Determination of the Sampling Rate Variation for SKC 575 Series Passive Samplers

Date: April 1998 Chemist: Warren Hendricks

Organic Methods Evaluation Branch
OSHA Salt Lake Technical Center
Salt Lake City, UT 84115-1802

INTRODUCTION

The purpose of this work was to determine sampling rate variation for SKC 575 Series Passive Samplers.

The concept of sampling rate variation in diffusive sampling was established in previous work and it was defined as the equivalent of sampling pump error in active sampling (Ref. 1). Tests were conducted to determine sampling rate variation for 3M 3520 diffusive samplers in that work. These tests must be performed for each unique design of sampler because the variation is presumed to be a function of sampler design. The tests are based on exposing a specific design of sampler to a five-component solvent mixture in a 16-run factorial test. The solvent mixture is composed of different classes of chemicals anticipated to provide a difficult, but realistic, challenge to the sampler. The factorial test involves sample collection from test atmospheres composed of 16 different combinations of high and low levels of analyte concentrations, high and low relative humidities, high and low face velocities, long and short sampling times, and parallel and perpendicular sampler orientation to the air flow in a test chamber. Effects of each environmental factor on sampler performance, and of possible factor interactions, are revealed by this test. This factorial test is based on that described in the very comprehensive NIOSH Protocol for the Evaluation of Passive Monitors (Ref. 2).

Sampling rates are determined for each component of the solvent mixture for each run of the 16-run factorial test. A relative standard deviation (RSD) is calculated from the 16 independently determined sampling rates for each constituent of the mixture. The RSDs are pooled if they are homogenous. The pooled RSD is a measure of sampling rate variation for several very different classes of chemicals under several very different environmental conditions. The sampling rate variation is assumed to apply to other chemicals, and to apply to other sampling conditions that approximate those tested. Sampling rate variation will have to be re-determined if the design for a particular sampler is changed. (Ref. 1)

Analytical results from any sampling method contain both sampling and analytical error components. Sampling and analytical error must be considered when analytical results are compared to an exposure limit as in OSHA compliance monitoring. OSHA personnel employing active sampling methods use SAE (sampling and analytical error) factors along with sample results to help determine if a particular exposure limit has been exceeded. SAEs are unique for each chemical hazard. Each include a fixed sampling error component (±5% sampling pump error), and a variable analytical error component that is periodically updated from analysis of SLTC QC Division samples. Use of the sampling rate variation for a particular diffusive sampler together with QC Division data for that sampler will permit similar treatment of analytical results from tested diffusive samplers as from active samplers.

REAGENTS

Methyl ethyl ketone (MEK), Aldrich Chemical Company, 99+%, Lot 11619 CX.

2-Propanol (IPA), Fisher Scientific, Optima grade, Lot 962286.

Methylene chloride (MeCL), Fisher Scientific, Optima grade, Lot 946571.

Toluene (Tol), Fisher Scientific, Optima grade, Lot 924028.

Butyl Acetate (BA), Sigma-Aldrich Chemical Co., 99.7% HPLC Grade, Lot 13091 CN.

A solvent mixture was prepared in the following ratio: 184-mL MEK, 306-mL IPA, 54-mL MeCL, 234-mL Tol, and 222-mL BA. This mixture was used to generate test atmospheres, and to prepare test samples and analytical standards.

Sample desorbing solution. The solution was composed of 60/40 (v/v) N,N-dimethylformamide (DMF) and carbon disulfide with internal standard (0.25 µL/mL p-cymene). This solution was prepared from reagent grade chemicals of various lot numbers. The desorbing solution was observed to darken and to develop an unpleasant odor upon standing for long time periods. The quantity of solvent prepared at any one time was limited to only that sufficient to last for two-three weeks.

SAMPLING MEDIA

SKC Passive Samplers, SKC 575-001, Lot 656, containing 350 mg of charcoal.

Anasorb® 747 adsorbent tubes, SKC 226-83, Lot 268, 200/400-mg sections, and SKC 226-81A, Lot 645, 70/140-mg sections. These sampling tubes were used as an independent means to monitor concentrations of test atmospheres.

