METHYL METHACRYLATE

Method no.:	94
Matrix:	Air
Target concentration:	100 ppm (410 mg/m^3)
Procedure:	Samples are collected by drawing air through glass sampling tubes containing coconut shell charcoal coated with 4-tert-butylcatechol. Samples are desorbed with toluene and analyzed by GC using a flame ionization detector.
Recommended air volume and sampling rate:	3 L at 0.05 L/min
Reliable quantitation limit:	151 ppb (617 µg/m^3)
Standard error of estimate at the target concentration:	5.85%
Special requirements:	Samples should be stored at reduced temperature when not in transit.
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

Date: June 1992

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1. General Discussion

1.1 Background

1.1.1 History

The OSHA Salt Lake Technical Center has in the past used NIOSH Method 2537 for the sampling and analysis of methyl methacrylate (MME) (Ref. 5.1). The NIOSH method specified collection using a jumbo XAD-2 tube (6-mm i.d. × 8-mm o.d. × 11.0 cm, 400 mg/200 mg) which had to be shipped to the laboratory at dry ice temperature. The method also specified desorption with carbon disulfide and analysis by GC with a flame ionization detector (FID). To eliminate the dry ice temperature shipment requirement, coconut shell charcoal coated with 4-*tert*-butylcatechol (TBC) was utilized for sampling. TBC has been previously used in OSHA methods to inhibit the polymerization of reactive compounds. (Refs. 5.2 - 5.4)

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

The vapor of MME can irritate the nose and throat at air concentrations of about 170 ppm. Concentrations above 2000 ppm are intolerable. High concentrations of vapor may cause headache, drowsiness, dizziness, difficulty in breathing, and at very high levels, loss of consciousness. Death by pulmonary edema has occurred. (Ref. 5.5)

Vapor concentrations above 170 ppm will irritate the eyes and may generate tearing. Liquid can cause considerable irritation or burns to the eyes. Skin contact with liquid MME may produce irritation or burns. Allergic skin sensitization can occur over time. Sensitized persons may have a severe reaction to low doses which do not affect unsensitized persons. Ingestion of MME irritates the mouth and stomach; causes nausea, vomiting, dizziness and drowsiness; and may produce liver and kidney damage. (Ref. 5.5)

The International Agency for Research on Cancer reports that there is inadequate data to support evidence for carcinogenicity of MME in humans or animals. (Ref. 5.6)

In the Code of Federal Regulations, the final rule limits in Table Z-1-A specify a TWA of 100 ppm (410 mg/m^3). (Ref. 5.7)

1.1.3 Workplace exposure

In 1979, 307 million kilograms of MME were produced in the United States (Ref. 5.8). MME is used in the manufacture of acrylic plastics (Lucite and Plexiglass), latex house paints, building materials, automobile parts, lubricating oil additives, polishes and coatings, adhesives, sealants, dental implants, hard contact lenses, bone cement, bone replacements (artificial hips), corneal implants, and ultraviolet cured inks in the printing industry. (Ref. 5.5)

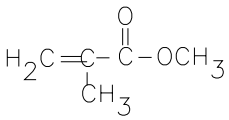
1.1.4 Physical properties and other descriptive information (Ref. 5.5.)

CAS no.:	80-62-6
molecular weight:	100.12
melting point:	-48°C
boiling point:	100°C
chemical formula:	$C_5H_8O_2$
vapor pressure: (at 20°C)	4.67 kPa (35 mmHg)
density: (at 20°C)	0.936 g/mL
self-ignition temperature:	421°C
flash point: (open cup)	10°C
lower explosive limit:	1.7%
upper explosive limit:	8.2%
odor threshold:	<1 ppb

solubility: slightly soluble in water; soluble in most organic solvents

synonyms: methylacrylic acid, methyl ester; methyl alpha-methyl acrylate; methyl 2-methyl-2-propenoate; 2-methyl propenoic acid, methyl ester; MME; Pegalan

structure:



