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Sampling and Analytical Methods / Propylene Glycol Monomethyl Ethers/Acetates (Organic Method #99)

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Propylene Glycol Monomethyl Ethers/Acetates

1-Methoxy-2-Propanol (1M2P)

2-Methoxy-1-Propanol (2M1P)

1-Methoxy-2-Propyl Acetate (1M2PA)

2-Methoxy-1-Propyl Acetate (2M1PA)

Related Information: [Chemical Sampling - 1-Methoxy-2-Propanol \(1M2P\)](#), [2-Methoxy-1-Propanol \(2M1P\)](#), [1-Methoxy-2-Propyl Acetate \(1M2PA\)](#), [2-Methoxy-1-Propyl Acetate \(2M1PA\)](#)

Method no.:	99
Matrix:	Air
Procedure:	Samples are collected by drawing air through standard size (6-mm o.d., 100/50-mg sections) coconut shell charcoal tubes. Samples are desorbed with 95/5 (v/v) methylene chloride/methanol and analyzed by gas chromatography using a flame ionization detector. Samples should be desorbed in the presence of a drying agent such as magnesium sulfate or Drierite®.
Recommended air volume and sampling rate:	10 L at 0.1 L/min

	1M2P	2M1P	1M2PA	2M1PA
Target concentration:	100 ppm (368 mg/m ³)	1 ppm (3.7 mg/m ³)	100 ppm (540 mg/m ³)	1 ppm (5.4 mg/m ³)
Reliable quantitation limit:	20 ppb (74 µg/m ³)	20 ppb (74 µg/m ³)	20 ppb (108 µg/m ³)	20 ppb (108 µg/m ³)
Standard error of estimate at the target concentration:	5.3%	5.5%	5.1%	5.4%

Special requirement: Samples for 1M2PA and 2M1PA should be refrigerated upon receipt by the laboratory to minimize hydrolysis.

Status of method: Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

Date: April 1993

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1. General Discussion

1.1. Background

1.1.1. History

Over the past several years there has been an increase in the number of samples submitted to the OSHA Salt Lake Technical Center for propylene glycol ethers and their acetates. This is likely due to the increased usage of these solvents as substitutes for ethylene glycol ethers and their acetates, which have been associated with the potential to cause adverse reproductive effects in both male and female workers. (Ref. 5.1.) Now there is evidence that 2M1P and 2M1PA may exhibit analogous toxicities. (Ref. 5.2.) 2M1P and 2M1PA are present as impurities in technical grade 1M2P and 1M2PA respectively. OSHA has adopted a TWA-PEL of 100 ppm for 1M2P, but has no PEL for the other three analytes. (Ref. 5.3.) Because 1M2P and 1M2PA have similar toxic properties, a target concentration of 100 ppm was chosen for these two analytes. Based on their potential reproductive toxicity, a target concentration of 1 ppm was chosen for 2M1P and 2M1PA.

There were no reported air monitoring procedures in the literature for these propylene glycol ethers/acetates. A method has now been evaluated based on previous evaluations done at the OSHA Salt Lake Technical Center for a number of ethylene glycol ether/acetates, which are chemically similar to these compounds. (Refs. 5.4.-5.5.) Sampling is done by drawing workplace air through sampling tubes containing coconut shell charcoal. The analytes are adsorbed by the charcoal and the tubes are sent to the laboratory for analysis. The analytes are desorbed from the charcoal with a 95/5 (v/v) methylene chloride/methanol mixture, and are quantitated by GC using flame ionization detection.

As was found for the acetates of ethylene glycol ethers (Refs. 5.4.-5.5.), the acetates of propylene glycol ethers are also prone to hydrolysis after being adsorbed onto charcoal. The average loss due to hydrolysis was about 3% for 1M2PA (100 ppm for 10 L) and 11% for 2M1PA (1 ppm for 10 L) when generated samples were stored for 15 days at ambient temperatures. When generated samples were refrigerated for 15 days, the average loss was about 1% for 1M2PA and 4% for 2M1PA. The slow rate of loss due to hydrolysis indicates it is unnecessary to ship samples under reduced temperatures, but they should be refrigerated at the lab until analyzed.

As was the case for some of the other previously evaluated ethylene glycol ethers (Ref. 5.4.), the use of a drying agent such as magnesium sulfate is needed to improve the desorption efficiency of 1M2P and 2M1P from charcoal. Because MgSO₄ can only be purchased as a powder, periodically the syringe used for sample injections into the GC may be plugged by MgSO₄ suspended in solution. This can be avoided by centrifuging the samples or by allowing the powder to settle out before analysis. Two granular drying agents, 20-40 mesh Drierite® (anhydrous calcium sulfate) and 10-60 mesh sodium sulfate, were tested as alternatives. Sodium sulfate was found to be ineffective. Drierite® was effective, but 400 mg must be used versus 125 mg of MgSO₄. If Drierite® is used, samples need not be centrifuged and can be analyzed immediately after the desorption period.

Many solvent vapors collected on charcoal and analyzed at the OSHA Salt Lake Technical Center are desorbed with 99/1 (v/v) carbon disulfide/N,N-dimethyl-formamide (CS₂/DMF) instead of methylene chloride/methanol. Because it is possible that these propylene glycol ether/acetates may be used with other solvents in the workplace, CS₂/DMF was investigated as an alternative desorbing solvent. For the target concentrations studied, CS₂/DMF is an acceptable alternative for 1M2PA and 2M1PA samples if MgSO₄ is added to the desorption vials. Desorption with CS₂/DMF is not acceptable for 1M2P and 2M1P samples because there is considerable differences in desorption efficiencies from wet and dry charcoal, even if MgSO₄ is used. Also, the desorption efficiency for 2M1P is less than 75% from wet charcoal.

It is felt that there will always be some amount of corresponding alcohol present in samples containing acetates because the acetates may be partially hydrolyzed in the air before collection or on the charcoal after collection. For example, 1M2P would be found in samples containing 1M2PA, and 2M1P would be found in 2M1PA samples. Also, the alcohols could be present as contaminants in the corresponding technical grade acetates. For these reasons, it would be wise to analyze for the alcohols in acetate samples. Thus, unless it is absolutely necessary to desorb with CS₂/DMF for analysis of other co-collected solvents, samples for 1M2PA and 2M1PA should also be desorbed with methylene chloride/methanol in the presence of MgSO₄ or Drierite® to facilitate the analysis of the corresponding alcohols.

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

These propylene glycol ethers/acetates have low acute toxicity, with the main effect at high doses being central nervous system depression. There are no reported studies on the carcinogenicity of these compounds. 1M2P and 1M2PA appear to lack reproductive toxicity, with the critical effect being irritation of the eyes and mucous membranes. 2M1PA is embryotoxic and teratogenic in laboratory animals. It is very likely that 2M1P is also teratogenic because the common metabolite of 2M1P and 2M1PA is 2-methoxypropionic acid. 3-Methoxypropionic acid, which is a structural isomer of 2-methoxypropionic acid, causes growth retardation and abnormalities in post-implantation rat embryo cultures. This is analogous to the effects of methoxyacetic acid, which is a common metabolite of 2-methoxyethanol and 2-methoxyethyl acetate. (Ref. 5.2.)

