

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM

Proficiency Analytical Testing (PAT) Program (November 23, 1998)

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PAT ROUND 135, NOVEMBER 1998

A total of 1196 laboratories were enrolled in the PAT Program with 1098 laboratories submitting results on round 135. Of the 1098 laboratories submitting results, 925 used the Internet data entry system (www.aiha.org/proftest.htm). Table I lists the reference values, performance limits, and participants for each sample type in the PAT Program. Table II presents the summary of the PAT proficiency ratings for each analytical area.

DIFFUSIVE SAMPLER ROUNDS 128-134

AIHA requested the National Institute for Occupational Safety and Health (NIOSH) to study PAT diffusive sampler data and various protocols for diffusive sampler evaluation to determine if PAT diffusive sampler performance limits could be improved. Results from Rounds 128-134 of the PAT Diffusive Sampler Program were studied. To examine the bias, the overall means of the reported results for each diffusive sampler, compound and sample combinations were calculated. A ratio of the overall mean to the calculated concentration was then used as an index to represent the relative difference of a diffusive sampler to the calculated concentration for that particular compound and sample combination. The ratios of 1.18 and 0.82 represent the upper and lower limits relative to the calculated concentration. A ratio of 1 indicates no bias of a particular diffusive

sampler to the calculated concentration for that compound sample combination.

BIAS AND PRECISION

Based on the index, the ratio of overall mean to the calculated concentration, 3M diffusive samplers exhibit an overall negative bias. Laboratories analyzing 3M diffusive samplers were likely to report values which were lower than the calculated concentrations. However, these results were mostly inside the performance limits (Figure 1). In contrast, results from Assay Technology (AT) and SKC diffusive samplers present an overall positive bias. Laboratories analyzing AT or SKC diffusive samplers were likely to report values higher than the calculated concentrations. The bias may be attributed to a combination of both the types of samplers and the particular labs analyzing that sampler. Some of the reported values were extremely high or low, especially for results from AT or SKC diffusive samplers. To better present the data graphically, and for more meaningful mean values, some of the extreme values were statistically treated.

The relative standard deviations (RSD) for each sample and compound combination were calculated. Results from 3M diffusive sampler consistently had lower RSDs. The RSD ranges for 3M samples were 11-17% (Figure 2). Results from AT and SKC diffusive samplers consistently had higher RSDs. The RSDs ranged from, 6-25% for AT samplers, and 5-26% for SKC samplers.

The following aspects were addressed by David Bartley and Mary Ellen Cassinelli of NIOSH/DPSE.

COMPARISON OF ACTIVE SAMPLERS WITH CALCULATED VALUES

Establishing the true or reference concentration within the exposure chamber is important to judge the accuracy of laboratories fairly. One approach proposed by OSHA is that the calculated concentration is regarded as the "benchmark," although an independent estimate is required and must be within 5% of the calculated estimate.⁽¹⁾ If these estimates differ, then a third independent estimate is required to establish the reference concentration through agreement with one of the other independent estimates. Unpublished policy within NIOSH for evaluating direct reading instruments, requires test concentrations measured through two independent methods to agree within 5%. Alternatively, the traditional NIOSH protocol for evaluating diffusive samplers⁽²⁾ requires averaging at least two independent methods (possibly including calculated estimates) with at least four samples per method. Finally, the Comité Européen de Normalisation (CEN)⁽³⁾ has adopted a looser requirement: calculated and independent measurements must agree within $\pm 10\%$.

Clearly, no consensus has yet been reached in establishing the reference concentrations. However, data from the PAT Program's experiments to date suggest an alternative. The data indicate that the active sampler results on average follow the diffusive sampler results better than the calculated or generated concentration. Furthermore, as far as is known, the diffusive versus active analytical errors are independent. These facts suggest that the active results may be the most accurate in establishing the reference concentration.

