# LESSONS LEARNED FROM PERFORMANCE EVALUATION STUDIES

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## ABSTRACT

Performance Evaluation (PE) samples are routinely utilized by both the regulatory and regulated communities to demonstrate a laboratory's proficiency in performing a given analytical method. PE samples are submitted to laboratories for a wide variety of regulatory programs and are typically prepared in deionized water, clean soil, or other prepared media. The laboratory's reported results are compared to the known identities and concentrations of target analytes in the PE samples. The evaluation of the laboratory's performance is typically based upon the percentage of analytes the laboratory successfully recovered within a defined range of acceptance limits. However, typically executed PE studies do not provide an indication of the laboratory's ability to successfully identify and quantitate target analytes in a complex matrix or test other non-analytical aspects of the laboratory's operation.

This presentation will focus on the authors' experience in conducting PE studies for a multi-state pipeline project and will present the findings relative to these studies. Information gleaned from the PE studies relative to the evaluation of the laboratory's performance will be discussed. Furthermore, observations regarding the laboratories' performance in analyzing multi-phasic samples will be presented.

## INTRODUCTION

Performance Evaluation (PE) samples are test samples that are prepared by spiking known concentrations of select analytes into a well-characterized matrix. Typically, PE samples are made in a single matrix such as an aqueous, solid, or an oil matrix. PE samples can be distributed as single-blind or as double-blind samples. For single-blind PE samples, the laboratory is informed that they will be receiving a test sample. In the case of double-blind PE samples, the test samples are given fictitious sample identifications and are submitted concurrently with other project samples to the laboratory. That is, for double-blind PEs samples, the laboratory does not know that the fictitiously labeled PE sample is a test sample. Typically, PE samples are utilized to determine a laboratory's accuracy as it relates to the execution of a particular analytical methodology.

The authors have participated in the maintenance of a number of corporate laboratory programs in the capacity of performing quality assurance/quality control (QA/QC) oversight for these programs. In these roles, the authors have had experience in procuring, distributing, and evaluating the results from PE studies. However, this paper will focus on the lessons learned from one particular project.

As the QA/QC oversight contractor on a 19,000 mile pipeline that stretches across nine of the United States, quarterly PE samples have been submitted to the seven project laboratories for approximately three years. At the onset of the project, a laboratory specification manual was prepared that identified prescribed SW-846 preparative and analytical methods for the program execution. Where method ambiguities existed, program-specific method requirements were established. In addition, the laboratory specification manual listed the target analytes, associated reporting limits, QC requirements (including frequency, QC limits, acceptance criteria and corrective action), and data deliverable specifications (electronic and hard copy). By establishing a corporate laboratory specification manual that all seven project laboratories were required to follow, data inconsistencies were minimized and data comparability was enhanced.

Typically, PE samples are utilized to demonstrate method proficiency based upon the accuracy of the laboratory-reported results compared to the known certified values. However, more information can be gleaned from a PE study than a laboratory's demonstration of method proficiency, particularly in the case when a laboratory specification manual is utilized for a laboratory program and when full data package deliverables are requested to substantiate the reported analytical results. Information relative to the evaluation of the laboratory's technical and administrative services, sample login and receipt, data package preparation, method compliance, and quality assurance can also be evaluated.[1] In addition, in this particular project, the authors were able to utilize the ongoing PE studies to identify laboratory specific trends, program specific trends, and to determine overall precision amongst the project laboratories.[2] These trends have been utilized to provide feedback to the project laboratories to enhance their overall performance.

Careful consideration was given to the preparation of the PE samples for the subject project. The intent was to test the project laboratories' ability to analyze samples that were similar in matrix and composition to the project samples for the analyses of interest. As such, the PE samples were custom-prepared by a reputable PE vendor for the analytes of interest (volatiles, polyaromatic hydrocarbons [PAHs], polychlorinated biphenyls [PCBs], and metals [including mercury]). The PE samples were soil samples that were carefully manufactured by mixing clay and sand in proper proportion and sieve size such that the real world matrix would be stable, homogeneous, and suitable for application of the spiked analytes. The PE samples were also moistened with deionized water to make a multi-phasic test sample (*viz*., moist soil). The analytes were spiked into the PE samples at a concentration roughly three to five times the reporting limits. The project reporting limits were based upon state cleanup action levels.

Since the PE samples were custom-made for the subject project, verification of the manufacturing process was important. Prior to distribution, the PE vendor verified (at their own production facility) that the recoveries of the spiked analytes in the PE samples were acceptable for distribution to the project laboratories. In addition to the distribution of the custom-made pre-moistened soil PE samples to the project laboratories, the PE samples were submitted to three referee laboratories, with one of these referee laboratories receiving the PE samples in triplicate. Use of the referee laboratories allowed for additional independent verification of the manufacturing process. It should be noted that the project laboratory specification manual was distributed to the referee laboratories to prescriptively follow for the analysis of the PE samples.

All PE samples were carefully shipped to the project laboratories and referee laboratories simultaneously. The PE samples were shipped via overnight courier in an iced cooler, under Chain-of-Custody. For single-blind PE sample rounds, the bottleware for the PE samples was provided by the PE provider. For double-blind PE sample rounds, the bottleware for the PE samples originated from the project laboratory via a request from the project sampling teams.

PE sample results are typically evaluated by comparing the laboratory-reported result to the certified true value and determining the accuracy of the reported analytical results as a percentage relative to the true or certified value. For the subject project, the PE sample results were evaluated in this manner and in two other ways. The first way was to compare the laboratory-reported result to the mean result of the referee laboratories and determine a percentage. The second way was to compare the laboratory-reported result to the historical average result and determine a percentage. The historical average result was based upon the large database of results obtained from the PE supplier for the analyte of interest from previous PE samples that they prepared and distributed in a similar manner.

The limits utilized for evaluating the PE samples were comparable to matrix spike limits typically observed for the analytical methods. That is, for the volatile organic analysis, the recovery limits of 70-130% were utilized. For the PAH analysis, recovery acceptance limits of 30-130% were utilized. For the PCB fraction, recovery acceptance limits of 60-130% were utilized. For the metals fraction, recovery acceptance limits of 75-125% were utilized. Finally, for the mercury fraction, recovery acceptance limits of 80-120% were utilized.