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

1.2 Limit-defining parameters

1.2.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 0.343 ng per injection (1.0-µL injection with a 5.4:1 split). This is the amount of analyte that will produce a peak with height that is approximately 5 times the height of a nearby contaminant peak. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 1.852 µg per sample. This is the amount of analyte spiked on the sampling device that, upon analysis, produces a peak similar in size to that of the detection limit of the analytical procedure. This detection limit corresponds to an air concentration of 151 ppb (617 µg/m^3). (Section 4.2)

1.2.3 Reliable quantitation limit

The reliable quantitation limit is 1.852 µg per sample. This is the smallest amount of analyte which can be quantitated within the requirements of a recovery of at least 75% and a precision (±1.96 SD) of ±25% or better. This reliable quantitation limit corresponds to an air concentration of 151 ppb (617 µg/m^3). (Section 4.3)

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

1.2.4 Instrument response to the analyte

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

1.2.5 Recovery

The recovery of MME from samples used in the 15-day ambient storage test remained above 83.4%. (Section 4.5, regression line of Figure 4.5.1)

1.2.6 Precision (analytical procedure)

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1 and 2 times the target concentration is 0.0041. (Section 4.6)

1.2.7 Precision (overall procedure)

The precision at the 95% confidence level for the 15-day ambient temperature storage test is ±11.5%. (Section 4.7) This includes an additional ±5% for sampling error.

1.2.8 Reproducibility

Six samples, liquid-spiked with MME, and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after 7 days of storage at 2°C. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.8)

1.3 Advantages

1.3.1 The sampling device is smaller and more conveniently sized than the large XAD-2 tube recommended by NIOSH Method 2537.

1.3.2 The sampler may be shipped at ambient temperatures. The large XAD-2 tube had to be shipped at dry ice temperature.

2. Sampling Procedure

2.1 Apparatus

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

2.1.2 Samples are collected with 4-mm i.d. × 6-mm o.d. × 7.0 cm glass sampling tubes packed with two sections of coconut shell charcoal that has been coated with TBC, 10% by weight. The front section contains 110 mg and the back section contains 55 mg of TBC-coated coconut shell charcoal. The sections are held in place with glass wool plugs. For this evaluation, tubes were purchased from SKC, Inc. (catalog no. 226-73).

2.2 Reagents

No sampling reagents are required.

2.3 Technique

2.3.1 Immediately before sampling, break off the ends of the TBC-coated coconut shell charcoal tube. All tubes should be from the same lot.

2.3.2 Attach the sampling tube to the sampling pump with flexible tubing. It is desirable to utilize sampling tube holders which have a protective cover to shield the employee from the sharp, jagged end of the sampling tube. Position the tube so that sampled air passes through the 110-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 Attach the sampler vertically with the 110-mg section pointing downward, in the worker's breathing zone so it does not impede work performance or safety.

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

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

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

2.3.8 Ship any bulk samples separate from the air samples.

2.3.9 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

The sampling capacity of the front section of a TBC-coated coconut shell charcoal sampling tube was tested by sampling from a dynamically generated test atmosphere of MME (144 ppm or 589

mg/m^3). The sample was collected at 0.05 L/min and the relative humidity was 80%. The 5% breakthrough occurred after sampling for 164 min or 8.19 L. (Section 4.9)

2.5 Desorption efficiency

2.5.1 The average desorption efficiency from TBC-coated coconut shell charcoal adsorbent is 96.1% over the range of 0.5 to 2 times the target concentration. (Section 4.10.1)

2.5.2 Desorbed samples remain stable for at least 25 h. (Section 4.10.2)

2.5.3 The desorption efficiency was determined at lower concentrations, down to 2% of the target concentration. This was done because the desorption efficiency decreased to 76% at the RQL. The desorption efficiency did not decrease over the concentration range studied. (Section 4.10.3)

2.5.4 Desorption efficiencies should be confirmed periodically because differences may occur due to variations in sampler lots, desorption solvent, and operator technique.

2.6 Recommended air volume and sampling rate

2.6.1 For TWA samples, the recommended air volume is 3 L collected at 0.05 L/min (1-h samples).

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

2.6.3 When short-term samples are required, the reliable quantitation limit becomes larger. For example, the reliable quantitation limit is 0.60 ppm (2.47 mg/m^3) when 0.75 L of air is collected.

