Research Report

Validation of Tetrachloroethylene using the
SKC 575-001 Diffusive Sampler

Abstract

A sampling method for Tetrachloroethylene (Perchloroethylene) in air has been validated for concentration levels from 10 to 200 ppm and for exposure times from 7.5 minutes to 12 hours. The 575-001 sampler was desorbed in 2 mL of carbon disulfide and analyzed by gas chromatography with flame ionization detection (FID).

The analytical recovery over the range of 10 to 200 ppm was 100.8 % with a relative standard deviation (RSD) of 3.6 %.

The sampling rate was 13.1 mL/min with an RSD of 4.0 %. This rate was confirmed by the precision and accuracy experiments, which gave a rate of 12.9 mL/min and consisted of 124 sampling results.

Samples can be stored for 14 days at either room or refrigerator temperatures. Temperatures over a range of 10 to 40 c did not affect the sampling rate of tetrachloroethylene.

Validation was conducted as outlined in the NIOSH protocol and in the ANSI/ISEA 104-1998 Standard.

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Background

Tetrachloroethylene (perchloroethylene) is used in dry-cleaning and textile processing. Potential symptoms of overexposure include eye and nose irritation, dizziness, and liver damage. It has an ACGIH and OSHA guideline of 100 ppm based on an 8-h TWA.

The purpose of this study was to validate the 575-001 diffusive sampler for monitoring tetrachloroethylene over a range of 10 to 200 ppm. Critical parameters include analytical recovery, sampling rate and capacity, storage, reverse diffusion, and temperature and humidity effects. Preliminary testing with the SKC diffusive samplers indicates that they can operate over a face velocity range of 10 to 150 cm/sec.

Tetrachloroethylene and carbon disulfide were obtained from Aldrich Chemical Company and were of 99 % purity or higher. A dynamic atmosphere was generated using a syringe pump and filtered air streams to generated various concentrations. The system is described in Appendix A and shown in Figure 1. The atmosphere was fed into an exposure chamber. The diffusive samplers were exposed on a rotating bracket inside the chamber to simulate wind velocity.

Analytical recoveries for the samplers were conducted by injecting a known amount of tetrachloroethylene into the back of each sampler. The samplers were capped, allowed to equilibrate overnight and analyzed the next day to determine the analytical recovery. The tests were conducted at mass loadings equivalent to an 8-h TWA sample based on a calculated sampling rate (13.9 mL/min) at 0.1, 0.5, 1.0, and 2.0 x PEL under dry conditions.

The sampling rate and capacity was conducted at 2 x PEL (200 ppm) for periods from 7.5 minutes up to 12 hours at 80 % RH and 25 c.

The reverse diffusion experiment was conducted at 2 x PEL and at 80 % RH (25 c). In this experiment, 20 samplers were exposed to 4 hours at 200 ppm tetrachloroethylene; 10 of these samplers were taken out for analysis. The other 10 were then left in the chamber and exposed to 0 ppm tetrachloroethylene for the remaining 4 hours.

The storage study consisted of exposing 30 samplers to 100 ppm (1 x PEL) tetrachloroethylene at 80 % RH (25 c) for a 4 hour period. Ten of these samplers were analyzed the next day. Of the remaining 20 samplers, 10 were stored at ambient temperatures and 10 stored in the refrigerator. The length of storage was 14 days.

The factorial experiments are outlined in Table 6. The precision and accuracy calculations are defined by taking data from the following experiments: the 4,6 and 8 hour sampling rate and capacity, the reverse diffusion tests, runs 1, 2, 3, 4, 13, 14, 15, and 16 of the factorial, the storage data, and the temperature experiments.

All diffusive samplers were desorbed in 2 mL of carbon disulfide and shaken on a flatbed shaker for 30 minutes. The extracts were then analyzed by flame ionization detection gas chromatography. A chromatogram is shown in Figure 2. The analytical limit of detection was 5 micrograms per badge.

SKC constantly reviews these data and conducts experiments to provide the most precise sampling rate. The rate published in these validation reports is the correct rate.

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Table 1

Analytical Recovery
Perchloroethylene

PEL	mg spiked	mg recovered	% Recovery
0.1	0.584	0.590	101
		0.581	99.5
		0.584	100
		0.594	98.3
0.5	2.92	2.81	96.1
		2.88	98.6
		2.98	102
		2.79	96.6
1.0	5.86	5.91	101
		5.96	102
		5.82	99.4
		5.82	99.4
2.0	11.7	13.1	112
		11.9	102
		12.1	104
		11.8	101
		Mean	100.8
		Std.Dev.	3.63
		RSD	3.6 %