1.1.3. Workplace exposure

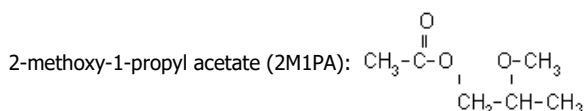
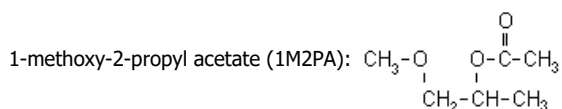
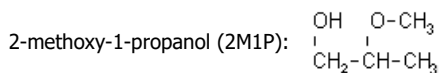
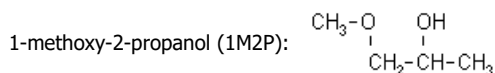
Propylene glycol ethers/acetates are used industrially as solvents for paints, lacquers, resins, oils and fats. Their use has increased considerably since 1985. This is probably because they are used as substitutes for the chemically similar ethylene glycol ethers, which have been associated with reproductive toxicity. From NIOSH occupational surveys done from 1981-1983, it is estimated that 329,000 people are potentially exposed to 1M2P and about 306,000 are potentially exposed to 1M2PA. 2M1P and 2M1PA, which are also known as the beta isomers, are typically present as impurities in varying amounts in 1M2P and 1M2PA respectively. (Ref. 5.2.)

1.1.4. Physical properties (Ref. 5.2. unless otherwise noted)

Property	1M2P	2M1P	1M2PA	2M1PA
CAS number:	107-98-2	1589-47-5	108-65-6	70657-70-4
molecular weight:	90.12	90.12	132.16	132.16
melting point (°C):			<-67	
boiling point (°C):	119.6	130 (Ref. 5.6.)	145.8	
flash point (°C):	38		42.2	
vapor pressure (kPa):	1.6 @ 25°C		0.5 @ 20°C	
vapor density (25°C, air=1):	3.11		4.55	
liquid density (25°C/4°C):	0.917	0.938 (Ref. 5.6.)	0.97	
description:	colorless liquid	colorless liquid	colorless liquid	colorless liquid
miscibility with water:	complete	complete	≈19% (w/w)	

Note: Commercial grade 1M2P contains mainly (95-99%) 1M2P, with the remainder (1-5%) being 2M1P. Technical 1M2PA consists mainly of (≈95%) 1M2PA, with remainder largely being 2M1PA. (Ref. 5.2.)

structural formulae:



synonyms:

1-methoxy-2-propanol- 1M2P; methoxypropanol, alpha isomer; propylene glycol monomethyl ether; propylene glycol methyl ether; PGME

2-methoxy-1-propanol- 2M1P; methoxypropanol, beta isomer; propylene glycol monomethyl ether; propylene glycol methyl ether; bPGME

1-methoxy-2-propyl acetate- 1M2PA; methoxypropyl acetate, alpha isomer; propylene glycol monomethyl ether acetate; propylene glycol methyl ether acetate; PGMEA

2-methoxy-1-propyl acetate- 2M1PA; methoxypropyl acetate, beta isomer; propylene glycol monomethyl ether acetate; propylene glycol methyl ether acetate; bPGMEA

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 limits of the analytical procedure are 48, 50, 71, and 71 pg per injection (1.0-μL injection with a 15:1 split) for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. These are the amounts of each analyte that will give peaks with heights approximately 5 times the height of baseline noise. (Section 4.1.)

1.2.2. Detection limit of the overall procedure

The detection limits of the overall procedure are 0.73, 0.75, 1.1, and 1.1 μg per sample for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. These are the amounts of each analyte spiked on the sampling device that, upon analysis, produce a peak similar in size to that of the respective detection limit of the analytical procedure. These detection limits correspond to air concentrations of 20 ppb (74 μg/m³), 20 ppb (74 μg/m³), 20 ppb (108 μg/m³), and 20 ppb (108 μg/m³) for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. (Section 4.2.)

1.2.3. Reliable quantitation limit

The reliable quantitation limits are 0.73, 0.75, 1.1, and 1.1 µg per sample for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. These are the smallest amounts of each analyte that can be quantitated within the requirements of recoveries of at least 75% and precisions (± 1.96 SD) of $\pm 25\%$ or better. These reliable quantitation limits correspond to air concentrations of 20 ppb (74 µg/m³), 20 ppb (74 µg/m³), 20 ppb (108 µg/m³), and 20 ppb (108 µg/m³) for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. (Section 4.3.)

The reliable quantitation limits and detection limits reported in the method are based upon optimization of the GC for the smallest possible amounts of each 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 target concentrations is linear for all four analytes. (Section 4.4.)

1.2.5. Recovery

The recovery of 1M2P, 2M1P, 1M2PA, and 2M1PA from samples used in a 15-day storage test remained above 100%, 94%, 97%, and 90% respectively when the samples were stored at ambient temperatures. (Section 4.5., from regression lines shown in Figures 4.5.1.2., 4.5.2.2., 4.5.3.2. and 4.5.4.2.)

1.2.6. Precision (analytical procedure)

The pooled coefficients of variation obtained from replicate determinations of analytical standards at 0.5, 1, and 2 times the target concentrations are 0.0025, 0.0045, 0.0025, and 0.0041 for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. (Section 4.6.)

1.2.7. Precision (overall procedure)

The precisions at the 95% confidence level for the ambient temperature 15-day storage tests are ± 10.3 , ± 10.8 , ± 10.0 , and $\pm 10.5\%$ for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. These include an additional $\pm 5\%$ for sampling error. (Section 4.7.)

1.2.8. Reproducibility

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

2. Sampling Procedure

2.1. Apparatus

2.1.1. Samples are collected using a personal sampling pump calibrated to within $\pm 5\%$ of the recommended flow rate with a sampling tube in line.

2.1.2. Samples are collected with solid sorbent sampling tubes containing coconut shell charcoal. Each tube consists of two sections of charcoal separated by a urethane foam plug. The front section contains 100 mg of charcoal and the back section, 50 mg. The sections are held in place with glass wool plugs in a glass tube 70-mm \times 4-mm i.d. \times 6-mm o.d. For this evaluation, SKC Inc. (Eighty Four, PA) charcoal tubes (catalog number 226-01, Lot 120) were used.

2.2. Reagents

None required

2.3. Technique

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

2.3.2. Connect the sampling tube to the sampling pump with flexible, non-crimpable tubing. It is desirable to utilize a sampling tube holder that shields the employee from the sharp, jagged end of the sampling tube. Position the tube so that sampled air first passes through the 100-mg section.