2.7 Interferences (sampling)

2.7.1 It is not known if any compounds will severely interfere with the collection of MME on TBC-coated coconut shell charcoal. In general, the presence of other contaminant vapors in the air will reduce the capacity of TBC-coated coconut shell charcoal to collect MME.

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 A GC equipped with a flame ionization detector (FID). A Hewlett-Packard 5890 Gas Chromatograph equipped with a 7673A Autosampler and an FID was used in this evaluation.

3.1.2 A GC column capable of separating MME and the internal standard from the desorbing solvent and any potential interferences. A 60-m × 0.32-mm i.d. SPB-1 (4.0-µm film thickness) capillary column (Supelco, Inc.) was used in this evaluation.

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

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

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

3.2 Reagents

3.2.1 Methyl methacrylate (MME). Reagent grade or better should be used. The BAKER grade used in this evaluation was purchased from JT Baker Chemical Co. (Phillipsburg, NJ).

3.2.2 Toluene. Reagent grade or better should be used. The Burdick & Jackson B&J Brand High Purity Solvent used in this evaluation was purchased from Baxter Healthcare Corp. (Muskegon, MI).

3.2.3 Desorbing solution. The desorbing solution is prepared by adding 20 µL of an appropriate internal standard to 1 L of toluene. Benzene (reagent grade) was used in this evaluation as the internal standard and was purchased from EM Science (Gibbstown, NJ).

3.3 Standard preparation

3.3.1 Prepare concentrated stock standards of MME in toluene. Prepare working analytical standards by injecting microliter amounts of concentrated stock standards into 2-mL vials containing 1 mL of desorbing solution delivered from the same dispenser used to desorb samples. For example, to prepare a target level standard, inject 10 µL of a stock solution containing 123.5 mg/mL of MME in toluene into 1 mL of desorbing solution.

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

3.4 Sample preparation

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

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

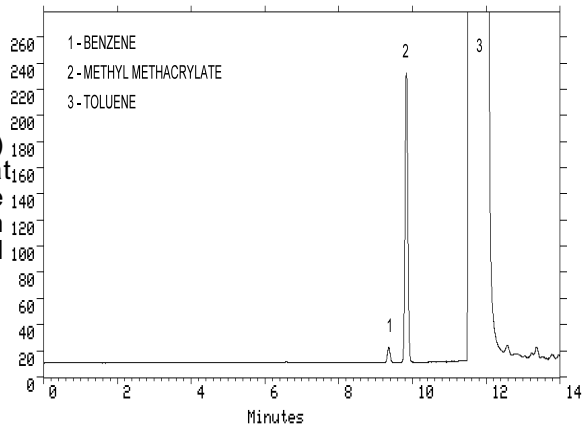
3.4.3 Shake the vials vigorously several times during the next 30 min to ensure complete desorption.

3.5 Analysis

3.5.1 Analytical conditions

<u>GC conditions</u>

zone temperatures:	250°C (injector) 300°C (detector)
column program:	initial temp at 120°C, increase temp at 5°C/min to 150°C, hold for 5 min
column gas flow:	1.35 mL/min (hydrogen)
septum purge:	1.5 mL/min (hydrogen)
injection size:	1.0 µL (5.4:1 split)
column:	60 m × 0.32-mm i.d. capillary SPB-1 (4.0-µm film thickness)
retention times:	9.8 min (MME) 9.3 min (benzene)



<u>FID conditions</u>

hydrogen flow:	36.5 mL/min

air flow: 444 mL/min
nitrogen makeup flow: 46.5 mL/min

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

3.6 Interferences (analytical)

3.6.1 Any compound that produces an FID response and has a similar retention time as the analyte or internal standard is a potential interference. If any potential interferences were reported, they should be considered before samples are desorbed. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

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

3.7 Calculations

The amount of analyte per milliliter is obtained from the appropriate calibration curve in terms of micrograms per milliliter uncorrected for desorption efficiency. The back (55-mg) section is analyzed primarily to determine the extent of sample saturation during sampling. If any analyte is found on the back section, it is added to the amount on the front section. This total amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulae.

