

Laboratory Evaluation of AT549 Diffusive Air Sampler using Dynamically-Generated HCFC Test Atmospheres

in accordance with guidelines described in ANSI/ISEA 104-1998 (R2009)

Prepared by: C.R. Manning, PhD, CIH

Date: 1 Mar 2012

These tests for Evaluation of Diffusive Air Samplers were conducted within the guidelines described in ANSI 104-1998 (R2009).

1. Test Apparatus & Method

Vapor exposures of HCFC 134a (1,1,1,2-tetrafluoroethane, CAS 811-97-2), HCFC 245fa (1,1,3,3,3-pentafluoropropane, CAS 460-73-1), and HCFC-1233zd (1E)-1-chloro-3,3,3-trifluoroprop-1-ene, CAS 2730-43-0) were created by dynamic dilution from a pressure bottle containing 100% of the Analyte (as a compressed liquid-gas mixture). The 100% analyte was acquired from the bottle at a fixed rate via mass flow controller (MFC), dynamically mixed with flow-controlled input air provided by the Miller-Nelson 501 atmosphere conditioner, then passed through an inert acrylic chamber containing Diffusive Samplers under test. MFC Flow was verified by calibration, and exposure concentrations were verified by charcoal tube samples continuously drawn from locations in the chamber bracketing the Samplers under test. Active and diffusive samplers were analyzed by Gas Chromatography on 0.32mm x 60 M dual capillary columns (Restek RT-1 and RT-volatiles).

2. De-Sorption Efficiency (DE)

Analyte recovery and de-sorption efficiency determined by analysis (Method AT549) of charcoal wafers "spiked" from standard analyte solutions in methylene chloride (dichloromethane). Samplers were tested at "spike" levels corresponding to expected levels of exposure at 0.5-2 times the OEL (Occupational Exposure Limit).

3. Determination of the Effect of Concentration and Time on Sampling Rate

(Verification of Diffusive Sampling Rate) Samplers were exposed to exposure concentrations in Chambers as described in Section 1, then analyzed by Method AT549. Exposures were applied to Samplers at 0.5-2 times the OEL. Results for the three HCFCs are shown in Figs. 3A, 3B, and 3C, respectively. The average Sampling Rate measured is reported for each HCFC.

4. Bias Due to Reverse Diffusion

Samplers were subject to an Exposure Pulse (> OEL) with duration of 25% of the Recommended Sampling Time (RST). Half of exposed Samplers were then subject to a Zero Exposure Period (ZEP) for the duration (75%) of the RST, while the other half were analyzed immediately (or stored at -20°C until analysis). Recovery of analyte from Samplers subject to the ZEP were compared with recovery of analyte from Samplers analyzed immediately. The difference between these two recoveries is taken as the extent of Reverse Diffusion

For Samplers subject to a Zero Exposure Period of 6 hours, recoveries were with 90-112%, compared to Samplers analyzed immediately after Exposure. Thus, no significant Bias Due to Reverse Diffusion was observed. Results for the 3 HCFCs are shown in Figs. 5A, 5B, and 5C.

5. Background (Blank) Determination

Unexposed Samplers analyzed by Method AT549 to determine background Analyte levels (if any) on the Sampler prior to sampling. No background peaks were detectable (< 0.1 µg).

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6. Effects of Air Velocity & Orientation

Samplers were exposed to atmospheres for 2-4 hrs at 1-2 times the OEL in a Chamber such that linear velocities of 15, 50, and 150 cm/sec, respectively, were generated. Samplers were placed in each zone with *50% of samplers placed normal to and 50% of Samplers perpendicular to the flow direction*. When data from different locations and flows were compared (representing normal air velocity and orientation variation in workplaces), no significant differences were found among the groups indicating the *absence of a significant effect of Air Velocity & Orientation on Sampling Rate*. *This test, performed previously on the Sampler using analytes other than the ones in this study, was not repeated in this study.*

7. Effect of Temperature & Humidity

Samplers were exposed to atmospheres for 2-4 hrs at 1-2 times the OEL in several Chamber runs in which nearly identical exposures were applied with variations in temperature and humidity as follows: 22°C/50%RH, 10°C/50%RH, 30°C/30%RH, 30°C/70% RH. Data from the four conditions (representing normal temperature & humidity variation) showed no significant differences among the groups indicating the *absence of an effect of Temperature & Humidity on Sampling Rate in the range 10-30°C and 30-70% RH*. *This test, performed previously on the Sampler using analytes other than the ones in this study, was not repeated in this study.*

8. Effect of Sampler Storage After Sampling (Analyte Stability)(Holding Time)

Several identical sets of Samplers were exposed as in Section 1 to Analyte concentrations at 0.5-2.0 times the OEL at 20-25°C. One set of Samples was stored at -20°C as a control, then compared to others sets of concomitantly-exposed Samplers after storage at indicated conditions and intervals. HCFC245fa, HCFC134a, and HCFC 1233zd were found to be stable on the AT549 Sampler for a 1 week Holding Time at Room Temperature. Results in Table 8.

