Laboratory Validation of AT575 Nitrous Oxide Vapor Sampler

Prepared by: C.R. Manning, PhD, CIH & B Quarles, PhD Date: 5 June 2017
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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_2O) 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.[1] 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).

[1] *Miles D. LaHue, Herman D. Axelrod, James P. Lodge. *Anal. Chem.*, **1971**, *43* (8), pp 1113–1115.

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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)	(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).

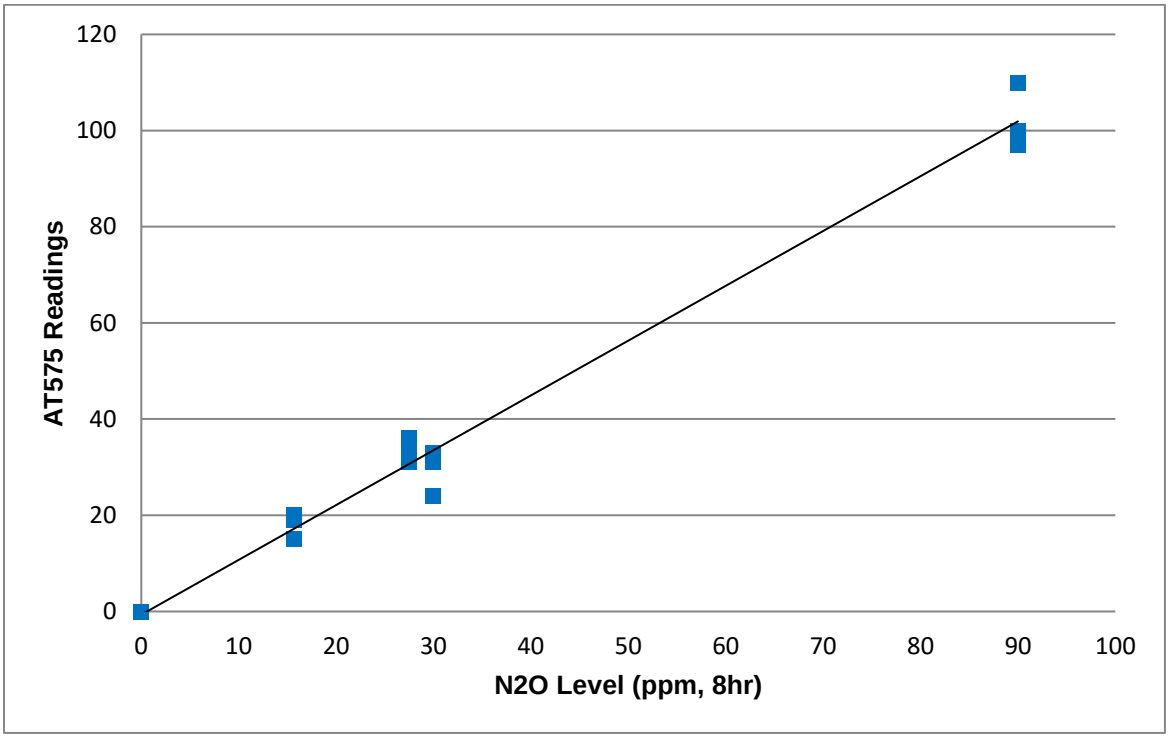

Figure 1. N_2O exposure data at three concentration levels.

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_2O 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

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for the 1-hour test as a reference set. The times were chosen to simulate a normal 8-hour work shift in a humid environment. The results are shown below in Table 3.

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

Sample Description	Amt Found (μg)	Volume (L)	Time (min)	Concentration (ppm)
Humidity Exp-Condiitoned-1	7.2	0.0528	60	76
Humidity Exp-Condiitoned-2	5.6	0.0528	60	59
Humidity Exp-Condiitoned-3	7	0.0528	60	74
Humidity Exp-Condiitoned-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 (μg)	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 (μg)	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. Storage Studies

Storage studies were undertaken to evaluate the best conditions for storing exposed samplers to ensure no analyte is lost during storage and prior to analysis. Because nitrous oxide is such a small and volatile gas, a silicone seal wafer was added to the 575AT badge casing to produce a better seal.

Molecular sieves were spiked with known amounts of nitrous oxide and allowed to sit in a closed, glass vial for 30 minutes. The spiked nitrous oxide molecular sieves were then transferred to a 575AT badge casing and properly assembled to resemble a standard 575AT monitor. The spiked samplers were stored at both room temperature (RT), in the freezer (-18 °C) and analyzed at 1 and 2 week intervals. A set of badges were analyzed immediately after spiking to serve as reference values.

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Recovery of the spiked badges was at or above 100% for both RT and freezer storage. A summary of the findings can be found in table 5.

Table 5. Results from storage study of spiked 575AT monitors at room temperature and freezer conditions.

Holding Time	Temperature	Avg Qty ug/mL	% Recovery
Initial	RT	1.25	-
1 week	RT	1.25	100
1 week	-18 °C	1.26	101
2 weeks	RT	1.25	100
2 weeks	-18 °C	1.29	103

8. 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 should be returned to the laboratory for analysis within two weeks of sampling in the foil pouch. 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.