APPARATUS

Adsorbent tube media were desorbed for one hour on a Labquake C415-110 Shaker set in the tube rotator mode.

Passive samplers were desorbed for one hour using a SKC 1-225-01 Sorbent Extractor with rack.

Samples were analyzed by gas chromatography. A Hewlett-Packard Model 5890 Series II GC equipped with a Chem-Station, an automatic sample injector, and an FID were used. Separations were performed on a Restek Stabilwax® (60-m × 0.32-mm i.d. × 1.00-µm df) column. The injection volume was 1 µL with either a 50 to 1 or a 5 to 1 split. The GC was temperature programmed from 40 to 220 °C in three ramps: 40 to 90 °C at 5 °C/min, 90 to 170 °C at 10 °C/min, and 170 to 220 °C at 15 °C/min. The GC oven was maintained at 40°C for 1 min following injection, and held at 220 °C for 5 min following completion of the temperature program. The hydrogen carrier gas flow rate was 1.2 mL/min, the nitrogen auxiliary gas flow rate was 30.5 mL/min, the septum purge flow rate was 2.1 mL/min, the detector hydrogen gas flow rate was 31.0 mL/min, and the detector air gas flow rate was 375 mL/min. A Waters Millennium Chromatography Manager system was used for data collection.

Samples were collected from dynamically-generated test atmospheres that were prepared using an apparatus constructed from stainless steel. The apparatus consisted of two chambers that were connected in series, and designed to permit simultaneous exposure of a large number of samplers to the same test atmosphere at two significantly different face velocities.

Humid air (for use with controlled test atmospheres) was generated using a Miller-Nelson Model HCS 301 Flow-Temperature-Humidity Control System. This system was equipped with a 500 L/min mass flow controller.

Relative humidity and temperature of the test atmospheres within the exposure apparatus were monitored with an EG&G Model 911 Dew-All Digital Humidity Analyzer. Comparison readings were taken periodically with a Solomat MPM 500e meter equipped with a Model 355RHX humidity/temperature probe. The probes were calibrated by the manufacturer.

Dilution airflow rates (50-360 L/min) were measured with an Equimeter No. 750 gas meter. The meter readings for several different flow rates were compared to those of a Singer DTM 115 gas meter (that had been tested by the local natural gas distributor and found to be accurate) that was connected in series before the Equimeter. Both meters gave very similar readings.

The solvent mixture was metered into the system with an Isco 100 DM syringe pump equipped with a cooling/heating jacket and an insulation cover package. The pump was operated in the constant flow mode. The temperature of water in the cooling/heating jacket was maintained at 23°C with a Forma Scientific Model 2006 CH/P Bath and Circulator.

Solvent vapors were generated by pumping the liquid through a short length of 0.0625-inch o.d. polyether-ether-ketone (PEEK) tubing into a vapor generator where it evaporated into the dilution air stream (Figure 1). The vapor generator consisted of a 10-cm length of 0.25-inch o.d. glass tubing with a small hole in the side. The hole was just large enough for the PEEK tubing to be inserted. The glass tubing was placed inside a 0.5-inch stainless steel Swagelok® tee wrapped with heating tape. The PEEK tubing entered the third port of the tee through an adaptor and was inserted about 0.125 inch (approximately in the center) into the glass tubing through the small hole. Solvent was pumped through the PEEK tubing into the glass tubing. The liquid flow rate was such that liquid did not accumulate in the evaporation tube. The entire dilution air stream passed through the tee and swept generated vapors into the remainder of the apparatus.