**RESULTS**

During the last two quarters of 1998, two single-blind PE sample studies were conducted for the subject project. The results for the two studies are tabulated as follows. The first table in each of the two PE studies is a comparison of the laboratory-reported results against the certified true value. The second set of tables in each of the two PE studies is a comparison of the laboratory-reported results against the mean referee-reported results. The third set of tables in each of the two PE studies is a comparison of the laboratory-reported results against the historical average (as previously discussed).

**ROUND 1.** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)***[4] | ***True Value*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 50 | 43 | 86.00% | 51 | 102.00% | 21 | 42.00% | 26 | 52.00% | 63 | 126.00% | 37 | 74.00% | 50 | 100.00% |
| 1,1,1-trichloroethane (µg/kg) | 75 | 62 | 82.67% | 75 | 100.00% | 29 | 38.67% | 33 | 44.00% | 79 | 105.33% | ND | | 27 | 36.00% |
| 1,1,2,2-tetrachloro-ethane (µg/kg) | 97 | 40 | 41.24% | 92 | 94.85% | 53 | 54.64% | 76 | 78.35% | 3 | | 110 | 113.40% | 45 | 46.39% |
| carbon tetrachloride (µg/kg) | 79 | 53 | 67.09% | 71 | 89.87% | 30 | 37.97% | 32 | 40.51% | 63 | 79.75% | 53 | 67.09% | 33 | 41.77% |
| chlorobenzene (µg/kg) | 63 | 58 | 92.06% | 77 | 122.22% | 27 | 42.86% | 43 | 68.25% | 72 | 114.29% | 82 | 130.16% | 59 | 93.65% |
| ethylbenzene (µg/kg) | 25 | 24 | 96.00% | 23 | 92.00% | 10 | 40.00% | 16 | 64.00% | 31 | 124.00% | 30 | 120.00% | 24 | 96.00% |
| 4-methyl-2-pentanone (µg/kg) | 247 | 170 | 68.83% | 230 | 93.12% | 190 | 76.92% | 110 | 44.53% | 230 | 93.12% | 290 | 117.41% | 82 | 33.20% |
| tetrachloroethene (µg/kg) | 44 | 45 | 102.27% | 48 | 109.09% | 18 | 40.91% | 18 | 40.91% | 51 | 115.91% | 49 | 111.36% | 48 | 109.09% |
| trichloroethene (µg/kg) | 35 | 38 | 108.57% | 57 | 162.86% | 21 | 60.00% | 20 | 57.14% | 69 | 197.14% | 31 | 88.57% | 33 | 94.29% |
| total xylenes (µg/kg) | 198 | 190 | 95.96% | 260 | 131.31% | 85 | 42.93% | 130 | 65.66% | 230 | 116.16% | 267 | 134.85% | 210 | 106.06% |
| Aroclor 1248 (µg/kg) | 342 | 55 | 16.08% | 310 | 90.64% | 240 | 70.18% | 200 | 58.48% | 270 | 78.95% | 329 | 96.20% | 264 | 77.19% |
| anthracene (µg/kg) | 3,510 | 71 | | 880 | 25.07% | 820 | 23.36% | 82 | | 500 | 14.25% | 470 | 13.39% | 1,300 | 37.04% |
| chrysene (µg/kg) | 2,040 | 490 | 24.02% | 1,300 | 63.73% | 560 | 27.45% | 170 | | 840 | 41.18% | 1,300 | 63.73% | 1,500 | 73.53% |
| benzo(k)fluoranthene (µg/kg) | 2,610 | 590 | 22.61% | 1,700 | 65.13% | 660 | 25.29% | 160 | | 1,400 | 53.64% | 1,800 | 68.97% | 2,100 | 80.46% |
| benzo(a)pyrene (µg/kg) | 4,370 | 380 | | 1,200 | 27.46% | 750 | 17.16% | 180 | | 1,400 | 32.04% | 1,000 | 22.88% | 2,000 | 45.77% |
| indeno(1,2,3-cd)pyrene (µg/kg) | 2,880 | 380 | 13.19% | 1,100 | 38.19% | 690 | 23.96% | 180 | | 710 | 24.65% | 1,800 | 62.50% | 2,000 | 69.44% |
| fluoranthene (µg/kg) | 3,720 | 780 | 20.97% | 2,000 | 53.76% | 900 | 24.19% | 370 | | 1,100 | 29.57% | 2,400 | 64.52% | 2,600 | 69.89% |
| naphthalene (µg/kg) | 4,600 | 1,300 | 28.26% | 2,400 | 52.17% | 1,500 | 32.61% | 980 | 21.30% | 1,800 | 39.13% | 1,500 | 32.61% | 2,700 | 58.70% |
| antimony (mg/kg) | 135 | 32.0 | 23.70% | 46.9 | 34.74% | 57.5 | 42.59% | 66.1 | 48.96% | 31.3 | 23.19% | 68.4 | 50.67% | 47.0 | 34.81% |
| arsenic (mg/kg) | 14.5 | 9.7 | 66.90% | 14.7 | 101.38% | 10.9 | 75.17% | 14.5 | 100.00% | 14.6 | 100.69% | 16.4 | 113.10% | 14.4 | 99.31% |
| barium (mg/kg) | 496 | 280 | 56.45% | 451 | 90.93% | 359 | 72.38% | 406 | 81.85% | 348 | 70.16% | 476 | 95.97% | 401 | 80.85% |
| beryllium (mg/kg) | 6.06 | 4.1 | 67.66% | 5.8 | 95.71% | 4.7 | 77.56% | 5.9 | 97.36% | 5.5 | 90.76% | 6.3 | 103.96% | 5.6 | 92.41% |
| cadmium (mg/kg) | 21.3 | 12 | 56.34% | 20.5 | 96.24% | 14.3 | 67.14% | 18.6 | 87.32% | 10.4 | 48.83% | 21.0 | 98.59% | 17.5 | 82.16% |
| chromium (mg/kg) | 234 | 150 | 64.10% | 225 | 96.15% | 182 | 77.78% | 229 | 97.86% | 204 | 87.18% | 255 | 108.97% | 220 | 94.02% |
| lead (mg/kg) | 543 | 340 | 62.62% | 505 | 93.00% | 372 | 68.51% | 501 | 92.27% | 442 | 81.40% | 555 | 102.21% | 472 | 86.92% |
| mercury (mg/kg) | 36.5 | 28.0 | 76.71% | 21.6 | 59.18% | 20.0 | 54.79% | 34.3 | 93.97% | 30.7 | 84.11% | 34.5 | 94.52% | 30.4 | 83.29% |
| nickel (mg/kg) | 292 | 150 | 51.37% | 269 | 92.12% | 186 | 63.70% | 241 | 82.53% | 186 | 63.70% | 273 | 93.49% | 239 | 81.85% |
| silver (mg/kg) | 413 | 140 | 33.90% | 114 | 27.60% | 275 | 66.59% | 344 | 83.29% | 275 | 66.59% | 428 | 103.63% | 233 | 56.42% |

