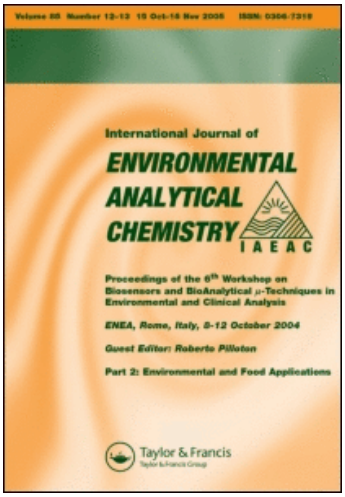

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EVALUATION OF CANDIDATE PROCEDURES FOR THE PREPARATION OF AUDIT MATERIALS FOR ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS

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Audit materials for the SemiVOST method (SW-846 Sampling Method 0010 and Analytical Method 8270) and Standard Operating Procedures for preparation of these audit materials have been developed and are now available from the U.S. Environmental Protection Agency (EPA). The audit materials consist of spiked XAD-2® sorbent. Two procedures were considered: gaseous spiking and liquid spiking. Standard Operating Procedures (SOPs) were prepared based on experience in preparing and analyzing the audit materials. An interlaboratory study involving three laboratories was planned to evaluate the ruggedness and transferability of the standard operating procedures. The initial interlaboratory study was unsuccessful in obtaining a complete data set; however it did demonstrate that a two-week hold time before sorbent extraction did not decrease recoveries of the spiked semivolatile organic compounds. The SOPs were revised after the first interlaboratory study, and a second study involved four laboratories. Three laboratories prepared the audit materials according to the SOPs and all four laboratories analyzed the spiked samples. The complete set of analytical data was statistically evaluated to judge the effectiveness of the SOPs in preparing the audit materials. EPA procedures for preparing audit standards had not been available previously.

Keywords: Semivolatile organic compounds; audit materials; EPA procedures

INTRODUCTION

Title III of the Clean Air Act Amendments of 1990 identifies 189 toxic analytes that the EPA, state, and local air pollution control agencies must regulate and include in State and local permits for all major stationary sources. With a crite-

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rion of boiling point ≥ 100°C to define a semivolatile organic compound, approximately 100 of the Clean Air Act analytes can be classified as semivolatile organic compounds (SVOCs).

Sampling methods for organic compounds in stationary sources very frequently focus on the collection of these compounds on a solid sorbent such as XAD-2® (a styrenedivinylbenzene copolymer), Carbosieve® (a carbon-based sorbent), or Tenax® (a phenylene oxide polymer). Analysis of the sorbed compounds usually requires thermal desorption or extraction procedures to separate the SVOCs from the sorbent and introduce the analytes into the analytical system. Because of the large number of semivolatile organic analytes listed in the Clean Air Act Amendments, development and validation of individual sampling and analytical methods for each analyte are not practical due to either technical considerations or cost. Multi-analyte methods are far more cost-effective than single-analyte methods, and many future regulations or permits will contain:

- Broadly applicable sampling and analytical test methods for multiple toxic SVOCs; and
- Some requirements for on-site certification that the sampling and analytical methodology has accurately measured the Title III SVOC emissions from the stationary source being tested.

The sampling and analytical methodology most commonly applied to the determination of SVOCs in gaseous stationary source emissions is the SemiVOST, which consists of:

SW-846 sampling Method 0010
SW-846 sample preparation proposed Method 3542
SW-846 analytical Method 8270.

The Method 0010 sampling train includes a filter to trap particulate matter and XAD-2® to collect semivolatile organic compounds. Proposed Method 3542 describes in detail the Soxhlet extraction of the filter and XAD-2®, with the associated sampling train glassware rinses, and the separatory funnel extraction of any condensate collected in the sampling train. Method 8270 describes gas chromatography-mass spectrometry (GC-MS) analysis of the methylene chloride extracts of the sampling train components. The SemiVOST does not have a specific list of applicable SVOCs; the methodology may be applied to any SVOC meeting the boiling point criterion. However, this lack of a specific list of applicable analytes raises the question of applicability of the methodology to any specific analyte: if SemiVOST were applied to test for Compound X, would the compound be observed if present in the emission stream?

