

## Laboratory Validation of AT575 Nitrous Oxide Vapor Sampler

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

### AT575 Nitrous Oxide Vapor Sampler

Assay Technology's 575 nitrous oxide (N<sub>2</sub>O) sampler consists of activated molecular sieves encased within a 4-port polypropylene sampling grid fitted with a polyester screen and contained within a polypropylene sampler body.

The 5A activated molecular sieves allow only molecules with a diameter less than 5 Angstroms to pass through. The 4-port sampling grid allows for a sensitive detection limit without overloading the media (capacity). The use of molecular sieves to trap nitrous oxide and subsequent gas chromatography analysis has been reported previously.<sup>1</sup> Nitrous oxide collected on the media is desorbed with water in a vial. The headspace of the vial, containing the desorb nitrous oxide gas, is then analyzed by gas chromatography with an electron capture detector (GC/ECD; AT SOP L575).

#### 1. Test Apparatus & Method

Exposures of nitrous oxide vapor were generated by dynamic dilution from a gas cylinder containing 10.0% nitrous oxide in ammonia. The analyte concentration was delivered into the air stream from the cylinder as at fixed rate via a mass flow controlled (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 the exposure concentrations were monitored by collecting gas samples from the chamber throughout testing; samples were direct-injected into the GC/ECD for immediate analysis. An average of the collected gas samples was used as a reference value. Diffusive samplers were analyzed according to the in-house ATSOP L575 method.

#### 2. Desorption Efficiency (DE)

Analyte recovery and desorption efficiency were determined by analysis of diffusive samplers spiked by a nitrous oxide gas concentration. Samplers were tested at three spike levels in quadruplicate.

The DE determined for AT575 samplers was 67.6%

#### 3. Determination of the Effect of Concentration and Time on Sampling Rate (verification of diffusive sampling rate)

Giving consideration to the overall capacity of the molecular sieves in the sampler, efforts were made to design the sampler with a low sampling rate, without sacrificing detection sensitivity in the analysis. Assay Technology employs the use of several polypropylene grids in different samplers. By selecting a grid with only 4 holes, the sampling rate was found to be less than 1 mL/min, thereby giving the AT575 sampler the ability to sample for long periods of time without concern of overloading the capacity of the sieves (Table 1).

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<sup>1</sup> \*Miles D. LaHue, Herman D. Axelrod, James P. Lodge. *Anal. Chem.*, **1971**, 43 (8), pp 1113–1115.

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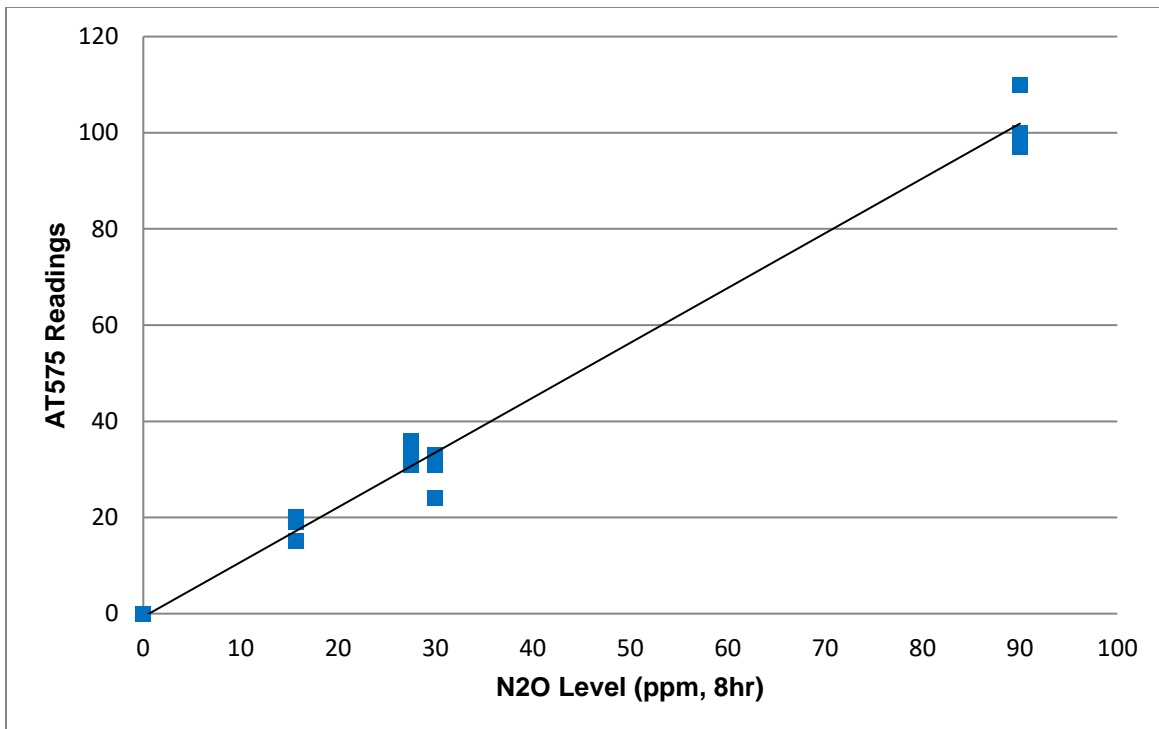
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**Table 1.** Demonstration of sampling capacity using different grids

