Sep-Pak XPoSure Aldehyde Sampler

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I. INTRODUCTION

Waters Sep-Pak® XPoSure™ Aldehyde Samplers are convenient, reproducible sampling devices for quantifying formaldehyde concentrations in the workplace and indoor air within a range of 0.001 to 5 parts per million (ppmv).
a. Sep-Pak XPoSure Aldehyde Sampler Description and Features

- Sep-Pak XPoSure Aldehyde Samplers contain acidified 2,4-Dinitrophenylhydrazine (DNPH)-coated silica, packed in Waters Sep-Pak Plus cartridges.
- The samplers are constructed from high-purity and high-density polyethylene components, triaxially-compressed packed beds and Luer fittings equipped with end caps and plugs.
- The samplers are designed for flow rates of up to 1.5 L/min with typical personal pumps.
- The gold-colored aluminum compression ring on the Sep-Pak XPoSure Aldehyde Sampler allows for easy identification.

II. USING THE SEP-PAK XPOSURE ALDEHYDE SAMPLER

a. Theory of Operation

Sep-Pak XPoSure Aldehyde Samplers trap aldehydes in air by reacting them with acidified 2,4-dinitrophenylhydrazine (DNPH), forming stable hydrazone derivatives. The derivitization reaction, as shown in Figure 2, takes place during sample collection. The derivatives are later eluted and analyzed using HPLC.

![Figure 2. Derivitization Reaction.](image)

b. Preventing Contamination

Contamination is most likely to occur during sample collection. Before eluting the derivatives, clean all glassware by rinsing with acetonitrile and heating to 60 °C in a vacuum oven for at least 30 minutes. Eluting the samples in a nitrogen-purged glove box further reduces the risk of contamination.

The acetonitrile you use to elute the DNPH derivatives can also be a source of contamination. Even HPLC-grade acetonitrile may have unacceptable levels of carbonyl contaminants and should be stored in a carbonyl-free environment. A concentration of 10 µg/L of any aldehyde or ketone contaminant will add 0.1 µg to the blank values determined for the DNPH-derivatives per cartridge. Follow the procedure in Appendix A, Measuring Acetonitrile Purity, to pre-qualify your acetonitrile.

![Figure 1. Cutaway View.](image)
c. Collecting the Sample

**Caution:** Beware of unintentional exposure of the samplers and eluted samples to aldehyde and ketone sources. Laboratory air often holds high concentrations of acetone. Labeling inks and adhesives as well as packaging containers (including vials with plastic caps) are all possible sources of contamination.

Sampling methods have been developed and validated for both 15 minute short-term exposure limit (STEL), and in 8 hour personal exposure limit (PEL) measurements following the National Institute for Occupational Safety and Health (NIOSH) guidelines. XPoSure Aldehyde Samplers have been tested under controlled laboratory conditions. Table 2 lists the equipment needed to collect air samples using XPoSure Aldehyde Samplers. The recommended measurement range for formaldehyde is:

<table>
<thead>
<tr>
<th>Measure</th>
<th>Formaldehyde Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>STEL</td>
<td>0.022 to 2 ppmv</td>
</tr>
<tr>
<td>PEL</td>
<td>0.01 to 1 ppmv</td>
</tr>
</tbody>
</table>

**Table 2: Sample Collection Equipment**

<table>
<thead>
<tr>
<th>Suggested Personal Pump Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operating Range</td>
</tr>
<tr>
<td>Compensation Range</td>
</tr>
<tr>
<td>Flow Control</td>
</tr>
<tr>
<td>Flow Indicator</td>
</tr>
</tbody>
</table>

* A flow calibrator may also be required.

The background levels of aldehydes and ketones in the sampler determine the sensitivity of the method. The volume of air passed through the sampler must be large enough for the quantity of DNPH derivatives formed to be several times greater than the background level. The United States Environmental Protection Agency (US EPA) recommends that this level be at least 10 times the background level. Table 3 lists the sampler background specifications.