For example, the problems with o-xylene in Round 130 would be minimized. Specifically in the case of Level 2 of Round 130, 73% of the AT and 64% of the SKC o-xylene estimates were rated *unacceptable*. Many of the ratings of *unacceptable* would be affected by a -8% bias in the reference concentration, if such bias exists. For example, if the active sorbent tube concentration estimates were used as the reference concentration (possibly eliminating a -8% bias), then the *unacceptable* rating fractions are found to drop to 9 and

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TABLE I. Reference Values, Performance Limits, and Participants for Each Sample Type; PAT Round 135 (November 1998)

Contaminant	Sample Number	No. of Labs	Reference Value	RSD (%)	Performance Limits		No. of Outliers
					Lower	Upper	
Cadmium (mg)	1	296	0.0076	4.6	0.00659	0.00869	17
	2	296	0.0142	4.2	0.01246	0.01601	19
	3	296	0.0048	5.1	0.00408	0.00553	19
	4	296	0.0095	4.4	0.00829	0.0108	16
Lead (mg)	1	302	0.0577	4.2	0.0505	0.0650	26
	2	302	0.0194	5.4	0.0162	0.0225	25
	3	302	0.0772	4.0	0.0679	0.0865	24
	4	302	0.0476	4.3	0.0414	0.0538	20
Zinc (mg)	1	292	0.173	4.5	0.1498	0.1962	33
	2	292	0.0681	5.1	0.0577	0.0786	31
	3	292	0.1348	4.6	0.1162	0.1534	30
	4	292	0.0975	4.8	0.0834	0.1115	27
Silica (mg)	1	79	0.0880	17	0.0429	0.1331	9
	2	79	0.0645	18.6	0.0285	0.1005	4
	3	79	0.0915	16.9	0.04507	0.1379	5
	4	79	0.0803	17.5	0.03815	0.1224	11
Asbestos/fibers (chrysotile) (f/mm ²) (man-made fiber)	1	946	104	20	51	176	189
	2	946	165	20	81	279	180
	3	946	93	20	46	158	207
	4	946	82	20	40	139	68
Methyl ethyl ketone (mg)	1	280	0.1484	8.2	0.1120	0.1849	17
	2	280	0.8016	6.1	0.6539	0.9492	22
	3	280	0.5108	6.4	0.4122	0.6095	21
	4	280	0.3042	7.0	0.2407	0.3678	25
Methyl isobutyl ketone (mg)	1	280	0.0899	9.5	0.0643	0.1155	14
	2	280	0.1851	7.7	0.1421	0.2280	21
	3	280	0.3745	6.7	0.2989	0.4501	19
	4	280	0.5431	6.4	0.4389	0.6472	27

29%, respectively. The 3M *unacceptable* rate would also drop, from 13 to 10%.

FUTURE PAT DIFFUSIVE SAMPLE PROGRAM CHANGES

NIOSH researchers recommend that active sorbent tubes be used to establish the reference concentration of a generated atmosphere. The AIHA PAT subcommittee of the Lab accreditation committee has accepted the following recommendations for the PAT diffusive sampler Round 138 in July 1999. The reference concentration of the test atmosphere will be determined from the active sorbent tubes. At least five measurements, distributed through the chamber, should be made for each generation run.⁽⁴⁾ The calculated concentration values would serve as a quality control check. If the active and calculated concentrations differ by more than 10% the run would be voided. The RSD for the active samplers should be less than 5%, and individual active sampler results should be plotted to ensure that no important trend exists in analyte concentration

across the generator. These recommendations are essentially an adaptation of the CEN⁽³⁾ criteria, and should result in a reduction in the number of outliers experienced by participants. Grab samples, taken with a gas-tight syringe, will be collected periodically throughout each diffusive sampler batch generation. The data will be analyzed and compared with the active sampler data to determine if any future improvement in reference values can be made. While some protocols require better active sampler to calculated value agreement, it is unclear that better agreement can be achieved consistently.

Before the final criteria for the diffusive sampler program is determined, the data

from several generator runs must be examined, and the various aspects of each as well as other protocols must be considered.

