# IX.  APPENDIX I


## SAMPLING AND ANALYTICAL METHOD FOR BGE, IGE, AND PGE


The following generalized sampling and analytical method for these glycidyl ethers is adapted from the NIOSH validated methods for these compounds [75,76,77]. If certain parameters are changed, such as solvents and gas-chromatograph operating conditions, it may also be suitable for other glycidyl ethers.


## Principle of the Method

A known volume of air is drawn through a charcoal tube to collect the organic vapors. The charcoal is then transferred to a small, stoppered sample container and desorbed with carbon disulfide. An aliquot of the desorbed sample is injected into a gas chromatograph. The area of the resulting peak is determined and compared with areas obtained from the injection of standards.


## Range and Sensitivity

This method was validated for each glycidyl ether at the limits presented in Table IX-1, but it is capable of measuring much smaller amounts if the desorption efficiency is adequate. Desorption efficiency must be determined over the range used.

TABLE IX-1

RANGE, PRECISION, AND ACCURACY OF THE GAS CHROMATOGRAPH
ANALYSIS OF GLYCIDYL ETHERS

| Glycidyl Ether | Temperature (C) and Pressure (mmHg) | Validated Range (mg/cu m) | Probable Range (mg/cu m) | Coefficient of Variation | Standard Deviation* (mg/cu m) | Reference |
|---|---|---|---|---|---|---|
| BGE | 22 at 767 | 133-542 | 30-810 | 0.074 | 20 | 75 |
| IGE | 21 at 763 | 121-484 | 25-720 | 0.067 | 16 | 76 |
| PGE | 22 at 766 | 31-121 | 6-180 | 0.057 | 3.4 | 77 |

*At current OSHA limit

The upper limit of the range of the method is dependent on the adsorptive capacity of the charcoal tube. This capacity varies with the concentration of a particular glycidyl ether and of other substances in the air. Experimental data on breakthrough are listed in Table IX-2.

TABLE IX-2

BREAKTHROUGH DATA FOR CHARCOAL-TUBE SAMPLING OF GLYCIDYL ETHERS

| Glycidyl Ether | Amount in first section (mg) | Influent Test Atmosphere (mg/cu m) | Sampling Rate (liters/ min) | Break- through Time (min) | Ref- erence |
|---|---|---|---|---|---|
| BGE | 23 | 530 | 0.183 | 240* | 75 |
| IGE | 21 | 480 | 0.183 | 240* | 76 |
| PGE | 25 | 112 | 0.93 | 240* | 77 |

*No breakthrough in time given

## Interferences

When the amount of water in the air is so great that condensation actually occurs in the charcoal tube, organic vapors will not be trapped efficiently. Preliminary experiments using toluene indicate that high humidity severely decreases breakthrough volume.

When two or more compounds are known or suspected to be present in the air, such information, including the suspected identities of the compounds, should be transmitted with the sample. It must be emphasized that any compound that has the same retention time as the glycidyl ether at the operating conditions described in this method is an interference. Retention-time data on a single column cannot be considered proof of chemical identity. If the possibility of interference exists, separation conditions (column packing, temperature, etc) must be changed to circumvent the problem.

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## Precision and Accuracy

The Coefficients of Variation (CVT) for the analytical and sampling method are listed in Table IX-1. The standard deviation at the OSHA standard level is also included in the table. It should be noted, however, that CVT's and standard deviations at environmental limits recommended in this document are not currently available.

## Advantages and Disadvantages of the Method

The sampling method uses a small, portable sampling device that involves no liquids. Interferences are minimal, and most of those which do occur can be eliminated by altering chromatographic conditions. The tubes are analyzed by means of a quick instrumental method. The method can also be used for the simultaneous analysis of two or more substances suspected to be present in the same sample simply by changing gas-chromatographic conditions from isothermal to a temperature-programmed mode of operation.

One disadvantage of the sampling method is that the amount of sample that can be taken is limited by the number of milligrams that the tube will hold before overloading. When the sample value obtained from the backup section of the charcoal trap exceeds 25% of that found on the front section, the possibility of sample loss exists. The precision of the method is affected by the reproducibility of the pressure drop across the tubes. This drop will affect the flowrate and cause the volume to be imprecise, because the pump is usually calibrated for one tube only.

Apparatus

(a)     An  approved  and  calibrated personal sampling pump whose flow can be determined within ±5% at the recommended flowrate.

