Laboratory Validation of AT545 Methanol Sampler

Prepared by: MDR Peralta, PhD Date: March 2022

AT545 Methanol Sampler

Assay Technology's 545 methanol (MeOH) sampler consists of bead shaped activated carbon encased within a 19-port polypropylene sampling grid fitted with a polyester screen and contained within a polypropylene sampler body.

The activated carbon absorbs MeOH which is extracted with a dimethyl formamide (DMF) and carbon disulfide (CS_2) mixture and analyzed by gas chromatography with a flame ionization detector (GC-FID). The 19-port sampling grid allows for air monitoring of methanol without overloading the media (capacity). The use of bead shaped activated carbon and subsequent gas chromatography analysis by a DMF/CS_2 mixture has been reported previously.[1]

1. Test Apparatus & Method

Vapor exposures of MeOH were created by dynamic dilution from a liquid phase containing the pure analyte. The liquid analyte was injected into a heated round bottom containing a flowing stream of air at a fixed rate via syringe pump (Harvard), then dynamically mixed with flow-controlled input air provided by the Miller-Nelson 501 atmosphere conditioner. The controlled mixture was passed through an inert acrylic chamber containing diffusive samplers under test. Flows were verified by calibration, and exposure concentrations monitored via a fourier transform infrared spectrometer (FTIR) and verified by charcoal tube samplers mounted in the chamber and bracketing the samplers under test. Active and diffusive samplers were desorbed using a 70% DMF/ 30% CS_2 solvent mixture and analyzed by GC-FID, using a similar method to OSHA Method 5001 V1.2.

2. Desorption Efficiency (DE)

Desorption efficiency (analyte recovery) was determined by spiking quadruplicate media samples at three different levels and measured over two columns on a GC-FID. The DE for AT545 was found to be 107.1% Data from the DE analysis can be found in Table 1.

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Table 1. Desorption efficiency data for AT545 monitors

	Column 1		Column 2	
Spike Level 1	Amt Recovered (ug/mL)	DE	Amt Recovered (ug/mL)	DE
Liquid Spike (no media)	30.42		31.72	
Spike 1	33.76	111.0%	35.07	110.5%
Spike 2	34.73	114.2%	36.27	114.3%
Spike 3	32.32	106.3%	34.00	107.2%
Spike 4	34.09	112.0%	35.83	113.0%
	Spike Level Average DE:	110.9%	Spike Level Average DE:	111.3%
Spike Level 2	Amt Recovered (ug/mL)	DE	Amt Recovered (ug/mL)	DE
Liquid Spike (no media)	85.02		85.15	
Spike 1	85.60	100.7%	86.47	101.5%
Spike 2	91.79	108.0%	93.54	109.9%
Spike 3	82.18	96.7	84.82	99.6%
Spike 4	92.00	108.2%	94.11	110.5%
	Spike Level Average DE:	103.4%		105.4%
Spike Level 3	Amt Recovered (ug/mL)	DE	Amt Recovered (ug/mL)	DE
Liquid Spike (no media)	134.45		131.74	
Spike 1	128.80	95.8%	130.00	98.7%
Spike 2	139.67	103.9%	144.42	109.6%
Spike 3	145.36	108.1%	148.12	112.4%
Spike 4	142.32	105.9%	147.42	111.9%
	Spike Level Average DE:	103.4%		108.2%

AT545 DE	107.1%

3. Verification of Diffusive Sampling Rate

Methanol exposures were performed as described in Section 1. Sampling rates were determined from evaluation of AT545 samplers compared to reference samples of charcoal tubes. Exposures were applied to samplers in the vicinity 0.5x of the OSHA permissible exposure limit (PEL) for four hours. Results are reported in Table 2. The sampling rate for MeOH was found to be 4.5 mLPM.

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Table 2. Data for sampling rate verification

Sample ID	Sample Description	Sampling Time	Reference Concentration (sampling tubes)	MeOH Found on Sampler	Concentration Found by Badge	Comparison to Ref Value
	(ppm)	(min)	(ppm)	(µg)	(ppm)	(% Recovery)
21041198	AT545	240	61.5	86.6	61.2	99%
21041199	AT545	240	61.5	83.1	58.7	95%
21041200	AT545	240	61.5	94.4	66.7	108%
21041201	AT545	240	61.5	89.2	63.0	102%
21041202	AT545	240	61.5	88.4	62.5	102%
21041203	AT545	240	61.5	84.5	59.7	97%
				Avg:	62.0	101%
				CV:	5%	5%

4. Background (Blank) Determination

Unexposed samplers were analyzed to determine background analyte levels (if any) on the sampler prior to sampling. No significant background peaks were detected during a quality control assessment, all levels were below our QC requirement of 3 µg.