One way to evaluate the performance of the SemiVOST method is to audit the entire SemiVOST sampling and analytical procedure by dynamically spiking a

solution of SVOCs into a sampling train in the field while the sampling process is occurring. This technique, following EPA Method 301 guidelines, has been applied to perform method validation of the SemiVOST for the Clean Air Act semivolatile organic analytes. However, the EPA Method 301 poses stringent statistical requirements for the validation of a method: use of quadruple sampling trains, with dynamic spiking in two of the four sampling trains, for a minimum of six complete sampling runs, is required. Very few field samplers have the equipment, procedures, and skills required to perform method validation using dynamic spiking techniques, and the cost of the procedure is very high (typically $50,000 – $150,000 for a complete test).

A historical and widely-accepted method to perform on-site certification of sampling and analytical methodology is the use of spiked sorbent media containing known quantities of the SVOCs of interest. This type of procedure does not provide a performance audit of the entire methodology (sampling is excluded from testing), but will test the sample preparation and analysis in the laboratory. Several candidate SOPs for placing semivolatile organic compounds onto solid media for subsequent use as audit materials have been developed in ERG's current work for EPA. Procedures were developed for liquid and gaseous spiking of SVOCs onto Tenax®, liquid spiking onto sampling train filters, and liquid and gaseous spiking onto XAD-2®. Spiked Tenax® tubes were analyzed by thermal desorption followed by GC-MS. Spiked XAD-2® and spiked filters were prepared for analysis by solvent extraction, followed by sample concentration and GC-MS analysis. Standard operating procedures were subsequently prepared for liquid and gaseous spiking of XAD-2® and liquid spiking onto Tenax®.

In a subsequent set of experiments, the three SOPs were evaluated by interlaboratory testing. Using the SOPs, the ERG laboratory prepared spiked sorbent samples. These spiked sorbent samples were analyzed by ERG and two other laboratories. The SOPs were then given to a second laboratory to prepare spiked sorbent materials to be analyzed by all four laboratories. This interlaboratory study did not achieve the objective of evaluating the SOPs for ruggedness and transferability to a second laboratory because one laboratory did not perform the analyses in the first round (spiked samples prepared by ERG), and spiking errors by the second laboratory resulted in a lack of analytical data from all of the laboratories in the second round of spiking and analysis.

The first interlaboratory study did demonstrate, however, that spiked Tenax® analyzed by thermal desorption (the routine analytical mode for Tenax®) is not adequate for meeting EPA Quality Assurance requirements for an audit material because of lack of reproducibility of the analytical results. Also, during a time parameter test, one set of samples was prepared and analyzed immediately and a

second set was prepared and analyzed two weeks later. Statistical analysis showed that the additional two weeks did not affect the analytical results.

A second and more rigorous interlaboratory study was planned and executed to evaluate the applicability of liquid and gaseous spiking of XAD-2® for generation of audit materials for SVOCs.

EXPERIMENTAL

The SOPs describe the procedures for preparing an appropriate spiking solution and performing the actual spiking, documenting those activities, and providing an analytical demonstration of the successful preparation. A set of applicable SVOCs was selected and included in the SOP. These SVOCs were Clean Air Act analytes or analogs, known on the basis of previous laboratory experiments to show a high and reproducible recovery (> 90%) from XAD-2®, to be stable in methylene chloride solution, and to produce good chromatographic response and peak shape for analysis. The compounds were chosen to avoid a serious challenge to laboratory technique or instrument sensitivity since the goal of the audit materials is to evaluate standard laboratory procedures. Also, the compounds listed in the SOP are used as examples. In the actual preparation of audit samples for a field study, the analyte list would be tailored to the requirements of the sampling and analytical program.

Laboratory experience had demonstrated that analytical detection limits for the compounds selected or similar compounds would be in the range of 20–50 μg/mL, so 30 μg/mL was used as a reasonable estimated value for detection limit. The SemiVOST method sample preparation procedures require a final extract volume of 5 mL, so the minimum detection limit was estimated as 150 μg. Spiking solutions were prepared in methylene chloride at a level of 5 times the minimum detection limit (i.e., 750 μg spiked). A second higher level of 10 times the lower level was initially selected for the preparation of spiked sorbent samples, but since this spiking level would produce a sample approximately ten times higher than the highest point of the typical GC-MS calibration range, the higher range was lowered to 1250 μg. This high level would still require dilution for successful analysis.