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

2.4.1. Sampler capacity is determined by measuring how much air can be sampled before breakthrough of analyte through the sampler occurs, 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 1M2P breakthrough was performed by monitoring the effluent (with a total hydrocarbon analyzer) from sampling tubes containing only the 100-mg section of charcoal while sampling at 0.2 L/min from an atmosphere containing 204 ppm of analyte. The atmosphere was at approximately 80% relative humidity and 20-25°C. The average 5% breakthrough volume from three determinations was 33.8 L (RSD=2.1%). A similar test was done for 1M2PA at 199 ppm. The average 5% breakthrough volume from three determinations was 24.9 L (RSD=6.8%).

2.4.2. Similar studies as in 2.4.1. were done for 1M2P at 2.0 ppm and 1M2PA at 2.2 ppm. Both atmospheres were sampled for more than 6 h (>72 L) with no breakthrough detected. 1M2P and 1M2PA were used in these studies instead of 2M1P and 2M1PA because sufficient quantities of the latter analytes were not available. It is felt that the breakthrough volume for 2M1P would approximate that of 1M2P and the breakthrough volume of 2M1PA would approximate that of 1M2PA.

2.5. Desorption efficiency

2.5.1. Desorption with 95/5 (v/v) methylene chloride/methanol

2.5.1.1. The average desorption efficiencies of 1M2P, 2M1P, 1M2PA, and 2M1PA from dry Lot 120 charcoal are 100.4%, 99.7%, 101.8%, and 101.4% respectively over the range of 0.5 to 2 times the target concentrations. (Section 4.9.)

2.5.1.2. Desorbed samples from Section 2.5.1. remain stable for at least 24 h. (Section 4.10.)

2.5.1.3. The desorption efficiencies at the target concentrations from wet charcoal are essentially the same as from dry charcoal when MgSO₄ is used. The desorbed samples are stable for at least 24 h. (Section 4.11.) The use of MgSO₄ is recommended for 1M2P and 2M1P samples, but is optional for 1M2PA and 2M1PA samples.

2.5.2. Desorption with 99/1 (v/v) carbon disulfide/N,N-dimethylformamide (CS₂/DMF)

2.5.2.1. The average desorption efficiencies of 1M2P, 2M1P, 1M2PA, and 2M1PA from dry Lot 120 charcoal are 86.3%, 80.8%, 98.4%, and 98.0% respectively over the range of 0.5 to 2 times the target concentrations. (Section 4.12.)

2.5.2.2. All of the analytes with the exception of 2M1P were sufficiently stable for the target concentration samples from Section 2.5.2.1. The average desorption efficiency for 2M1P dropped from 82.2% to 75.8% in 24 h. (Section 4.13.)

2.5.2.3. The average desorption efficiencies at the target concentrations from wet charcoal are 79.5% for 1M2P and 70.5% for 2M1P when MgSO₄ is used. These compare to 87.4% and 82.2% respectively from dry charcoal. The desorbed 1M2P samples are stable for at least 24 h, while the average desorption efficiency dropped to 64.3% for the 2M1P samples. The optional use of CS₂/DMF (with MgSO₄) would be acceptable for 1M2P samples but not for 2M1P samples at or around the studied loadings. (Section 4.14.)

The average desorption efficiencies at the target concentrations for 1M2PA and 2M1PA from wet charcoal using 99/1 CS₂/DMF as the desorbing solvent (with MgSO₄) are essentially the same as from dry charcoal. The desorbed samples are stable for at least 24 h. (Section 4.14.)

2.6. Recommended air volume and sampling rate

2.6.1. For TWA samples, the recommended air volume is 10 L collected at 0.1 L/min (100-min samples). The recommended air volume is significantly lower than the breakthrough volumes, but 10 L was chosen to provide a reasonable safety margin in case other solvent vapors are present in the sampled air. Also, 10 L is commonly recommended for solvent vapors collected on charcoal tubes.

2.6.2. For short-term samples, the recommended air volume is 3 L collected at 0.20 L/min (15-min samples).

2.6.3. When short-term samples are required, the reliable quantitation limits become larger. For example, the quantitation limits are 67 ppb when 3 L is sampled.

2.7. Interferences (sampling)

2.7.1. It is not known if any compound(s) will severely interfere with the collection of any of the four analytes on charcoal. In general, the presence of other contaminant vapors in the air will reduce the capacity of charcoal to collect the analytes.

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

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.

2.8.2. Wear eye protection when breaking the ends of the charcoal 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 5890A Gas Chromatograph equipped with a 7673A Automatic Sampler was used.

3.1.2. A GC column capable of separating the analyte of interest from the desorbing solvent, internal standard and any interferences. A 30-m × 0.32-mm i.d. fused silica Stabilwax-DA® column with a 1-μm df (Cat. no. 11054, 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 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. 1-Methoxy-2-propanol, 2-methoxy-1-propanol, 1-methoxy-2-propyl acetate, and 2-methoxy-1-propyl acetate, reagent grade or of known purity. Lot HB062777 1M2P from Aldrich Chemical Company (Milwaukee, WI), 2M1P from Dow Chemical (Midland, MI), and mixtures of 1M2PA and 2M1PA from Aldrich (Lot 03019HY) and Frinton Laboratories (Vineland, NJ) were used in this evaluation. The Aldrich 1M2P contains some 2M1P as a contaminant and the Dow Chemical 2M1P contains some 1M2P. A 98.97/1.03 (w/w) 1M2P/2M1P mixture was prepared by mixing the appropriate proportions of these two solvents. The mixture was assayed by GC. The density of the mixture was determined to be 0.917 g/mL (RSD=0.1%, n=4) at 24°C by weighing 10.00 mL of the mixture contained in tared volumetric flasks. A 99.01/0.99 (w/w) 1M2PA/2M1PA mixture was similarly prepared from the Aldrich and Frinton mixtures. The density of this mixture was determined to be 0.961 g/mL (RSD=0.17%, n=4) at 24°C.

3.2.2. Anhydrous magnesium sulfate or 20-40 mesh Drierite® (anhydrous calcium sulfate), reagent grade. Chempure Lot M172 KDHM magnesium sulfate was used in this evaluation.

3.2.3. Methylene chloride, chromatographic grade. Burdick and Jackson Lot BB551 was used in this evaluation.

3.2.4. Methanol, chromatographic grade. Fisher Lot 913607 was used in this evaluation.

3.2.5. A suitable internal standard, reagent grade. "Quant Grade" 2-heptanol from Polyscience Corporation (Niles, IL) was used in this evaluation.

3.2.6. The desorbing solvent consists of 95/5 (v/v) methylene chloride/methanol containing an internal standard at a concentration of 1 µL/mL.

3.2.7. GC grade nitrogen, air, and hydrogen.

3.3. Standard preparation

3.3.1. Prepare standards by injecting microliter amounts of analytes into vials containing 1.0 mL of desorbing solvent delivered from the same dispenser used to desorb samples. For example, to prepare a standard of 1M2P and 2M1P, inject 4.00 µL of a 98.97/1.03 (w/w) 1M2P/2M1P mixture (density = 0.917) into a vial containing 1.0 mL of desorbing solvent. This standard contains 3630 µg of 1M2P and 37.78 µg of 2M1P per sample.