**Table 3: Sampler Background Specifications**

<table>
<thead>
<tr>
<th>Compound</th>
<th>µg DNPH Derivatives per Sampler</th>
<th>µg Carbonyl Compounds per Sampler</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde</td>
<td>&lt;0.45</td>
<td>&lt;0.06</td>
</tr>
<tr>
<td>Adetaldehyde</td>
<td>&lt;0.75</td>
<td>&lt;0.15</td>
</tr>
<tr>
<td>Acetone</td>
<td>&lt;1.5</td>
<td>&lt;0.38</td>
</tr>
<tr>
<td>Othersa</td>
<td>&lt;0.75</td>
<td>-</td>
</tr>
</tbody>
</table>

* Individually, as acetone-DNPH.

Sample Collection – STEL (0.02 to 2 ppmv)

To collect the STEL air sample:

1. Calibrate the sampling pump with a representative sampler in line. Set the flow to 1.5 L/min. Figure 3 shows the flow rate through a sampler versus applied vacuum. Once calibrated, remove and store this representative sampler for future calibrations.
2. Take a fresh sample from its pouch. Remove and save the end cap and plugs.
3. Connect the sampler to a pump with flexible plastic tubing. The sampler is bidirectional (flow can be in either direction).
4. Draw air for 15 minutes, yielding a sample volume of 22.5 liters.
5. Re-seal the sampler with its end cap and plug.
6. Store the sampler in the pouch provided with appropriate identification. Seal the pouch by folding the edge over twice and stapling it shut. Avoid exposing the samplers to heat.

A 22.5 L air sample is sufficient for quantifying formaldehyde in the range of 0.02 to 2 ppmv. Formaldehyde concentrations lower than 0.02 ppmv in air will require longer sampling times and a larger air sample. Conversely, formaldehyde concentrations that exceed 2.0 ppmv will require shorter sampling times or reduced sampling flow rates in order to avoid overloading the sampler and obtaining nonlinear results.

**Note:** The maximum recommended sampler capacity is 2.3 µmoles total carbonyl species. This calculates to 50% of the DNPH consumed. Contaminated air may contain significant concentrations of other aldehydes and ketones and the total may exceed the capacity of the sampling device. Follow the procedure in Appendix C, Measuring Breakthrough for more information.
Sample Collection – PEL (0.01 to 1.0 ppmv)
To collect the PEL air sample:

1. Calibrate the sampling pump with a representative sampler in line. Set the flow to 100 mL/min. Figure 3 shows the flow rate through a sampler versus applied vacuum. Once calibrated, remove and store this representative sampler for future calibrations.

2. Take a fresh sample from its pouch. Remove and save the end cap and plugs.

3. Connect the sampler to a pump with flexible plastic tubing. The sampler is bidirectional (flow can be in either direction).

4. Draw air for 8 hours, yielding a sample volume of 48 liters.

5. Reseal the sampler with its end cap and plug.

6. Store the sampler in the pouch provided with appropriate identification. Seal the pouch by folding the edge over twice and stapling it shut. Avoid exposing the samplers to heat.

A 48 L air sample is sufficient for quantifying formaldehyde in the range of 0.01 to 1 ppmv. Formaldehyde concentrations lower than 0.01 ppmv in air will require longer sampling times and a larger air sample. Conversely, formaldehyde concentrations that exceed 1.0 ppmv will require shorter sampling times or reduced sampling flow rates in order to avoid overloading the sampler and obtaining nonlinear results.

Note: The maximum recommended sampler capacity is 2.3 µmoles total carbonyl species. This calculates to 50% of the DNPH consumed. Contaminated air may contain significant concentrations of other aldehydes and ketones and the total may exceed the capacity of the sampling device. Follow the procedure in Appendix C, Measuring Breakthrough for more information.

d. Eluting the Derivatives from the Sampler
To elute the derivatives from the sampler:

1. Remove the sampler from the stapled pouch.

2. Elute the DNPH derivatives from the sampler with pre-qualified acetonitrile directly into a 10 mL volumetric flask. Use a flow rate of <3 mL/min. Higher flow rates (>3 mL/min) can result in reduced recovery.

3. Cap the volumetric flask and mix by inverting it several times.

4. Analyze the eluate using HPLC.

Note: Since background levels may change during storage, always compare samples to a blank sampler from the same lot, stored under the same conditions.