The SOP for liquid injection presents a detailed discussion on the technique required to accurately and reproducibly inject liquid into a nominal 40 g bed of clean XAD-2® in the SemiVOST sampling module. The standard procedure for gaseous injection of analytes onto the sorbent bed provides a diagram of a device designed and constructed for performing flash evaporation of SVOCs in methyl-

ene chloride solution onto the bed of XAD-2® in the SemiVOST sampling module. Because the XAD-2® sampling module connects to the sampling train by a ball joint, modification of the standard injection port of a gas chromatograph was required.

The following program design was used for the second interlaboratory study:

- Select three other laboratories to participate in the interlaboratory evaluation of the SOPs;
- Select the list of analytes to be spiked on the sorbents for the interlaboratory study.;
- Using the two SOPs prepared in the ERG laboratory, prepare spiked sorbents for analysis by all four participating laboratories.
- Perform sample preparation and analysis of the audit materials prepared in the ERG laboratory;
- Supply cleaned glassware to the second laboratory to perform spiking using the SOPs;
- Supply an aliquot of the ERG spiking solution for use as a check sample for the second spiking laboratory, along with standard operating procedures and any other specific instructions.
- All four laboratories prepare and analyze samples spiked by the second laboratory;
- Supply cleaned glassware to the third laboratory to perform spiking using the SOPs;
- Supply an aliquot of the ERG spiking solution for use as a check sample for the third spiking laboratory, along with SOPs and any other specific instructions;
- Collect data from all four participating laboratories, review, and submit consolidated data set to EPA for statistical evaluation.

Sample preparation and analytical procedures for the laboratories analyzing the spiked samples were not specified, but EPA Proposed Method 3542 for sample preparation was recommended (and supplied to the laboratories) and Method 8270 for analysis was recommended. A final extract volume of 5 mL was, however, specified. Laboratories were advised of the high spiking level so that the surrogate compounds required for Method 8270 could be spiked at appropriate levels and two analyses would not be required to obtain surrogate compound recoveries and accurate quantitative analysis of the compounds of interest. Each laboratory used their own current internal procedures for preparation and quality control of the XAD-2® sorbent and for filling the XAD-2® sampling modules (supplied by ERG), and for preparing spiking solutions and instrument calibration solutions (a calibration range was recommended but not required) from neat

chemicals. A control sample supplied by ERG was available for reference to verify preparation of spiking solutions. Laboratories were not required to use exactly the spiking levels specified, but a range was specified and laboratories were required to be within the range. Additional sorbent was supplied by each spiking laboratory for use as a laboratory blank to monitor contamination in the sample preparation and analysis process.

Detailed instructions were provided for packing and shipping the spiked sorbents, as well as returning glassware so that the glassware could be cleaned and made available to the next laboratory to perform spiking. Analytical data were reported according to the standard procedures for data reporting in each laboratory. Recoveries of surrogate compounds were reported, but data were not corrected for surrogate recoveries.

RESULTS AND DISCUSSION

All analytical data were statistically analyzed to evaluate the ability of the external laboratories to accurately and reproducibly execute the procedures described in the SOPs.

Statistical Analysis

The purpose of this study was to examine the effects on recovery of spiked compounds of the medium by which spiking of the sorbent was achieved (flash evaporation [i.e., gaseous spike] vs. direct liquid solution). Percent recovery (or lack of recovery) is expressed as a percent difference in the following equation:

$$R = 100 \times \frac{\text{Measured Amount}(\mu g) - \text{Spiked Amount}(\mu g)}{\text{Spiked Amount}(\mu g)} \quad (1)$$

All individual measurements were transformed to a percent difference prior to statistical analysis. Typically, percent differences in the range of ± 10% represent very good (better than 90%) recovery. On the other hand, measurements that differ at least an order of magnitude from the spike have percent differences outside the range (–90%, 900%). A large difference is often the result of gross mistakes such as decimal point errors during transcription or calculation, or possible contamination in the laboratory. In this study, two measurements were excluded from the statistical analysis as potential outliers. Both values-one above 4400% and the other above 700% – were toluene measurements, both resulting from laboratory contamination as demonstrated by the analysis of the blanks included with the spiked samples.

