

# Laboratory Evaluation of 3M™ Ethylene Oxide Monitor 3550+, 3551+

These tests for evaluation of diffusive air samplers were conducted within the guidelines described in ANSI 104-1998 (R2009).

The 3M™ Ethylene Oxide Monitor 3551+ and 3550+ (with prepaid analysis) consist of a wafer of activated carbon in a PTFE binder that is treated with hydrogen bromide. The wafer is encased in a polypropylene sampler body with a 76-port polypropylene sampling grid.

When ethylene oxide is absorbed, it reacts with the hydrogen bromide to form 2-bromoethanol; the 2-bromoethanol is then extracted and analyzed by GC/ECD (modified ASTM D5578 method).

## 1. Test Apparatus & Method

Vapor exposures of ethylene oxide (CAS 75-21-8) were created by dynamic dilution from a gas cylinder containing 1.0% ethylene oxide in nitrogen. The analyte was acquired from the cylinder at a fixed rate via mass flow controller (MFC), dynamically mixed with flow-controlled input air provided by the Miller-Nelson 501 atmosphere conditioner, and then passed through an inert acrylic chamber containing diffusive samplers under test. MFC flow was verified by calibration, and exposure concentrations were verified by HBr-treated tube samples continuously drawn from locations in the chamber bracketing the samplers under test.

## 2. Desorption Efficiency (DE)

Desorption efficiency (analyte recovery) was determined by analysis (Method AT555) of HBr-treated charcoal wafers spiked from standard solutions of 2-bromoethanol. The test is conducted at three levels in quadruplicates. The desorption efficiency calculated from this test was 98.1% (Table 1).

**Table 1.** Desorption Efficiency results for 3551+

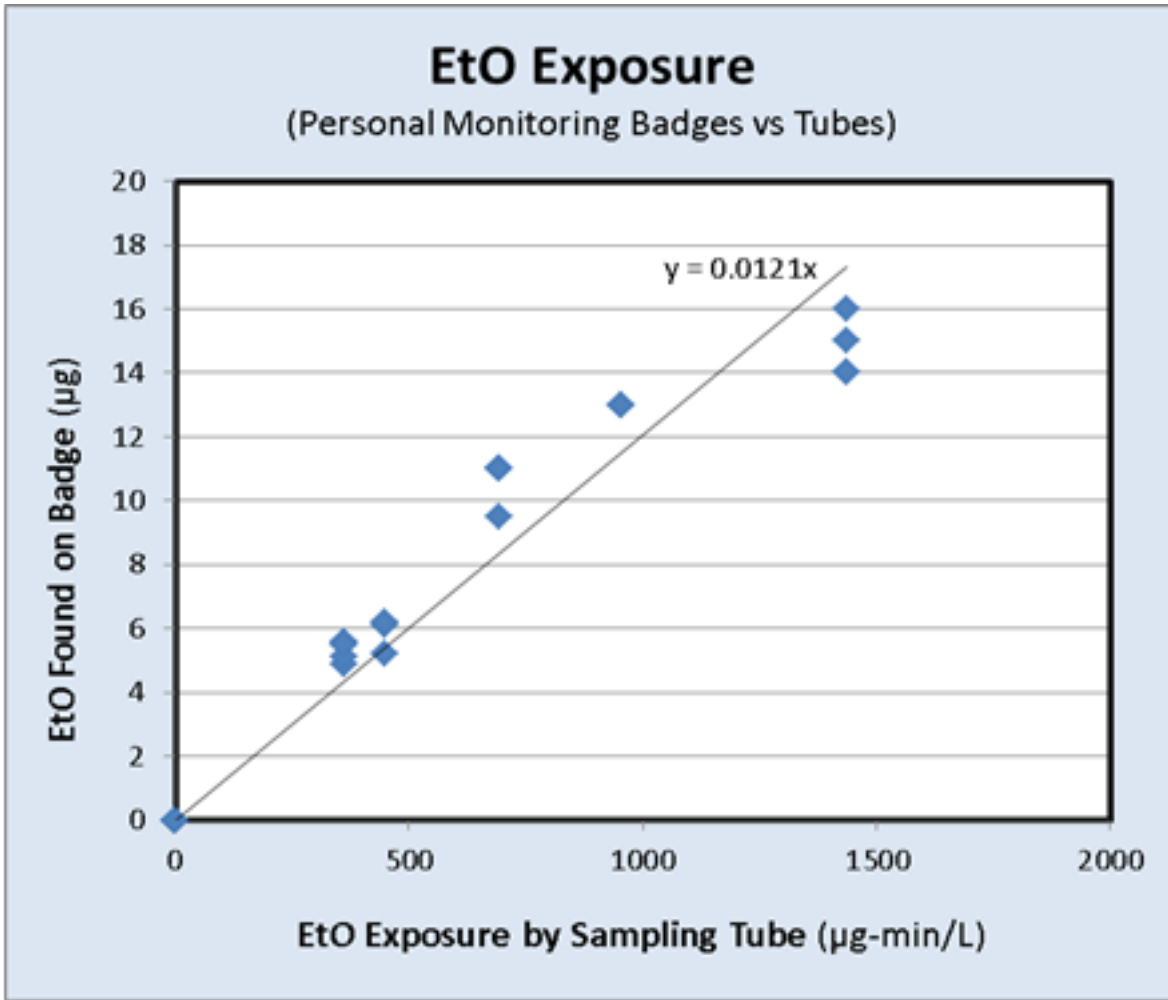
| Spike Level 1           | Amt Recovered (µg/mL) | DE    |
|-------------------------|-----------------------|-------|
| Liquid Spike (no media) | 1.45                  |       |
| Spike 1                 | 1.39                  | 95.9% |
| Spike 2                 | 1.41                  | 97.4% |
| Spike 3                 | 1.40                  | 96.7% |
| Spike 4                 | 1.38                  | 95.6% |
|                         | Average DE            | 96.4% |