**ROUND 1 (Cont.)** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)[4]*** | ***Referee Mean*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 28 | 43 | 153.57% | 51 | 182.14% | 21 | 75.00% | 26 | 92.86% | 63 | 225.00% | 37 | 132.14% | 50 | 178.57% |
| 1,1,1-trichloroethane (µg/kg) | 39 | 62 | 158.97% | 75 | 192.31% | 29 | 74.36% | 33 | 84.62% | 79 | 202.56% | ND | | 27 | 69.23% |
| 1,1,2,2-tetrachloro-ethane (µg/kg) | 51 | 40 | 77.97% | 92 | 179.34% | 53 | 103.31% | 76 | 148.15% | 3 | | 110 | 214.42% | 45 | 87.72% |
| carbon tetrachloride (µg/kg) | 39 | 53 | 136.95% | 71 | 183.46% | 30 | 77.52% | 32 | 82.69% | 63 | 162.79% | 53 | 136.95% | 33 | 85.27% |
| chlorobenzene (µg/kg) | 40 | 58 | 143.92% | 77 | 191.07% | 27 | 67.00% | 43 | 106.70% | 72 | 178.66% | 82 | 203.47% | 59 | 146.40% |
| ethylbenzene (µg/kg) | 16 | 24 | 150.00% | 23 | 143.75% | 10 | 62.50% | 16 | 100.00% | 31 | 193.75% | 30 | 187.50% | 24 | 150.00% |
| 4-methyl-2-pentanone (µg/kg) | 90 | 170 | 188.89% | 230 | 255.56% | 190 | 211.11% | 110 | 122.22% | 230 | 255.56% | 290 | 322.22% | 82 | 91.11% |
| tetrachloroethene (µg/kg) | 24 | 45 | 189.87% | 48 | 202.53% | 18 | 75.95% | 18 | 75.95% | 51 | 215.19% | 49 | 206.75% | 48 | 202.53% |
| trichloroethene (µg/kg) | 21 | 38 | 183.57% | 57 | 275.36% | 21 | 101.45% | 20 | 96.62% | 69 | 333.33% | 31 | 149.76% | 33 | 159.42% |
| total xylenes (µg/kg) | 138 | 190 | 137.98% | 260 | 188.82% | 85 | 61.73% | 130 | 94.41% | 230 | 167.03% | 267 | 193.90% | 210 | 152.51% |
| Aroclor 1248 (µg/kg) | 242 | 55 | 22.73% | 310 | 128.10% | 240 | 99.17% | 200 | 82.64% | 270 | 111.57% | 329 | 135.95% | 264 | 109.09% |
| anthracene (µg/kg) | 820 | 71 | | 880 | 107.32% | 820 | 100.00% | 82 | 10.00% | 500 | 60.98% | 470 | 57.32% | 1,300 | 158.54% |
| chrysene (µg/kg) | 1,447 | 490 | 33.86% | 1,300 | 89.84% | 560 | 38.70% | 170 | 11.75% | 840 | 58.05% | 1,300 | 89.84% | 1,500 | 103.66% |
| benzo(k)fluoranthene (µg/kg) | 1,737 | 590 | 33.97% | 1,700 | 97.87% | 660 | 38.00% | 160 | | 1,400 | 80.60% | 1,800 | 103.63% | 2,100 | 120.90% |
| benzo(a)pyrene (µg/kg) | 1,755 | 380 | 21.65% | 1,200 | 68.38% | 750 | 42.74% | 180 | 10.26% | 1,400 | 79.77% | 1,000 | 56.98% | 2,000 | 113.96% |
| indeno(1,2,3-cd)pyrene (µg/kg) | 1,575 | 380 | 24.13% | 1,100 | 69.84% | 690 | 43.81% | 180 | 11.43% | 710 | 45.08% | 1,800 | 114.29% | 2,000 | 126.98% |
| fluoranthene (µg/kg) | 2,250 | 780 | 34.67% | 2,000 | 88.89% | 900 | 40.00% | 370 | 16.44% | 1,100 | 48.89% | 2,400 | 106.67% | 2,600 | 115.56% |
| naphthalene (µg/kg) | 2,325 | 1,300 | 55.91% | 2,400 | 103.23% | 1,500 | 64.52% | 980 | 42.15% | 1,800 | 77.42% | 1,500 | 64.52% | 2,700 | 116.13% |
| antimony (mg/kg) | 47 | 32.0 | 68.09% | 46.9 | 99.79% | 57.5 | 122.34% | 66.1 | 140.64% | 31.3 | 66.60% | 68.4 | 145.53% | 47.0 | 100.00% |
| arsenic (mg/kg) | 12.1 | 9.7 | 80.17% | 14.7 | 121.49% | 10.9 | 90.08% | 14.5 | 119.83% | 14.6 | 120.66% | 16.4 | 135.54% | 14.4 | 119.01% |
| barium (mg/kg) | 402 | 280 | 69.65% | 451 | 112.19% | 359 | 89.30% | 406 | 101.00% | 348 | 86.57% | 476 | 118.41% | 401 | 99.75% |
| beryllium (mg/kg) | 5.40 | 4.1 | 75.93% | 5.8 | 107.41% | 4.7 | 87.04% | 5.9 | 109.26% | 5.5 | 101.85% | 6.3 | 116.67% | 5.6 | 103.70% |
| cadmium (mg/kg) | 15.2 | 12 | 78.95% | 20.5 | 134.87% | 14.3 | 94.08% | 18.6 | 122.37% | 10.4 | 68.42% | 21.0 | 138.16% | 17.5 | 115.13% |
| chromium (mg/kg) | 194 | 150 | 77.32% | 225 | 115.98% | 182 | 93.81% | 229 | 118.04% | 204 | 105.15% | 255 | 131.44% | 220 | 113.40% |
| lead (mg/kg) | 476 | 340 | 71.37% | 505 | 106.00% | 372 | 78.09% | 501 | 105.16% | 442 | 92.78% | 555 | 116.50% | 472 | 99.08% |
| mercury (mg/kg) | 34.5 | 28.0 | 81.16% | 21.6 | 62.61% | 20.0 | 57.97% | 34.3 | 99.42% | 30.7 | 88.99% | 34.5 | 100.00% | 30.4 | 88.12% |
| nickel (mg/kg) | 208 | 150 | 72.18% | 269 | 129.45% | 186 | 89.51% | 241 | 115.98% | 186 | 89.51% | 273 | 131.38% | 239 | 115.01% |
| silver (mg/kg) | 190 | 140 | 73.53% | 114 | 59.87% | 275 | 144.43% | 344 | 180.67% | 275 | 144.43% | 428 | 224.79% | 233 | 122.37% |

NOTE:

ND - Not Detected.