Figure 1 presents a dotplot[1] which shows the percent differences for each medium (circles for gas and plus signs for liquid) for each compound at both low and high spike levels. The averages were based on the following linear model describing the percent differences for each compound at each spike level:

$$R_{ijkl(m)} = \mu_{(m)} + M_{i(m)} + P_{j(m)} + PM_{ij(m)} + L_{k(m)} + ML_{ik(m)} + PL_{jk(m)} + PML_{ijk(m)} + E_{ijkl(m)} \quad (2)$$

where the subscript (m) indicates that we are dealing with data for spike level m only; $R_{ijkl(m)}$ is the 1^{th} replicate percent difference within the k^{th} laboratory(L) during the j^{th} phase(P) using the i^{th} medium(M). The parameter $\mu_{(m)}$ represents the overall mean. The different phases represent the preparation of audit samples by different laboratories (i.e., Phase 1 includes spiked samples prepared by Laboratory 1). The combination of letters in the model represent interactions between different variables in the experiment. For example, the term $ML_{ik(m)}$ measures the extent to which different laboratories disagree in their assessment of medium differences (gas vs. liquid spiking) at spike level m. Measurement error is represented by the term $E_{ijkl(m)}$. Three replicate measurements were made by each of four laboratories on samples prepared during three different phases using two media (gas and liquid) at two different spike levels. Each point in Figure 1 represents the average of 36 values and is estimated by equation (3):

$$\bar{R}_{i(m)} = \sum_{jkl(m)}^{36} \frac{R_{ijkl(m)}}{36}. \quad (3)$$

Referring to Figure 1, the average percent recovery of both media changes from compound to compound. However, the difference between gas and liquid media remains fairly stable across compounds for both high and low spikes. Averaged over compounds, the liquid medium is 15 percentage points higher than the gas medium at the high spike and 14 percentage points higher at the low spike. The overall average percent recovery for the liquid medium was 5% less than the high spike and 11% less than the low spike. The average recovery for the gas medium was 20% less than the high spike and 25% less than the low spike.

Statistically comparing the two media using the difference between their average recovery for each compound involves estimating uncertainty based on the variation in the measurements. The total variance of the mean for each medium at each different level and compound is made up of components of variance corresponding to the random variables in the linear model and represented here by equation(4):

$$\sigma^2_{Tot} = \frac{\sigma^2_E}{36} + \frac{\sigma^2_{PML}}{12} + \frac{\sigma^2_{PL}}{12} + \frac{\sigma^2_{PM}}{3} + \frac{\sigma^2_P}{3} + \frac{\sigma^2_{ML}}{4} + \frac{\sigma^2_L}{4}. \quad (4)$$