(b)     Charcoal  tubes:  glass tube with both ends flame sealed, 7-cm long with a 6-mm outer diameter and a 4-mm inner diameter,  containing  two sections  of  20/40  mesh activated charcoal separated by a 2-mm portion of urethane foam.  The activated charcoal is prepared from coconut shells  and is  fired at 600 C prior to packing.  The adsorbing section contains 100 mg of charcoal, the backup section 50 mg.  A 3-mm portion of urethane foam  is placed  between  the outlet end of the tube and the backup section.  A plug of silylated glass wool is placed in front of the adsorbing  section.   The pressure  drop  across  the  tube  must be less than 1 inch of mercury at a flowrate of 1 liter/minute.

(c)     Gas chromatograph equipped with a flame ionization detector.

(d)     Column  (10-foot  x  1/8-inch stainless steel) packed with 10% FFAP on 80/100 mesh, acid-washed DMCS Chromosorb W.

(e)     An  electronic  integrator  or  some other suitable method for measuring.

(f)     Microliter  syringes:   $10-\mu l$,  and other convenient sizes for making standards.

(g)     Pipets:   0.5-ml  delivery  pipets or 1.0-ml type graduated in 0.1-ml increments.

(h)     Volumetric  flasks:   10-ml  or  convenient  sizes  for making standard solutions.

(i)     Sample  containers:  1-ml, with glass stoppers or Teflon-lined caps.

## Reagents

(a)    BGE, IGE, or PGE, reagent grade.

(b)    Carbon disulfide, chromatographic quality.

(c)    Nitrogen, purified.

(d)    Hydrogen, prepurified.

(e)    Filtered compressed air.


## Sampling Procedure

(a)    Calibration of Personal Pumps.    Each personal pump must be calibrated with a representative charcoal tube in the line, as shown in Figure XIV-1.    This will minimize errors associated with uncertainties in the sample volume collected.

(b)    Collection and Shipping of Samples.

(1)    Immediately before sampling, break the ends of the tube to provide an opening at least one-half the internal diameter of the tube (2 mm).

(2)    The smaller section of charcoal is used as a backup and should be positioned nearest the sampling pump.

(3)    The charcoal tube should be placed in a vertical position during sampling to minimize channeling through the charcoal.

(4)    Air being sampled should not be passed through any hose or tubing before entering the charcoal tube.

(5)    The sample size and sampling rate for BGE, PGE, and IGE should be 15 liters sampled at 1 liter/minute.    The sampling rates and sample sizes have been changed from those reported for BGE [75] and PGE [77] and the sample size for IGE [76].    This was done to adapt the methods

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to sample for ceiling rather than for TWA concentrations. These changes should not affect the collection efficiency of the method and should provide an adequate amount of sample for analysis, but they have yet to be tested.

(6)    The temperature and pressure of the atmosphere being sampled should be recorded. If pressure reading is not available, record the elevation.

(7)    The charcoal tubes should be capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

(8)    One tube should be handled in the same manner as the sample tube (break, seal, and transport), except that no air is drawn through this tube. This tube should be labeled as a blank.

(9)    Capped tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

(10)    A sample of the bulk material should be submitted to the laboratory in a glass container with a Teflon-lined cap. This sample should not be transported in the same container as the charcoal tubes.

## Analysis of Samples

All glassware used for the laboratory analysis should be washed with detergent and thoroughly rinsed with tap water and distilled water.

(a)    Preparation of Samples. In preparation for analysis, remove the plastic cap used to close the tube after sample collection and remove and discard the glass wool. The charcoal in the first (larger) section is transferred to a 1-ml stoppered sample container. The separating sections

of foam are removed and discarded; the second section is transferred to another container. These two sections are then analyzed separately.

(b) Desorption of Samples. Prior to analysis of BGE, IGE, or PGE, 0.5 ml of carbon disulfide is pipetted into each sample container. (All work with carbon disulfide should be performed in a hood because of its high toxicity.) Desorption should be done for 30 minutes. Tests indicate that this is adequate if the sample is stirred occasionally during this period.

(c) Gas-chromatographic Conditions. The typical operating conditions for the gas chromatograph are listed in Table IX-3.