The following is a description of the arrangement of the apparatus that was placed in a walk-in hood: Liquid from which vapors were to be dynamically generated was pumped with a precision Isco syringe pump (an identical pump and a small solvent mixing tee was available when its use was desired) into a heated manifold where it evaporated. The generated vapors were swept from the manifold with dilution air. Stainless steel tubing (0.5-inch o.d.) connected with stainless steel Swagelok® fittings was used to transfer the test atmosphere. The dilution air was humidified (if desired) using a Miller-Nelson Flow-Temperature-Humidity controller. The vapor/dilution air mixture then passed into a 3×24-inch stainless steel mixing chamber that could be removed from the system. The test atmosphere next passed through 0.5-inch ball valves where it could be either diverted to waste, or directed into the exposure chambers. An additional ball valve allowed the chambers to be purged with room air. The transfer tubing diameter was increased from 0.5 inch to 1 inch at this point using a Swagelok® adaptor attached to the chamber inlet. Tube and fitting diameter was increased to 1 inch after this fitting to help reduce any increased pressure to ambient. The 1-inch o.d. chamber inlets have small stainless steel deflectors to help insure that the test atmosphere completely fills the sampling chambers. Stainless steel screens were placed inside the chambers for the same purpose. This design should cause air flow through the chambers to be somewhat turbulent. A Gast Model R1102 blower was used to move the test atmosphere through the apparatus. A gate valve was used to help regulate pressure by adding make up air to the blower. Pressure within the chambers was monitored with an Omega Technologies Company Model Px 212-060 AV Pressure Transducer connected to an Omega Technologies Company Model DP 41-S Strain Gauge Meter. The pressure transducer was calibrated daily

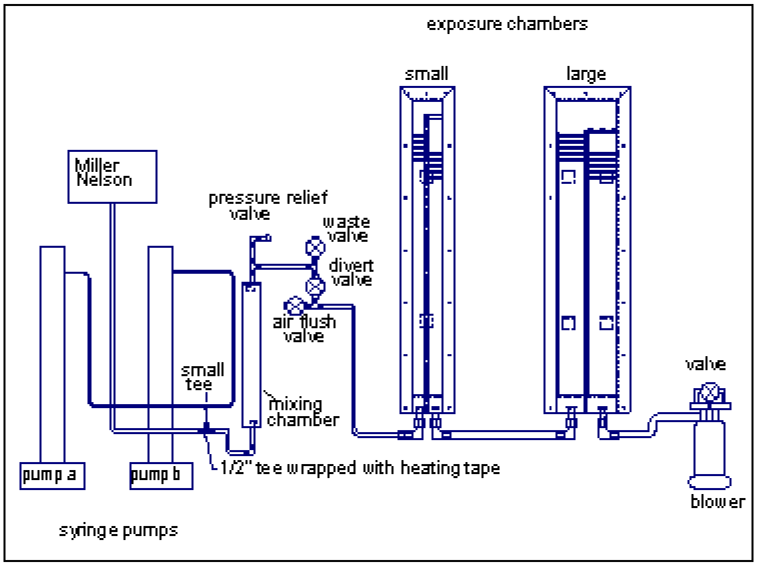

Figure 1. Test atmosphere generation and sample collection apparatus.

with a Princo Nova Full Range Mercurial Barometer. Face velocities of the test atmospheres were calculated by dividing the volumetric flow of each atmosphere by the cross-sectional area available for air flow in each chamber. The cross-sectional area available for air flow was the cross-sectional area of each chamber reduced by the cross-sectional areas of the samplers. Humidity and temperature were monitored only within the large chamber because of access port limitations. This arrangement of the apparatus is shown in Figure 1.

EXPERIMENTAL

Desorption Efficiency

Desorption efficiency experiments were performed by spiking SKC 575-001 Passive Samplers, and front sections of Anasorb® 747 adsorbent tubes with the solvent mixture. The following masses were those studied: MEK- 12, 206, and 2056 µg; IPA- 19, 333, and 3333 µg; MeCL- 6, 99, and 992 µg; Tol- 16, 283, and 2823 µg; and BA- 16, 273, and 2727 µg. Anasorb® 747 desorption samples were prepared by transferring the front sections of sampling tubes into 4-mL glass vials, spiking each sample with the solvent mixture, and storing the samples at ambient temperature overnight. The samples were desorbed with 2 mL of desorption solvent for one hour on a tube rotator the next day. Diffusive samplers were prepared by sealing the samplers with the O-rings and covers included with the sampler, cutting off the ends of the two protruding tubes of each sampler, spiking each sampler with the solvent mixture through one of the tubes, and then sealing the tubes with the manufacturer-supplied plugs. Samples were allowed to stand six hours before desorption. Six hours, rather than overnight, was used because low desorption efficiencies were obtained for MEK when samples allowed to stand overnight. The low recoveries were attributed to instability of MEK on the charcoal surface. These samples were desorbed for one hour in-situ with 2 mL of desorbing solvent using a SKC 1-225-02 Sorbent Extractor with rack. The sampler was secured by clipping it to a rail of the SKC Sorbent Extractor rack, and then the solvent was added through the port nearest the outside edge of the sampler with a 2-mL volumetric pipet. The tip of the pipet just fit inside the sampler port. The desorption solvent was transferred from the desorbed sampler to a GC auto-sampler vial using the manufacturer-supplied PTFE tube and the port nearest the outside edge of the sampler.