**ROUND 1 (Cont.)** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)[4]*** | ***Historical Average*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 51 | 43 | 84.81% | 51 | 100.59% | 21 | 41.42% | 26 | 51.28% | 63 | 124.26% | 37 | 72.98% | 50 | 98.62% |
| 1,1,1-trichloroethane (µg/kg) | 76 | 62 | 82.01% | 75 | 99.21% | 29 | 38.36% | 33 | 43.65% | 79 | 104.50% | ND | | 27 | 35.71% |
| 1,1,2,2-tetrachloro-ethane (µg/kg) | 100 | 40 | 40.20% | 92 | 92.46% | 53 | 53.27% | 76 | 76.38% | 3 | | 110 | 110.55% | 45 | 45.23% |
| carbon tetrachloride (µg/kg) | 80 | 53 | 66.08% | 71 | 88.53% | 30 | 37.41% | 32 | 39.90% | 63 | 78.55% | 53 | 66.08% | 33 | 41.15% |
| chlorobenzene (µg/kg) | 65 | 58 | 89.37% | 77 | 118.64% | 27 | 41.60% | 43 | 66.26% | 72 | 110.94% | 82 | 126.35% | 59 | 90.91% |
| ethylbenzene (µg/kg) | 26 | 24 | 92.66% | 23 | 88.80% | 10 | 38.61% | 16 | 61.78% | 31 | 119.69% | 30 | 115.83% | 24 | 92.66% |
| 4-methyl-2-pentanone (µg/kg) | 264 | 170 | 64.39% | 230 | 87.12% | 190 | 71.97% | 110 | 41.67% | 230 | 87.12% | 290 | 109.85% | 82 | 31.06% |
| tetrachloroethene (µg/kg) | 45 | 45 | 99.12% | 48 | 105.73% | 18 | 39.65% | 18 | 39.65% | 51 | 112.33% | 49 | 107.93% | 48 | 105.73% |
| trichloroethene (µg/kg) | 34 | 38 | 111.44% | 57 | 167.16% | 21 | 61.58% | 20 | 58.65% | 69 | 202.35% | 31 | 90.91% | 33 | 96.77% |
| total xylenes (µg/kg) | 207 | 190 | 91.79% | 260 | 125.60% | 85 | 41.06% | 130 | 62.80% | 230 | 111.11% | 267 | 128.99% | 210 | 101.45% |
| Aroclor 1248 (µg/kg) | 287 | 55 | 19.16% | 310 | 108.01% | 240 | 83.62% | 200 | 69.69% | 270 | 94.08% | 329 | 114.63% | 264 | 91.99% |
| anthracene (µg/kg) | 1,820 | 71 | | 880 | 48.35% | 820 | 45.05% | 82 | | 500 | 27.47% | 470 | 25.82% | 1,300 | 71.43% |
| chrysene (µg/kg) | 1,510 | 490 | 32.45% | 1,300 | 86.09% | 560 | 37.09% | 170 | 11.26% | 840 | 55.63% | 1,300 | 86.09% | 1,500 | 99.34% |
| benzo(k)fluoranthene (µg/kg) | 1,920 | 590 | 30.73% | 1,700 | 88.54% | 660 | 34.38% | 160 | | 1,400 | 72.92% | 1,800 | 93.75% | 2,100 | 109.38% |
| benzo(a)pyrene (µg/kg) | 2,590 | 380 | 14.67% | 1,200 | 46.33% | 750 | 28.96% | 180 | | 1,400 | 54.05% | 1,000 | 38.61% | 2,000 | 77.22% |
| indeno(1,2,3-cd)pyrene (µg/kg) | 2,330 | 380 | 16.31% | 1,100 | 47.21% | 690 | 29.61% | 180 | | 710 | 30.47% | 1,800 | 77.25% | 2,000 | 85.84% |
| fluoranthene (µg/kg) | 2,610 | 780 | 29.89% | 2,000 | 76.63% | 900 | 34.48% | 370 | 14.18% | 1,100 | 42.15% | 2,400 | 91.95% | 2,600 | 99.62% |
| naphthalene (µg/kg) | 2,460 | 1,300 | 52.85% | 2,400 | 97.56% | 1,500 | 60.98% | 980 | 39.84% | 1,800 | 73.17% | 1,500 | 60.98% | 2,700 | 109.76% |
| antimony (mg/kg) | 42 | 32.0 | 76.19% | 46.9 | 111.67% | 57.5 | 136.90% | 66.1 | 157.38% | 31.3 | 74.52% | 68.4 | 162.86% | 47.0 | 111.90% |
| arsenic (mg/kg) | 12.0 | 9.7 | 80.83% | 14.7 | 122.50% | 10.9 | 90.83% | 14.5 | 120.83% | 14.6 | 121.67% | 16.4 | 136.67% | 14.4 | 120.00% |
| barium (mg/kg) | 451 | 280 | 62.08% | 451 | 100.00% | 359 | 79.60% | 406 | 90.02% | 348 | 77.16% | 476 | 105.54% | 401 | 88.91% |
| beryllium (mg/kg) | 5.01 | 4.1 | 81.84% | 5.8 | 115.77% | 4.7 | 93.81% | 5.9 | 117.76% | 5.5 | 109.78% | 6.3 | 125.75% | 5.6 | 111.78% |
| cadmium (mg/kg) | 18.1 | 12 | 66.30% | 20.5 | 113.26% | 14.3 | 79.01% | 18.6 | 102.76% | 10.4 | 57.46% | 21.0 | 116.02% | 17.5 | 96.69% |
| chromium (mg/kg) | 220 | 150 | 68.18% | 225 | 102.27% | 182 | 82.73% | 229 | 104.09% | 204 | 92.73% | 255 | 115.91% | 220 | 100.00% |
| lead (mg/kg) | 479 | 340 | 70.98% | 505 | 105.43% | 372 | 77.66% | 501 | 104.59% | 442 | 92.28% | 555 | 115.87% | 472 | 98.54% |
| mercury (mg/kg) | 28.0 | 28.0 | 100.00% | 21.6 | 77.14% | 20.0 | 71.43% | 34.3 | 122.50% | 30.7 | 109.64% | 34.5 | 123.21% | 30.4 | 108.57% |
| nickel (mg/kg) | 249 | 150 | 60.24% | 269 | 108.03% | 186 | 74.70% | 241 | 96.79% | 186 | 74.70% | 273 | 109.64% | 239 | 95.98% |
| silver (mg/kg) | 340 | 140 | 41.18% | 114 | 33.53% | 275 | 80.88% | 344 | 101.18% | 275 | 80.88% | 428 | 125.88% | 233 | 68.53% |