TABLE IX-3

TYPICAL GAS CHROMATOGRAPH CONDITIONS FOR GLYCIDYL ETHERS

| Glycidyl Ether | Column Packing | Gas Flow (ml/min) | | | Temperature (C) | | | Ref- erence |
|---|---|---|---|---|---|---|---|---|
| | | Carrier Nitrogen (at 60 psig) | Hydrogen* (at 24 psig) | Air* (at 50 psig) | In- jec- tor | Mani- fold | Col- umn | |
| BGE | 10% FFAP on 80/100 mesh, acid-washed DMCS Chromosorb W | 50 | 65 | 500 | 180 | 275 | 130 | 75 |
| IGE | " | " | " | " | 205 | 270 | 115 | 76 |
| PGE | " | " | " | " | 230 | 265 | 90 | 77 |

*Flow to detector

(d) Injection. The first step in the analysis is the injection of the sample into the gas chromatograph. To eliminate difficulties arising from blowback or distillation within the syringe needle, one should employ the solvent-flush injection technique. The 10-$\mu$l syringe is first flushed with solvent several times to wet the barrel and plunger. Draw 3 $\mu$l of solvent into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the solvent, and the plunger is pulled back about 0.2 $\mu$l to separate the solvent flush from the sample with a pocket of air to be used as a marker. The needle is then immersed in the sample, and a 5-$\mu$l aliquot is withdrawn, taking into consideration the volume of the needle, since the sample in the needle will be completely injected. After the needle is removed from the sample and prior to injection, the plunger is pulled back 1.2 $\mu$l to minimize evaporation of the sample from the tip of the needle. Observe that the sample occupies 4.9-5.0 $\mu$l in the barrel of the syringe. Duplicate injections of each sample and standard should be made. No more than a 3% difference in area is to be expected.

(e) Area Measurement. The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement, and preliminary results are read from a standard curve prepared as discussed below.


Determination of Desorption Efficiency

(a) Importance of Determination. The desorption efficiency of a particular compound can vary from one laboratory to another and also from one batch of charcoal to another. Thus, it is necessary to determine at

least once the percentage of the specific compound that is removed in the desorption process, provided the same batch of charcoal is used.

(b)      Procedure for Determination.  Activated charcoal equivalent to the amount in the first section of the sampling tube (100 mg) is measured into a 2.5-inch, 4-mm inner diameter glass tube, flame-sealed at one end. This charcoal must be from the same batch as that used in obtaining the samples and can be obtained from unused charcoal tubes.  The open end is capped with Parafilm.  A known amount of the glycidyl ether is injected directly into the activated charcoal with a microliter syringe, and the tube is capped with more Parafilm.  The amount injected is equivalent to that present in a 15-liter liter air sample for BGE, IGE, and PGE, respectively, at the selected level.  Six tubes at each of three levels (0.5, 1, and 2 times the recommended standard) are prepared in this manner and allowed to stand at least overnight to assure complete adsorption of the glycidyl ether onto the charcoal.  These tubes are referred to as the samples.  A parallel blank tube is also prepared.  The sample and blank tubes are desorbed and analyzed in exactly the same manner as the sampling tube described in Analysis of Samples.  Two or three standards are prepared by injecting the same volume of compound into 0.5 ml of carbon disulfide with the same syringe used in the preparation of the samples.  (All work with carbon disulfide should be performed in a hood because of its high toxicity.)  These are analyzed with the samples.  The desorption efficiency (DE) equals the average weight in mg recovered from the tube divided by the weight in mg added to the tube, or:

$$\text{DE} = \frac{\text{Average weight (mg) recovered}}{\text{Weight (mg) added}}$$

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The desorption efficiency is dependent on the amount of glycidyl ether collected on the charcoal. The desorption efficiency is plotted against the weight of glycidyl ether found.

## Calibration and Standards

It is convenient to express concentrations of standards in terms of mg/0.5 ml of carbon disulfide, because samples are desorbed in this amount of carbon disulfide. The density of the glycidyl ether is used to convert mg into $\mu$l for easy measurement with a microliter syringe. A series of standards, varying in concentration over the range of interest, is prepared and analyzed under the same gas-chromatographic conditions and during the same time period as the unknown samples. Curves are established by plotting concentration in mg/0.5 ml vs peak area. Note: Since no internal standard is used in the method, standard solutions must be analyzed at the same time that the analysis of samples is done. This will minimize the effect of known day-to-day variations and variations during the same day in the gas-chromatographic detector response.