$$mg/m^3 = \frac{A \times B}{C \times D}$$

where A = micrograms of analyte per milliliter
B = desorption volume
C = liters of air sampled
D = desorption efficiency

$$ppm = \frac{24.46 \times mg/m^3}{MW}$$

where 24.46 = molar volume (liters) at 101.3 kPa (760 mmHg) and 25°C
MW = 100.12

3.8 Safety precautions (analytical)

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

3.8.2 Avoid skin contact and inhalation of all chemicals.

3.8.3 Wear safety glasses, gloves and a lab coat at all times while working with chemicals.

4. Backup Data

4.1 Detection limit of the analytical procedure

The injection size recommended in the analytical procedure (1-µL, 5.4:1 split) was used to determine the detection limit of the analytical procedure. The detection limit of the analytical procedure is 0.343 ng on-column. This was the amount of analyte that gave a peak with a height about 5 times the height of a nearby contaminant peak. This detection limit was determined by analysis of a standard containing 1.852 µg/mL of MME.

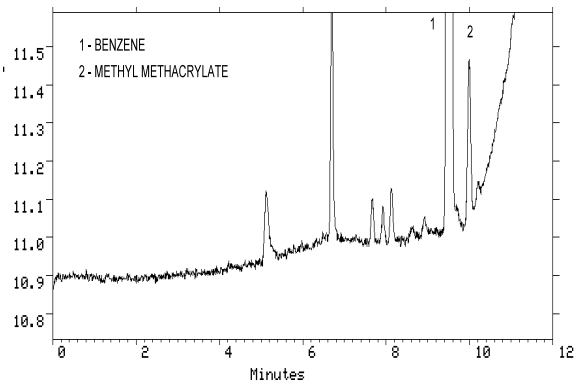


Figure 4.1. Detection limit of the analytical procedure.

4.2. Detection limit of the overall procedure

The detection limit of the overall procedure is 1.852 µg per sample (151 ppb or 617 µg/m^3). The injection size listed in the analytical procedure (1.0 µL, 5.4:1 split) was used to determine the detection limit of the overall procedure. Six vials containing 110 mg of TBC- coated coconut shell charcoal were liquid-spiked with 1.852 µg of MME. The samples were stored at ambient temperature and were desorbed about 8 h after being spiked.

Table 4.2
Detection Limit of the Overall Procedure for MME

sample no.	µg spiked	µg recovered
1	1.852	1.470
2	1.852	1.406
3	1.852	1.404
4	1.852	1.370
5	1.852	1.359
6	1.852	1.434

4.3 Reliable quantitation limit data

The reliable quantitation limit is 1.852 µg per sample (151 ppb or 617 µg/m^3). The injection size listed in the analytical procedure (1.0 µL, 5.4:1 split) was used to determine the reliable quantitation limit. Six vials containing 110 mg of TBC- coated coconut shell charcoal were liquid- spiked with a toluene solution containing MME. Because the recovery of the analyte from the spiked samples was greater than 75% and had a precision of ±25% or better, the detection limit of the overall procedure and reliable quantitation limit are the same.

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

percent recovered	statistics
79.4	
75.9	$\overline{X}$ = 76.0
75.8	SD = 2.2
74.0	Precision = (1.96)(±2.2)
73.4	= ±4.3
77.4	

4.4 Instrument response to the analyte

The instrument response to MME over the range of 0.5 to 2 times the target concentration is linear with a slope of 819 (in ISTD-corrected area counts per microgram per milliliter). The precision of the response to the analyte was determined by multiple injections of standards. The data below is presented graphically in Figure 4.4.

