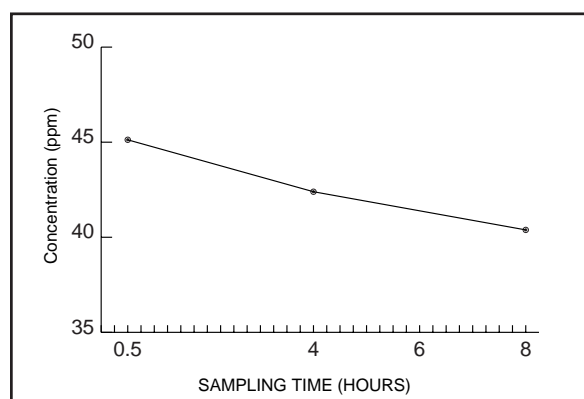
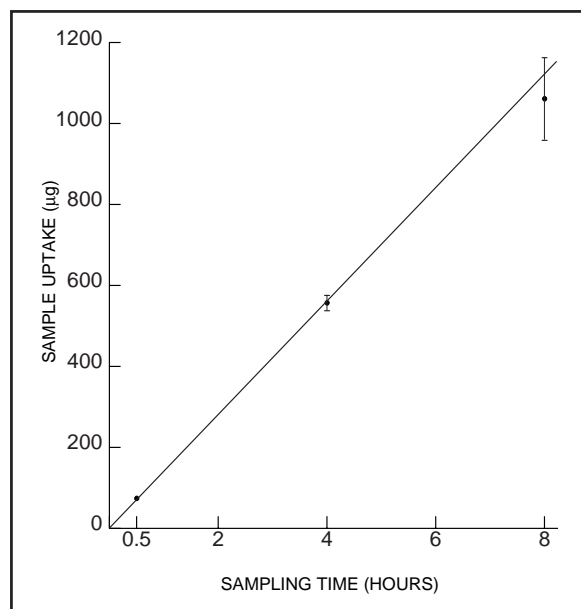
Expose samplers (4 per time period) for 1/8, 1/4, 1/2, 1, 2, 4, 6, 8, 10 and 12 h to 2 x STD, 80% RH and 20 cm/s face velocity. Plot concentration vs. time exposed. Determine MRST and SRST.

Interpretation of Results

Verify sampling rate. State useful range at 80% RH and 2 x STD. Capacity - sample loading corresponding to the downward break in conc. vs. time curve from constant concentration. SRST-time linear uptake rate achieved. MRST - 0.67 x capacity (1 analyte) MRST-0.33 x capacity (multi-analyte).

Results:

Time (hrs)	Uptake μg	Mean μg	RSD %	DE Corr μg	Conc. (ppm)
0.50	76.4 73.1 74.9				
4.0	75.1 549.8 564.3 572.1	74.8	1.9	74.1	45.1
8.0	564.6 1059 1003 1111 1114	562.7	1.7	557.1	42.4
		1072	4.8	1061	40.4



The mean sampling rate was 13.1 ml/min (3.29 μg/ppm/hr) and was calculated as the mean of all the exposure periods, based on a reference concentration of 42.6 ppm.

Appendix A

Atmosphere Generation Apparatus

The instrument is designed to expose a known concentration of a chemical hazard to a passive sampler under controlled conditions of: 1. Concentration, 2. Temperature, 3. Humidity, 4. Wind Velocity Effect, 5. Time, and 6. Up to four multicomponent hazards.

Description

The instrument consists of:

1. An exposure chamber in which the wind velocity effects are controlled by internal rotating holders.
2. An air supply and purification train such that dry air is blended with saturated air under desired temperature conditions so as to provide air at a known flow and selectable humidity.
3. An injection system composed of precision motor driven syringes in which 1 to 4 chemical hazards can be injected into the flow system and in which the temperature of the injectors is closely controlled.
4. An electrical control system that controls the entire instrument operation.
5. The chamber concentration can be verified by either solid sorbent sampling tubes actively sampled or by gas analysis of the gas phase. The particular verification method used will depend on the analyte of interest.

Means are also included to check the relative humidity.