## Calculations

The weight in mg corresponding to each peak area is read from the standard curve. No volume corrections are needed, because the standard curve is based on mg/0.5 ml of carbon disulfide and the volume of sample injected is identical with the volume of the standards injected. Corrections for the blank must be made for each sample:

$$mg = mg \text{ sample} - mg \text{ blank}$$

where:

mg sample = mg found in front section of sample tube

mg blank  = mg found in front section of blank tube

A similar procedure is followed for the backup sections.

Add the weights found in the front and backup sections to get the total weight in the sample.

Read the desorption efficiency from the curve for the amount found in the front section. Divide the total weight by this desorption efficiency to obtain the corrected mg/sample.

$$\text{Corrected mg/sample} = \frac{\text{Total weight}}{\text{DE}}$$

Determine the volume (in liters) of air sampled at ambient conditions based on the appropriate information, such as flowrate in liters/minute multiplied by sampling time. If a pump using a rotameter for flowrate control was used for sample collection, a pressure and temperature correction must be made for the indicated flowrate when the pump was calibrated under substantially different conditions than those that exist during sampling. The expression for the correction is:

$$\text{Corrected volume} = f \times t \left( \sqrt{\frac{P1}{P2} \times \frac{T2}{T1}} \right)$$

where:
  f = flowrate during sampling
  t = sampling time
  P1 = pressure during calibration of samplimg (mmHg)
  P2 = pressure of air sampled (mmHg)
  T1 = temperature during calibration of sampling pump (K)
  T2 = temperature of air sampled (K)

The concentration of the glycidyl ether in the air sampled can be expressed in mg/cu m.

$$\text{mg/cu m} = \frac{\text{Corrected mg x 1,000 (liter/cu m)}}{\text{Air volume sampled (liters)}}$$

Another method of expressing concentration is ppm:

$$\text{ppm} = \text{mg/cu m x } \frac{24.45}{\text{MW}} \text{ x } \frac{760}{\text{P}} \text{ x } \frac{\text{T + 273}}{298}$$

where:

   P    = pressure (mmHg) of air sampled
   T    = temperature (C) of air sampled
24.45   = molar volume (liters/mole) at 25 C and 760 mmHg
  MW    = molecular weight (g/mole) of the glycidyl ether
 760    = standard pressure (mmHg)
 298    = standard temperature (K)

## X.  APPENDIX II


### SAMPLING AND ANALYTICAL METHOD FOR AGE


The  following method for AGE is adapted from the draft report of the NIOSH validated method [78].  If certain parameters are  changed,  such  as the  solvent  and  the  gas-chromatographic operating conditions, it may be suitable for other glycidyl ethers.


### Principle of the Method

A  known volume of air is drawn through a Tenax-GC resin tube to trap the organic  vapors  present.   The  sampling  tube  consists  of  a  front adsorbing  section  and  a  backup  section.   The  resin  in  each tube is transferred to a vial and the  AGE  is  desorbed  with  diethyl  ether  and analyzed by gas chromatography.


### Range and Sensitivity

This  method  was  validated  over  the  range of 19-87 mg/cu m at an atmospheric temperature of 17 C and atmospheric pressure of 752 mmHg  using a  3-liter sample volume.  This sample volume is based on two-thirds of the 5% breakthrough capacity determined at 90% relative humidity when  sampling a  test  atmosphere at 2 times the OSHA standard (45 mg/cu m).  This method is capable of measuring much smaller amounts if the  desorption  efficiency is adequate.  Desorption efficiency must be determined over the range used.

The upper limit of the range of the method is dependent on the adsorptive capacity of the Tenax-GC resin tube. This capacity can vary with the concentrations of AGE and other substances in the air.

## Interferences

When two or more compounds are known or suspected to be present in the air, such information, including the suspected identities of the compounds, should be transmitted with the sample. It must be emphasized that any compound that has the same retention time as AGE at the operating conditions described in this method is an interference. Retention-time data on a single column cannot be considered as proof of chemical identity. If the possibility of interference exists, separation conditions (column packing, temperature, etc) must be changed to circumvent the problem.

## Precision and Accuracy

The Coefficient of Variation (CVT) for the total analytical and sampling method in the range of 19-87 mg/cu m was 0.058. This value corresponds to a 2.6 mg/cu m standard deviation at the OSHA standard level (45 mg/cu m).