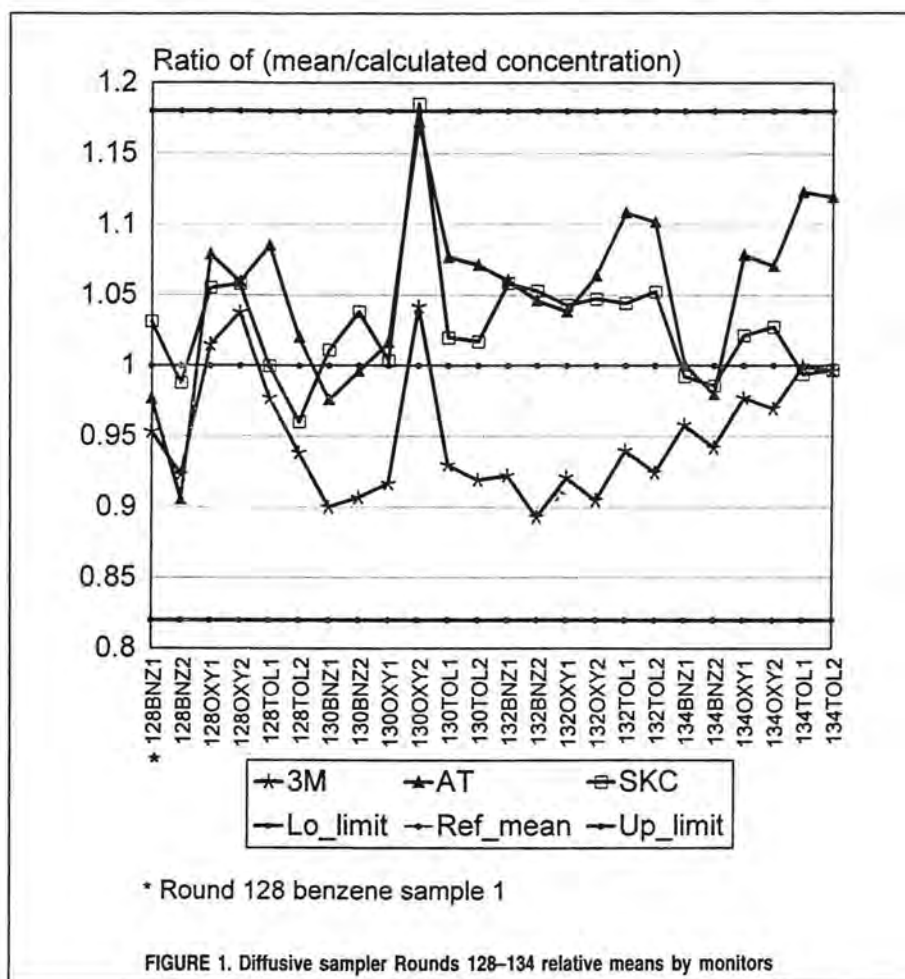
PAT ROUND 136 JANUARY 1999

PAT Round 136 was sent to participating laboratories on January 1, 1999. For this round, the organic solvents were benzene, o-xylene and toluene and the metals were cadmium, chromium, and lead. Silica had talc and coal mine dust background and asbestos/fibers were amosite with one man-made fiber sample.

TABLE II. PAT Proficiency Ratings Based on Rounds 132 to 135 (September 1998–November 1998)

Contaminant	Number of Labs Rated	Number of Labs Rated Proficient	Percent Labs Rated Proficient
Metals	292	273	93.5
Silica	79	78	98.7
Asbestos/fibers	946	857	90.6
Organic solvents	280	256	91.4

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BACKGROUND

The Proficiency Analytical Testing (PAT) Program is managed by the American Industrial Hygiene Association (AIHA) in Fairfax, Virginia. The PAT Program provides

quality control reference samples to approximately 1200 occupational health and environmental laboratories in 17 countries. Although one objective of the PAT Program is to evaluate the analytical ability of participating laboratories, the primary objective is

TABLE III. Current Sets of Samples in PAT Program

Metals	cadmium	
	chromium	
	lead	
	zinc	
Silica	quartz	
Asbestos/fibers	amosite	
	chrysotile	
	man-made fibers	
Organic solvents	benzene	methyl ethyl ketone
	n-butyl acetate	methyl isobutyl ketone
	chloroform	tetrachloroethylene
	1,2-dichloroethane	toluene
	p-dioxane	1,1,1-trichloroethane
	ethyl acetate	trichloroethylene
	isopropanol	o-xylene
	methanol	

to assist these laboratories in improving their laboratory performance.

Each calendar quarter (designated as a round), samples are mailed to participating laboratories, and the data are analyzed to evaluate laboratory performance on a series of analyses. Each mailing and subsequent data analysis is completed in time for participants to obtain repeat samples and to correct analytical problems before the next calendar quarter starts. The PAT Program currently includes four sets of samples as shown in Table III. A mixture of 3 of the 4 possible metals, and 1 to 3 of the 15 possible organic solvents are rotated for each round. Fibers alternate between amosite and chrysotile asbestos and man-made fibers; no fiber mixtures are provided. Each set consists of four concentrations and a blank. The metals, silica, and fiber samples are on filters, and the organic solvents are on charcoal, carbon molecular sieve, or silica gel tubes. The organic solvent set also includes five blank tubes for desorption efficiency determination. Every other round includes two diffusive samplers with benzene, o-xylene, and toluene.