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 section of the samples to separate vials. Discard the glass tubes and plugs.

3.4.2. For 1M2P and 2M1P samples and for 1M2PA and 2M1PA samples to be analyzed for 1M2P and 2M1P respectively, add about 125 mg of anhydrous magnesium sulfate or 400 mg of 20-40 mesh Drierite® to each vial.

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

3.4.4. Immediately cap the vials and shake them periodically for about 30 min.

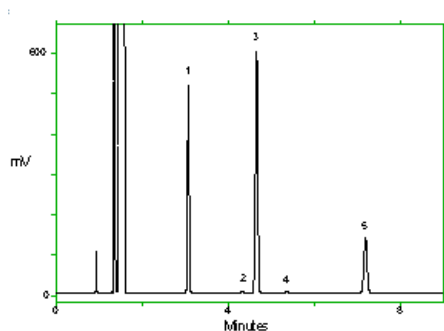
3.4.5. If magnesium sulfate is used as the drying agent, centrifuge the vials or allow time for the powder to settle out to avoid plugging the syringe used for GC injections.

3.5. Analysis

3.5.1. GC conditions

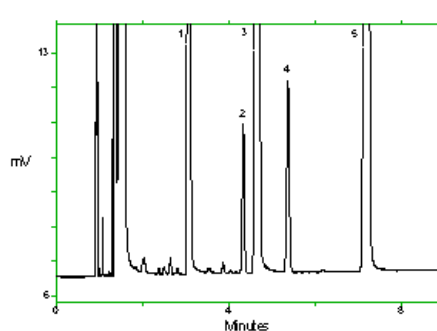
zone temperatures:	column-	95°C
	injector-	175°C
	detector-	200°C
gas flows:	hydrogen (carrier)-	3.0 mL/min (60 kPa head pressure)
	nitrogen (makeup)-	37 mL/min
	hydrogen (flame)-	33 mL/min
	air-	390 mL/min
signal range:		0
injection volume:		1.0 µL (with a 15:1 split)
column:		30-m × 0.32-mm i.d. fused silica, Stabilwax-DA®, 1-µm df
retention times:	1M2P-	3.1 min
	2M1P-	4.4 min
	1M2PA-	4.7 min
	2M1PA-	5.4 min
	2-heptanol-	7.2 min (internal standard)

chromatograms at the target concentrations:



Chromatogram of the analytes at the target concentrations with 1M2P and 1M2PA at approximately full scale.

Key: (1) 1M2P, (2) 2M1P, (3) 1M2PA, (4) 2M1PA, (5) 2-heptanol.



Chromatogram of the analytes at the target concentrations with 2M1P and 2M1PA at approximately full scale.

Key: (1) 1M2P, (2) 2M1P, (3) 1M2PA, (4) 2M1PA, (5) 2-heptanol.

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 analyte 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 responds on a flame ionization detector and has the same general retention time of the analyte or internal standard is a potential interference. Possible interferences should be reported to the laboratory with submitted samples by the industrial hygienist. These interferences 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. Analyte identity should be confirmed by GC/mass spectrometry.

3.7. Calculations

The analyte concentration for samples is obtained from the appropriate calibration curve in terms of micrograms of analyte per sample, uncorrected for desorption efficiency. The air concentration is calculated using the following formulae. The back (50-mg) section is analyzed primarily to determine if there was any breakthrough from the front (100-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 on the blank.

(μg of analyte per sample)

$$\text{mg/m}^3 = \frac{\text{---}}{(\text{L of air sampled})(\text{desorption efficiency})}$$

where desorption efficiencies = 1.00 for 1M2P, 1.00 for 2M1P,
1.02 for 1M2PA, 1.01 for 2M1PA

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

where 24.46 is the molar volume at 25°C and 101.3 kPa (760 mmHg) and molecular weights =

90.12 for 1M2P and 2M1P,
132.16 for 1M2PA and 2M1PA

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 when 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 detection limits of 48, 50, 71, and 71 pg per injection were determined by making injections (1.0 μL with a 15:1 split) of 726, 754, 1066, and 1066 $\text{pg}/\mu\text{L}$ standards for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. These amounts were judged to produce peaks with heights approximately 5 times the baseline noise.

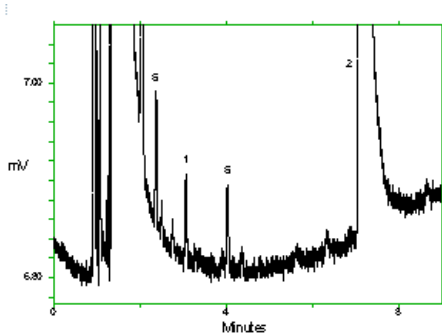


Figure 4.1.1. Detection limit chromatogram for 1M2P. Key: (1) 1M2P, (2) 2-heptanol, (S) contaminants in desorbing solution.

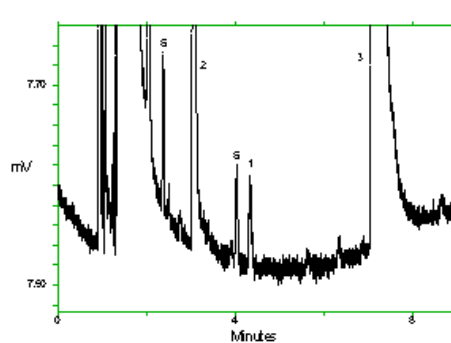


Figure 4.1.2. Detection limit chromatogram for 2M1P. Key: (1) 2M1P, (2) 2-heptanol, (S) contaminants in desorbing solution.

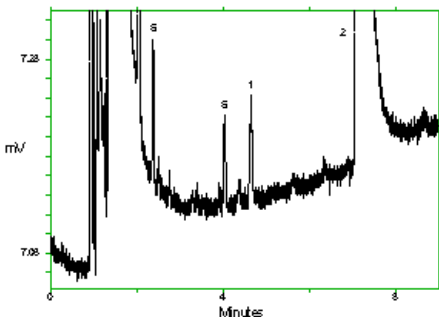


Figure 4.1.3. Detection limit chromatogram for 1M2PA. Key: (1) 1M2PA, (2) 2-heptanol, (S) contaminants in desorbing solution.

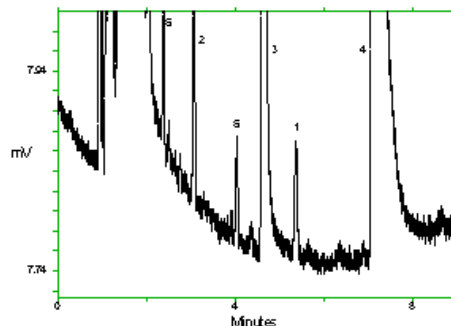


Figure 4.1.4. Detection limit chromatogram for 1M2PA. Key: (1) 1M2PA, (2) 2-heptanol, (S) contaminants in desorbing solution.