On the average, the concentrations obtained at the OSHA standard level (22 mg/cu m) using the overall sampling and analytical method were 0.5% lower than the "true" concentrations in a limited number of laboratory experiments. Any difference between the "found" and "true" concentrations may not represent a bias in the sampling and analytical method but rather a random variation from the experimentally determined "true" concentration. Therefore, no recovery correction should be applied to the final result.

The data are based on validation experiments using the internal standard method.


## Advantages and Disadvantages

The sampling device is small, portable, and involves no liquids. Interferences are minimal, and most of those which do occur can be eliminated by altering chromatographic conditions. The tubes are analyzed by means of a quick, instrumental method.

One disadvantage of the method is that the amount of sample that can be taken is limited by the number of mg that the tube will hold before overloading. When an atmosphere at 90% relative humidity containing 92 mg/cu m of AGE was sampled at 0.8 liter/minute, 5% breakthrough was observed after 15 minutes (capacity = 12 liters or 1.1 mg). The sample size recommended is less than the 5% breakthrough capacity at 90% relative humidity for a test atmosphere at 2 times the OSHA standard (90 mg/cu m) to minimize the probability of overloading the sampling tube.

The precision of the method is affected by the reproducibility of the pressure drop across the tubes. This drop will affect the flowrate and cause the volume to be imprecise, because the pump is usually calibrated for one tube only.


## Apparatus

(a)   A calibrated personal sampling pump whose flow can be determined within ±5% at the recommended flowrate.

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(b)    Resin    tubes.    Glass    tube with both ends flame-sealed, 10-cm long with 8-mm outer diameter and 6-mm inner diameter, containing two sections of 35/60 mesh Tenax-GC resin. The adsorbing section contains 100 mg of resin, the backup section 50 mg. A small wad of silylated glass wool is placed between the front adsorbing section and the backup section; a plug of silylated glass wool is also placed in front of the adsorbing section and at the end of the backup section. Since the pressure drop across the tube must be less than 25 mmHg at a flowrate of 1 liter/minute, it is necessary to avoid overpacking with glass wool.

(c)    Gas chromatograph equipped with a flame ionization detector.

(d)    Column (20-foot x 1/8-inch stainless steel) packed with 10% FFAP stationary phase on 100/120 mesh Supelcoport.

(e)    An electronic integrator or some other suitable means of measuring peak areas.

(f)    Sample containers with Teflon-lined caps, 5-ml.

(g)    Microliter syringes, 10-$\mu$l and 500-$\mu$l, and other convenient sizes for making standards and for taking sample aliquots for dilution.

(h)    Pipets, 2-ml, delivery type.

(i)    Volumetric flasks, 1-ml and 10-ml or convenient sizes for making standard solutions and dilution of samples.

Reagents

(a)    Diethyl ether, anhydrous.

(b)    AGE, 99%.

(c)    Isoamyl alcohol or other suitable internal standard. The appropriate solution of the internal standard is prepared in ether.

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(d)    Hexane. This is used to prepare solutions of AGE for preparing the analytical samples for desorption efficiency determination.

(e)    Nitrogen, purified.

(f)    Hydrogen, prepurified.

(g)    Air, filtered compressed.


## Sampling Procedure

(a)    Calibration of Personal Pumps.    Each personal pump must be calibrated with a representative Tenax-GC resin tube in line, as shown in Figure XIV-1.    This will minimize errors associated with uncertanties in the sample volume collected.

(b)    Collection and Shipping of Samples.

(1)    Immediately before sampling, break the two ends of the resin tube to provide an opening at least one-half the internal diameter of the tube (3 mm).

(2)    The section containing 50 mg of resin is used as a backup and should be positioned nearest the sampling pump.

(3)    The resin tube series should be placed in a vertical position during sampling to minimize channeling through the resin.

(4)    Air being sampled should not be passed through any hose or tubing before entering the resin tube.

(5)    A sample size of 3 liters is recommended. Sample at a flowrate of 0.2 liter/minute for 15 minutes. The flowrate should be known with an accuracy of at least ±5%.

(6)     The temperature and pressure of the atmosphere being sampled should be recorded.  If pressure reading is not available, record the elevation.

(7)     The resin tube should be labeled appropriately and capped with plastic caps.  Under no circumstances should rubber caps be used.