| No. of Sampling Ports | Sample Capacity ( L of air) | Sampling Rate (L/min) | Effective Sampling Time ( min ) | Effective Sampling Time ( day ) |
|-----------------------|-----------------------------|-----------------------|---------------------------------|---------------------------------|
| 76                    | 5                           | 0.016                 | 313                             | 0.22                            |
| 19                    | 5                           | 0.005                 | 1250                            | 0.87                            |
| 4                     | 5                           | 0.0008                | 6250                            | 4.34                            |

Once the 4-port grid was selected for use in the nitrous oxide sampler, experiments were conducted to evaluate performance over a range of concentrations (Figure 1).



**Figure 1.** N<sub>2</sub>O exposure data at three concentration levels.

Verification of badge performance and sampling rates was conducted in 2014 with additional tests. Two levels of nitrous oxide were used for chamber studies. The results showed accordance with the established sampling rate and previous sampling data (Table 2).

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**Table 2.** N<sub>2</sub>O exposure data at two concentration levels, 2014 reverification study

### Exp A - Reference concentration = 98 ppm

| Sample Description | Amt Foun (ug) | Volume (L) | Time (min)     | Concentration (ppm) |
|--------------------|---------------|------------|----------------|---------------------|
| Exp A-100 -1       | 19            | 0.106      | 120            | 98                  |
| Exp A-100 -2       | 20            | 0.106      | 120            | 100                 |
| Exp A-100 -3       | 18            | 0.106      | 120            | 95                  |
| Exp A-100 -4       | 19            | 0.106      | 120            | 98                  |
| Exp A-100 -5       | 19            | 0.106      | 120            | 100                 |
|                    |               |            | Average        | 98.2                |
|                    |               |            | CV             | 2%                  |
|                    |               |            | % of Reference | 0.2%                |

### Exp B - Reference Concentration = 231 ppm

| Sample Description | Amt Foun (ug) | Volume (L) | Time (min)     | Concentration (ppm) |
|--------------------|---------------|------------|----------------|---------------------|
| Exp B-200 - 1      | 33            | 0.106      | 120            | 170                 |
| Exp B-200 - 2      | 38            | 0.106      | 120            | 200                 |
| Exp B-200 - 3      | 41            | 0.106      | 120            | 226                 |
| Exp B-200 - 4      | 44            | 0.106      | 120            | 260                 |
| Exp B-200 - 5      | 32            | 0.106      | 120            | 170                 |
|                    |               |            | Average        | 205.2               |
|                    |               |            | CV             | 19%                 |
|                    |               |            | % of Reference | 11%                 |

#### 4. Background (Blank) Determination

Unexposed samplers were analyzed by Method AT L575 to determine background analyte levels (if any) on the sampler prior to sampling. No background response was detectable (< 0.4 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 on sampling rate in the range 0 – 50°C. Because the chosen molecular sieve size for this application is large enough to collect water, a more humid sampling environment could potentially affect the sampling rate/capacity of the 575 sampler. In order to verify that the sampler could function as claimed at a higher humidity, a study was conducted.

In this experiment, four samplers were opened in the acrylic test chamber while air humidified to 80% RH was passed through the chamber at 60 LPM. The samplers were allowed to condition for 7 h, then were exposed to a nitrous oxide concentration for 1 h. Fresh, unexposed badges were placed in the chamber for the 1-hour test as a reference set. The times were chose to simulate a normal 8-hour work shift in a humid environment. The results are shown below in Table 3.