| <b>Spike Level 2</b>    | <b>Amt Recovered (µg/mL)</b> | <b>DE</b> |
|-------------------------|------------------------------|-----------|
| Liquid Spike (no media) | 2.72                         |           |
| Spike 1                 | 2.71                         | 99.6%     |
| Spike 2                 | 2.70                         | 99.3%     |
| Spike 3                 | 2.69                         | 98.8%     |
| Spike 4                 | 2.70                         | 99.3%     |
|                         | Average DE                   | 99.2%     |
|                         |                              |           |
| <b>Spike Level 3</b>    | <b>Amt Recovered (µg/mL)</b> | <b>DE</b> |
| Liquid Spike (no media) | 5.34                         |           |
| Spike 1                 | 5.31                         | 99.4%     |
| Spike 2                 | 5.30                         | 99.1%     |
| Spike 3                 | 5.27                         | 98.5%     |
| Spike 4                 | 5.25                         | 98.2%     |
|                         | Average DE                   | 98.8%     |
|                         |                              |           |
|                         | 3551+ DE:                    | 98.1%     |

### 3. Verification of Diffusive Sampling Rate

Per internal quality control specifications, periodic testing of 3551+ samplers is conducted to verify sampler performance and sampling rates (set from experimental data). Table 2 and Figure 1 show data from several of these tests between 2014 and 2015.

Sampling Rate (corresponding to the least squares slope of the regression line) correlates well with previous studies. Scatter in results is attributed to variable recovery for analysis of tubes which has been documented for sampling tubes (ORBO-78 and SKC 126-178).



**Figure 1.** Comparison of 3551+ and tube sampling for sampling rate verification.

**Table 2.** Comparison of amount of ethylene oxide collected by 3551+ and tubes

| Sample Name     | Amt Found (µg) | Sampling Time (min) | Amt Found (ppm) |
|-----------------|----------------|---------------------|-----------------|
| 2 ppm tube - 1  | 9.1            | 120                 | 2.20            |
| 2 ppm tube - 2  | 8.7            | 120                 | 2.10            |
| 2 ppm tube - 3  | 7.6            | 120                 | 1.90            |
| 2 ppm tube - 4  | 9.1            | 120                 | 2.10            |
|                 |                | Average             | 2.1             |
|                 |                | CV                  | 6%              |
| 3551+/2 PPM - 1 | 6.1            | 120                 | 2.28            |
| 3551+/2 PPM - 2 | 6.2            | 120                 | 2.31            |
| 3551+/2 PPM - 3 | 5.2            | 120                 | 1.94            |

