

Laboratory Validation of AT584 Ammonia Sampler

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AT584 Ammonia Vapor Sampler

Assay Technology's 584 ammonia vapor sampler consists of a fiberglass wafer treated with sulfuric acid encased within a 76-port polypropylene sampling grid and contained within a polypropylene sampler body.

In an acid-base reaction between ammonia and sulfuric acid, ammonium ion is collected on the wafer, which can be recovered and analyzed via a modified OSHA 188 method using an ion selective electrode (ISE).

1. Test Apparatus & Method

Vapor exposures of ammonia were created by dynamic dilution from a gas cylinder containing 5.0% ammonia in nitrogen. The analyte concentration was delivered into the air stream 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 active sampling tubes (silica gel treated with sulfuric acid), sampled from locations in the chamber bracketing the samplers under test.

2. Desorption Efficiency (DE)

Desorption efficiency (analyte recovery) was determined by analysis (Method AT584) of sulfuric acid-treated wafers spiked from ammonia-nitrogen standard solutions. Samplers were tested at spike levels corresponding to expected levels of exposure near to the OSHA PEL of 1.0 ppm.

The desorption efficiency calculated from this test was 88%.

3. Verification of Diffusive Sampling Rate

Per internal quality specifications, samplers are evaluated periodically to assess performance and to compare to reference sampling tubes. The degree to which results for the AT584 samplers agree with results for the reference tubes is the essence of the evaluation.

In the most recent laboratory chamber testing protocol of AT584 samplers for verification of diffusive sampling rate, the samplers show agreement within an average of $\pm 3\%$ of reference tube samples. The tests were conducted as described in Section 1, using two separate chamber exposures at two different concentrations, corresponding to an 8-hr TWA of 0.5 and 1 times the OSHA PEL (50 ppm). Table 1 shows the results from the study.

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Table 1. 2016 evaluation of AT584 samplers compared to reference tubes at two concentration levels

Sample Description	Chemical Analyte	Qty (ug)	Volume (L)	Time (min)	Concn (ppm)
Level A Tube-1	AMMONIA	341	2.3	120	210
Level A Tube-2	AMMONIA	308	2.28	120	190
Level A Tube-3	AMMONIA	265	2.06	120	180
Average					193
CV					8%
Sample Description	Chemical Analyte	Qty (ug)	Volume (L)	Time (min)	Concn (ppm)
10B15 - LL5348	AMMONIA	442	3.46	120	180
10B15 - LL5245	AMMONIA	482	3.46	120	200
10B15 - LL5276	AMMONIA	456	3.46	120	190
10B15 - LL5933	AMMONIA	460	3.46	120	190
10B15 - LL5568	AMMONIA	467	3.46	120	190
Average					190
CV					4%
Variance vs Sampling Tube					2%
Sample Description	Chemical Analyte	Qty (ug)	Volume (L)	Time (min)	Concn (ppm)
Level B Tube-1	AMMONIA	126	2.3	120	79
Level B Tube-2	AMMONIA	132	2.28	120	83
Level B Tube-3	AMMONIA	126	2.06	120	87
Average					83
CV					5%
10B15 - LL5425	AMMONIA	218	3.46	120	91
10B15 - LL5892	AMMONIA	223	3.46	120	92
10B15 - LL5154	AMMONIA	194	3.46	120	80
10B15 - LL5490	AMMONIA	189	3.46	120	79
10B15 - LL5530	AMMONIA	204	3.46	120	85
Average					85.4
CV					7%
Variance vs Sampling Tube					-3%

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4. Background (Blank) Determination

Unexposed samplers were analyzed by Method AT584 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. Analyte Stability (storage post-sampling)

Stability tests have also been conducted with the AT584 samplers. After a two-hour exposure to ammonia, 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 -10°C (freezer), as a reference point for storage temperature (freezing should effectively be the same as samples sent immediately for analysis). Two different challenge levels were used for the stability tests. Table 2 shows stability data on the AT584 samplers from 2016.

Sample Description	Chemical Analyte	Qty (ug)	Concentration (ppm)	% of Initial
Level A - Initial Average	Ammonia	461	190	
Level A - Avg 1 week storage at RT	Ammonia	417	172	91%
Level A - Avg 2 week storage at RT	Ammonia	455	190	100%
Level A - Avg 2 week storage at -10oC	Ammonia	447	184	97%
Sample Description	Chemical Analyte	Qty (ug)	Concentration (ppm)	% of Initial
Level B - Initial Average	Ammonia	206	85	
Level B - Avg 1 week storage at RT	Ammonia	218	91	106%
Level B - Avg 2 week storage at RT	Ammonia	238	98	115%
Level B - Avg 2 week storage at -10oC	Ammonia	244	100	117%

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As can be seen from the results, once ammonia has been collected on the AT584 sampler, it is stable over a two-week hold period. This validates that recommendation that samples may be stored at room temperature after sampling and returned to the lab up to two weeks after sampler use.

7. Summary Comments

Sampler AT584 has been evaluated for sampling ammonia. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at $\leq 25\%$ at PEL; $\leq 25\%$ at STEL and action level.

Sampling Time	15 minutes – 8 hours
Air Velocity	15-150 cm/sec
Temperature	0 – 50°C
Humidity	10 – 80% RH

For maximum shelf life, the product should be stored under refrigerated conditions, but does *not* need to be stored under refrigerated conditions after sampling.

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

It is recommended that AT584 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.