NOTE:

ND - Not Detected.

**ROUND 2.** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)[4]*** | ***True Value*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 149 | 100 | 67.11% | 84 | 56.38% | 46 | 30.87% | 110 | 73.83% | 120 | 80.54% | 76 | 51.01% | 88 | 59.06% |
| chlorobenzene (µg/kg) | 44 | 38 | 85.78% | 31 | 69.98% | 24 | 54.18% | 36 | 81.26% | 37 | 83.52% | 28 | 63.21% | 32 | 72.23% |
| 1,2-dichloroethane (µg/kg) | 78 | 68 | 87.63% | 51 | 65.72% | 34 | 43.81% | 68 | 87.63% | 74 | 95.36% | 51 | 65.72% | 54 | 69.59% |
| ethylbenzene (µg/kg) | 108 | 87 | 80.56% | 67 | 62.04% | 44 | 40.74% | 86 | 79.63% | 85 | 78.70% | 68 | 62.96% | 70 | 64.81% |
| tetrachloroethene (µg/kg) | 61 | 42 | 68.63% | 33 | 53.92% | 16 | 26.14% | 30 | 49.02% | 37 | 60.46% | 21 | 34.31% | 33 | 53.92% |
| toluene (µg/kg) | 89 | 69 | 77.44% | 58 | 65.10% | 37 | 41.53% | 72 | 80.81% | 72 | 80.81% | 57 | 63.97% | 57 | 63.97% |
| 1,1,2-trichloroethane (µg/kg) | 53 | 52 | 98.30% | 41 | 77.50% | 32 | 60.49% | 51 | 96.41% | 46 | 86.96% | 38 | 71.83% | 38 | 71.83% |
| trichloroethene (µg/kg) | 129 | 88 | 68.22% | 69 | 53.49% | 33 | 25.58% | 83 | 64.34% | 90 | 69.77% | 61 | 47.29% | 73 | 56.59% |
| total xylenes (µg/kg) | 328 | 260 | 79.27% | 200 | 60.98% | 140 | 42.68% | 240 | 73.17% | 250 | 76.22% | 210 | 64.02% | 210 | 64.02% |
| Aroclor 1254 (µg/kg) | 181 | 150 | 82.87% | 130 | 71.82% | 160 | 88.40% | 110 | 60.77% | 130 | 71.82% | 140 | 77.35% | 170 | 93.92% |
| benzo(b)fluroanthene (µg/kg) | 4,240 | 3600 | 84.91% | 2700 | 63.68% | 3500 | 82.55% | 670 | 15.80% | 2900 | 68.40% | 5,000 | 117.92% | 3100 | 73.11% |
| benzo(k)fluoranthene (µg/kg) | 1,910 | 1600 | 83.77% | 1,200 | 62.83% | 1400 | 73.30% | 350 | 18.32% | 1,500 | 78.53% | 2,100 | 109.95% | 1,400 | 73.30% |
| benzo(a)pyrene (µg/kg) | 4,170 | 2900 | 69.54% | 2,200 | 52.76% | 2300 | 55.16% | 470 | 11.27% | 2,500 | 59.95% | 3,800 | 91.13% | 2,500 | 59.95% |
| chrysene (µg/kg) | 2,420 | 2300 | 95.04% | 1,700 | 70.25% | 1700 | 70.25% | 490 | 20.25% | 1700 | 70.25% | 2,700 | 111.57% | 1,800 | 74.38% |
| fluorene (µg/kg) | 3,290 | 2800 | 85.11% | 2,300 | 69.91% | 3000 | 91.19% | 740 | 22.49% | 2,800 | 85.11% | 3,300 | 100.30% | 2,600 | 79.03% |
| naphthalene (µg/kg) | 3,780 | 2,400 | 63.49% | 2,100 | 55.56% | 2,100 | 55.56% | 1000 | 26.46% | 2,100 | 55.56% | 2,300 | 60.85% | 2,300 | 60.85% |
| phenanthrene (µg/kg) | 1,570 | 1400 | 89.17% | 1,100 | 70.06% | 1400 | 89.17% | 380 | 24.20% | 1,400 | 89.17% | 1,600 | 101.91% | 1,200 | 76.43% |
| pyrene (µg/kg) | 4,940 | 3900 | 78.95% | 2,700 | 54.66% | 4100 | 83.00% | 1400 | 28.34% | 3,600 | 72.87% | 5,500 | 111.34% | 3,500 | 70.85% |
| antimony (mg/kg) | 65 | 0.0 | | 58.3 | 90.11% | 12.2 | 18.86% | 19.4 | 29.98% | 24 | 37.09% | 24.2 | 37.40% | 27.7 | 42.81% |
| arsenic (mg/kg) | 23.2 | 21 | 90.52% | 23.8 | 102.59% | 21.7 | 93.53% | 21.1 | 90.95% | 20.8 | 89.66% | 21.8 | 93.97% | 24.2 | 104.31% |
| barium (mg/kg) | 385 | 330 | 85.71% | 351 | 91.17% | 331 | 85.97% | 346 | 89.87% | 255 | 66.23% | 413 | 107.27% | 336 | 87.27% |
| beryllium (mg/kg) | 18.20 | 15 | 82.42% | 17.3 | 95.05% | 16.6 | 91.21% | 16.8 | 92.31% | 11.9 | 65.38% | 19.0 | 104.40% | 18.1 | 99.45% |
| cadmium (mg/kg) | 19.4 | 16 | 82.47% | 19.4 | 100.00% | 16.7 | 86.08% | 17.6 | 90.72% | 5.8 | 29.90% | 23.3 | 120.10% | 18.4 | 94.85% |
| chromium (mg/kg) | 191 | 160 | 83.77% | 190 | 99.48% | 169 | 88.48% | 178 | 93.19% | 190 | 99.48% | 181 | 94.76% | 197 | 103.14% |
| lead (mg/kg) | 511 | 410 | 80.23% | 494 | 96.67% | 426 | 83.37% | 448 | 87.67% | 391 | 76.52% | 519 | 101.57% | 480 | 93.93% |
| mercury (mg/kg) | 25.0 | 17.0 | 68.00% | 15.7 | 62.80% | 24.7 | 98.80% | 21.8 | 87.20% | 18.4 | 73.60% | 27.5 | 110.00% | 25.9 | 103.60% |
| nickel (mg/kg) | 222 | 170 | 76.58% | 206 | 92.79% | 186 | 83.78% | 198 | 89.19% | 66.8 | 30.09% | 272 | 122.52% | 219 | 98.65% |
| silver (mg/kg) | 391 | 130 | 33.25% | 164 | 41.94% | 326 | 83.38% | 339 | 86.70% | 208 | 53.20% | 333 | 85.17% | 361 | 92.33% |