Table 2

Reverse Diffusion
200 ppm Perchloroethylene

micrograms

	Exposed 4 hours to Analyte Concentration	Exposed 4 hours to Analyte Concentration, and 4 hours to Zero Analyte
	3836	4283
	3843	3868
	3895	4151
	4255	4420
	4354	4268
	4317	3988
	4363	4382
	4177	4259
Mean	4131	4202
SD	233.4	190.5
RSD	5.6 %	4.5 %

Table 3

Sampling Rate and Capacity
200 ppm Perchloroethylene, 80% RH

Time	μg collected	mL/min
0.125	145.6	14.3
	140.3	13.8
	141.5	13.9
	139.3	13.7
0.23	246.2	13.1
	252.2	13.5
	252.8	13.5
	253.3	13.6
0.50	546.1	13.4
	517.9	12.8
	529.8	13.1
	536.4	13.2
1.0	1050	12.9
	1132	13.9
	1143	14.0
	1089	13.4
2.0	2028	12.5
	2063	12.7
	2127	13.1
	2137	13.1
4.0	4210	12.9
	3866	11.9
	4142	12.8
	4106	12.6
6.0	6202	12.7
	5900	12.0
	6131	12.5
	6088	12.5
8.0	8064	12.4
	8287	12.8
	8398	12.9
	8397	12.9

Table 3 (con't)

Sampling Rate and Capacity

Time	μg collected	mL/min
10	10,517	12.9
	10,572	13.0
	10,553	13.0
	10,917	13.4
12.0	12871	13.2
	12997	13.3
	12243	12.5
	12905	13.2
Mean		13.1
SD		0.55
RSD		4.0 %

Table 4

Temperature Effects
50 ppm Perchloroethylene

micrograms

	<u>10 c</u>	<u>25 c</u>	<u>40 c</u>
	984.1	1042	1152
	1105	1137	1295
	1086	1098	1274
	1086	1303	1210
	1030	1082	1203
	1118	1144	1185
	1151	1162	1226
	1053	1117	1174
	1117	1140	1208
	1126	1067	
Mean	1086	1129	1214
SD	50.3	71.9	45.8
RSD	4.6 %	6.4 %	3.8 %

Table 5

**Factorial Summary
As Outlined in NIOSH Protocol
4 Determinations at Each Run Level**

Run Number	mL/min
1	15.5
2	13.3
3	12.6
4	12.1
5	12.1
6	11.0
7	9.9
8	11.1
9	8.35
10	14.6
11	9.77
12	10.8
13	14.2
14	12.3
15	14.2
16	12.7
Mean	12.1
SD	1.95
RSD	16.1 %

Table 6

Summary of Factorial Testing Procedures

Run#	PEL	Time (hr)	FV (cm/sec)	RH	Interference	Orientation
1	0.1	8	150	10	no	Per.
2	2.0	0.5	20	10	no	Per.
3	0.1	0.5	150	80	no	Par.
4	2.0	8.0	20	80	no	Par.
5	0.1	8.0	20	10	yes	Par.
6	2.0	0.5	150	10	yes	Par.
7	0.1	0.5	20	80	yes	Per.
8	2.0	8.0	150	80	yes	Per.
9	2.0	0.5	20	80	yes	Par.
10	0.1	8.0	150	80	yes	Par.
11	2.0	8.0	20	10	yes	Per.
12	0.1	0.5	150	10	yes	Per.
13	2.0	0.5	150	80	no	Per.
14	0.1	8.0	20	80	no	Per.
15	2.0	8.0	150	10	no	Par.
16	0.1	0.50	20	10	no	Par.

FV= Face Velocity
Par.= parallel
Per.= perpendicular

Table 7

Storage Study
100 ppm Perchloroethylene, 80% RH

micrograms

	Day 1	Day 14 (Ambient)	Day 14 (Refrigerator)
	1993	2166	1864
	2171	2076	1922
	1974	2166	2038
	1979	2109	2158
	2031	2160	2221
	1968	2078	2120
	2180	1963	2127
	2029	2074	2051
	2084	2082	2228
	2112		
Mean	2052	2097	2081
SD	80.3	64.4	125.4
RSD	3.9 %	3.1 %	6.0 %

Appendix A

Atmosphere Generation Apparatus

The instrument is designed to expose a known concentration of a chemical hazard to a passive sampler under controlled conditions of: 1. Concentration, 2. Temperature, 3. Humidity, 4. Wind Velocity Effect, 5. Time, and 6. Up to four multicomponent hazards.

Description

The instrument consists of:

1. an exposure chamber in which the wind velocity effects are controlled by internal rotating holders,

2. an air supply and purification train such that dry air is blended with saturated air under desired temperature conditions so as to provide air at a known flow and selectable humidity,

3. an injection system composed of precision motor driven syringes in which 1 to 4 chemical hazards can be injected into the flow system and in which the temperature of the injectors is closely controlled,

4. an electrical control system that controls the entire instrument operation,

5. the chamber concentration can be verified by either solid sorbent sampling tubes actively sampled or by gas analysis of the gas phase. The particular verification method used will depend on the analyte of interest.