4.2. Detection limit of the overall procedure

The detection limits of the overall procedure of 0.73, 0.75, 1.1, and 1.1 µg per sample were determined by analyzing six samples for each analyte that had been spiked with 0.726, 0.754, 1.066, and 1.066 µg of 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. The detection limits of the overall procedure correspond to air concentrations of 20 ppb (74 µg/m³), 20 ppb (74 µg/m³), 20 ppb (108 µg/m³), and 20 ppb (108 µg/m³) for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively.

Table 4.2.1.
Detection Limit of the Overall Procedure for 1M2P

sample no.	µg spiked	µg recovered
1	0.726	0.690
2	0.726	0.686
3	0.726	0.710
4	0.726	0.702
5	0.726	0.724
6	0.726	0.702

Table 4.2.2.
Detection Limit of the Overall Procedure for 2M1P

sample no.	µg spiked	µg recovered
1	0.754	0.713
2	0.754	0.756
3	0.754	0.742
4	0.754	0.753
5	0.754	0.720
6	0.754	0.760

Table 4.2.3.
Detection Limit of the Overall Procedure for 1M2PA

sample no.	µg spiked	µg recovered
1	1.066	1.048
2	1.066	1.093
3	1.066	1.086
4	1.066	1.102
5	1.066	1.017
6	1.066	1.144

Table 4.2.4.
Detection Limit of the Overall Procedure for 2M1PA

sample no.	µg spiked	µg recovered
1	1.066	1.079
2	1.066	1.140
3	1.066	1.098
4	1.066	1.075
5	1.066	1.099
6	1.066	1.069

4.3. Reliable quantitation limit

The reliable quantitation limits of 0.73, 0.75, 1.1, and 1.1 g per sample were determined by analyzing six samples for each analyte that had been spiked with 0.726, 0.754, 1.066, and 1.066 µg of 1M2P, 2M1P, 1M2PA, and 2M1PA respectively. The reliable quantitation limits correspond to air concentrations of 20 ppb (74 µg/m³), 20 ppb (74 µg/m³), 20 ppb (108 µg/m³), and 20 ppb (108 µg/m³) for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively.

Table 4.3.1.
Reliable Quantitation Limit for 1M2P
(Based on samples and data of Table 4.2.1.)

percent recovered	statistics
95.0	
94.5	mean = 96.7
97.8	SD = 1.9
96.7	Precision = (1.96)(±1.9)
99.7	= ±3.7
96.7	

Table 4.3.2.
Reliable Quantitation Limit for 2M1P
(Based on samples and data of Table 4.2.2.)

percent recovered	statistics
94.6	
100.3	mean = 98.2
98.4	SD = 2.6
99.9	Precision = (1.96)(±2.6)
95.5	= ±5.1
100.8	

Table 4.3.3.

Table 4.3.4.

Reliable Quantitation Limit for 1M2PA
(Based on samples and data of Table 4.2.3.)

percent recovered	statistics
98.3	
102.5	mean = 101.5
101.9	SD = 4.1
103.4	Precision = (1.96)(±4.1)
95.4	= ±8.0
107.3	

Reliable Quantitation Limit for 2M1PA
(Based on samples and data of Table 4.2.4.)

percent recovered	statistics
101.2	
106.9	mean = 102.6
103.0	SD = 2.4
100.8	Precision = (1.96)(±2.4)
103.1	= ±4.7
100.3	

4.4. Instrument response to the analyte

The instrument response to the analytes over the range of 0.5 to 2 times the target concentrations was determined from multiple injections of analytical standards. The response is linear for all four analytes with slopes (in ISTD-corrected area counts per microgram of analyte per sample) of 275, 290, 291, and 290 for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively.

Table 4.4.1.
Instrument Response to 1M2P

× target concn	0.5×	1×	2×
µg/sample	1815	3630	7260
ppm	49.3	98.5	197
area counts	503710	1005400	2005600
	501140	1004600	2001400
	504620	1006400	1991800
	502310	1003300	2002700
	504370	1004000	1992000
	503980	1001500	1997800
mean	503360	1004200	1998600

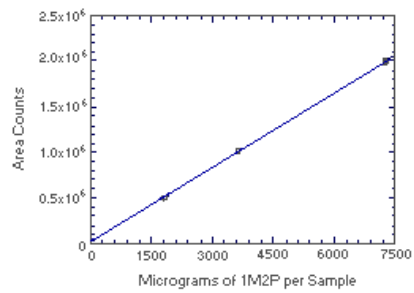


Figure 4.4.1. Instrument response to 1M2P. (Slope = 275)

Table 4.4.2.
Instrument Response to 2M1P

× target concn	0.5×	1×	2×
µg/sample	18.89	37.78	75.56
ppm	0.51	1.03	2.05
area counts	5534	11000	22048
	5490	11055	21910
	5532	11018	21957
	5534	10987	22031
	5544	10980	21782
	5539	10881	21909
mean	5529	10987	21940

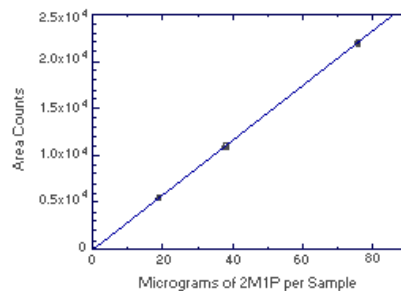


Figure 4.4.2. Instrument response to 2M1P. (Slope = 290)

Table 4.4.3.
Instrument Response to 1M2PA

× target concn	0.5×	1×	2×
µg/sample			
ppm			
area counts			
mean			

× target concn	0.5×	1×	2×
μg/sample	2664	5328	10657
ppm	49.3	98.6	197
<hr/>			
area counts	751990	1544600	3086000
	757280	1541100	3073400
	755840	1547500	3089900
	753060	1535200	3076200
	753250	1540700	3083300
	755340	1538500	3084000
mean	503360	1541300	3082100

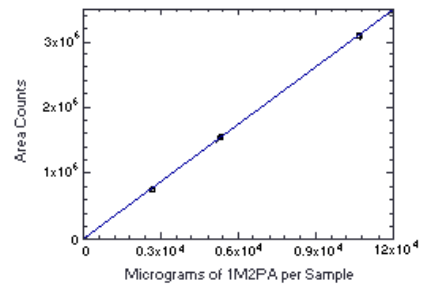


Figure 4.4.3. Instrument response to 1M2PA. (Slope = 291)

Table 4.4.4.
Instrument Response to 2M1PA

× target concn	0.5×	1×	2×
μg/sample	26.64	53.28	106.6
ppm	0.49	0.99	1.97
<hr/>			
area counts	7615	15492	30869
	7575	15425	30848
	7639	15513	31009
	7627	15386	30759
	7678	15470	30804
	7646	15314	30871
mean	7630	15433	30860

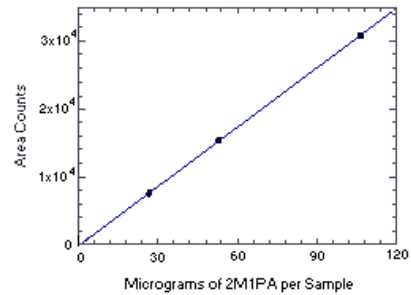


Figure 4.4.4. Instrument response to 2M1PA. (Slope = 290)

4.5. Storage test

Storage samples were generated by sampling from atmospheres containing the analytes at the target concentrations. 1M2P and 2M1P were generated in the same atmosphere and 1M2PA and 2M1PA were generated together in another atmosphere. For each set of 36 samples, 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 20-25°C. Six samples from each set, three from refrigerated and three from ambient storage, were analyzed in three-day intervals over a period of fifteen days. The results are given in Tables 4.5.1.- 4.5.4. and shown graphically in Figures 4.5.1.1., 4.5.1.2., 4.5.2.1., 4.5.2.2., 4.5.3.1., 4.5.3.2., 4.5.4.1., and 4.5.4.2.