9. Sampler Package Integrity

Ethylene Oxide Samplers (Monitor 502) in sealed packaging exposed to >10 ppm ethylene oxide for >2 hours, then analyzed as directed in the Instructions for Use. Results from analysis were not significantly different from results for un-exposed Samplers (blank values) demonstrating the integrity of Sampler packaging. *This result with ethylene oxide (which has highest permeability through plastics and pinholes of all analytes tested) is applicable to all Samplers manufactured by Assay Technology and packaged in its standard aluminum foil pouch.*

10. Summary Comments

Sampler AT549 has been evaluated for sampling HCFC134a, HCFC 245fa, and HCFC 1233zd. The overall system accuracy expressed as Maximum Total Error (95% confidence) is estimated at $\leq 20\%$

Concentration Range	0.1-2.0 times the OEL
Sampling Time	0.25 - 8 hour
Air Velocity	15-150 cm/sec
Temperature	Room Temperature



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Humidity 30-70% RH

Based on estimated Sampler-to-Sampler variation of $\pm 7\%$, Laboratory Variation of $\pm 3\%$, and Exposure Chamber Variation of Error $\pm 5\%$, less than 1% of the Maximum Total Error is attributed to Bias (i.e. systematic error).

It is recommended that Sampler 549 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 here.

The recommended maximum Holding Times after sampling (including shipment to the lab) for HCFC134a (1,1,1,2-tetrafluoroethane), HCFC245fa (1,1,3,3,3-pentafluoropropane) HCFC-1233zd (1-chloro-3,3,3-trifluoroprop-1-ene) are 7 days at room temperature. The Holding Times could possibly be extended if further studies were conducted.

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Fig. 3A
EXPOSURE TEST RESULTS
Effect of Concentration on Sampling Rate
HCFC-245fa (1,1,1,3,3-pentafluoropropane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Badge Amt Found (µg)	Tube Amt Found (µg)	Ref Conc'n (ppm)	Sampling Rate (mL/min)
1120552	3	600	308	9271	694	0.67
1120552	3	300	129	4406	323	0.60
1120552	3	150	78	2605	160	0.62
2010487	3	75	36	254	82	0.67
1120641	6	600	139	5145	368	0.57
2010491	8	600	588	15940	1509	0.59
2010491	4	300	32	48	78	0.61
2002020	8	300	35	307	89	0.60

Mean Sampling Rate = 0.61

Fig. 4A
EXPOSURE TEST RESULTS
Reverse Diffusion (Analyte Loss Due to Overloading)
HCFC-245fa (1,1,1,3,3-pentafluoropropane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Exposure Treatment	Post-Exp Treatment	Badge Amt Found (µg)	% Recovery of Ref (%)
1120641	3	600	Exposed 120 min at 600 ppm	Capped 360 min in Freezer	139	100%
1120641	3	600	Exposed 120 min at 600 ppm	Expose 360 min at Zero Conc'n	155	112%
2002020	3	300	Exposed 120 min at 300 ppm	Capped 360 min in Freezer	39	100%
2002020	3	300	Exposed 120 min at 300 ppm	Expose 360 min at Zero Conc'n	35	91%

Fig. 8A
EXPOSURE TEST RESULTS
Effect of Sampler Storage on Analyte Recovery
HCFC-245fa (1,1,1,3,3-pentafluoropropane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Storage Temp (°C)	Storage Time (days)	Badge Amt Found (µg)	% Recovery of Ref (%)
2010491	3	600	-20	7	566	100%
2010749	3	600	4	14	512	90%
2010484	3	600	20-25	7	609	108%
2010756	3	600	20-25	14	497	88%
2010596	3	300	-20	7	32	100%
2010886	3	300	4	14	29	92%
2010600	3	300	20-25	7	39	122%
2010884	3	300	20-25	14	27	86%

