Development of a Fast, Cost-Effective Laboratory Analysis for a PCB-Remediation Project as an Alternative to Field Kits or Conventional Laboratory Techniques

Glenna J. Reuel
Canviro Analytical Laboratories Ltd., 50 Bathurst Dr., Waterloo, Canada. N2V 2C5
Charles W. Mitz
CH2M HILL Engineering Ltd., 180 King St. South, Waterloo, Canada. N2J 1P8

Introduction

In the dynamic field of environmental analysis, the demands presented to the laboratory are constantly changing. Field test kits are now being considered as an alternative to laboratory testing[1], due to two perceived advantages; cost and turnaround time. In cooperation with CH2M HILL Engineering Ltd., CANVIRO developed a method which put laboratory testing on a competitive basis with the field test kits in terms of price and speed of analysis, and gave the client advantages which could not be achieved through field testing.

Background

In the autumn of 1994, CANVIRO was approached by CH2M HILL to provide an analytical technique capable of producing fast, reliable test results for PCB's in soil with a detection limit of 1 ppm. This method would provide analytical support to a remediation project, and would have a high volume sample throughput.

The remediation included the delineation and removal of a layer of sludge that had accumulated in a wastewater pond over a period of some 40 years. Preliminary investigations indicated that the sludge contained elevated, spatially variable levels of polychlorinated biphenyls (PCBs), occasionally exceeding 50 mg/kg - the threshold level for definition as a PCB waste in Ontario[2]. Because the expense of treating PCB-contaminated materials was approximately 10 times greater than the balance of the sludge, a remedial plan was adopted to map the zones of PCB-contaminated sludge on the basis of chemical testing and to excavate and treat these "hot spots" separately from the balance of the material. Both the delineation and excavation would take place during winter when the pond was temporarily drained.

Analytical Requirements

Due to the limited time available for remediation, emphasis was placed on laboratory turnaround time and the reliability of the analytical results. A single false positive result could add significantly to the cost of remediation. To meet these demanding requirements, CH2M HILL evaluated a number of alternative analytical techniques including conventional laboratory analysis and a number of field test kits based on organohlorine detection and immunoassay techniques. A useful description of field test kits is given by Thompson and Novgod[3].

Several of the kits were evaluated using thoroughly homogenized sludge samples that had been previously characterized by ultrasonic extraction followed by gas chromatographic/mass spectrometric analysis. The PCB concentration in the sludge samples ranged from 2 mg/kg to 87 mg/kg. For the assessment, each sample was relabelled and all evaluation work was carried out under double blind conditions, i.e. neither the testing lab nor the engineer knew the identities of the samples.

Initial testing revealed a number of problems with the various test kits: Kits using organochlorine detection methods were found to be susceptible to interferences from a wide variety of chlorinated compounds. Immunoassay kits required expensive accessories such as a spectrophotometer to quantify the PCB concentrations. Each kit is calibrated only for a specific arochlor with total PCBs estimated based on the assumption that arochlor responses remain constant. Use of the kits required thorough homogenization of the samples, moisture content determination, and use of a number of reagents, pipettes, micro-pipettes, and tubes. All of these operations are difficult to carry out under field conditions, especially during the cold Canadian winter.

To address the requirements of the remediation project, CANVIRO developed a cost-effective extraction procedure to enable the laboratory to provide accurate results under controlled laboratory conditions with a one day turnaround time. The extraction method, referred to within the lab as "Shake and Shoot", speeds the extraction time over the conventional sonication by allowing the simultaneous extraction of a large number of samples. For the evaluation, a 10 g sediment sample was spiked with a surrogate (2,4,6-Tribromobiphenyl) in a 40 mL volatile bottle. After the addition of acetone, the bottle was slowly tumbled for two hours on an apparatus normally used for leachate testing. An aliquot of the organic extract was diluted to 1.00 mL with toluene and d10-Anthracene was added as an internal standard. The extracts were analyzed by GC/MS using

a Hewlett-Packard (HP) 5890 GC and either an HP 5970 or HP 5972 mass selective detector. The short analytical column allowed an analysis time of 8 minutes. The method measures individual arochlors, which allows a comparison of their relative ratios as a secondary determination of analysis accuracy.

Evaluation of Alternative Analytical Techniques

Some of the data that CH2M HILL considered in the choice of analytical method are presented on Figure 1. Test results are presented for conventional laboratory analysis, "shake and shoot" analysis (both analyzed at CANVIRO), and an immunoassay kit (analyzed by the distributor of the kits) each of which is compared to a "reference value" established by the previously mentioned characterization. Additionally, two samples were analyzed by external environmental testing laboratories. The results obtained from the organochlorine determination test kit are not included in the graph, as the kit failed to even detect three samples with PCB concentrations greater than 50 mg/kg, and was therefore determined as unsuitable for this application.

Based on the limited number of data points represented by the graph, there appears to be good agreement between the various testing methods evaluated. External laboratory tests were done on two samples and these agreed well with the reference values. Of the test kits evaluated, only immunoassay performed well - though it was found to be somewhat more prone to outliers than the laboratory analyses. As a result of the comparative evaluation, CH2M HILL selected the "Shake and Shoot" method for the sludge remediation project.