Table 4.5.1.
Storage Test Data for 1M2P

days of storage	% recovery (refrigerated)			% recovery (ambient)		
0	102.3	101.2	100.6	102.3	101.2	100.6
0	101.6	100.2	100.1	101.6	100.2	100.1
3	101.4	100.5	101.3	99.1	99.7	98.9
6	99.2	99.8	100.3	99.4	98.9	99.4
9	101.8	102.7	102.1	102.9	102.8	103.4
12	99.5	99.4	99.6	99.0	98.5	99.0
15	98.8	99.0	100.0	102.6	103.3	102.3

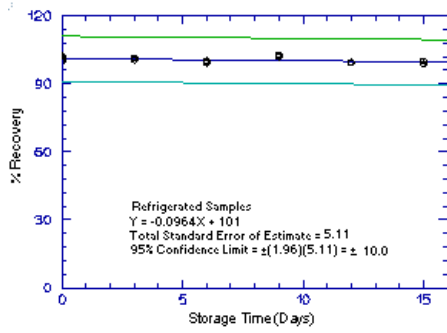


Figure 4.5.1.1. Refrigerated 1M2P storage samples.

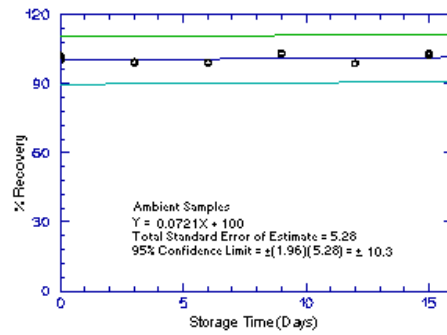


Figure 4.5.1.2. Ambient 1M2P storage samples.

Table 4.5.2.
Storage Test Data for 2M1P

days of storage	% recovery (refrigerated)			% recovery (ambient)		
0	105.1	98.8	96.7	105.1	98.8	96.7
0	100.9	103.0	100.4	100.9	103.0	100.4
3	101.8	95.9	98.0	103.2	100.1	96.8
6	96.4	98.8	98.4	95.9	98.2	97.2
9	95.0	95.7	95.7	94.8	93.8	94.6
12	98.5	96.6	97.3	97.4	97.5	97.2
15	94.5	92.3	96.9	95.8	94.3	96.0

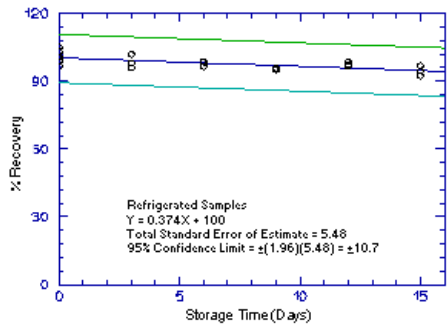


Figure 4.5.2.1. Refrigerated 2M1P storage samples.

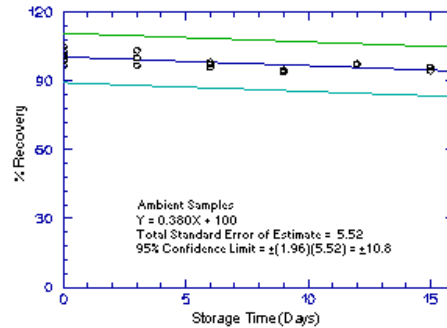


Figure 4.5.2.2. Ambient 2M1P storage samples.

Table 4.5.3.
Storage Test Data for 1M2PA

days of storage	% recovery (refrigerated)			% recovery (ambient)		
0	101.1	100.5	98.4	101.1	100.5	98.4
0	99.2	98.8	99.8	99.2	98.8	99.8
3	96.4	97.5	97.6	97.2	97.3	97.2
6	98.4	97.3	97.7	98.8	98.6	99.1
9	96.6	96.8	92.8	98.7	98.8	98.1
12	96.9	97.5	97.0	98.8	98.6	98.7
15	99.2	99.1	100.0	97.8	97.6	95.6

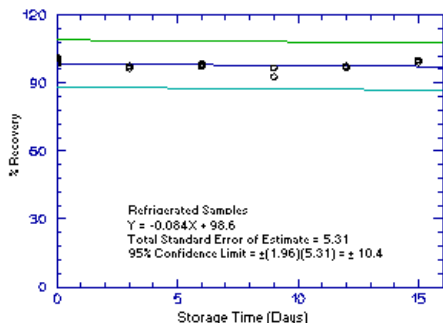


Figure 4.5.3.1. Refrigerated 1M2PA storage samples.

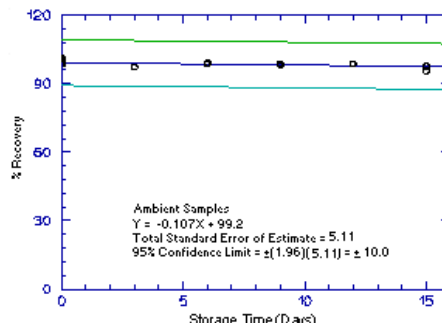


Figure 4.5.3.2. Ambient 1M2PA storage samples.

Table 4.5.4.
Storage Test Data for 2M1PA

days of storage	% recovery (refrigerated)			% recovery (ambient)		
0	101.3	100.8	98.4	101.3	100.8	98.4
0	99.0	101.3	100.0	99.0	101.3	100.0
3	95.8	96.0	96.9	93.8	92.9	94.0
6	96.1	96.8	96.6	94.6	93.8	95.5
9	95.0	94.5	91.2	93.8	94.2	93.3
12	95.3	95.5	93.8	92.5	93.4	93.3
15	95.0	95.7	96.6	91.2	89.7	87.0

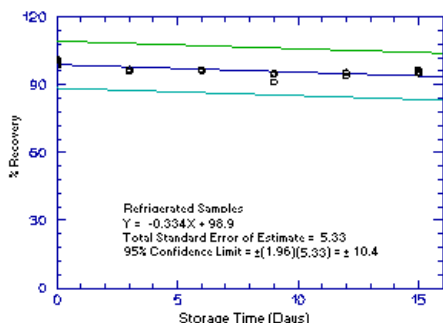


Figure 4.5.4.1. Refrigerated 2M1PA storage samples.