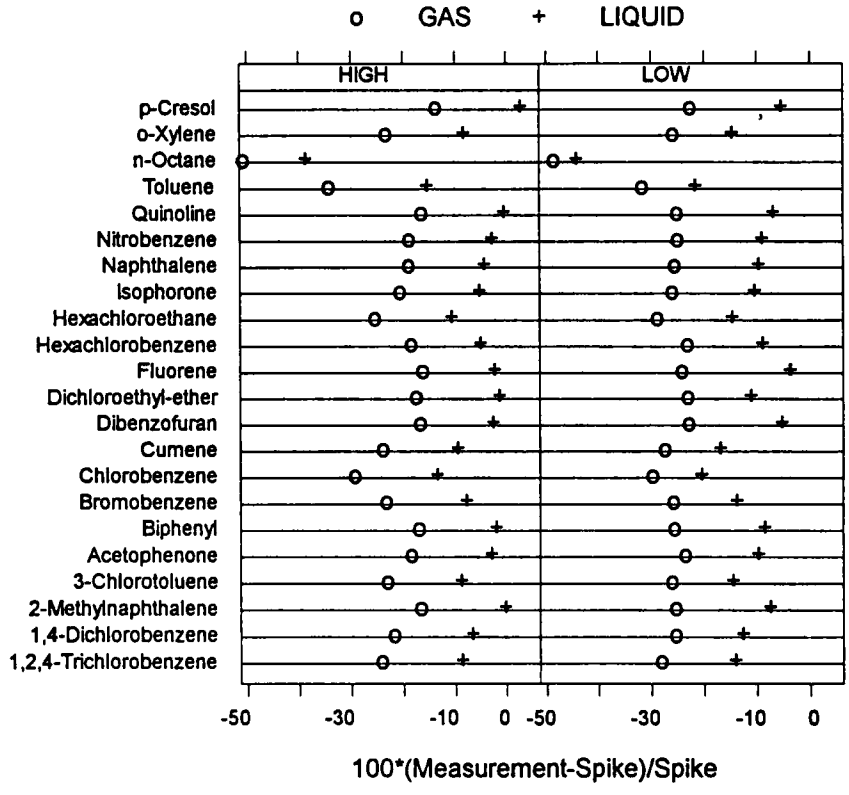

FIGURE 1 Dotplots of Means for Gas and Liquid Media at Low and High Spikes

Even under the best of conditions a variation generally exists between replicate measurements of different samples of the same audit material. Using Analysis of Variance techniques, estimates of the variance components making up the total variance for a medium (i.e., gas vs. liquid spiking) average were determined at low and high spikes for each compound. Considering for the moment only the variation between replicate measurements within each laboratory (i.e., $\sigma^2_E/36$), the variance associated with each point in Figure 1 at the high spike is 13.9. This variance was determined by estimating $\sigma^2_E/36$ for each individual compound and then averaging over all compounds. For individual compounds the variance ranged from 6.8 (*n*-octane) to 19.8 (*p*-cresol). The average standard error is 3.7 percentage points and the 95% confidence interval (48 degrees of freedom) for the mean is ± 7.5 percentage points. The critical value for the statistical separation between gas and liquid spiking media is 10.6 percentage points. Therefore,

taking into account only the uncertainty in the replicates, the difference of 15 percentage points between media at the high spike is real.

The estimate of variance associated with the low spike level is 2.5 with a range depending on the compound of 1.6 (*n*-octane) to 4.2 (hexachlorobenzene). The average standard error is 1.6 percentage points and the 95% confidence interval is ± 3.2 percentage points. The critical value for differences between medium averages at the low spike level is 4.5 percentage points. So, there also appears to be a real difference between spiking media at the low spike based only on the variation among the replicates.

One of the reasons for separately analyzing the data at different spike levels is to examine the effects of levels on the variance of replicate measurements within the same laboratory. The variance at the high level was approximately 6 times (13.9/2.5) greater than at the low level. This ratio is marginally significant for a p-value of 0.06 based on an F-test with 48 degrees of freedom in the numerator and denominator. Another way of looking at this outcome is that the measurement method is relatively more efficient at the low level since a single measurement at the low level is as precise as the average of 6 measurements at the high level.

Up to this point discussion of uncertainties in the data has been strictly limited to the variation between replicate measurements within a laboratory. Inference was limited to only those laboratories participating in the study. In order to broaden inference to cover a larger population of laboratories capable of both preparing and measuring samples, the laboratories in this experiment were considered a random sample from the larger population. Estimates of variation between laboratoriés, between phases, plus interaction variation between laboratories and phases in addition to two-way and three-way interaction between media, phases, and laboratories were combined with the within-laboratory replicate variation to produce an estimate of "total" variation for a medium average. This value is represented by equation (4). The results indicate where most of the uncertainty in both the preparation and measurement of audit samples lies and where more control in the process is required.

The total variance associated with each dot (representing a gaseous vs. liquid medium average) in Figure 1 is estimated to be 94.5 for the high spike level. The standard error is 9.7 percentage points and the 95% confidence interval (4 degrees of freedom) is ± 27.0 percentage points. The total variance of each dot at the low spike level is 84.4. The standard error is 9.1 percentage points and the 95% confidence interval (4 degrees of freedom) is ± 25.0 percentage points. For both spike levels, the critical difference for comparing spiking media is approximately ± 38.0 percentage points. The conclusion is that we cannot detect a differ-

ence between gaseous and liquid spiking media using this broader interpretation of uncertainty in the audit procedure.

For the high level spike with a total variance of σ^2_{Tot}= 94.5, the variation between replicate measurements ($\sigma^2_E/36$) accounts for only 15% of the total. Variation between laboratories ($\sigma^2_L/4$) accounts for 20% of the total. The variation in the difference between media from laboratory to laboratory ($\sigma^2_{ML}/4$) accounts for 26% of the total variance and the difference between laboratories from phase to phase ($\sigma^2_{PL}/12$) contributed 30% of the total variation. Variation between phases ($\sigma^2_P/3$) accounts for only 2% of the total. The remaining 7% total variation is attributed to differences between media across phases ($\sigma^2_{PM}/4$). It appears that most of the variation at the high spike is due to differences between laboratories plus the way these differences vary across spiking media and across phases (successive sequences of spiking and analysis). These percent contributions of the different components of variation to the total given here represent an average across compounds.