|                 |                |                     |              |
|-----------------|----------------|---------------------|--------------|
|                 |                | Average             | 2.2          |
|                 |                | CV                  | 9%           |
| Sample Name     | Amt Found (µg) | Sampling Time (min) | Amount (ppm) |
| 4 ppm tube - 1  | 18             | 120                 | 4.20         |
| 4 ppm tube - 2  | 18             | 120                 | 4.30         |
| 4 ppm tube - 3  | 18             | 120                 | 4.50         |
| 4 ppm tube - 4  | 19             | 120                 | 4.30         |
|                 |                | Average             | 4.3          |
|                 |                | CV                  | 3%           |
| 3551+/4 PPM - 1 | 13             | 120                 | 4.85         |
| 3551+/4 PPM - 2 | 13             | 120                 | 4.85         |
| 3551+/4 PPM - 3 | 13             | 120                 | 4.85         |
|                 |                | Average             | 4.8          |
|                 |                | CV                  | 0%           |
|                 |                |                     |              |
| Sample Name     | Amt Found (µg) | Sampling Time (min) | Amount (ppm) |
| 6 ppm tube - 1  | 28             | 120                 | 6.70         |
| 6 ppm tube - 2  | 26             | 120                 | 6.00         |
| 6 ppm tube - 3  | 27             | 120                 | 6.80         |
| 6 ppm tube - 4  | 29             | 120                 | 6.60         |
|                 |                | Average             | 6.5          |
|                 |                | CV                  | 6%           |
| 3551+/6 PPM - 1 | 14             | 120                 | 5.22         |
| 3551+/6 PPM - 2 | 14             | 120                 | 5.22         |
| 3551+/6 PPM - 3 | 15             | 120                 | 5.60         |
| 3551+/6 PPM - 4 | 16             | 120                 | 5.97         |
|                 |                | Average             | 5.3          |
|                 |                | CV                  | 4%           |

## 4. Background (Blank) Determination

Unexposed samplers were analyzed by Method AT555 to determine background analyte levels (if any) on the sampler prior to sampling. No background peaks were detectable (< 0.1 µg).

## 5. Atmospheric Effects

Air Velocity & Orientation – Previous studies demonstrated that there is no significant effect of air velocity and orientation on sampling rate

Temperature and Humidity – Previous studies demonstrated the absence of an effect of temperature and humidity on sampling rate in the range 0 – 50°C and 10 – 80% RH.

## 6. Reverse Diffusion

A reverse diffusion study was performed with the media. After exposure to EtO for two hours, five samples were removed from the chamber (initial) and six samples were left in the chamber. Those samplers were left open with just air flow from the chamber fan passing by for an additional six hours. They were collected and closed after a total time of 8 hours.

Analysis of the samplers removed immediately following the two-hour test and the reverse diffusion samples indicated that these samplers don't experience a significant amount of reverse diffusion of an 8-hr workday simulation (Table 3).

**Table 3.** Comparison of data from initial samples and reverse diffusion samples

| <b>Initial Samples</b>   |                |                 |
|--|----------------|-----------------|
| Sample Name  | Amt Found (µg) | Amt Found (ppm) |
| INITIAL -1   | 10.7           | 4.0             |
| INITIAL -2   | 10.5           | 3.9             |
| INITIAL -3   | 9.8            | 3.7             |
| INITIAL -4   | 10.3           | 3.8             |
| INITIAL -5   | 10.5           | 3.9             |
| Average  | 10.4           | 3.9             |
| CV   | 3%             | 3%              |
| <b>Reverse Diffusion Samples (2 h with EtO , 6 h air only)</b>         |                |                 |
| Sample Name  | Amt Found (µg) | Amt Found (ppm) |
| REVERSE DIFFUSION -1   | 10.4           | 3.9             |
| REVERSE DIFFUSION -2   | 10.0           | 3.7             |
| REVERSE DIFFUSION -3   | 10.2           | 3.8             |
| REVERSE DIFFUSION -4   | 10.1           | 3.8             |
| REVERSE DIFFUSION -5   | 10.0           | 3.7             |
| REVERSE DIFFUSION -6   | 9.8            | 3.6             |
| Average  | 10.1           | 3.8             |
| CV   | 2%             | 3%              |
| <b>% Difference of Initial Samples and Reverse Diffusion Samples =</b> | <b>2.8%</b>    |                 |