Sampling Rate and Capacity

The sampling rate and capacity for each component of the solvent mixture was determined by exposing diffusive samplers (in sets of four) to the mixture for increasing time periods. Typical concentrations of test atmospheres and OSHA PELs for the components of the solvent mixture are shown in Table 1. Mixture concentrations at this level are referred to as 1×, and one-tenth of these concentrations as 0.1×, in this report. Unless otherwise noted, the sampler orientation was parallel to the flow direction of the test atmosphere.

Table 1
Typical Concentrations of Test Atmospheres

	MEK	IPA	MeCL	Tol	BA
concn (µg/L)	535	865	260	725	695
OSHA PEL (µg/L)	590	980	87	755	710

The average relative humidity, temperature, and face velocity of the test atmospheres was 76%, 24°C, and 0.24 m/s respectively. Six active samples were collected for each run. Five of the active samplers were large Anasorb® 747 sampling tubes, and one was a small tube that was employed because of sampling chamber access port limitations. The sampling rate for the active samplers was 30 mL/min.

Reverse diffusion experiments were performed by sampling the 1× concentration for one-half the total sampling time, and then sampling clean humid air for the remainder of the sampling time. Ten diffusive samplers were exposed to the solvent mixture for 128 min, five were removed and analyzed, and then the remaining five were exposed to clean humid air for an additional 120 min. The relative humidity, temperature, and face velocity of the test atmosphere was 84%, 21 °C, and 0.10 m/s respectively. In another experiment, eight samplers were exposed to the 1× mixture for 60 min, four samplers were removed, and then the remaining four were exposed to clean humid air for an additional 60 min. The relative humidity, temperature, and face velocity of the test atmosphere was 75%, 22 °C, and 0.21 m/s respectively.

Several experiments were performed to determine the effects of face velocity of the test atmosphere past the samplers. Face velocity in the test chamber was varied by changing the flow rate of the dilution air. The concentration of the mixture was 1×, and the average relative humidity was 78% at 23 °C.

Factor Tests

A 16-run factor test was performed using a modified version (Ref. 1) of the NIOSH Factor Test (Ref. 2). NIOSH has identified six factors that can affect sampler performance. These factors are analyte concentration, exposure time, face velocity, relative humidity, interferant, and monitor orientation. Sixty-four experimental runs (26) would be required to fully evaluate combinations of each factor at two levels. NIOSH recognized that this would be an excessive number of tests, and has devised a 16-run fraction of the full factorial that is capable of revealing any of these factors having a significant effect, free of two-factor interactions, on sampler performance. Some two and three-factor interactions can also be screened by this design. The test is based on the comparison of each factor effect to experimental error so that the significance of that effect can be determined. Experimental conditions are shown in Table 2. Interferant was provided by the components of the solvent mixture, for example: if MEK was being examined then IPA, MeCL, Tol, and BA were the interferents, and the levels were declared either high or low.