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**Table 3.** Results from humidity experiments on AT575 samplers; 7 h preconditioning followed by 1 h testing

| Sample Description         | Amt Found ( $\mu\text{g}$ ) | Volume (L) | Time (min) | Concentration (ppm) |
|----------------------------|-----------------------------|------------|------------|---------------------|
| Humidity Exp-Conditioned-1 | 7.2                         | 0.0528     | 60         | 76                  |
| Humidity Exp-Conditioned-2 | 5.6                         | 0.0528     | 60         | 59                  |
| Humidity Exp-Conditioned-3 | 7                           | 0.0528     | 60         | 74                  |
| Humidity Exp-Conditioned-4 | 5.8                         | 0.0528     | 60         | 61                  |
| Average                    |                             |            |            | 67.5                |
| CV                         |                             |            |            | 13%                 |
| Humidity Exp -New-1        | 6.1                         | 0.0528     | 60         | 64                  |
| Humidity Exp -New-2        | 5.2                         | 0.0528     | 60         | 55                  |
| Humidity Exp -New-3        | 7.3                         | 0.0528     | 60         | 77                  |
| Humidity Exp -New-4        | 6.5                         | 0.0528     | 60         | 68                  |
| Average                    |                             |            |            | 66                  |
| CV                         |                             |            |            | 14%                 |
| % difference               |                             |            |            | 2%                  |

As can be seen from the data, although humidity could affect sampler performance, preconditioned badges were found to sample with an average of 2% of badges that were not exposed to excess moisture.

### 6. Reverse Diffusion

One consideration in using passive samplers is the reverse diffusion of the analyte off of the media over the sampling period. In a reverse diffusion chamber test, some samplers are left sampling beyond the gas challenge, while others are collected and stored immediately. The extent to which the samplers being tested for signs of reverse diffusion match the initial samples collected is the basis for evaluation. For the AT575 samplers, an experiment was done where 14 samplers were exposed to a nitrous oxide concentration for 1 h. Seven samplers were collected immediately following the exposure and seven were allowed to remain open while clean air controlled by the Miller-Nelson instrument flowed through the chamber for an additional 7 h to simulate a badge being worn for a full 8-h work shift. All the badges were then analyzed. The results can be seen in Table 4.

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**Table 4.** Results from reverse diffusion experiment on AT575; 1 h test followed by 7 h clean air for reverse diffusion samples

| Sample Description | Amount found ( $\mu\text{g}$ ) | Concentration (ppm) |
|--------------------|--------------------------------|---------------------|
| 575 Initial - 1    | 3.1                            | 33                  |
| 575 Initial - 2    | 3                              | 32                  |
| 575 Initial - 3    | 3.3                            | 34                  |
| 575 Initial - 4    | 3.1                            | 32                  |
| 575 Initial - 5    | 3.3                            | 35                  |
| 575 Initial - 6    | 3.8                            | 40                  |
| 575 Initial - 7    | 3.6                            | 38                  |
| Average            | 3.3                            | 35                  |
| CV                 | 9%                             | 9%                  |

| Sample Description    | Amount found ( $\mu\text{g}$ ) | Concentration (ppm) |
|-----------------------|--------------------------------|---------------------|
| 575 Rev Diffusion - 1 | 3                              | 31                  |
| 575 Rev Diffusion - 2 | 3                              | 31                  |
| 575 Rev Diffusion - 3 | 3.5                            | 37                  |
| 575 Rev Diffusion - 4 | 3.2                            | 33                  |
| 575 Rev Diffusion - 5 | 3.7                            | 39                  |
| 575 Rev Diffusion - 6 | 4                              | 42                  |
| 575 Rev Diffusion - 7 | 3.8                            | 40                  |
| Average               | 3.5                            | 36                  |
| CV                    | 12%                            | 12%                 |

As the data shows, there is not significant reverse diffusion of nitrous oxide off the molecular sieves once collected during sampling.

### 7. Summary Comments

Sampler AT575 has been evaluated for sampling nitrous oxide. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at  $\leq 25\%$

|                  |                          |
|------------------|--------------------------|
| Sample Capacity: | 70 ppm for 8-hour sample |
| Sampling Time    | 1 – 8 hours              |
| Air Velocity     | 15 – 150 cm/sec          |
| Temperature      | 0 – 50°C                 |
| Humidity         | 10 – 80% RH              |

The samplers must be returned to the laboratory for analysis within one week of sampling, sealed in the return container and return envelope provided by Assay Technology.

It is recommended that AT575 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.