## 7. Analyte Stability (storage post-sampling)

Stability tests have also been conducted with the 3551+ samplers. After a two-hour exposure to ethylene oxide, samplers were collected from the chamber. Five badges were sent the next day for analysis (initial). The remaining samplers were stored in the standard foil return envelope. Three different groups of five samplers each were separated: 1-week and 2-week samples stored at room temperature and 2-week samples stored at -20°C (freezer), as a reference point for storage temperature (freezing should effectively stay the same as initial samples). Two different challenge levels were used for the stability tests. Tables 4 and 5 show stability data for 2- and 4-ppm EtO exposures.

**Table 4.** Stability results for 2-ppm EtO challenge

| <b>Initial Collection</b>                 |                |                 |
|---|----------------|-----------------|
| Sample Name                               | Amt Found (µg) | Amt Found (ppm) |
| 2 PPM INITIAL -1                          | 2.93           | 1.1             |
| 2 PPM INITIAL -2                          | 2.91           | 1.1             |
| 2 PPM INITIAL -3                          | 2.9            | 1.1             |
| 2 PPM INITIAL -4                          | 2.76           | 1.0             |
| 2 PPM INITIAL -5                          | 2.97           | 1.1             |
| Average                                   | 2.9            | 1.1             |
| CV  | 3%             | 4%              |
| <b>1 week - Room Temperature Storage</b>  |                |                 |
| Sample Name                               | Amt Found (µg) | Amt Found (ppm) |
| 2 PPM 1 week -1                           | 2.39           | 0.89            |
| 2 PPM 1 week -2                           | 2.68           | 1.00            |
| 2 PPM 1 week -3                           | 2.64           | 0.99            |
| 2 PPM 1 week -4                           | 2.61           | 0.97            |
| 2 PPM 1 week -5                           | 2.56           | 0.96            |
| Average                                   | 2.6            | 1.0             |
| CV  | 4%             | 4%              |
| <b>2 weeks - Room Temperature Storage</b> |                |                 |
| Sample Name                               | Amt Found (µg) | Amt Found (ppm) |
| 2 PPM 2 week -1                           | 2.6            | 0.97            |
| 2 PPM 2 week -2                           | 2.73           | 1.00            |
| 2 PPM 2 week -3                           | 2.63           | 0.98            |
| 2 PPM 2 week -4                           | 2.55           | 0.95            |
| 2 PPM 2 week -5                           | 2.48           | 0.93            |

|                                  |                |                           |
|----------------------------------|----------------|---------------------------|
| Average                          | 2.6            | 1.0                       |
| CV                               | 4%             | 3%                        |
| <b>2 weeks - Freezer Storage</b> |                |                           |
| Sample Name                      | Amt Found (µg) | Amt Found (ppm)           |
| 2 PPM 2 week -1                  | 2.61           | 0.97                      |
| 2 PPM 2 week -2                  | 2.69           | 1.00                      |
| 2 PPM 2 week -3                  | 2.64           | 0.98                      |
| 2 PPM 2 week -4                  | 2.72           | 1.00                      |
| 2 PPM 2 week -5                  | 2.63           | 0.98                      |
| Average                          | 2.7            | 1.0                       |
| CV                               | 2%             | 1%                        |
| <b>Loss upon storing</b>         |                |                           |
|                                  | Average (µg)   | % Difference from Initial |
| Initial                          | 2.9            |                           |
| 1 Week - RT                      | 2.6            | <b>-10%</b>               |
| 2 Weeks - RT                     | 2.6            | <b>-10%</b>               |
| 2 Weeks - Freezer                | 2.7            | <b>-7%</b>                |