Figure 1
Atmosphere Generation Apparatus

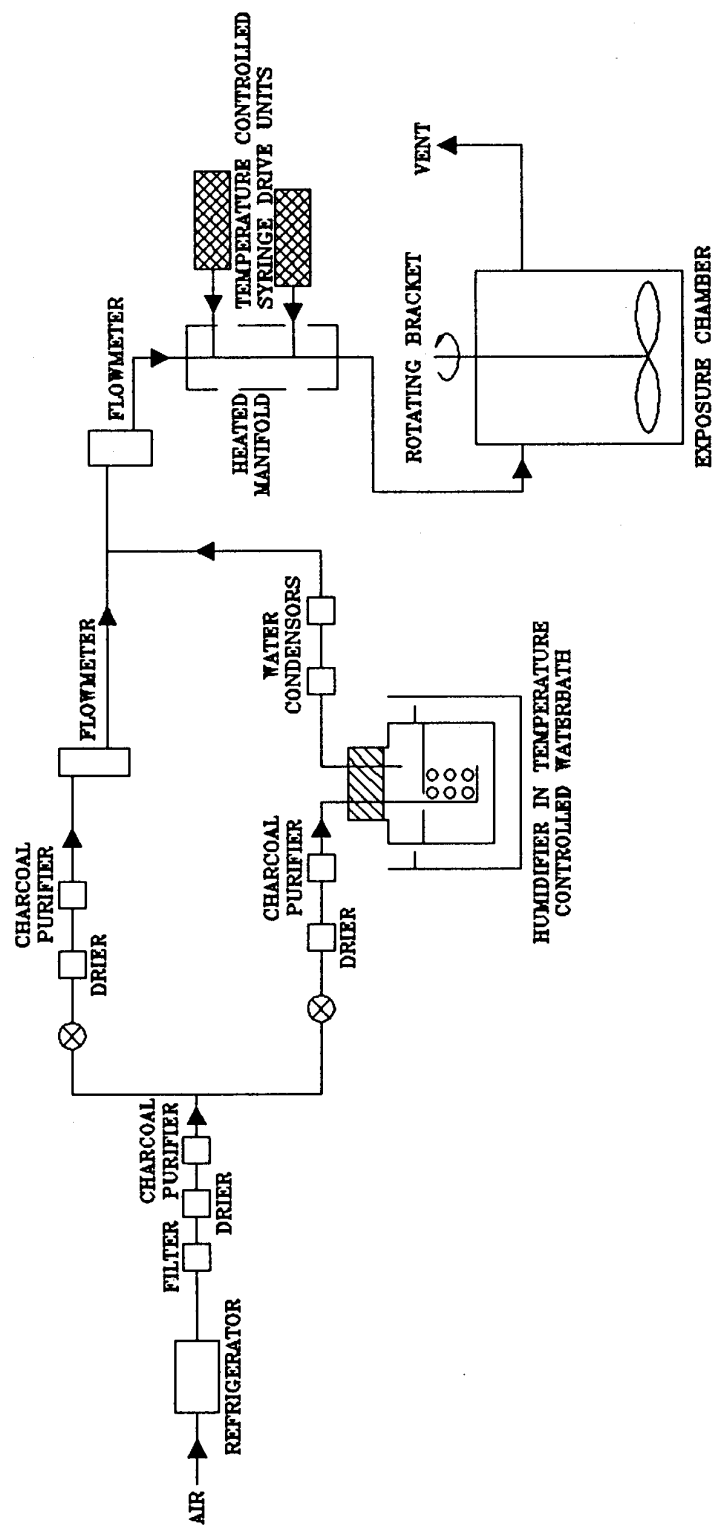
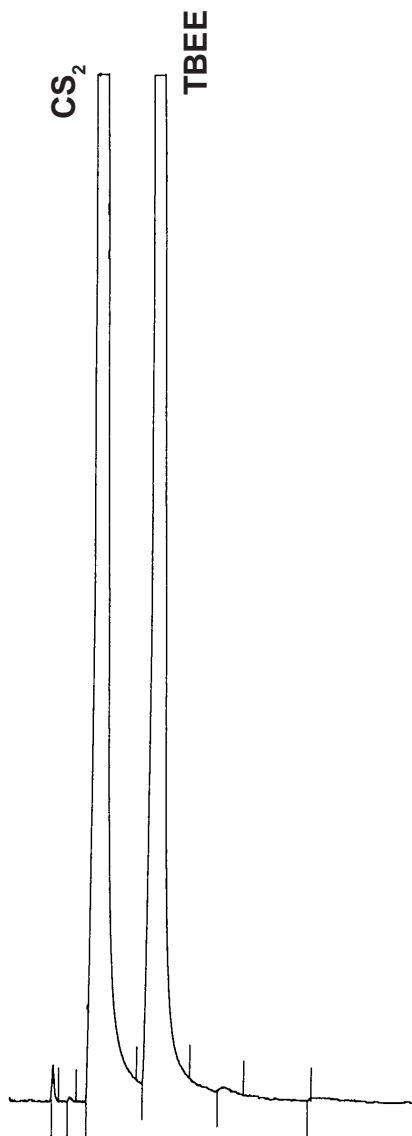


Figure 2
Analytical Instrument

Sample Chromatogram
TBEE in CS₂



GC Conditions

Column: 10' x 1/8" 10% SP-1000
on 80/100 Supelcoport®

Temperatures: Column: 50° C
FID: 150° C

Carrier gas: N₂ at 20 ml/min

Injection: 1.75 µL

Abbreviations

C	Celsius
CL	Confidence Level
cm	centimeter
ml	milliliter
min	minute
g	gram
GC-FID	Gas Chromatography-Flame Ionization Detector
h	hour
L	Liter
LOD	Limit of Detection
MRST	Maximum Recommended Sampling Time
N.S.	Not Significant
PEL	Permissible Exposure Limit
RH	Relative Humidity
TLV	Threshold Limit Value
TWA	Time-Weighted Average
RSD	Relative Standard Deviation
SD	Standard Deviation
SRST	Shortest Recommended Sampling Time
STD	appropriate exposure standard (OSHA PEL, ACGIH TLV, or NIOSH recommended standard)
S	Second
S_r	pooled relative standard deviation
V	Volume

Trademarks

Anasorb is a registered trademark of SKC Inc.

Tedlar is a registered trademark of DuPont Corporation.

Porapak is a registered trademark of Waters Associates, Inc.

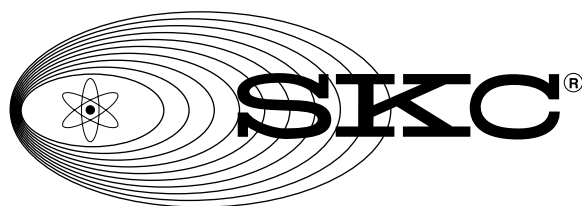
Supelcoport is a registered trademark of Supelco, Inc.

TLV is a registered trademark of ACGIH.

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**Validation of Ethyl tert-Butyl Ether
(tert-Butyl Ethyl Ether, TBEE)
using
SKC Passive Sampler
575-001**



**SKC Inc.
863 Valley View Road
Eighty Four, PA 15330**

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