Table 2
Experimental Design Of The Factor Test

run no.	analyte concn	RH (%, °C)	inter level	time (min)	lin vel (m/s)	sampler orien	run no.	analyte concn	RH (%, °C)	inter level	time (min)	lin vel (m/s)	sampler orien
1	0.1×	7, 21	low	120	1.7	perp	9	1×	81, 21	high	30	0.1	paral
2	1×	14, 22	low	31	0.1	perp	10	0.1×	64, 24	high	120	1.4	paral
3	0.1×	78, 23	low	30	1.5	paral	11	1×	14, 22	high	121	0.1	perp
4	1×	81, 21	low	121	0.1	paral	12	0.1×	11, 23	high	30	1.8	perp
5	0.1×	13, 21	high	120	0.1	paral	13	1×	78, 22	low	30	1.8	perp
6	1×	12, 22	high	30	1.5	paral	14	0.1×	85, 21	low	121	0.1	perp
7	0.1×	80, 21	high	31	0.1	perp	15	1×	8, 22	low	121	1.5	paral
8	1×	73, 22	high	120	1.8	perp	16	0.1×	14, 21	low	30	0.1	paral

inter = interferant, perp = perpendicular, paral = parallel

Precision

Relative standard deviations of percent recovery for active samples and of sampling rates for diffusive samplers were calculated for runs 2, 4, 13, and 15 of the Factor Test, for the 6-hour sampling rate and capacity test, and for the first set of reverse diffusion (0.21 m/s) samples for SKC diffusive and active samplers. These runs were selected because similar runs were performed in previous work for 3M Organic Vapor Monitors (OVMs), and it is useful to compare results for the samplers. The data were pooled for each component of the solvent mixture for each sampler, after first testing that the data were homogenous by the Cochran Test at the 95% confidence level. The following data were not homogenous and not included in the pools. SKC diffusive: MeCL result for factor test 13. 3M: MeCL result for factor test 4. Active: Tol and BA results for reverse diffusion test.

RESULTS and DISCUSSION

Desorption Efficiency

The results of the desorption efficiency experiments are summarized in Table 3. All samples were desorbed with a 60/40 mixture of DMF and carbon disulfide. This mixture is used to prevent two-phase samples caused by the presence of water. Desorption of each component of the solvent mixture from Anasorb® 747 was high and constant over the studied range. Desorption of MEK, however, from the charcoal diffusive samplers was only about 82% when the samplers were allowed to stand overnight after spiking. Desorption of MEK was about 101, 96, and 93% when samples were allowed to stand about 40,130, and 360 min after spiking. The decreasing recoveries for MEK were attributed to the documented instability and desorption problems of ketones on charcoal (Ref. 3). The desorption efficiency results from the six-hour standing time for diffusive samplers were used for this study because the delay between sampling and analysis of samples was less than six hours in most cases. Desorption of the other components of the solvent mixture from diffusive samplers was high and constant, even when the samples were allowed to stand overnight.

Table 3
Desorption Efficiency (Percent)

	MEK	IPA	MeCL	Tol	BA
140 mg 747	100.4	100.9	102.5	98.1	100.5
400 mg 747	103.7	106.5	105.8	96.3	104.3
SKC Sampler	92.9	102.6	100.9	95.0	102.1

Sampling Rate and Capacity

Sampling rates were calculated by dividing mass collected (corrected for desorption efficiency) by sampling time multiplied by the actual concentration of the test atmosphere (sampling rate = μg/(min × μg/L)). Sampling rate, in L/min, was converted to mL/min, the same units often used for adsorbent tubes. Theoretical concentrations were calculated from the test atmosphere generator apparatus operation parameters. The actual concentrations of the test atmospheres were determined by the analysis of adsorbent tube samples. The average of adsorbent tube results was about 95% of the theoretical amounts for this work. The same sampling times were used for both active and diffusive sampling. All samples were analyzed as soon as possible after collection.

Sampling rate and capacity results are presented in Table 4 and in Figure 2. The sampling rates were determined at ambient temperature and pressure, and were converted to their equivalent at 25 °C and 760 mmHg.