**ROUND 2 (Cont.)** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)[4]*** | ***Historical Average*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 152 | 100 | 65.79% | 84 | 55.26% | 46 | 30.26% | 110 | 72.37% | 120 | 78.95% | 76 | 50.00% | 88 | 57.89% |
| chlorobenzene (µg/kg) | 45 | 38 | 83.70% | 31 | 68.28% | 24 | 52.86% | 36 | 79.30% | 37 | 81.50% | 28 | 61.67% | 32 | 70.48% |
| 1,2-dichloroethane (µg/kg) | 80 | 68 | 85.53% | 51 | 64.15% | 34 | 42.77% | 68 | 85.53% | 74 | 93.08% | 51 | 64.15% | 54 | 67.92% |
| ethylbenzene (µg/kg) | 112 | 87 | 77.68% | 67 | 59.82% | 44 | 39.29% | 86 | 76.79% | 85 | 75.89% | 68 | 60.71% | 70 | 62.50% |
| tetrachloroethene (µg/kg) | 63 | 42 | 66.88% | 33 | 52.55% | 16 | 25.48% | 30 | 47.77% | 37 | 58.92% | 21 | 33.44% | 33 | 52.55% |
| toluene (µg/kg) | 90 | 69 | 76.41% | 58 | 64.23% | 37 | 40.97% | 72 | 79.73% | 72 | 79.73% | 57 | 63.12% | 57 | 63.12% |
| 1,1,2-trichloroethane (µg/kg) | 55 | 52 | 94.89% | 41 | 74.82% | 32 | 58.39% | 51 | 93.07% | 46 | 83.94% | 38 | 69.34% | 38 | 69.34% |
| trichloroethene (µg/kg) | 127 | 88 | 69.29% | 69 | 54.33% | 33 | 25.98% | 83 | 65.35% | 90 | 70.87% | 61 | 48.03% | 73 | 57.48% |
| total xylenes (µg/kg) | 342 | 260 | 76.02% | 200 | 58.48% | 140 | 40.94% | 240 | 70.18% | 250 | 73.10% | 210 | 61.40% | 210 | 61.40% |
| Aroclor 1254 (µg/kg) | 150 | 150 | 100.00% | 130 | 86.67% | 160 | 106.67% | 110 | 73.33% | 130 | 86.67% | 140 | 93.33% | 170 | 113.33% |
| benzo(b)fluroanthene (µg/kg) | 2,780 | 3600 | 129.50% | 2700 | 97.12% | 3500 | 125.90% | 670 | 24.10% | 2900 | 104.32% | 5,000 | 179.86% | 3100 | 111.51% |
| benzo(k)fluoranthene (µg/kg) | 1,470 | 1600 | 108.84% | 1,200 | 81.63% | 1400 | 95.24% | 350 | 23.81% | 1,500 | 102.04% | 2,100 | 142.86% | 1,400 | 95.24% |
| benzo(a)pyrene (µg/kg) | 2,470 | 2900 | 117.41% | 2,200 | 89.07% | 2300 | 93.12% | 470 | 19.03% | 2,500 | 101.21% | 3,800 | 153.85% | 2,500 | 101.21% |
| chrysene (µg/kg) | 1,840 | 2300 | 125.00% | 1,700 | 92.39% | 1700 | 92.39% | 490 | 26.63% | 1700 | 92.39% | 2,700 | 146.74% | 1,800 | 97.83% |
| fluorene (µg/kg) | 2,240 | 2800 | 125.00% | 2,300 | 102.68% | 3000 | 133.93% | 740 | 33.04% | 2,800 | 125.00% | 3,300 | 147.32% | 2,600 | 116.07% |
| naphthalene (µg/kg) | 2,150 | 2,400 | 111.63% | 2,100 | 97.67% | 2,100 | 97.67% | 1000 | 46.51% | 2,100 | 97.67% | 2,300 | 106.98% | 2,300 | 106.98% |
| phenanthrene (µg/kg) | 1,140 | 1400 | 122.81% | 1,100 | 96.49% | 1400 | 122.81% | 380 | 33.33% | 1,400 | 122.81% | 1,600 | 140.35% | 1,200 | 105.26% |
| pyrene (µg/kg) | 3,550 | 3900 | 109.86% | 2,700 | 76.06% | 4100 | 115.49% | 1400 | 39.44% | 3,600 | 101.41% | 5,500 | 154.93% | 3,500 | 98.59% |
| antimony (mg/kg) | 21 | 0.0 | | 58.3 | 276.30% | 12.2 | 57.82% | 19.4 | 91.94% | 24 | 113.74% | 24.2 | 114.69% | 27.7 | 131.28% |
| arsenic (mg/kg) | 19.3 | 21 | 108.81% | 23.8 | 123.32% | 21.7 | 112.44% | 21.1 | 109.33% | 20.8 | 107.77% | 21.8 | 112.95% | 24.2 | 125.39% |
| barium (mg/kg) | 350 | 330 | 94.29% | 351 | 100.29% | 331 | 94.57% | 346 | 98.86% | 255 | 72.86% | 413 | 118.00% | 336 | 96.00% |
| beryllium (mg/kg) | 15.00 | 15 | 100.00% | 17.3 | 115.33% | 16.6 | 110.67% | 16.8 | 112.00% | 11.9 | 79.33% | 19.0 | 126.67% | 18.1 | 120.67% |
| cadmium (mg/kg) | 16.5 | 16 | 96.97% | 19.4 | 117.58% | 16.7 | 101.21% | 17.6 | 106.67% | 5.8 | 35.15% | 23.3 | 141.21% | 18.4 | 111.52% |
| chromium (mg/kg) | 180 | 160 | 88.89% | 190 | 105.56% | 169 | 93.89% | 178 | 98.89% | 190 | 105.56% | 181 | 100.56% | 197 | 109.44% |
| lead (mg/kg) | 451 | 410 | 90.91% | 494 | 109.53% | 426 | 94.46% | 448 | 99.33% | 391 | 86.70% | 519 | 115.08% | 480 | 106.43% |
| mercury (mg/kg) | 19.2 | 17.0 | 88.54% | 15.7 | 81.77% | 24.7 | 128.65% | 21.8 | 113.54% | 18.4 | 95.83% | 27.5 | 143.23% | 25.9 | 134.90% |
| nickel (mg/kg) | 190 | 170 | 89.47% | 206 | 108.42% | 186 | 97.89% | 198 | 104.21% | 66.8 | 35.16% | 272 | 143.16% | 219 | 115.26% |
| silver (mg/kg) | 322 | 130 | 40.37% | 164 | 50.93% | 326 | 101.24% | 339 | 105.28% | 208 | 64.60% | 333 | 103.42% | 361 | 112.11% |