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Fig. 3B
EXPOSURE TEST RESULTS
Effect of Concentration on Sampling Rate
HCFC-134a (1,1,1,2-tetrafluoroethane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Badge Amt Found (µg)	Tube Amt Found (µg)	Ref Conc'n (ppm)	Sampling Rate (mL/min)
1120553	3	500	402	12119	2520	0.84
1120553	3	1000	1713	21265	5040	0.67
1120553	3	2000	3026	38024	7592	0.79
2010294	4	1000	1676	49402	5173	0.64
2010295	4	500	971	6553	2998	0.64
2010490	4	250	341	4048	895	0.75
2010298	4	1000	1845	16023	6960	0.52
2010298	4	1000	963	9707	3403	0.56
2010594	4	500	515	3536	1618	0.63

Mean Sampling Rate = 0.67

Fig. 4B
EXPOSURE TEST RESULTS
Reverse Diffusion (Analyte Loss Due to Overloading)
HCFC-134a (1,1,1,2-tetrafluoroethane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Exposure Treatment	Post-Exp Treatment	Badge Amt Found (µg)	% Recovery of Ref (%)
2010298	3	1000	Exposed 120 min at 1000 ppm	Capped 360 min in Freezer	1845	100%
2010298	3	1000	Exposed 120 min at 1000 ppm	Expose 360 min at Zero Conc'n	1740	94%
2020017	3	500	Exposed 120 min at 500 ppm	Capped 360 min in Freezer	406	100%
2020017	3	500	Exposed 120 min at 500 ppm	Expose 360 min at Zero Conc'n	444	109%

Fig. 8B
EXPOSURE TEST RESULTS
Effect of Sampler Storage on Analyte Recovery
HCFC-134a (1,1,1,2-tetrafluoroethane)

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Storage Temp (°C)	Storage Time (days)	Badge Amt Found (µg)	% Recovery of Ref (%)
2010493	3	1000	-20	7	963	100%
2010748	3	1000	4	14	922	96%
2010486	3	1000	20-25	7	713	74%
2010752	3	1000	20-25	14	706	73%
2010594	3	500	-20	7	515	100%
2010888	3	500	4	14	432	84%
2010599	3	500	20-25	7	502	97%
2010883	3	500	20-25	14	293	57%

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Fig. 3C
EXPOSURE TEST RESULTS
Effect of Concentration on Sampling Rate
HCFC-1233zd [(1E)-1-chloro-3,3,3-trifluoroprop-1-ene]

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Badge Amt Found (µg)	Tube Amt Found (µg)	Ref Conc'n (ppm)	Sampling Rate (mL/min)
2010296	4	1000	2626	77753	5897	0.69
2010813	4	75	13	128	37	0.53
2010873	4	40	11	76	24	0.68
2010297	8	1000	588	77753	1179	0.77
2010970	4	150	18	132	39	0.70
1120639	8	2000	1154	39161	3827	0.66
2020015	4	300	17	305	69	0.38
2010843	4	150	21	234	71	0.46

Mean Sampling Rate = 0.61

Fig. 4C
EXPOSURE TEST RESULTS
Reverse Diffusion (Analyte Loss Due to Overloading)
HCFC-1233zd [(1E)-1-chloro-3,3,3-trifluoroprop-1-ene]

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Exposure Treatment	Post-Exp Treatment	Badge Amt Found (µg)	% Recovery of Ref (%)
2010297	3	1000	Exposed 120 min at 1000 ppm	Capped 360 min in Freezer	595	100%
2010297	3	1000	Exposed 120 min at 1000 ppm	Expose 360 min at Zero Conc'n	582	98%
1120639	3	2000	Exposed 120 min at 2000 ppm	Capped 360 min in Freezer	1135	100%
1120639	3	2000	Exposed 120 min at 2000 ppm	Expose 360 min at Zero Conc'n	1173	103%

Fig. 8C
EXPOSURE TEST RESULTS
Effect of Sampler Storage on Analyte Recovery
HCFC-1233zd [(1E)-1-chloro-3,3,3-trifluoroprop-1-ene]

Lab No.	No. of Samplers	Nominal Exposure (ppm)	Storage Temp (°C)	Storage Time (days)	Badge Amt Found (µg)	% Recovery of Ref (%)
2020013	3	300	-20	7	15	100%
	3	300	4	14	16	101%
2020013	3	300	20-25	7	17	110%
	3	300	20-25	14	17	108%
2020015	3	150	-20	7	18	100%
	3	150	4	14	20	111%
2020015	3	150	20-25	7	21	116%
	3	150	20-25	14	13	73%