(8)     With each batch of 10 samples, one resin tube that has been handled in the same manner as the sample tubes (break, seal, and transport), except that no air is sampled through it, should be submitted. This tube should be labeled as a blank.

(9)     Capped resin tubes should be packed tightly and padded before they are shipped to minimize breakage during shipping.

## Analysis of Samples

All glassware used for the laboratory analysis should be washed with detergent and thoroughly rinsed with tap water and distilled water.

(a)     Preparation of Samples.  In preparation for analysis, remove the plastic caps used to cover tube after sample collection, and remove and discard the glass wool.  The resin in the front 100-mg section is transferred to a 5-ml screw-capped sample container.  The separating section of glass wool is removed and discarded.  The second 50-mg section is transferred to another container.  These two sections are analyzed separately.

(b)     Desorption of Sample.  Prior to analysis, 2.0 ml of ether is pipetted into each sample container.  Samples should be desorbed for 30 minutes.  Tests indicate that this is adequate if the sample is agitated

occasionally during this period. The sample vials should be capped as soon as the solvent is added to minimize volatilization. For the internal standard method, desorb using 2.0 ml of internal standard solution in ether.

(c) Gas-chromatographic Conditions. The typical operating conditions for the gas chromatograph are:

    (1)    Nitrogen carrier gas flow, 30 ml/minute (60 psig).

    (2)    Hydrogen gas flow to detector, 30 ml/minute (25 psig).

    (3)    Air flow to detector, 300 ml/minute (60 psig).

    (4)    Injector temperature, 200 C.

    (5)    Manifold temperature (detector), 280 C.

    (6)    Column temperature, 150 C.

A retention time of approximately 10.0 minutes is to be expected for the analyte using these conditions and the recommended column. The internal standard elutes between ether and the AGE.

(d) Injection of Samples. A 2-$\mu$l aliquot of the sample solution is injected into the gas chromatograph. The solvent-flush method or other suitable alternative, such as an automatic sample injector, can be used provided that duplicate injections of a solution agree well. No more than a 3% difference in area is to be expected.

(e) Measurement of Area. The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement, and preliminary results are read from a standard curve.

Determination of Desorption Efficiency

(a) Importance of Determination. The desorption efficiency of a particular compound can vary from one laboratory to another and also from

one batch of Tenax-GC to another. Thus, it is necessary to determine the percentage of the specific compound that is removed in the desorption process for the particular batch of resin used for sample collection and over the concentration range of interest. The desorption efficiency must be at least 75% at the OSHA standard level.

(b) Preparation for Determination. Desorption efficiency must be determined over the sample concentration range of interest. To determine the sample concentration range that should be tested, the samples are analyzed first. Then the analytical samples are prepared based on the relative amount of AGE found in the samples. The desorption efficiency must be determined at least twice for each concentration of AGE found in the samples.

The analytical samples are prepared as follows: Tenax-GC, equivalent to the amount in the front section (100 mg), is measured into a 5-ml screw-capped vial. This resin must be from the same batch as that used in obtaining the samples.

A known amount of a solution of AGE in hexane (spiking solution) is injected directly into the resin by means of a microliter syringe. Adjust the concentration of the spiking solution so that no more than a 10-$\mu$l aliquot is used to prepare the analytical samples.

For the validation studies conducted to determine the precision and accuracy of this method, six analytical samples at each of the three concentration levels (0.5, 1, and 2 times the OSHA standard of 45 mg/cu m) were prepared by adding an amount of AGE equivalent to that present in a 3-liter sample at the selected level. A stock solution containing 67.34 mg of AGE/ml of hexane was prepared. One-, 2-, and 4-$\mu$l aliquots of the

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solution were added to Tenax-GC resin tubes to produce solutions of 0.5, 1, and 2 times the OSHA standard level. The analytical samples were allowed to stand at least overnight to assure complete adsorption of the analyte onto the resin. A parallel blank tube was treated in the same manner except that no sample was added to it.

The procedure described can be used to prepare the analytical samples that are analyzed to determine desorption efficiency over the concentration range of interest.

(c) Procedure for Determination. The analytical samples and the blank are desorbed and analyzed as described in Analysis of Samples. Calibration standards are prepared by adding the appropriate volume of spiking solution to 2.0 ml of ether with the same syringe used in the preparation of the samples. Standards should be prepared at the same time that the sample analysis is done and should be analyzed with the samples.