The components of variance for the low level spike are distributed in a completely different manner across the total variance of 84.4. The variation between replicates accounts for only 3% of the total. On the other hand, laboratory differences account for 59% of the total and phase-to-phase differences account for nearly 28% of the total. The interaction between media and laboratories accounts for only 4% of the total variance, while all other components combined account for less than 6% of the total. It appears that most of the uncertainty at the low spike is due to laboratory differences during both the measurement process and the preparation of the samples. Again, these percent contributions at the low spike represent an average across compounds.

To get a better understanding of how media differ across laboratories and spiking/analysis sequences at low and high spikes, Figure 2 and 3 show multiway dotplots of medium averages for all laboratory-phase combinations. Each point on the graphs represents a single *test result* defined as the average percent difference for 3 replicate measurements. The within-laboratory variance associated with each point in Figure 2 (low spike) is 30.2. The standard error is 5.5 percentage points and the 95 % confidence limit (48 degrees of freedom) is ± 11 percentage points. The critical difference between media for any specified laboratory-phase-compound combination at the low spike is 15.6 percentage points. The within-laboratory variance associated with each point in Figure 3 (high spike) is 167.0. The standard error is 12.9 percentage points and the 95% confidence interval is ± 26.0 percentage points. The critical value for comparing media at a specified laboratory-phase combination is 36.7 percentage points. These critical differences could also apply to the comparisons between two test results from the same medium. In any case, these comparisons reflect the uncer-

tainty of a single test result measured and prepared under tightly specified conditions.

Referring to Figure 2 and 3 it is not difficult to see how the differences between spiking media can vary from laboratory-to-laboratory, from phase-to-phase, and across different laboratory-phase combinations to contribute to the total uncertainty of a single test result. To compare test results for any two different laboratories for a given phase or between two phases for a given laboratory also requires a broader interpretation of uncertainty. The total uncertainty associated with randomly selecting a single test result from any laboratory xphase combination is given by the following equation expressing the total variance as the sum of its individual components:

$$\sigma^2_{Tot} = \frac{\sigma^2_E}{3} + \sigma^2_{PML} + \sigma^2_{PL} + \sigma^2_{PM} + \sigma^2_P + \sigma^2_{ML} + \sigma^2_L. \quad (5)$$

As before, the assumption to expand the scope of inference is that the laboratories in this study are representative of a larger population of laboratories capable of preparing and measuring audit samples using the methods described in this paper. Under this broader interpretation of uncertainty, the total variance associated with each point in Figure 2 is 360.5. The standard error is 19.0 percentage points and the 95% confidence interval (4 degrees of freedom) is ± 53.2 percentage points. The total variance for each point in Figure 3 is 707.2. The standard error is 26.6 percentage points and the 95% confidence interval is ± 74.5 percentage points.

The critical value for comparing any two test results at the nigh spike is 105.3 percentage points and for the low spike is 75.1 percentage points. This critical difference is not necessarily limited to comparing test results from two media but also applies to comparing two test results in general. Such a comparison deals more with "specifications" for an individual test result under a much broader interpretation of uncertainty. For example, the critical values for comparing two test results from different media also applies to comparing two test results from the same medium measured in two different laboratories or prepared during two different phases. Obviously, not specifying which laboratory prepares the samples and which laboratories make the measurements inflates the uncertainty associated with each test result regardless of which medium is used.

SUMMARY

Limiting the outcome of this experiment to only those laboratories participating in the study, it must be concluded that the liquid spiking media produce a signifi-