Table 4.4
Instrument Response to MME

× target concn µg/mL	0.5× 617.5	1.0× 1235	2.0× 2470
area counts	512437	1022070	2025506
	512257	1017157	2037759
	514988	1014684	2027601
	515158	1026172	2039743
	517743	1025209	2027149
	515895	1026773	2038024
$\overline{X}$	514746	1022011	2032630

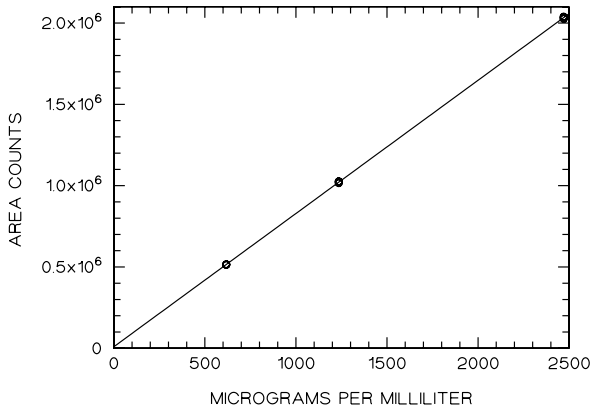


Figure 4.4. Calibration curve for MME.

4.5 Storage data

Storage samples were prepared by injecting 10 µL of a standard solution onto the TBC-coated coconut shell charcoal. The standard contained 123.5 mg/mL MME in toluene. Humid air, 80% RH, was drawn through the tubes for 1 h at 0.05 L/min. Thirty-six storage samples were prepared. Six samples were analyzed immediately. Fifteen tubes were stored at reduced temperature (12°C) and the other fifteen were stored in a closed drawer at ambient temperature (about 22°C). At 2 to 4 day intervals, three samples were selected from each of the two storage sets and analyzed. The results are listed below and shown graphically in Figures 4.5.1 and 4.5.2.

Table 4.5
Storage Test of MME

time (days)	percent recovery (ambient)			percent recovery (refrigerated)		
0	97.3	100.1	96.6	97.3	100.1	96.6
	90.5	98.8	90.2	90.5	98.8	90.2
3	91.6	92.0	97.6	94.8	100.5	100.9
6	88.1	88.2	90.2	95.9	95.0	94.7
9	85.0	86.2	85.6	92.6	92.9	94.0
13	86.1	82.5	84.7	93.0	95.9	96.2
15	86.3	84.6	87.0	92.2	93.1	95.0

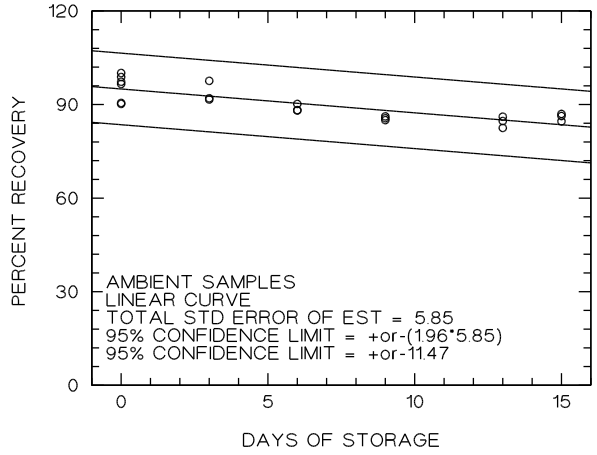


Figure 4.5.1. Ambient storage test for MME.

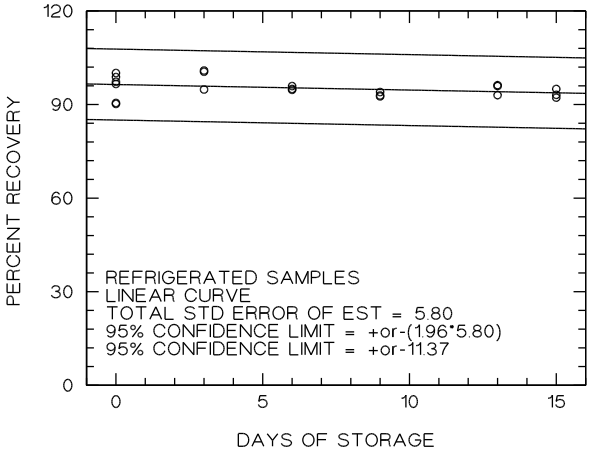


Figure 4.5.2. Refrigerated storage test for MME.