5. Atmospheric Effects

Air Velocity & Orientation – Previous studies demonstrated that there is no significant effect of air velocity and orientation on sampling rate for this specific monitor design.

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 for this specific monitor design.

6. Bias Due to Reverse Diffusion

Samplers were subject to an exposure pulse in the vicinity of the OSHA PEL for 30 minutes at 80%RH; approximately 6% of the recommended sampling time (RST). A set of 6 samplers were then removed and labeled as reference samplers. The remaining samplers were subject to a zero exposure period (ZEP) for the duration of the RST, 7.5 hrs. The reference samplers were stored at -20 °C for one day followed by analysis. Recovery of analyte from the samplers subject to the ZEP were compared with recovery of analyte from the reference samplers. Any difference between these two recoveries is taken as the extent of reverse diffusion. Both the exposure pulse and ZEP were performed at 80%RH.

The samplers subject to the ZEP of 7.5 hours had an average recovery of 86% of the reference samplers. Results are shown in Table 3.

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Table 3. Assessment of Reverse Diffusion

Sample ID	Sample Description	Sampling Time	MeOH Found on Sampler	Comparison to Ref Value
	(ppm)	(min)	(µg)	(% Recovery)
21041218	Reference Sampler	30	30.2	
21041219	Reference Sampler	30	31.6	
21041220	Reference Sampler	30	30.3	
21041221	Reference Sampler	30	32	
21041222	Reference Sampler	30	31.8	
21041223	Reference Sampler	30	29.6	
		Avg:	30.9	
		CV:	3%	
21041224	7.5 hr at Zero Exposure	480	26.5	86%
21041225	7.5 hr at Zero Exposure	480	26.9	87%
21041226	7.5 hr at Zero Exposure	480	24.6	80%
21041227	7.5 hr at Zero Exposure	480	26.8	87%
21041228	7.5 hr at Zero Exposure	480	28.4	92%
21041229	7.5 hr at Zero Exposure	480	25.8	83%
		Avg:	22.2	86%
		CV:	6%	5%

7. Capacity

Sample capacity was determined by exposing the media to a known amount of MeOH under controlled conditions and evaluating the airstream downstream of the media. Once 10% of the PEL of MeOH was detected downstream, it was determined the media had reached its capacity; no longer able to absorb MeOH. For the AT545 monitor, it was experimentally determined to have a MeOH capacity of 18.7 mg.

8. Storage Studies

Storage studies were undertaken to evaluate the best conditions for storing exposed samplers so as to ensure no analyte is lost during storage and prior to analysis. Sets of samplers were exposed to a high and low level of MeOH and stored at various temperatures and time periods: 1-week at room temperature (RT) and 2-weeks at RT. A set of samplers stored at -20 °C and analyzed within 2 days of initial exposure were used as reference samplers to which stored samplers could be compared against.

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Both set of stored samplers showed a MeOH recovery greater than 90% compared to the reference samplers, indicating no MeOH was lost during storage at RT. Results are shown in table 4 and are an average of 6 samplers per condition.

Table 4. Assessment of Storage Data over 2 weeks at Room Temperature: Average quantity recovered and % of initial collection.

Methanol Low Level

Holding Time	Avg Qty (ug, Avg)	% Recovery
Initial	29.1	
1 week - RT	28.3	97%
2 weeks - RT	30.0	103%

Methanol High Level

Holding Time	Avg Qty (ug)	% Recovery
Initial	672.5	
1 week – RT	675.8	100%
2 weeks - RT	643.0	96%

9. Summary Comments

Sampler AT545 has been evaluated for sampling methanol. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at ≤25% at the PEL.

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

The recommended maximum holding time after sampling is two weeks at room temperature.

It is recommended that AT545 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.

References

1. OSHA Method 5001. Organic Vapor Sampling Group 2 (OVSG-2) Alcohol Analytes Collected on Synthetic Charcoal Sorbent Tubes. OSHA, June 2021, Version 1.2.