Table 4
Sampling Rate and Capacity (mL/min)

time (min)	MEK	IPA	MeCL	Tol	BA
7.5	17.58	17.96	18.01	14.06	12.31
15	18.37	18.50	18.46	14.75	12.83
30	18.58	19.42	18.72	14.48	13.60
60	18.97	19.83	18.56	14.80	14.02
120	18.64	19.34	17.24	14.62	13.79
180	18.00	19.11	16.54	14.78	13.95
240	17.78	19.45	16.36	15.24	14.41
300	17.98	17.91	14.86	15.04	13.25
362	17.59	17.64	14.16	14.90	13.16
420	14.12	16.08	12.37	14.02	12.52

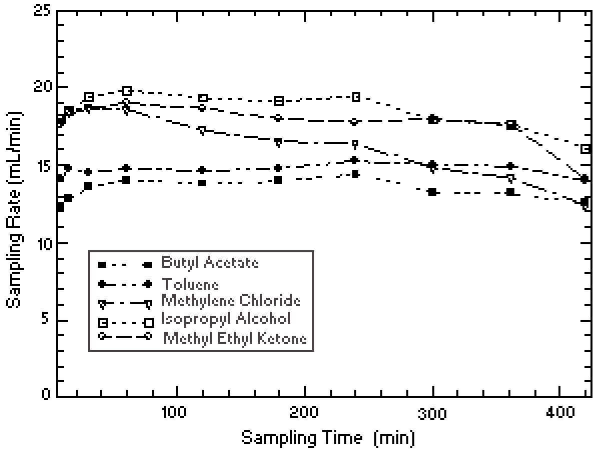

Figure 2. Samping rate and capacity.

The sampling rates for the mixture components, except methylene chloride, were fairly constant from 30 to 240 min. The capacity of the sampler for a component is presumed to be exceeded when the apparent sampling rate for that component decreases rapidly. The sampling rate for methylene chloride (and to a lesser degree, IPA and MEK) appeared to decrease after 60 min. The capacity of the sampler for methylene chloride was clearly exceeded after four hours, and perhaps even two hours. The sampler loading after two hours was approximately 6 mg of the mixture, including 0.6 mg of methylene chloride. The shortest recommended sampling time was set at 30 min, and the maximum recommended sampling time at 120 min for this work because of methylene chloride capacity limitations. Table 5 shows average sampling rates for 30 to 120 min. Eight-hour sampling rates (and validation levels) have been published by SKC in their

1998 catalog, and are included in Table 5. SKC determined these sampling rates in full (F), bi-level (Bl), and calculated (C) levels of validation. SKC defines these levels as follows: full, passed NIOSH protocol requirements; bi-level, a key member of a homologous series passed full, and the analyte passes NIOSH protocol for sampling rate, desorption efficiency, humidity effects, reverse diffusion, and storage stability; and calculated, use of the Hierschfelder Equation and internal dimensions of the sampler to calculate sampling rate. The biggest difference between SLTC and SKC sampling rates is for methylene chloride.

Table 5
Average Sampling Rate (mL/min) and RSD (Percent)

	MEK	IPA	MeCL	Tol	BA
30-120min	18.7	19.5	18.2	14.6	13.8
RSD (%)	1.1	1.3	4.5	1.1	1.5
SKC samp rates	17.1(Bl)	17.8(C)	14.7(F)	14.5(Bl)	12.7(C)

Experiments were performed to determine if the capacity of the sampler for methylene chloride had actually been exceeded, or if reverse diffusion had occurred. The net effect of exceeded capacity and of reverse diffusion is the same, analyte lost and not measured. This test is performed to detect the loss of a collected, but inadequately retained analyte. The sampling times in Table 6 are one-half the total test sampling time. For example: a reverse diffusion test for a two-hour sampling time requires one-half the samples be exposed to the mixture for one hour, and the other half be exposed to the mixture for 1 hour and then to clean, humid air for an additional hour. Reverse diffusion was measured as the percent ratio of mass recovered from samples exposed to both the solvent mixture and also to clean humid air relative to samples that had been exposed to only the solvent mixture. Table 6 shows that there was a loss of methylene chloride for the 60-min test, and a more significant loss in the 128-min test. MEK had a less severe loss in the 128-min test than methylene chloride. These results show that reverse diffusion could be a fairly large uncorrectable bias for methylene chloride if collected for long sampling times at the mixture concentrations used for this work. The SKC sampler has no back-up section.