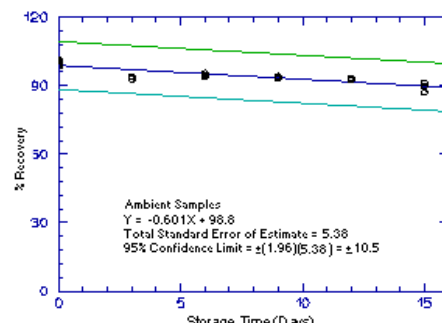


Figure 4.5.4.2. Ambient 2M1PA storage samples.

4.6. Precision (analytical procedure)

The precision of the analytical procedure for each analyte is the pooled coefficient of variation determined from replicate injections of standards. The coefficients of variation (CV) are calculated from the data from Tables 4.4.1.-4.4.4. The pooled coefficients of variation are 0.0025, 0.0045, 0.0025, and 0.0041 for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively.

Table 4.6.1.
Precision of the Analytical Method for 1M2P
(Based on the Data of Table 4.4.1.)

× target concn	0.5×	1×	2×
µg/sample	1815	3630	7260
ppm	49.3	98.5	197
mean	1352	1708	5728
CV	0.00269	0.00170	0.00287

Table 4.6.3.
Precision of the Analytical Method for 1M2PA

Table 4.6.2.
Precision of the Analytical Method for 2M1P
(Based on the Data of Table 4.4.2.)

× target concn	0.5×	1×	2×
µg/sample	18.89	37.78	75.56
ppm	0.51	1.03	2.05
mean	19.5	58.4	97.0
CV	0.00353	0.00532	0.00442

Table 4.6.4.
Precision of the Analytical Method for 2M1PA

(Based on the Data of Table 4.4.3.)

(Based on the Data of Table 4.4.4.)

× target concn	0.5×	1×	2×
µg/sample	2664	5328	10657
ppm	49.3	98.6	197

× target concn	0.5×	1×	2×
µg/sample	26.64	53.28	106.6
ppm	0.49	0.99	1.97

mean	2008	4354	6190
CV	0.00266	0.00282	0.00201

mean	34.4	74.4	84.7
CV	0.00451	0.00482	0.00274

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 no. 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 4.5.1.1. The data for Figures 4.5.1.2., 4.5.2.2., 4.5.3.2. and 4.5.4.2. were used to determine the SEEs of ±5.3%, ±5.5%, ±5.1%, and ±5.4% and the precisions of the overall procedure of ±10.3%, ±10.8%, ±10.0%, and ±10.5% for 1M2P, 2M1P, 1M2PA, and 2M1PA respectively.

4.8. Reproducibility

Six samples for each analyte collected from controlled test atmospheres (at about 80% R.H., 20-26°C, 86-88 kPa) were analyzed by a chemist unassociated with this evaluation. The samples were stored in a refrigerator for nine days before being analyzed.

Table 4.8.1.
Reproducibility Data for 1M2P

µg expected	µg found	% found	deviation
2861	2807	98.1	-1.9
2810	2750	97.9	-2.1
2773	2764	99.7	-0.3
2766	2750	99.4	-0.6
2848	2866	100.6	+0.6
3030	3050	100.7	+0.7

Table 4.8.2.
Reproducibility Data for 2M1P

µg expected	µg found	% found	deviation
29.10	28.18	96.8	-3.2
28.58	27.31	95.6	-4.4
28.20	27.70	98.2	-1.8
28.13	27.66	98.3	-1.7
28.97	28.83	99.5	-0.5
30.82	30.52	99.0	-1.0

Table 4.8.3.
Reproducibility Data for 1M2PA

µg expected	µg found	% found	deviation
5824	5583	95.9	-4.1
5721	5533	96.7	-3.3
5645	5517	97.7	-2.3
5630	5487	97.5	-2.5
5798	5711	98.5	-1.5
6168	6044	98.0	-2.0

Table 4.8.4.
Reproducibility Data for 2M1PA

µg expected	µg found	% found	deviation
58.24	54.81	94.1	-5.9
57.21	54.45	95.2	-4.8
56.45	54.20	96.0	-4.0
56.30	53.89	95.7	-4.3
57.98	55.98	96.6	-3.4
61.68	59.28	96.1	-3.9

4.9. Desorption efficiency [from dry charcoal using 95/5 (v/v) methylene chloride/methanol]

The average desorption efficiencies of 1M2P, 2M1P, 1M2PA, and 2M1PA are 100.4%, 99.7%, 101.8%, and 101.4% respectively over the range of 0.5 to 2 times the target concentrations. They were determined by injecting microliter amounts of stock standards into the front section of Lot 120 charcoal tubes. Eighteen samples were prepared, six samples for each concentration level listed in the following tables. The samples were stored in a refrigerator and analyzed the next day.

Table 4.9.1.
Desorption Efficiency Data for 1M2P and 2M1P

	desorption efficiency, %					
	1M2P			2M1P		
	0.5×	1×	2×	0.5×	1×	2×
× target concn	1815	3630	7260	18.89	37.78	75.56
µg/sample	49.3	98.5	197	0.51	1.03	2.05
ppm						
	100.8	99.6	99.9	99.9	99.7	99.4
	100.3	101.5	100.5	98.1	101.2	100.2
	99.5	100.0	100.1	97.5	101.8	97.7
	100.2	100.7	100.5	97.3	103.1	100.2
	101.5	100.4	99.7	99.5	97.4	98.7
	100.7	100.1	100.3	101.4	100.6	100.7
mean	100.5	100.4	100.2	99.0	100.6	99.5
grand mean		100.4			99.7	

Table 4.9.2.
Desorption Efficiency Data for 1M2PA and 2M1PA

	desorption efficiency, %					
	1M2PA			2M1PA		
	0.5×	1×	2×	0.5×	1×	2×
× target concn	2664	5328	10657	26.64	53.28	106.6
µg/sample	49.3	98.6	197	0.49	0.99	1.97
ppm						
	102.2	102.3	101.9	102.5	100.8	101.6
	101.6	102.1	101.5	102.2	101.9	100.9
	101.6	101.8	101.7	101.2	101.4	101.9
	101.9	101.9	102.2	100.8	100.0	102.6
	101.3	102.1	101.8	101.2	101.3	101.2
	101.6	101.8	101.6	101.1	101.8	101.6
mean	101.7	102.0	101.8	101.5	101.2	101.6
grand mean		101.8			101.4	

4.10. Stability of desorbed samples [from dry charcoal using 95/5 (v/v) methylene chloride/methanol]

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 after 24 Hours

desorption efficiency, %				
	1M2P	2M1P	1M2PA	2M1PA
	100.1	99.8	102.9	102.9
	101.1	100.6	102.2	101.9
	99.9	101.8	101.9	102.1
	101.2	100.2	102.2	101.1
	100.4	99.5	102.4	102.9
	101.0	102.4	102.0	100.0
mean	100.6	100.7	102.3	101.8

4.11. Desorption efficiency and stability of desorbed samples [from wet charcoal using 95/5 (v/v) methylene chloride/methanol]

Studies were done at the target concentrations to determine what effect the presence of water had on the desorption efficiency for the four analytes. This was done by injecting analytical standards into the front sections of charcoal tubes that previously had 10 L of 80% RH air drawn through them. The samples were reanalyzed 24 h later to check the stability of desorbed samples. Finally, magnesium sulfate was added to the desorbed samples and they were reanalyzed again.