If the internal standard method is used, prepare calibration standards by using 2.0 ml of ether containing a known amount of the internal standard.

The desorption efficiency (DE) equals the average weight in $\mu$g recovered from the tube divided by the weight in $\mu$g added to the tube, or:

$$DE = \frac{\text{Average weight } (\mu g) \text{ recovered}}{\text{Weight } (\mu g) \text{ added}}$$

The desorption efficiency may be dependent on the amount of AGE collected on the resin. Plot the desorption efficiency against the weight of AGE found. This curve is used to correct for adsorption losses.

## Calibration and Standards

(a)    Add  2.0  ml of ether (or 2.0 ml of internal standard solution in ether) to a 5-ml vial.  The same solution of AGE in hexane may  be  used to  prepare calibration standards, or microliter aliquots of pure AGE could be diluted to the appropriate volume for the standard  concentration  range of  interest.   The concentration of standards can be expressed in terms of $\mu$g of AGE/2.0 ml of ether.

(b)    A series of standards, varying in concentration over the range of interest, is prepared as described above and  analyzed  under  the  same gas-chromatographic  conditions  and  during  the  same  time period as the unknown samples.  Curves are established by plotting peak  area  (ordinate) against sample concentration in $\mu$g/2.0 ml.

For   the   internal   standard   method,   use   ether   containing   a predetermined amount of  the  internal  standard.   The  internal  standard concentration  used  was  approximately 70% of the concentration at 44 mg/cu m.  The area ratio of the AGE to that of the internal standard  is  plotted against the AGE concentration in $\mu$g/2.0 ml.

Note:   Whether  the external standard or internal standard method is used, standard solutions should be analyzed at the  same  time  the  sample analysis  is done.  This will minimize the effect of variations in the gas-chromatographic detector response.

## Calculations

Read  the  weight,  in  $\mu$g,  corresponding to each peak area from the standard curve.  No volume corrections are  needed,  because  the  standard curve  is based on $\mu$g/2.0 ml of ether, and the volume of sample injected is identical with the volume of the standards injected.

Corrections for the blank must be made for each sample:

$$\mu g = \mu g \text{ sample} - \mu g \text{ blank}$$

where:

$\mu g$ sample = $\mu g$ found in front (100-mg) sample section
$\mu g$ blank  = $\mu g$ found in front (100-mg) blank section

A similar procedure is followed for the backup (50-mg) section.

Read the desorption efficiency from the curve for the amount found in the front section of the tube. Divide the total weight by this desorption efficiency to obtain the corrected $\mu g$/sample.

$$\text{Corrected } \mu g/\text{sample} = \frac{\text{Weight } (\mu g) \text{ of front section}}{\text{DE}}$$

Add the amounts present in the front and backup sections for the same sample to determine the total weight in the sample.

(e)    Determine the volume in liters of air sampled at ambient conditions based on the appropriate information, such as flowrate in liters/minute multiplied by sampling time. If a pump using a rotameter for flowrate control was used for sample collection, a pressure and temperature correction must be made for the indicated flowrate. The expression for this correction is:

$$\text{Corrected volume} = f \times t \left( \sqrt{\frac{P1}{P2} \times \frac{T2}{T1}} \right)$$

where:

f  = flowrate during sampling
t  = sampling time
P1 = pressure during calibration of sampling pump (mmHg)
P2 = pressure of air sampled (mmHg)
T1 = temperature during calibration of sampling pump (K)
T2 = temperature of air sampled (K)

The concentration of the AGE in the air sampled can be expressed in mg/cu m, which is numerically equal to $\mu$g/liter:

$$mg/cu\ m = \frac{Corrected\ g}{Air\ volume\ sampled\ (liters)}$$

Another method of expressing concentration is ppm (corrected to standard conditions of 25 C and 760 mmHg):

$$ppm = mg/cu\ m\ x\ \frac{24.45}{MW}\ x\ \frac{760}{P}\ x\ \frac{T + 273}{298}$$

where:

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        P = pressure (mmHg) of air sampled
        T = temperature (C) of air sampled
    24.45 = molar volume (liters/mole) at 25 C and 760 mmHg
       MW = molecular weight of AGE
      760 = standard pressure (mmHg)
      298 = standard temperature (K)
```