**ROUND 2 (Cont.)** Summary of Laboratory Results and Recoveries

| | | Lab A | | Lab B | | Lab C | | Lab D | | Lab E | | Lab F | | Lab G | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Compound/Analyte (reporting units)[4]*** | ***Referee Mean*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** | ***Reported Value*** | ***Recovery*** |
| benzene (µg/kg) | 118 | 100 | 84.75% | 84 | 71.19% | 46 | 38.98% | 110 | 93.22% | 120 | 101.69% | 76 | 64.41% | 88 | 74.58% |
| chlorobenzene (µg/kg) | 44 | 38 | 85.78% | 31 | 69.98% | 24 | 54.18% | 36 | 81.26% | 37 | 83.52% | 28 | 63.21% | 32 | 72.23% |
| 1,2-dichloroethane (µg/kg) | 80 | 68 | 85.53% | 51 | 64.15% | 34 | 42.77% | 68 | 85.53% | 74 | 93.08% | 51 | 64.15% | 54 | 67.92% |
| ethylbenzene (µg/kg) | 97 | 87 | 89.69% | 67 | 69.07% | 44 | 45.36% | 86 | 88.66% | 85 | 87.63% | 68 | 70.10% | 70 | 72.16% |
| tetrachloroethene (µg/kg) | 45 | 42 | 93.75% | 33 | 73.66% | 16 | 35.71% | 30 | 66.96% | 37 | 82.59% | 21 | 46.88% | 33 | 73.66% |
| toluene (µg/kg) | 83 | 69 | 82.83% | 58 | 69.63% | 37 | 44.42% | 72 | 86.43% | 72 | 86.43% | 57 | 68.43% | 57 | 68.43% |
| 1,1,2-trichloroethane (µg/kg) | 54 | 52 | 96.65% | 41 | 76.21% | 32 | 59.48% | 51 | 94.80% | 46 | 85.50% | 38 | 70.63% | 38 | 70.63% |
| trichloroethene (µg/kg) | 92 | 88 | 95.65% | 69 | 75.00% | 33 | 35.87% | 83 | 90.22% | 90 | 97.83% | 61 | 66.30% | 73 | 79.35% |
| total xylenes (µg/kg) | 295 | 260 | 88.14% | 200 | 67.80% | 140 | 47.46% | 240 | 81.36% | 250 | 84.75% | 210 | 71.19% | 210 | 71.19% |
| Aroclor 1254 (µg/kg) | 183 | 150 | 81.97% | 130 | 71.04% | 160 | 87.43% | 110 | 60.11% | 130 | 71.04% | 140 | 76.50% | 170 | 92.90% |
| benzo(b)fluroanthene (µg/kg) | 2,880 | 3600 | 125.00% | 2700 | 93.75% | 3500 | 121.53% | 670 | 23.26% | 2900 | 100.69% | 5,000 | 173.61% | 3100 | 107.64% |
| benzo(k)fluoranthene (µg/kg) | 1,200 | 1600 | 133.33% | 1,200 | 100.00% | 1400 | 116.67% | 350 | 29.17% | 1,500 | 125.00% | 2,100 | 175.00% | 1,400 | 116.67% |
| benzo(a)pyrene _(µg/kg) | 2,220 | 2900 | 130.63% | 2,200 | 99.10% | 2300 | 103.60% | 470 | 21.17% | 2,500 | 112.61% | 3,800 | 171.17% | 2,500 | 112.61% |
| chrysene (µg/kg) | 1,780 | 2300 | 129.21% | 1,700 | 95.51% | 1700 | 95.51% | 490 | 27.53% | 1700 | 95.51% | 2,700 | 151.69% | 1,800 | 101.12% |
| fluorene (µg/kg) | 2,700 | 2800 | 103.70% | 2,300 | 85.19% | 3000 | 111.11% | 740 | 27.41% | 2,800 | 103.70% | 3,300 | 122.22% | 2,600 | 96.30% |
| naphthalene (µg/kg) | 2,400 | 2,400 | 100.00% | 2,100 | 87.50% | 2,100 | 87.50% | 1000 | 41.67% | 2,100 | 87.50% | 2,300 | 95.83% | 2,300 | 95.83% |
| phenanthrene (µg/kg) | 1,240 | 1400 | 112.90% | 1,100 | 88.71% | 1400 | 112.90% | 380 | 30.65% | 1,400 | 112.90% | 1,600 | 129.03% | 1,200 | 96.77% |
| pyrene (µg/kg) | 3,760 | 3900 | 103.72% | 2,700 | 71.81% | 4100 | 109.04% | 1400 | 37.23% | 3,600 | 95.74% | 5,500 | 146.28% | 3,500 | 93.09% |
| antimony (mg/kg) | 25 | 0.0 | | 58.3 | 236.99% | 12.2 | 49.59% | 19.4 | 78.86% | 24 | 97.56% | 24.2 | 98.37% | 27.7 | 112.60% |
| arsenic (mg/kg) | 21.8 | 21 | 96.33% | 23.8 | 109.17% | 21.7 | 99.54% | 21.1 | 96.79% | 20.8 | 95.41% | 21.8 | 100.00% | 24.2 | 111.01% |
| barium (mg/kg) | 344 | 330 | 95.93% | 351 | 102.03% | 331 | 96.22% | 346 | 100.58% | 255 | 74.13% | 413 | 120.06% | 336 | 97.67% |
| beryllium (mg/kg) | 15.80 | 15 | 94.94% | 17.3 | 109.49% | 16.6 | 105.06% | 16.8 | 106.33% | 11.9 | 75.32% | 19.0 | 120.25% | 18.1 | 114.56% |
| cadmium (mg/kg) | 17.4 | 16 | 91.95% | 19.4 | 111.49% | 16.7 | 95.98% | 17.6 | 101.15% | 5.8 | 33.33% | 23.3 | 133.91% | 18.4 | 105.75% |
| chromium (mg/kg) | 170 | 160 | 94.12% | 190 | 111.76% | 169 | 99.41% | 178 | 104.71% | 190 | 111.76% | 181 | 106.47% | 197 | 115.88% |
| lead (mg/kg) | 424 | 410 | 96.70% | 494 | 116.51% | 426 | 100.47% | 448 | 105.66% | 391 | 92.22% | 519 | 122.41% | 480 | 113.21% |
| mercury (mg/kg) | 23.8 | 17.0 | 71.43% | 15.7 | 65.97% | 24.7 | 103.78% | 21.8 | 91.60% | 18.4 | 77.31% | 27.5 | 115.55% | 25.9 | 108.82% |
| nickel (mg/kg) | 193 | 170 | 88.08% | 206 | 106.74% | 186 | 96.37% | 198 | 102.59% | 66.8 | 34.61% | 272 | 140.93% | 219 | 113.47% |
| silver (mg/kg) | 290 | 130 | 44.83% | 164 | 56.55% | 326 | 112.41% | 339 | 116.90% | 208 | 71.72% | 333 | 114.83% | 361 | 124.48% |