Means are also included to check the relative humidity.

Figure 1
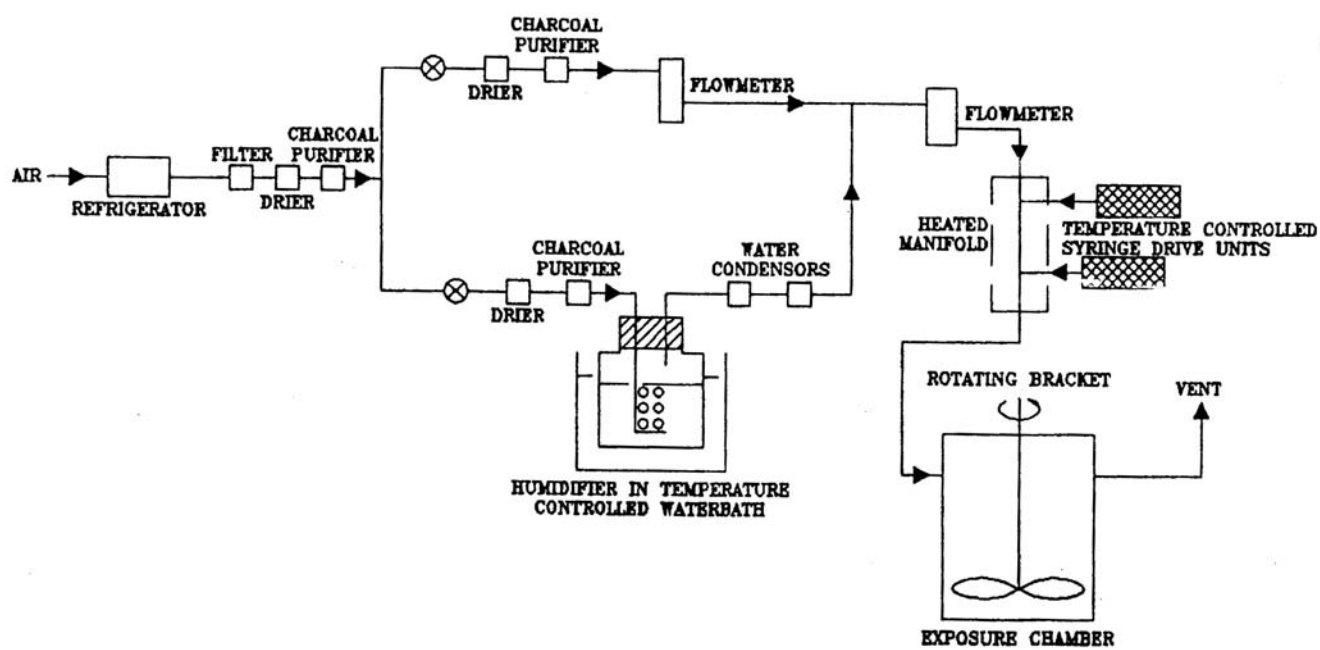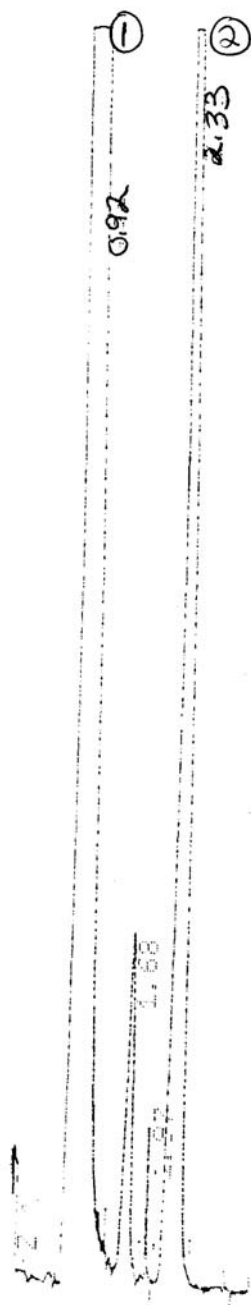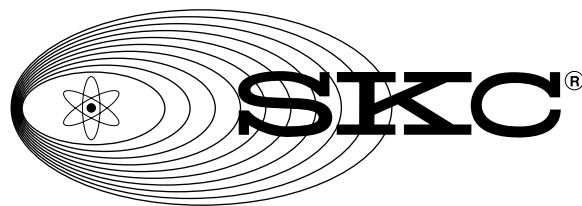
Atmosphere Generation Apparatus

Figure 2

Sample Chromatogram
Tetrachloroethylene in CS2

Peak Identification:

① Carbon Disulfide

② Tetrachloroethylene

Validation of Tetrachloroethylene using SKC Passive Sampler 575-001

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