III. ANALYZING THE DNPH DERIVATIVES

a. Operating Guidelines
To ensure success in your HPLC analysis:

- Use a pre-column filter between the injector and column.
- Use HPLC-grade water and HPLC-grade acetonitrile.
- Degas the mobile phases by simultaneously applying vacuum and ultrasound to the mobile phases for 30 seconds. If you are using a low-pressure mixing gradient system, sparging with helium may be necessary.
- Waters Symmetry® C18 columns are shipped containing water/acetonitrile. Before the first analysis, equilibrate the column with mobile phase at 1.3 mL/min for 10 minutes in mobile phase or until the baseline is stable. See Table 4 for separation conditions.
b. Performing the HPLC Analysis

To analyze the sample:

1. Prepare the standard solution of the DNPH derivatives that you need to quantify. The concentrations of the standards should be in the same range as the expected concentrations in the sample. To synthesize DNPH derivatives, see Appendix B.

2. Prepare a cartridge blank from the same sample lot as the cartridge used to collect the sample, using the sample procedure and same bottled solvent.

3. Analyze the standard solution to determine the response factor for each derivative. Due to the high linearity of the detector response, a single point calibration is sufficient for Waters detectors.

   *Note: Use an injection volume appropriate for your column.*

   *Inject ≤ 20 µL for a 3.9 x 150 mm Waters Symmetry C18 column, and ≤ 10 µL for a 3.0 x 75 mm Waters Symmetry C18 column.*

4. Analyze the cartridge blank to determine background levels. Ensure that the blank values are in the normal range (see Table 3). Figures 4 and 5 show a representative separation and blank sample, respectively.

5. Analyze the samples.

6. Subtract the blank values from the sample values. Run standards at regular intervals between samples.

---

Table 4: HPLC Separation Conditions

<table>
<thead>
<tr>
<th>Column</th>
<th>Waters Symmetry C18, 3.9 x 150 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mobile Phase</td>
<td>45:55 Acetonitrile/Water</td>
</tr>
<tr>
<td>Flow Rate</td>
<td>1.3 mL/min</td>
</tr>
<tr>
<td>Injection Volume</td>
<td>20 µL</td>
</tr>
<tr>
<td>Detection Wavelength</td>
<td>Absorbance at 360 nm (&lt;50 mV of baseline noise)</td>
</tr>
<tr>
<td>Limit of Detection for Formaldehyde</td>
<td>Based on a signal to noise ratio of 3 (55 picograms in 20 µL injection)</td>
</tr>
<tr>
<td>Limit of Quantification for Formaldehyde</td>
<td>Based on a signal to noise ratio of 10 (183 picograms in 20 µL injection)</td>
</tr>
</tbody>
</table>

---

![Figure 4. Isocratic Separation of C1-C3 Hydrazone Derivatives.](image)

![Figure 5: Typical Sampler Blank.](image)

c. Analyzing a Cartridge Blank

Analyze a blank to determine background levels. Since background levels may change during storage, always compare samples to blank cartridges from the same lot stored under the same conditions.

*Note: When preparing a blank sample, ensure that you use the exact bottled reagents that were used for the preparation of the sample.*

To prepare a cartridge blank:

1. Elute a fresh DNPH-Silica Sep-Pak cartridge from the same lot as the cartridges used to collect your sample.

2. Analyze the solution by HPLC using the same conditions as those used for the sample.

3. Multiply the concentration of each DNPH derivative by the volume of the eluate to determine the amount of background DNPH derivative.
d. Calculating Results
To calculate the aldehyde concentration in air:
1. Prepare a calibration line of peak area to standard concentration on µg/mL of analyte as carbonyl compound.
2. Use the calibration line to determine the analyte concentration in the blank and sample eluates.
3. Determine the mass of each analyte in the blank (Wb) in µg. This is done by multiplying each analyte concentration by the eluate volume in mL. Compare these values to the specifications listed in Table 3. If these values are higher than the specifications, contamination occurred during sample preparation or storage.
4. Determine the mass of each analyte in the sample (Ws) in µg. This is done by multiplying each analyte concentration by the eluate volume in mL.
5. Calculate the analyte concentration (C) in µg/L*. This is done by dividing the weight of the analyte in the air (Ws-Wb) by the volume of the air sampled (V) in liters.

\[ C = \frac{W_s - W_b}{V} \]

*The units, µg/L, is equivalent to mg/m 3. For converting µg/L to ppmv at 1 atm and 20 °C, see Appendix D.