Table 4.11.1.
Desorption of 1M2P from Wet Charcoal

	initial analysis	next day analysis	next day + MgSO4
% desorption	96.0	96.1	103.0
	96.4	96.9	101.9
	96.1	96.6	101.3
	96.7	97.4	102.3
	96.2	96.9	102.4
	96.2	97.0	101.9
mean	96.3	96.8	102.1

Table 4.11.2.
Desorption of 2M1P from Wet Charcoal

	initial analysis	next day analysis	next day + MgSO4
% desorption	92.3	95.3	102.6
	94.8	96.8	103.1
	94.6	94.7	103.2
	93.4	96.2	102.7
	93.3	94.8	102.8
	93.0	94.8	101.5
mean	93.6	95.4	102.6

Table 4.11.3.
Desorption of 1M2PA from Wet Charcoal

	initial analysis	next day analysis	next day + MgSO4
% desorption	102.7	102.5	102.2
	103.0	102.7	102.3
	103.0	102.6	102.7
	103.0	102.7	102.6
	103.5	103.5	102.5
	102.3	102.5	101.9

Table 4.11.4.
Desorption of 2M1PA from Wet Charcoal

	initial analysis	next day analysis	next day + MgSO4
% desorption	102.4	103.2	101.8
	103.2	102.5	103.4
	102.5	103.2	104.0
	101.9	103.8	103.0
	102.7	102.5	103.1
	100.7	103.6	101.4

4.12. Desorption efficiency [from dry charcoal using 99/1 (v/v) CS₂/DMF]

The average desorption efficiencies of 1M2P, 2M1P, 1M2PA, and 2M1PA are 86.3%, 80.8%, 98.4%, and 98.0% respectively over the range of 0.5 to 2 times the target concentrations. They were determined by injecting microliter amounts of stock standards into the front section of Lot 120 charcoal tubes. Eighteen samples were prepared, six samples for each concentration level listed in the following tables. The samples were stored in a refrigerator and analyzed the next day.

Table 4.12.1.
Desorption Efficiency Data for 1M2P and 2M1P

		desorption efficiency, %					
		1M2P			2M1P		
× target concn		0.5×	1×	2×	0.5×	1×	2×
μg/sample		1815	3630	7260	18.89	37.78	75.56
ppm		49.3	98.5	197	0.51	1.03	2.05
		84.1	87.6	89.2	81.0	80.0	83.6
		84.7	87.9	88.7	78.7	80.6	82.7
		84.8	88.8	83.4	79.1	84.1	80.5
		83.3	89.0	89.1	76.5	83.8	85.3
		84.1	84.4	88.6	76.5	83.7	81.9
		83.5	86.8	85.4	75.2	81.0	79.7
mean		84.1	87.4	87.4	77.8	82.2	82.3
grand mean		86.3			80.8		

Table 4.12.2.
Desorption Efficiency Data for 1M2PA and 2M1PA

		desorption efficiency, %					
		1M2P			2M1P		
× target concn		0.5×	1×	2×	0.5×	1×	2×
μg/sample		2664	5328	10657	26.64	53.28	106.6
ppm		49.3	98.6	197	0.49	0.99	1.97
		99.1	97.9	98.9	97.9	97.8	97.9
		98.8	98.6	98.6	98.2	98.9	97.9
		98.7	98.2	98.5	97.6	98.1	98.0
		98.8	97.5	98.6	98.7	97.5	97.7
		98.4	97.7	98.3	98.7	97.6	97.7
		98.3	98.0	98.9	98.0	98.0	98.4
mean		98.7	98.0	98.6	98.2	98.0	97.9
grand mean		98.4			98.0		

4.13. Stability of desorbed samples [from dry charcoal using 99/1 (v/v) CS₂/DMF]

The stability of desorbed samples was checked by reanalyzing the target concentration samples from Section 4.12. 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 4.13.
Stability of Desorbed Samples at the Target Concentration after 24 Hours

	desorption efficiency, %			
	1M2P	2M1P	1M2PA	2M1PA
	80.9	72.3	98.2	97.8
	81.4	72.4	98.1	97.9
	85.2	78.9	97.1	96.4
	84.9	77.4	97.6	97.5
	86.8	78.6	97.6	96.9
	82.7	75.1	97.6	97.2
mean	83.7	75.8	97.7	97.3

4.14. Desorption efficiency and stability of desorbed samples [from wet charcoal using 99/1 (v/v) CS₂/DMF]

Studies were done at the target concentrations to determine what effect the presence of water had on the desorption efficiency for the four analytes. This was done by injecting analytical standards into the front sections of charcoal tubes that previously had 10 L of 80% RH air drawn through them. The samples were reanalyzed 24 h later to check the stability of desorbed samples. Magnesium sulfate was added to one-half of the samples when they were desorbed.

Table 4.14.1.
Desorption of 1M2P from Wet Charcoal

	initial (no MgSO ₄)	next day (no MgSO ₄)	initial (with MgSO ₄)	next day (with MgSO ₄)
% desorption	50.9	48.0	79.5	78.9
	50.9	48.1	83.0	75.3
	50.8	49.7	76.2	79.7
mean	50.9	48.6	79.6	78.0

Table 4.14.2.
Desorption of 2M1P from Wet Charcoal

	initial (no MgSO ₄)	next day (no MgSO ₄)	initial (with MgSO ₄)	next day (with MgSO ₄)
% desorption	40.5	36.9	70.1	65.8
	40.4	37.7	74.7	59.1
	40.7	39.0	66.6	68.1
mean	40.5	37.9	70.5	64.3

Table 4.14.3.
Desorption of 1M2PA from Wet Charcoal

	initial (no MgSO4)	next day (no MgSO4)	initial (with MgSO4)	next day (with MgSO4)
% desorption	93.2	94.6	95.8	96.5
	94.5	94.3	96.6	95.8
	93.0	93.8	96.7	95.2
mean	93.6	94.2	96.4	95.8

Table 4.14.4.
Desorption of 2M1PA from Wet Charcoal

	initial (no MgSO4)	next day (no MgSO4)	initial (with MgSO4)	next day (with MgSO4)
% desorption	91.7	92.8	95.4	95.7
	93.0	92.5	96.2	94.9
	91.3	91.3	96.6	94.7
mean	92.0	92.2	96.1	95.1

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