In the first PE study, some general observations can be made. Lower recoveries were observed for the volatile fraction for laboratory C, D, and G. In addition, the project laboratories had higher recoveries in the volatiles fraction than the referee laboratories. Furthermore, the recoveries of the PAH fraction were somewhat on the low side across all laboratories, with laboratory D exhibiting very low PAH recoveries (<10%). In addition, laboratory A exhibited a low recovery for the PCB fraction. Finally, the recoveries for antimony were observed to be low for both the project laboratories and the referee laboratories.

At the conclusion of the first PE study, sanitized versions of the PE study results were provided to the project laboratories as a mechanism for feedback. The project laboratories were requested to investigate the origin of the problem areas that were identified in the study and take appropriate corrective action to identify and correct the problem. In the second PE study, some general observations can be made. Lower recoveries were observed for the volatile fraction for laboratory C, G, B, and F. The recoveries of the PAH fraction were greatly improved from the previous round, with exception of laboratory D. Laboratory D still exhibited very low recoveries for the PAH fraction. In addition, the recoveries of the PCB fraction were within acceptance limits (as previously defined) for all project laboratories. Furthermore, the recoveries for antimony were observed to be low for both the project laboratories and the referee laboratories.

**DISCUSSION**

There are several reasons why a PE result could be outside the defined acceptance limits. First, there could be a laboratory performance issue. This is usually observed when one laboratory performs very differently than all of the other project laboratories for a given fraction or analyte. This was observed to be the case for laboratory D for the PAH fraction. Second, there could be a method limitation for analyzing a sample for a given analyte. This was observed to be the case for antimony where both the project laboratories and the referee laboratories exhibited low recoveries for both rounds of PE samples. Furthermore, there could be a PE vendor preparation issue. This is usually observed when all laboratories exhibit recoveries that are not within the defined acceptance range for most of the analytes in a fraction. This was not observed to be the case for any of the PE samples issued to the project laboratories.

**LESSONS LEARNED/CONCLUSIONS**

There were some inherent issues regarding the addition of water to the soil PE samples. Typically, dried, pulverized sands are utilized for the solid matrix PE samples. Laboratories that perform well on the analysis of dried solid PE samples do not necessarily perform well on the analysis of the multi-phasic PE samples (the latter being samples that typically are submitted for analysis during environmental investigations), particularly for the PAH fraction. Laboratories that utilized a single-solvent extraction for the preparation of the pre-moistened PAH soil PE samples exhibited low recoveries. Laboratories that utilized a 1:1 mixture of methylene chloride/acetone for the preparation of the pre-moistened PAH soil PE samples exhibited recoveries within the project-defined acceptance limits. Another lesson learned is that often times laboratories pay close attention to the instrumentation and data review but may not carefully evaluate the chemistry inherently embedded in the prescribed method. As such, laboratory results can exceed the defined acceptance limits. For instance, between the first PE round and the second PE round, laboratory A identified that their volumetric glassware had not been calibrated in the manufacturer’s recommended frequency and the tolerance of the glassware they were using for the preparation of the PCB fraction was outside the tolerance specifications.

Another observation is that the percent moisture that is added to each fraction of the PE sample should be as consistent as possible. Often the laboratory will analyze an aliquot of sample from one of the designated analytical fractions for percent moisture and cross-apply that one determination to all fractions from dry-weight calculations. If the percent moisture is different for each fraction in the PE sample, the laboratory may unknowingly cross-apply an incorrect percent moisture to the other fractions. Similarly, it was observed that the addition of water to the volatile fraction of the soil PE sample resulted in a matrix that was not homogeneous. To overcome this problem that was identified through the course of the PE studies, it was determined that a special coring tool was required to properly subsample the volatile fraction of the PE sample to obtain acceptable (as previously defined) PE results.

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