IV. APPLICATION EXAMPLES
a. Formaldehyde in Laboratory Air - STEL
The sample in Figure 6 was collected in a chemical research laboratory using a portable sampling pump. The 22.5 liter air sample was collected at 1.5 L/min. The chromatogram shows 0.02 ppmv of formaldehyde. Under these chromatographic conditions the DNPH-glutaraldehyde diastereomers are resolved.

b. Glutaraldehyde in Laboratory Air – STEL
The sample in Figure 7 was collected in chemical research laboratory using a portable sampling pump. The 22.5 liter air sample was collected at 1.5 L/min. The chromatogram shows 0.02 ppmv of glutaraldehyde. Under these chromatographic conditions the DNPH-glutaraldehyde diastereomers are resolved.

V. STORAGE AND DISPOSAL OF THE SAMPLERS
a. Storing unused samplers
Always store any unused Waters Sep-Pak XPoSure Aldehyde Samplers in their protective pouches to prevent contamination.

Store the sealed pouches in a refrigerator at (4 °C ± 2 °C) for up to six months. Cartridges may be stored in their unopened pouches at room temperature (20 to 25 °C) for up to two weeks.

Background levels of hydrazone derivatives increase slightly with time and temperature. Before using cartridges exposed to high temperatures or stored longer than the recommended periods, run a blank.

b. Storing exposed samplers
Once a sampler has been used for collection, be careful to cap and seal it until it is time to elute it. Inadvertent exposure of an exposed cartridge can ruin a carefully collected sample. Elute the derivatives from the cartridge within two weeks. The sample eluates are stable at 4 °C ± 2 °C for up to one month.

c. Disposing of used cartridges
Dispose of used cartridges according to applicable government regulations.
VI. TROUBLESHOOTING

Table 5 describes solutions to problems that may arise while using the samplers. Most errors occur as a result of contamination during sample preparation. To resolve chromatographic problems not listed, refer to your HPLC system manual.¹

<table>
<thead>
<tr>
<th>Symptom</th>
<th>Possible Cause</th>
<th>Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>High carbonyl values in sampler blank</td>
<td>Contaminated acetonitrile</td>
<td>Certify acetonitrile quality prior to use, see Appendix A.</td>
</tr>
<tr>
<td></td>
<td>Contaminated glassware</td>
<td>Use only pre-cleaned glassware.</td>
</tr>
<tr>
<td></td>
<td>Air contamination during elution</td>
<td>Prepare sample in a glove box.</td>
</tr>
<tr>
<td></td>
<td>Sampler age and storage conditions</td>
<td>Replace samplers.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Refrigerate fresh samplers.</td>
</tr>
<tr>
<td>High formaldehyde levels in sampler blank</td>
<td>Coelution of formaldehyde with an impurity</td>
<td>Prepare a fresh mobile phase or decrease the acetonitrile content.</td>
</tr>
<tr>
<td>Broad peaks</td>
<td>Injection volume too high</td>
<td>Reduce the injection volume.</td>
</tr>
<tr>
<td></td>
<td>System or column failure</td>
<td>Consult the HPLC System manual.</td>
</tr>
</tbody>
</table>

VII. REFERENCES AND BIBLIOGRAPHY

1. Committee on Aldehydes, Board of Toxicology and Environmental Hazards, National Research Council, Formaldehyde and Other Aldehydes; National Academy Press, Washington, DC, 1981.
5. ASTM Method E411; Standard Test Method for Trace Quantities of Carbonyl Compounds with 2,4-Dinitrophenylhydrazine.

VIII. ORDERING INFORMATION

Waters Sep-Pak XPoSure Aldehyde Samplers are shipped in boxes of 20 individually-packaged cartridges. Pouches are supplied for storage after sampling.

