



Background and Current Status

To cite this article: (1999) Background and Current Status, *Applied Occupational and Environmental Hygiene*, 14:5, 292-296, DOI: [10.1080/104732299302864](https://doi.org/10.1080/104732299302864)

To link to this article: <https://doi.org/10.1080/104732299302864>



Published online: 30 Nov 2010.



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PAT Program

Background and Current Status

H. Amy Feng and Paul Schlecht, Column Editors

Introduction

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 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 in-

cludes four sets of samples as shown in Table I. A mixture of three of the four possible metals, and one to three of the fifteen 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, oxylene, 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 \pm 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 percent and a minimum relative standard deviation of 4 percent. For diffusive samplers, performance limits are based on the reference values \pm 3 standard deviations and the relative standard deviation is assumed to be 6 percent. 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 upon 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 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.⁽¹⁾

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 II lists the reference values, performance limits, and participants for each sample type in the PAT Program. Table III presents the summary of the

TABLE I
Current sets of samples in proficiency analytical testing (PAT) program

| | | |
|------------------|-------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------|
| Metals | Cadmium Chromium Lead Zinc | |
| Silica | Quartz | |
| Asbestos/fibers | Amosite Chrysotile Manmade fibers | |
| Organic solvents | Benzene n-Butyl acetate Chloroform 1,2-Dichloroethane p-Dioxane Ethyl acetate Isopropanol Methanol | Methyl ethyl ketone Methyl isobutyl ketone Tetrachloroethylene Toluene 1,1,1-Trichloroethane Trichloroethylene o-Xylene |

TABLE II
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 |

TABLE III
PAT proficiency ratings based upon 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 |

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 four 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

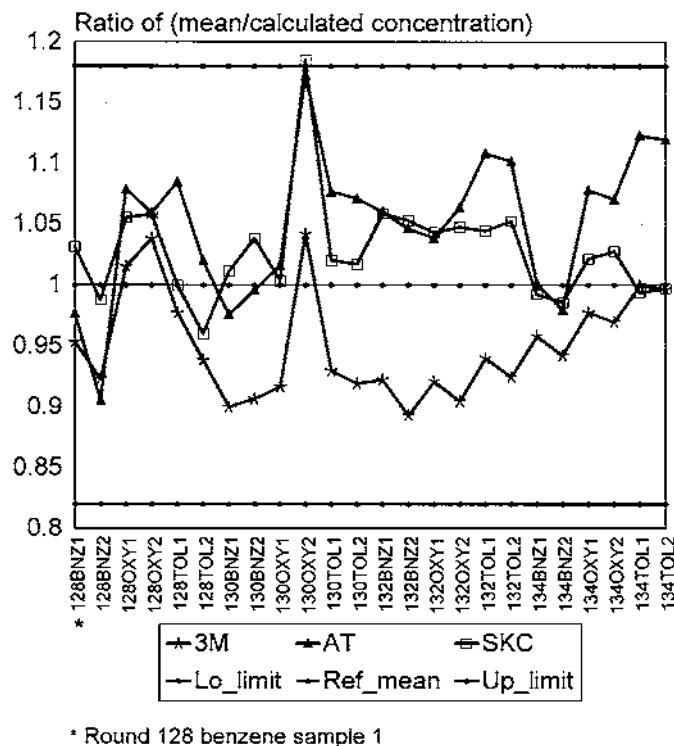


FIGURE 1
Diffusive sampler Rounds 128–134 relative means by samplers.

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 percent (Figure 2). Results from AT and SKC diffusive samplers consistently had higher RSDs. The RSDs ranged from, 6–25 percent for AT samplers, and 5–26 percent 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 percent of the calculated estimate.⁽²⁾ 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 percent. Alternatively, the traditional NIOSH protocol for evaluating diffusive samplers⁽³⁾ requires averaging of at least two independent methods (possibly including calculated estimates) with at least four samples per method. Finally, the Comité Européen de Normalisation (CEN)⁽⁴⁾ has adopted a looser requirement: calculated and independent measurements must agree within ± 10 percent.

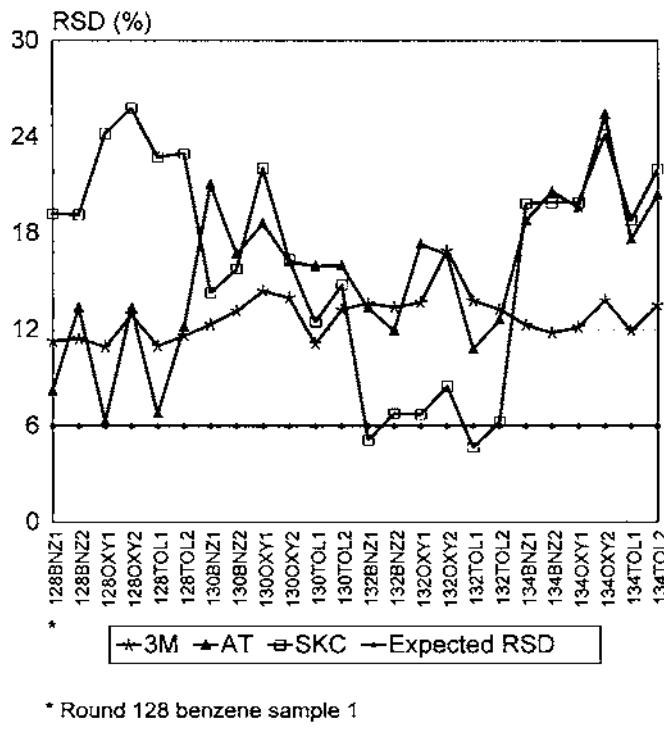


FIGURE 2
Diffusive sampler Rounds 128–134 relative standard deviation by samplers.

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 percent of the AT and 64 percent of the SKC o-xylene estimates were rated *unacceptable*. Many of the ratings of *unacceptable* would be affected by a - 8 percent 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 percent and 29 percent, respectively. The 3M *unacceptable* rate would also drop, from 13 percent to 10 percent.

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.⁽⁵⁾ The calculated concentration

values would serve as a quality control check. If the active and calculated concentrations differ by more than 10 percent, the run would be voided. The RSD for the active samplers should be less than 5 percent, 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⁽⁴⁾ 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. Although 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.

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EDITORIAL NOTE: H. Amy Feng and Paul Schlecht are with the National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering, Quality Assurance, and Statistics Activity.