4.6 Precision (analytical method)

The precision of the analytical procedure is defined as the pooled coefficient of variation determined from replicate injections of MME standards at 0.5, 1 and 2 times the target concentration. Based on the data of Table 4.4, the coefficient of variation (CV) for the three levels and the pooled coefficient of variation were calculated. The pooled coefficient of variation is 0.0041.

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

× target concn µg/mL	0.5× 617.5	1.0× 1235	2.0× 2470
SD[1]	2100	5049	6513
CV	0.0041	0.0049	0.0032

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 precision at the 95% confidence level is obtained by multiplying the SEE (with pump error included) by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). The 95% confidence intervals are drawn about their respective regression line in the storage graph as shown in Figure 4.5.1. The data for Figure 4.5.1 was used to determine the SEE of ±5.85% and the precision of the overall procedure of ±11.5%.

4.8 Reproducibility data

Six samples were prepared by injecting an aliquot of a toluene solution containing 116.3 mg/mL MME onto the TBC-coated coconut shell charcoal. Humid air, 80% RH, was drawn through the tubes for 1 h at 0.05 L/min to add water to the sampler matrix. The samples were given to a chemist unassociated with this study. The samples were analyzed after being stored for 7 days at 2°C in a refrigerator. Sample results were corrected for desorption efficiency. No sample result has a deviation greater than the precision of the overall procedure determined in Section 4.7, which is ±11.5%.

Table 4.8
Reproducibility Data for MME

µg spiked	µg recovered	percent recovered	percent deviation
1163	1054.2	90.6	-9.4
1163	1102.2	94.8	-5.2
1163	1120.7	96.4	-3.6
1163	1102.9	94.8	-5.2
1163	1117.9	96.1	-3.9
1163	1092.2	93.9	-6.1

4.9 Sampler capacity

An attempt to generate a dynamic test atmosphere was performed by injecting pure MME from a gas-tight syringe driven by a syringe pump at 2.11 mg/min into an air stream that was flowing at 2.45 L/min (21°C and 80% relative humidity). This should have generated an atmosphere of 210 ppm or 861 mg/m^3. The MME polymerized in the airstream to form a solid and only a small amount of the MME vaporized. To overcome this problem, the bottom of a U-tube was filled with glass beads coated with *p*-methoxy phenol. *p*-Methoxy phenol is commonly used to inhibit the polymerization of MME. By placing the end of the polytetrafluoroethylene needle in the glass beads, most of the MME to vaporize and resulted in an atmosphere of 589 mg/m^3 or 144% of the target concentration. This was determined by sampling the air stream with a TBC-coated coconut shell charcoal tube and analyzing the tube. The atmosphere was much higher than the earlier attempt but was still not at theoretically amount. Solid polymerized MME was still found among the glass beads and this was the reason for the lower than theoretical amount.

The sampling capacity of the front section of a TBC-coated coconut shell charcoal sampling tube was tested by sampling the second dynamically generated atmosphere at 0.048 L/min. A GC equipped with a gas sampling valve and an FID detector was used to analyze the downstream effluent from the tube periodically. The response was compared to the previously measured upstream air flow. After the downstream concentration had exceeded 8% of the upstream concentration, the sampling was stopped. The 5% breakthrough air volume was determined to be the point when the downstream concentration was 5% of the upstream concentration or 8.19 L.

Table 4.9
Breakthrough on the TBC-coated Charcoal Tube for MME

air volume (L)	sample time (min)	breakthrough (percent)
2.84	60	0.00
4.02	84	0.00
5.24	110	0.33
5.64	118	0.49
6.37	133	1.09
6.62	138	1.38
6.86	144	1.71
7.10	149	2.35
7.35	154	2.78
7.60	159	3.21
7.84	164	3.91
8.08	169	4.64
8.33	174	5.57
8.58	179	6.71
8.82	185	7.85