Field Validation

The detailed delineation program was carried out to identify zones of PCB waste material during the winter of 1995. This included the establishment of a grid over the pond at a spacing of 10 m or less, with over

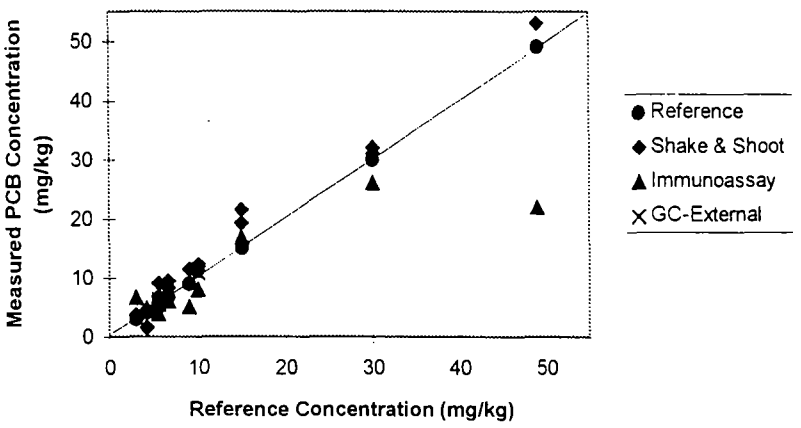

Figure 1-Results of Method Trials

350 locations being analyzed for PCB's and total solids.

Samples were submitted to the testing laboratory at the end of each day, and results were provided within 24 hours of submission by facsimile and electronic transfer. Electronic transfer of the analytical data facilitated the use of geostatistical contouring software to delineate zones of PCB waste. Figure 2 illustrates the interpreted PCB concentrations for a section of the pond. To confirm analytical precision, 17 duplicate samples were analyzed, yielding an average relative percent difference between duplicates of less than 10% with a correlation coefficient of 0.992.

Conclusions

Use of the "Shake and Shoot" procedure allowed the project to be carried out within schedule and without significant overexcavation of PCB materials. Laboratory analysis yielded additional benefits. Sample handling and analysis were carried out by highly trained personnel under well controlled conditions which included laboratory based quality control procedures such as lab blanks, spikes, and surrogate recoveries. The analytical technique gave concentrations for constituent arochlors as well as for total PCBs thereby allowing arochlor ratios to be used as a quality control technique to identify outliers and anomalous results. Finally, data were provided as electronic files which facilitated direct input into statistical analysis and contouring software.

The results obtained using the selected analytical procedure proved to be far superior to the field test kits originally considered for use. Analytical expense was equal to or lower than that for immunoassay when the additional staff time and accessories were factored into the total analytical cost.

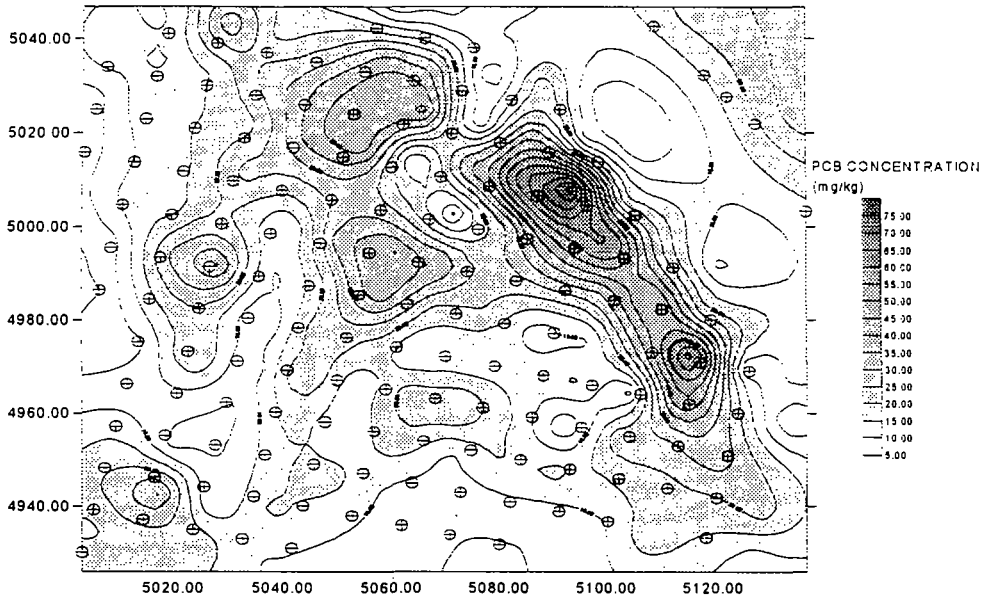

FIGURE 2-PCB Concentrations in Sludge

References

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2. Government of Ontario, Regulation 362 under the Environmental Protection Act, R.S.O. 1990.

3. Thompson, T., Novgod, J., (1994). On-Site Field Screening Kits, the use of immunoassay technology for fast test results.