IX. APPENDICES

a. Appendix A: Measuring Acetonitrile Purity

HPLC-grade acetonitrile may contain traces of aldehydes and ketones, and especially acetone. A concentration of 10 µg/L of an aldehyde or ketone in the acetonitrile adds 0.1 µg to the blank values determined for the DNPH-derivatives per cartridge. If acetonitrile is unacceptable for your application, contact your solvent supplier, to purify the acetonitrile. To purify acetonitrile, distill it from an acidified DNPH solution, using a procedure analogous to the one described in ASTM Method E411 for the purification of methanol.⁵

To measure acetonitrile purity:

1. Clean all glassware by rinsing with acetonitrile and heating in a 60 °C vacuum oven for at least 30 minutes.
2. Elute a fresh sampler with 3 mL acetonitrile.
3. Within 3 minutes, inject the eluate into the HPLC system to measure the concentration of DNPH derivatives.
4. Add 1 drop of concentrated HCl to the eluate, and allow to react at room temperature for 30 minutes.
5. Remeasure the concentration of DNPH derivatives by HPLC.
6. Calculate the difference in the concentration of each DNPH derivative measured in steps 3 and 5 to yield the contribution form the acetonitrile.
7. Calculate the percent hydrazone contributed by the acetonitrile relative to the background level. The value for any hydrazone should not exceed 25% of its value in the blank.
Example: Measuring Acetonitrile Purity

1. HPLC analysis of a fresh sampler shows the sample contains:

<table>
<thead>
<tr>
<th>Derivative</th>
<th>Contribution from Acetonitrile</th>
<th>Divided by Background Value</th>
<th>Times 100</th>
<th>Equals Percent Relative to Background</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde-DNPH</td>
<td>0.01</td>
<td>+ 0.08</td>
<td>x 100</td>
<td>= 12%</td>
</tr>
<tr>
<td>Acetaldehyde-DNPH</td>
<td>0.02</td>
<td>+ 0.12</td>
<td>x 100</td>
<td>= 17%</td>
</tr>
<tr>
<td>Acetone-DNPH</td>
<td>1.60</td>
<td>+ 0.40</td>
<td>x 100</td>
<td>= 400%</td>
</tr>
</tbody>
</table>

Since the percent for formaldehyde and acetaldehyde arising from the acetonitrile is less than 25% of the background level in the sampler, the acetonitrile is considered clean for these compounds. If the analysis considers only these compounds, the acetonitrile is acceptable.

However, the amount of acetone arising for the acetonitrile is 4 times the amount found in the background level. Therefore, it is suggested that this lot of acetonitrile may be unacceptable for use in the analysis of acetone.

2. Analysis of the concentrations of hydrazones after reacting with acid yields:

<table>
<thead>
<tr>
<th>Derivative</th>
<th>Concentration (µg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde-DNPH</td>
<td>0.08</td>
</tr>
<tr>
<td>Acetaldehyde-DNPH</td>
<td>0.12</td>
</tr>
<tr>
<td>Acetone-DNPH</td>
<td>0.40</td>
</tr>
<tr>
<td>All other hydrazones</td>
<td>&lt;0.05</td>
</tr>
</tbody>
</table>

3. The difference between the concentrations of hydrazone from steps 3 and 5 represents the amount of hydrazone contributed by the acetonitrile:

<table>
<thead>
<tr>
<th>Derivative</th>
<th>Concentration (µg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde-DNPH</td>
<td>0.09</td>
</tr>
<tr>
<td>Acetaldehyde-DNPH</td>
<td>0.14</td>
</tr>
<tr>
<td>Acetone-DNPH</td>
<td>2.00</td>
</tr>
<tr>
<td>All other hydrazones</td>
<td>&lt;0.05</td>
</tr>
</tbody>
</table>

4. The percent of the hydrazones contributed by the acetonitrile is:

<table>
<thead>
<tr>
<th>Derivative</th>
<th>Concentration after Reaction with Acid</th>
<th>Minus Concentration in Blank</th>
<th>Equals Contribution form Acetonitrile</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde-DNPH</td>
<td>0.09 µg/mL</td>
<td>- 0.08 µg/mL</td>
<td>= 0.01 µg/mL</td>
</tr>
<tr>
<td>Acetaldehyde-DNPH</td>
<td>0.14 µg/mL</td>
<td>- 0.12 µg/mL</td>
<td>= 0.02 µg/mL</td>
</tr>
<tr>
<td>Acetone-DNPH</td>
<td>2.00 µg/mL</td>
<td>- 0.40 µg/mL</td>
<td>= 1.60 µg/mL</td>
</tr>
</tbody>
</table>

b. Appendix B: Synthesizing the DNPH-Derivative Standards

High purity (99%) DNPH derivatives are commercially available or can be synthesized from DNPH supplied by Aldrich Chemical Company (70% DNPH and 30% water). To synthesize 98-99% pure hydrozones:

1. Prepare one liter of 2 M HCl solution: Add 172 mL concentrated reagent-grade hydrochloric acid (HCl) to a 1 L volumetric flask. Fill the flask to the mark with distilled deionized water.

2. Saturate the 2 M HCl solution with DNPH: Add 8 g DNPH and stir for one hour at 20 to 25 °C. Filter through a 0.45 µm hydrophilic membrane (HVLP) filter (Waters Part number: WAT200530).

3. Form the hydrazone derivative by adding a two-fold molar excess of reagent-grade aldehyde or ketone to the filtered 2 M HCl DNPH solution. Stir for 30 minutes to one hour at 20 to 25 °C.

4. Filter the hydrazone slurry. Wash the hydrazone with 50 mL 2 M HCl 3 times. Wash with 50 mL water 3 times. Dry the filter cake in an oven at 50 to 60 °C overnight.

5. Prepare a standard stock solution of the DNPH-derivatives by dissolving an accurately weighed amount in acetonitrile. Prepare a set of calibration standards using the stock solution. The concentration of the standards should be in the same range as the expected concentration of the samples. The solutions are stable for at least one month when stored in tightly-capped glass vials at 4 °C ± 2 °C.
c. Appendix C: Measuring Breakthrough

Note: If several aldehydes and ketones are present in significant concentration, estimate the maximum sample size from the total concentration of all species. Collection efficiency determinations are best made during times expected to yield peak formaldehyde concentrations. This will enable appropriate sampling rates and intervals to be selected to avoid breakthrough.

Figure 8 shows the predicted total carbonyl concentration versus the range of sample volumes.

![Image](image-url)

Figure 8. Total Carbonyl Concentration vs. Range of Sample Volumes.

To measure Waters Sep-Pak XPoSure Aldehyde Sampler for collection efficiency:

1. Connect two unused cartridges together by the Luer fittings and mark each cartridge for identification.
2. Connect the cartridges to a calibrated pump with a short length of flexible tubing.
3. Collect the sample.
4. Elute both cartridges and a third blank cartridge.
5. Analyze all three cartridges by HPLC.
6. Subtract the blank value from the values determined from the other two cartridges.
7. Calculate and sum of the total captured DNPH-derivative from both cartridges.
8. Divide the amount of DNPH-derivative determined from the first cartridge by the total amount determined form cartridges 1 and 2. Multiply by 100. This is the percentage of DNPH-derivatives captured on the first cartridge. This value should exceed 95%; otherwise, some of the sample broke through to the second cartridge.

**Example: Measuring Sample Breakthrough**

The expected concentration of formaldehyde is 0.66 ppmv (µL/L). Flow rate is 1.25 L/min for 80 minutes. A sample volume of 100 liters is collected. The theoretical quantity of carbonyl is:

\[
\text{Analyte ppmv \times Carbonyl \ molecule weight \times air volume = µg Carbonyl weight}
\]

This calculates to:

\[
0.66 \, \text{µL} \times 30.03 \, \text{g/mole} \times 100 \, \text{L} = 81 \, \text{µg formaldehyde}
\]

\[
\frac{24.46 \, \text{L/mole}}{}
\]

The actual results are shown in Table 7. To calculate the percent captured on the first sampler, divide the quantity captured on sampler 1 by the total quantity captured, then multiply by 100. Since this value is less than 95%, and the total carbonyl amount exceeded 2.3 µmoles, breakthrough occurred.