Table 6
Reverse Diffusion (Percent Ratio)

	MEK	IPA	MeCL	Tol	BA
60 min	100.5	100.7	95.7	100.7	102.2
120 min	94.9	97.0	91.9	98.8	98.6

The effects of increasing face velocity on sampling rates is shown in Figure 3. The most dramatic effects occur at low to medium velocities. The overall effect was similar to that observed for 3M OVMs (Ref. 1).

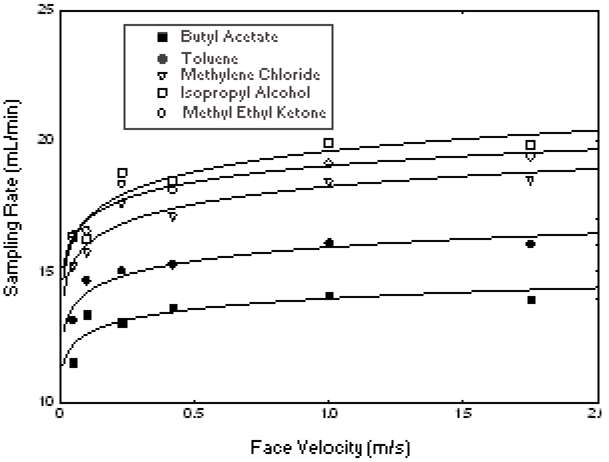

Figure 3. The effects of increasing face velocity on sampling rates.

Factor Test

The results of the factor test are presented in Table 7. The sampling rates were determined at ambient temperature and pressure, but are expressed at 25 °C and 760 mmHg. Average sampling rates and their percent RSDs are shown in Table 8. These percent RSDs were found to be homogenous by the Cochran Test. The pooled percent RSD, 8.7%, is the sampling rate variation for SKC 575-001 Passive Samplers as determined by this work.

Table 7
Factor Test Results (mL/min)

test	MEK	IPA	MeCL	Tol	BA	test	MEK	IPA	MeCL	Tol	BA
1	19.26	20.52	20.20	16.26	14.07	9	17.80	17.84	17.91	14.38	12.80
2	17.68	18.09	18.30	14.52	12.64	10	20.61	20.56	19.42	16.54	14.38
3	20.52	20.41	20.39	16.39	14.54	11	16.69	17.07	17.15	13.87	12.07
4	16.66	16.51	15.78	13.26	11.45	12	21.18	21.61	20.72	17.57	15.18
5	16.18	17.06	17.31	13.83	11.93	13	20.21	20.42	20.02	16.42	14.37
6	19.50	20.88	20.79	16.77	14.51	14	17.51	17.61	16.96	13.90	12.14
7	17.20	17.49	17.62	13.97	12.17	15	18.55	19.28	18.79	15.65	13.67
8	19.41	19.88	18.54	16.06	13.96	16	17.53	17.50	18.16	14.10	12.10

Table 8
Percent RSDs of Average Sampling Rates

	MEK	IPA	MeCL	Tol	BA
ave (mL/min)	18.53	18.92	18.63	15.22	13.25
RSD (%)	8.57	8.86	8.07	8.95	9.04
pooled RSD (%)			8.71		

The results in Table 7 were further analyzed to detect factor effects as described in Refs. 1 and 2. This analysis gave an experimental error for each component of the mixture. Minimum significant effect (MSE) was calculated for each component by multiplying the error by 2.26 (the t statistic at the 95% confidence level for nine degrees of freedom).

The average experimental error was slightly more than 1%, which is sufficiently low to reveal factor effects of about 3%. These results are shown in Table 9. The analysis also gave an effect result for each component of the solvent mixture for concentration, relative humidity, interferant, sampling time, face velocity, sampler orientation, and for some factor interactions, such as an interaction between concentration, relative humidity, and sampling time. The absolute value of the ratio of effect/MSE was calculated and is presented in Table 10. Any ratio above one is significant at the 95% confidence level, and that effect should be studied further in additional experiments. The results for interferant may be somewhat equivocal because the remainder of the mixture was considered the interference. That is IPA, MeCL, Tol, and BA were declared the interference in MEK tests; MEK, MeCL, Tol, and BA the interference for IPA...etc. Sampling time and face velocity had significant effects on the sampling rate for each component of the solvent mixture. Face velocity had the more pronounced effect. Relative humidity was also significant for methylene chloride. Concentration was essentially a significant factor for methylene chloride. The E5 interaction for methylene chloride is completely confounded among some interaction between concentration and sampling time or interferant and sampler orientation.