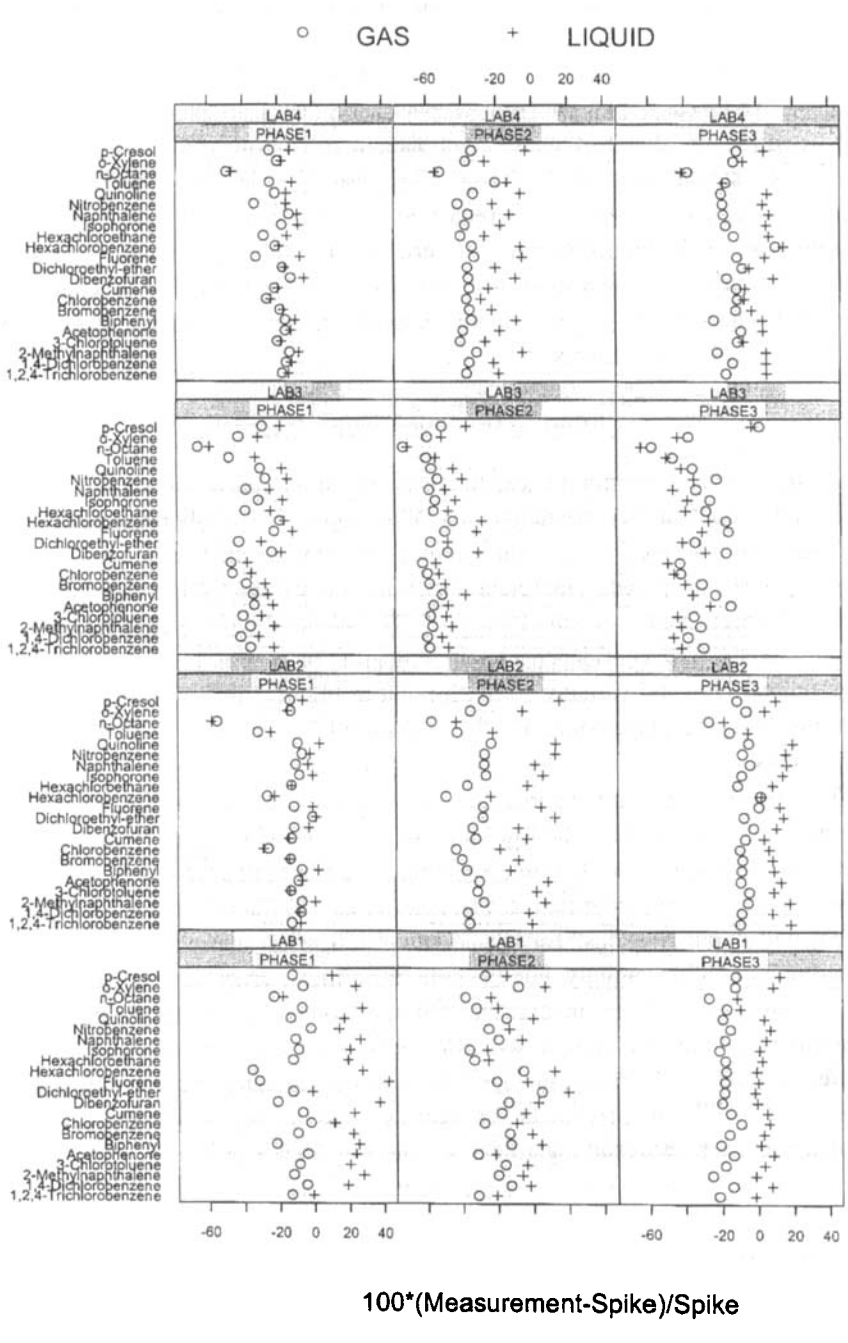

FIGURE 2 Multiway Dotplots of Means for Gas and Liquid Medium at Low Level Spike

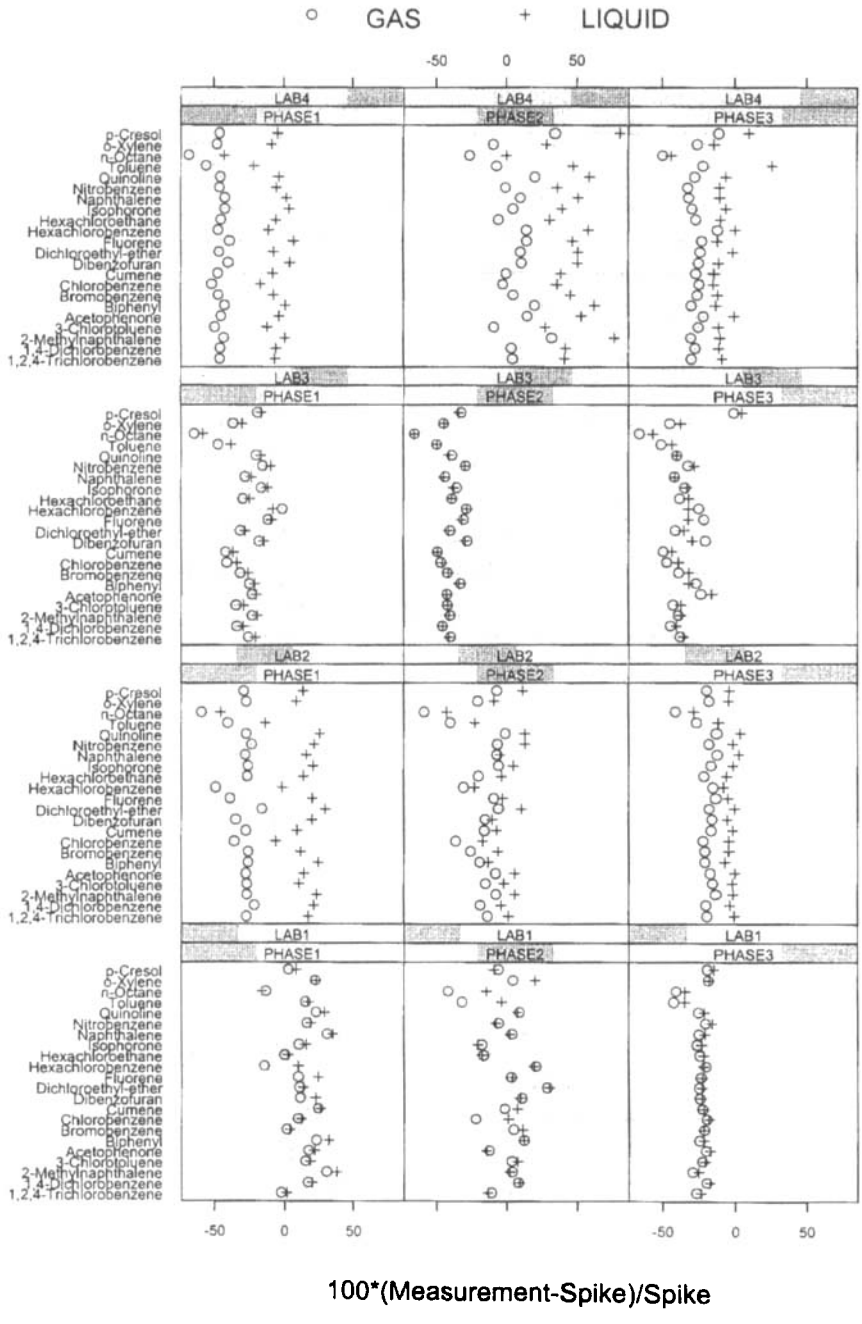

FIGURE 3 Multiway Dotplots of Means for Gas and Liquid Medium at High Level Spike

cantly higher recovery than the gas medium. However, this claim is invalid in the presence of additional uncertainty. Considering laboratories as random variables broadens the scope of inference to cover a larger population of laboratories. This broadening of scope necessarily inflates the variation and widens the confidence intervals so that the difference between media is no longer significant. The inherent differences between laboratories cannot be reduced by taking more measurements. This only applies to reducing the variation of a single test result. It can be argued that the variability among laboratories during both preparation and measurement must be included in determining the uncertainty which reflects the reliability of the method.

The spike levels have a definite effect on variation between replication errors. There is also a difference in the way the components of variance distribute across the total variance at different spike levels. At the low spike level, laboratories, phases, plus two-way and three-way interactions account for 97% of the total variation in medium averages. At the high spike level, this additional uncertainty accounts for 85% of the total variance associated with a medium average.

In this experiment, a test result is defined as the average of three replicate measurements of the same test material. At the low spike level, two test results prepared in the same laboratory and measured by a designated laboratory should not differ by more than 15.6 percentage points but 1 time in 20 due to chance alone. This observation also applies to two test results from different media. The same comparisons at the high spike can differ by as much as 36.7% without being significant. Under the broader interpretation of uncertainty the critical difference for a test result is 75.1 percentage points at the low spike, and 105.3 percentage points at the high spike.

CONCLUSION

Statistical evaluation of the data shows that the variances observed are due more to analysis and the spiking/analysis sequence than spiking techniques. The two SOPs are statistically acceptable methods for the preparation of audit and performance evaluation standards for SVOCs collected on XAD-2® sorbent. These SOPs are available from the National Exposure Research Laboratory, U. S. Environmental Protection Agency, Research Triangle Park, NC 27711.

Participation of the following laboratories in the interlaboratory studies is gratefully acknowledged: Air Toxics, Ltd., Environmental Science and Engineering, ManTech Environmental Technology, Inc., and Triangle Laboratories, Incorporated.

DISCLAIMER

Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the U. S. Environmental Protection Agency.

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