**Table 5.** Stability results for 4-ppm EtO challenge

|  |                |                 |
|--|----------------|-----------------|
| Sample Name                              | Amt Found (µg) | Amt Found (ppm) |
| 4 PPM INITIAL -1                         | 10.7           | 4.0             |
| 4 PPM INITIAL -2                         | 10.5           | 3.9             |
| 4 PPM INITIAL -3                         | 9.83           | 3.7             |
| 4 PPM INITIAL -4                         | 10.3           | 3.8             |
| 4 PPM INITIAL -5                         | 10.5           | 3.9             |
| Average                                  | 10.4           | 3.9             |
| CV                                       | 3%             | 3%              |
| <b>1 week - Room Temperature Storage</b> |                |                 |
| Sample Name                              | Amt Found (µg) | Amt Found (ppm) |
| 4 PPM 1 week -1                          | 9.71           | 3.6             |
| 4 PPM 1 week -2                          | 9.22           | 3.4             |

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|   |                |                           |
|---|----------------|---------------------------|
| 4 PPM 1 week -3                           | 9.63           | 3.6                       |
| 4 PPM 1 week -4                           | 9.6            | 3.6                       |
| 4 PPM 1 week -5                           | 9.53           | 3.6                       |
| Average                                   | 9.5            | 3.6                       |
| CV  | 2%             | 3%                        |
| <b>2 weeks - Room Temperature Storage</b> |                |                           |
| Sample Name                               | Amt Found (µg) | Amt Found (ppm)           |
| 4 PPM 2 week -1                           | 9.69           | 3.6                       |
| 4 PPM 2 week -2                           | 9.48           | 3.5                       |
| 4 PPM 2 week -3                           | 9.55           | 3.6                       |
| 4 PPM 2 week -4                           | 9.48           | 3.5                       |
| 4 PPM 2 week -5                           | 9.23           | 3.4                       |
| Average                                   | 9.5            | 3.5                       |
| CV  | 2%             | 2%                        |
| <b>2 weeks - Freezer Storage</b>          |                |                           |
| Sample Name                               | Amt Found (µg) | Amt Found (ppm)           |
| 4 PPM 2 week -1                           | 8.81           | 3.3                       |
| 4 PPM 2 week -2                           | 9.76           | 3.6                       |
| 4 PPM 2 week -3                           | 9.18           | 3.4                       |
| 4 PPM 2 week -4                           | 9.24           | 3.4                       |
| 4 PPM 2 week -5                           | 9.45           | 3.5                       |
| Average                                   | 9.3            | 3.4                       |
| CV  | 4%             | 3%                        |
| <b>Loss upon storing</b>                  |                |                           |
|   | Average        | % Difference from Initial |
| Initial                                   | 10.4           |                           |
| 1 Week - RT                               | 9.5            | <b>-8%</b>                |
| 2 Weeks - RT                              | 9.5            | <b>-8%</b>                |
| 2 Weeks - Freezer                         | 9.3            | <b>-10%</b>               |

As it can be seen, the loss for up to 2 weeks of storage at room temperature is, on average, 9%.



## 8. Summary Comments

Sampler 3551+ has been evaluated for sampling ethylene oxide. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at  $\leq 25\%$  at PEL;  $\leq 35\%$  at STEL and action level.

|                     |                                       |
|---------------------|---------------------------------------|
| Concentrating Range | 0.1 - 2.0 times the NIOSH REL of 2ppm |
| Sampling Time       | 15 min - 8 hr                         |
| Air Velocity        | 15 - 150 cm/sec                       |
| Temperature         | 0 - 50°C                              |
| Humidity            | 10 - 80% RH                           |

The recommended maximum holding time after sampling is 14 days at room temperature.

It is recommended that 3551+ samplers be used within the envelope of conditions specified above, but, in general, minor excursions outside these limits would be expected to have only minor effects. Longer or shorter sampling times are possible but have not been evaluated.

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