Table 9
Analysis of Factor Data

	MEK	IPA	MeCL	Tol	BA
error	0.29	0.21	0.20	0.16	0.16
MSE	0.66	0.48	0.45	0.36	0.35

Table 10
Analysis of Factor Data

	MEK	IPA	MeCL	Tol	BA
concn	0.66	0.72	0.97	0.56	0.37
RH	0.63	0.33	1.32	0.57	0.13
inter	0.12	0.53	0.24	0.86	0.71
time	1.27	1.49	2.70	1.64	1.64
vel	4.15	6.33	5.44	6.87	6.14
orien	0.34	0.69	0.26	0.57	0.43
interaction	none	none	E5:1.03	none	none

The ±8.7% pooled sampling rate variation for the SKC samplers is somewhat higher than the ±6.4% variation determined in similar experiments for 3M 3520 OVMs (Ref. 1). The two sets of data were subjected to the Cochran and F Tests. These tests are used to compare variances to determine if they differ significantly. The critical values for the Cochran and F Tests are 0.2086 and 2.86 at the 95% confidence level, respectively. The results of the tests are shown in Table 11. These tests detected no statistical difference in the variances. The F Test is used to compare two variances, while the Cochran Test is used to compare more than two variances to determine if they differ significantly. The Cochran Test is more appropriate for this work.

Table 11
Comparison of Sampling Rate Variations

	MEK	IPA	MeCL	Tol	BA
SKC	8.6	8.9	8.1	9.0	9.0
3M	5.6	7.5	6.9	6.3	5.6
Cochran	g statistic = 0.1380				
F Test	2.36	1.41	1.38	2.04	2.58

Precision

Relative standard deviations were calculated for each component of the solvent mixture for tests that were common to active, SKC diffusive, and 3M diffusive samplers. The results are presented in Table 12. The data shows that the precision was similar for all the samplers. Active and SKC diffusive samples were collected at the same time. 3M OVMs were collected in previous, but similar work.

Table 12
Precision Data (Percent RSD)

	MEK	IPA	MeCL	Tol	BA
active	2.0	2.1	2.0	1.7	2.0
SKC	2.7	2.3	2.0	2.3	2.4
3M	1.8	1.7	1.8	1.8	

CONCLUSIONS

The sampling rate variation for SKC 575-001 Passive Samplers is ±8.7% as determined by this work. No statistical difference between this result and the ±6.4% variation previously determined for 3M 3520 Organic Vapor Monitors was detected by the F and Cochran Tests. Sampling rate variation is assumed to be a function of the design of the sampler, and not dependent on the sorbent within the sampler. Therefore, ±8.7% variation is appropriate for use with SKC 575-001 (charcoal), 575-002 (Anasorb® 747), and 575-003 (Anasorb® 727) passive samplers. The sampling rate variation is further assumed to apply to chemicals other than those tested, and to other environmental conditions that approximate those tested.

It is not possible to assess reverse diffusion in SKC Passive Samplers used in field applications because they have no reference (back-up) section. Some chemicals, like methylene chloride, have been shown to have a potential for reverse diffusion that could be exacerbated by the presence of other chemicals. This would necessitate that methods for chemicals shown to have a likelihood for reverse diffusion have conservative sampling times.

The appropriate sampling rate variation will be used by OSHA as the sampling error component in SAE calculations for 3M OVM and SKC diffusive samplers. The analytical error component will be periodically updated from the analysis of QC Division samples. Each analyte will have a unique SAE. The treatment of SLTC analytical results for diffusive samples will parallel that for active samples for comparison of field sample results with OSHA exposure standards.

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