Collection efficiency for Waters Sep-Pak XPoSure Aldehyde Sampler is greater than 95% for air sampling rates of up to 1.5 L/min. The sampler may exhibit breakthrough if:
- The sampling flow rate is greater than 1.5 L/min
- The amount of sample collected is enough to react with more than 50% of the DNPH (~2.3 µmoles)
In the above example, only a single carbonyl source was present. Under many test conditions more than one carbonyl source may be present in significant concentrations. These other compounds will consume DNPH, effectively reducing the capacity of the sampler for the compound of interest. To assure that the capacity of the sampler has not been exceeded, compare the DNPH peak areas of the sample to a similarly eluted blank. The DNPH peak area in all samples must be no less than 50% of the DNPH peak area of the blank. This ensures the sampler capacity has not been exceeded.

Table 7: Breakthrough Example HPLC Results

<table>
<thead>
<tr>
<th>Sampler</th>
<th>Amount (µg)</th>
<th>Quantity Captured</th>
<th>Percent Captured on Sampler</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sampler 1</td>
<td>75.06</td>
<td>75.00</td>
<td>91.8</td>
</tr>
<tr>
<td>Sampler 2</td>
<td>6.72</td>
<td>6.66</td>
<td>8.2</td>
</tr>
<tr>
<td>Blank</td>
<td>0.06</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

d. Appendix D: Useful Conversion Factors

This appendix contains:
- Carbonyl to hydrazone conversion factors
- Equation for converting µg/L to ppmv
- Conversion factors: µg/L ↔ ppmv

Obtaining carbonyl concentrations in eluates and air samples required the use of several constants and conversion factors. The factors described in this appendix can be used when converting carbonyl weights to:
- Equivalent derivative weights for preparing standard solutions
- Volumes for reporting air samples in ppmv

d.1. Carbonyl to Hydrazine Conversion Factors

Table 8 lists the molecular weights (MW) for some carbonyl compounds. These values were used to derive the conversion factors listed in Table 9. Multiply the carbonyl or derivative weights by the appropriate factor for the desired conversion.

Table 8: Carbonyl and Hydrazine Molecular Weights

<table>
<thead>
<tr>
<th>Carbonyl Compounds</th>
<th>Carbonyl Compounds Molecular Weight, (MW_C)</th>
<th>Hydrazine Derivative Molecular Weight (MW_D)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde</td>
<td>30.03</td>
<td>210.15</td>
</tr>
<tr>
<td>Adetaldehyde</td>
<td>44.05</td>
<td>224.17</td>
</tr>
<tr>
<td>Adetone</td>
<td>58.08</td>
<td>238.20</td>
</tr>
<tr>
<td>Glutaraldehyde</td>
<td>100.12</td>
<td>460.36</td>
</tr>
</tbody>
</table>

d.2. Equation for converting µg/L to ppmv

Carbonyl concentrations can be converted from µg/L to ppmv (µL/L) by using the following expression:

\[
\text{(Result in ppmv)} = \left( \frac{\text{Result in µg/L}}{\text{MWC}} \right) \times 22.41 \times \frac{T_2}{T_1} \times \frac{P_1}{P_2}
\]

Where values are:
- 22.41 = Molar volume of an ideal gas at STP (273.15 °K and 1 atm), L/mole
- MWC = Molecular weight of carbonyl, g/mole
- T1 = Standard temperature, 273.15 °K
- T2 = Air sample temperature, K
- P1 = Standard pressure, 1 atm
- P2 = Air sample pressure, atm

d.3. Conversion Factors: µg/L to ppmv

Table 10 lists the factors for converting between µg/L and ppmv at 25 °C and 1 atm. Results are converted between µg/L (or mg/m³) and ppmv, by multiplying by the appropriate factor.

Table 10: Factors for Converting Between µg/L and ppmv at 25 °C and 1 atm.

<table>
<thead>
<tr>
<th>Carbonyl Compounds</th>
<th>ppmv → µg/L</th>
<th>µg/Lp → ppmv</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde</td>
<td>1.23</td>
<td>0.813</td>
</tr>
<tr>
<td>Adetaldehyde</td>
<td>1.80</td>
<td>0.555</td>
</tr>
<tr>
<td>Adetone</td>
<td>2.38</td>
<td>0.420</td>
</tr>
<tr>
<td>Glutaraldehyde</td>
<td>4.09</td>
<td>0.244</td>
</tr>
</tbody>
</table>