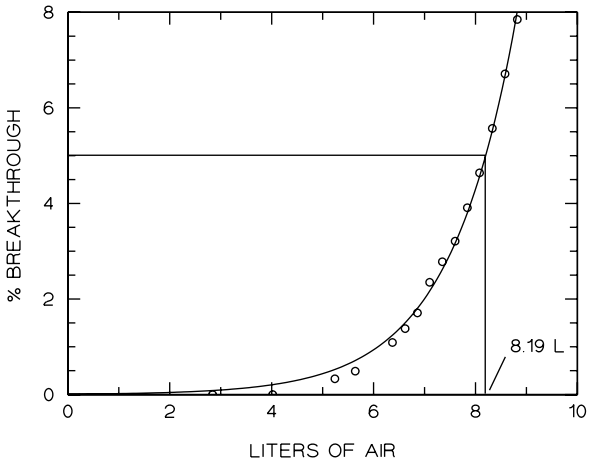


Figure 4.9. Breakthrough air volume for MME.

4.10 Desorption efficiency and stability of desorbed samples

4.10.1 Desorption efficiency

The desorption efficiency (DE) of MME was determined by liquid-spiking 110-mg portions of TBC-coated coconut shell charcoal with MME at 0.5, 1 and 2 times the target concentration. These samples were stored for 6 h at ambient temperature and then desorbed and analyzed. The average desorption efficiency over the studied range was 96.1%.

Table 4.10.1
Desorption Efficiency of MME

× target µg	0.5× 617.5	1.0× 1235	2.0× 2470
DE, %	96.8	96.7	96.6
	98.7	96.3	93.4
	95.3	96.7	94.8
	96.7	99.0	96.1
	94.4	95.8	94.4
	96.4	96.6	95.0
$\bar{X}$	96.4	96.8	95.0

4.10.2 Stability of desorbed samples

The stability of desorbed samples was investigated by reanalyzing the target concentration samples 25 h after initial analysis. The original analysis was performed and the vials were not recapped after injection. The samples were reanalyzed with fresh standards at the completion of the desorption efficiency test without removing the samples from the GC. The average recovery of the reanalysis, compared to the average recovery of the original analysis, was 97.2% (+0.4% change).

Table 4.10.2
Stability of Desorbed Samples for MME

initial recovery (percent)	recovery after 25 h (percent)	percent change
96.7	98.4	+1.7
96.3	96.4	+0.1
96.7	96.4	-0.3
99.0	98.2	-0.8
95.8	96.5	+0.7
96.6	97.4	+0.8

4.10.3 Linearity of desorption

The average desorption efficiency is 96.1% for MME but the desorption at the RQL, 0.15% of the target concentration, is 76.0%. This can infer that the desorption efficiency is not constant as the amount of MME on the sampler decreases. To determine the linearity of the desorption efficiency, a series of samplers were spiked over the range of 2 to 100% of the target concentration. The data from these samples resulted in a line that deviates only 2% from theoretical values. The desorption efficiency is linear throughout this range.

Table 4.10.3
Desorption Efficiency at Lower Concentrations

% of target concentration	µg spiked	µg recovered
2	24.7	29.6
4	49.4	50.6
6	74.1	71.6
8	98.8	92.6
10	123.5	114.9
20	247.0	240.8
40	494.0	492.8
60	741.0	739.8
80	988.0	1001.6
100	1235.0	1265.9

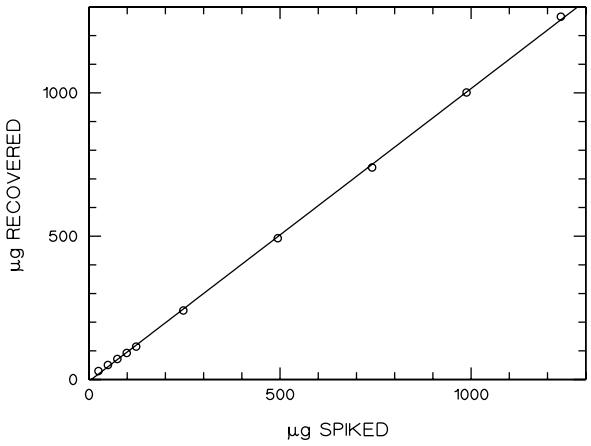


Figure 4.10.3. Linear of desorption over a wide range of concentrations.

5. References

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