Laboratories are evaluated for each analysis by comparing their reported results against an acceptable performance limit for each PAT Program sample the laboratory analyzes. After the data from all laboratories are collected and statistically treated, the mean of the collected data is calculated, and the performance limits equal the mean ± 3 standard deviations. The performance limits for all analytes (metals, silica, asbestos, and organic solvents) are calculated using a maximum relative standard deviation of 20% and a minimum relative standard deviation of 4%. For diffusive samplers, performance limits are based on the reference values ± 3 standard deviations and the relative standard deviation is assumed to be 6%. The reference value is the calculated value from the generation system. Data are acceptable if they fall within the performance limits. Data falling outside the performance limits are reported as outliers.

Laboratories are rated based on performance in the PAT Program over the last year (i.e., four calendar quarters), as well as on individual contaminant performance. Individual contaminants are metals, silica, asbestos/fibers, organic solvents and diffusive samplers. Individual contaminant performance is rated as (1) proficient if all

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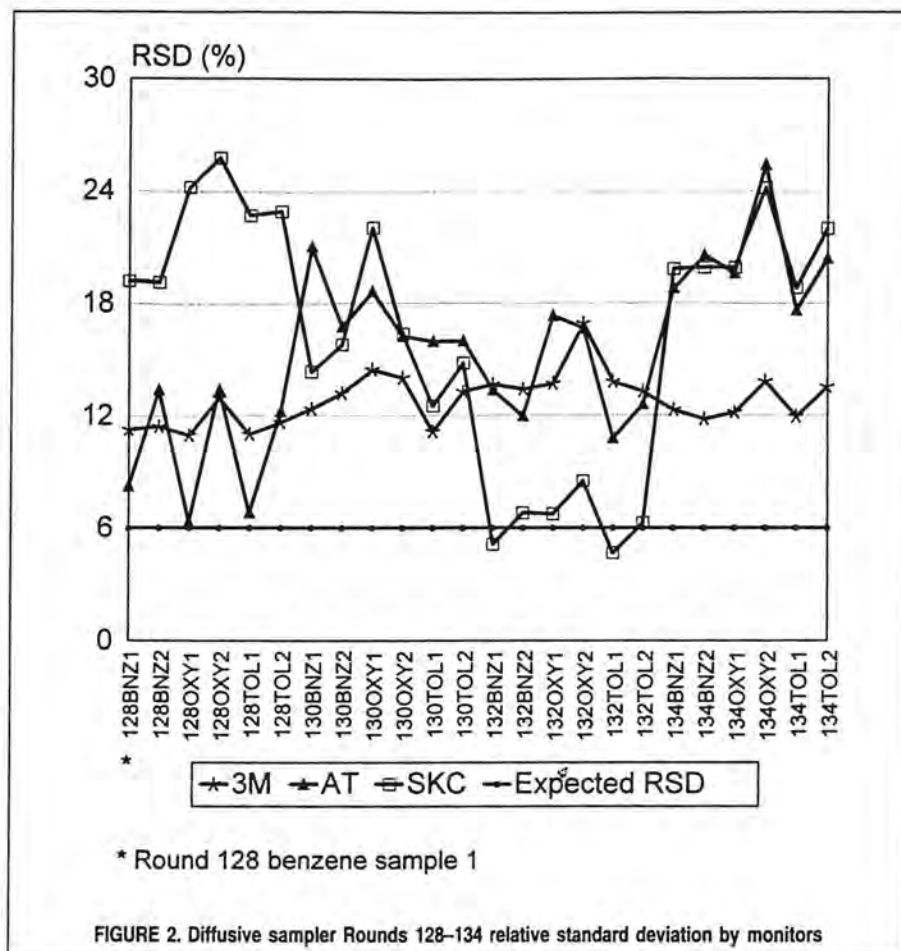


FIGURE 2. Diffusive sampler Rounds 128-134 relative standard deviation by monitors

results have been reported and all are classified as acceptable for the last two consecutive rounds; and (2) proficient in all other cases if three-fourths or more of the results reported in the last four (two rounds per diffusive samplers) consecutive rounds are classified as acceptable.⁽⁵⁾

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