

Laboratory Validation of AT592 Acrolein Sampler

Prepared by: C.R. Manning, PhD, CIH & B Quarles, PhD

Date: 12 June 2017

AT592 Acrolein Sampler

Assay Technology's 592 acrolein (propenal) sampler consists of a silica gel disc treated with 2-hydroxymethylpiperidine (HMP) encased within a 76-port polypropylene sampling grid and contained within a polypropylene sampler body.

The 2-hydroxymethylpiperidine reacts with acrolein to form a derivative. The derivative can be recovered via toluene extraction and then analyzed by a modified OSHA 52 method using gas chromatography with a nitrogen phosphorus detector (GC/NPD).

The use of HMP to collect acrolein vapor and analysis by GC/NPD has been the preferred method for acrolein sampling (OSHA 52 and NIOSH 2501). The AT592 sampler takes advantage of the HMP/acrolein chemistry in the form of a passive sampler, compared to active sampling sorbent tubes.

1. Test Apparatus & Method

Vapor exposures were created by dynamic dilution of acrolein in air. The analyte was injected into a flowing stream of air at a fixed rate via a syringe pump, and was 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 were verified by sampling tubes mounted in the chamber, bracketing the samplers under test. All tubes and samplers were analyzed by the modified OSHA 52 method.

2. Desorption Efficiency (DE)

Desorption efficiency (analyte recovery) was determined by spiking quadruplicate wafers at three different levels. The DE for AT592 was found to be 90.1% (Table 1).

Spike Level 1	Amt Recovered (ug)	DE
Liquid Spike (no media)	0.27	
Spike 1	0.27	100.8%
Spike 2	0.27	101.9%
Spike 3	0.26	98.1%
Spike 4	0.26	99.2%
	Spike Level Average DE:	100.0%
Spike Level 2	Amt Recovered (ug)	DE
Spike Level 2 Liquid Spike (no media)	Amt Recovered (ug) 0.59	DE
Spike Level 2 Liquid Spike (no media) Spike 1	Amt Recovered (ug) 0.59 0.52	DE 87.5%
Spike Level 2 Liquid Spike (no media) Spike 1 Spike 2	Amt Recovered (ug) 0.59 0.52 0.51	DE 87.5% 86.6%
Spike Level 2 Liquid Spike (no media) Spike 1 Spike 2 Spike 3	Amt Recovered (ug) 0.59 0.52 0.51 0.51	DE 87.5% 86.6% 86.0%
Spike Level 2 Liquid Spike (no media) Spike 1 Spike 2 Spike 3 Spike 4	Amt Recovered (ug) 0.59 0.52 0.51 0.51 0.51 0.51	DE 87.5% 86.6% 86.0% 86.3%

Table 1. Desorption Efficiency data for AT592 wafers



Laboratory Validation of AT592 Acrolein Sampler

Prepared by: C.R. Manning, PhD, CIH & B Quarles, PhD

Date: 12 June 2017

Spike Level 3	Amt Recovered (ug)	DE
Liquid Spike (no media)	1.17	
Spike 1	1.02	86.5%
Spike 2	1.00	84.8%
Spike 3	1.00	85.1%
Spike 4	0.92	78.7%
	Spike Level Average DE:	83.8%
	AT592 DE	90.1%

3. Verification of Diffusive Sampling Rate

Per internal quality specifications, samplers are evaluated periodically to assess performance and to compare to reference samples. The degree to which results for the AT592 samplers agree with results for concurrently sampled reference tubes is the essence of the evaluation.

In a 2016 laboratory chamber study of AT592 samplers for verification of diffusive sampling rate, the samplers show agreement within an average of approximately 15%. The test was conducted as described in Section 1. Table 2 shows the results from the study.

_	Sample ID	Analyte	Amt Found (µg)	Volume (L)	Time (min)	Concentration (ppm)
	AT592-1	ACROLEIN	13.6	0.642	75	9.2
	AT592-2	ACROLEIN	14.1	0.642	75	9.6
	AT592-3	ACROLEIN	13.4	0.642	75	9.1
	AT592-4	ACROLEIN	14.1	0.642	75	9.6
	AT592-5	ACROLEIN	14.1	0.642	75	9.6
	AT592-6	ACROLEIN	13	0.642	75	8.8
	AT592-7	ACROLEIN	13.7	0.642	75	9.3
	AT592-8	ACROLEIN	13.5	0.642	75	9.1
					Average	9.3
					CV	3%
					% difference of ref tubes	16%
-	Sample ID	Analyte	Amt Found (µg)	Volume (L)	Time (min)	Concentration (ppm)
	Ref Tube-1	ACROLEIN	27.7	1.43	75	8.4
	Ref Tube-2	ACROLEIN	25.9	1.39	75	000000000000
	Ref Tube-3	ACROLEIN	22.3	1.3	75	7.5
					Average	8.0

Table 2. 2016 data from sampling rate reverification study



Laboratory Validation of AT592 Acrolein Sampler

Prepared by: C.R. Manning, PhD, CIH & B Quarles, PhD

Date: 12 June 2017

4. Background (Blank) Determination

Unexposed samplers were analyzed to determine background analyte levels (if any) on the sampler prior to sampling. No background peaks were detectable ($<0.5 \mu g$).

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 $0 - 50^{\circ}$ C and $10 - 80^{\circ}$ RH.

6. Summary Comments

Sampler AT592 has been evaluated for sampling acrolein. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at \leq 25 % at PEL.

Capacity	80 ppm-hours
Sampling Time	6 – 12 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 two weeks at room temperature.

It is recommended that AT592 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.