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# Interlaboratory Comparison of Extraction Efficiency of Pesticides from Surface and Laboratory Water Using Solid-Phase Extraction Disks

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A continuation of an earlier interlaboratory comparison was conducted (1) to assess solid-phase extraction (SPE) using Empore disks to extract atrazine, bromacil, metolachlor, and chlorpyrifos from various water sources accompanied by different sample shipping and quantitative techniques and (2) to compare quantitative results of individual laboratories with results of one common laboratory. Three replicates of a composite surface water (SW) sample were fortified with the analytes along with three replicates of deionized water (DW). A nonfortified DW sample and a nonfortified SW sample were also extracted. All samples were extracted using Empore $C_{18}$ disks. After extraction, part of the samples were eluted and analyzed in-house. Duplicate samples were evaporated in a 2-mL vial, shipped dry to a central laboratory (SDC), redissolved, and analyzed. Overall, samples analyzed in-house had higher recoveries than SDC samples. Laboratory × analysis type and laboratory × water source interactions were significant for all four compounds. Seven laboratories participated in this interlaboratory comparison program. No differences in atrazine recoveries were observed from in-house samples analyzed by laboratories A, B, D, and G compared with the recovery of SDC samples. In-house atrazine recoveries from laboratories C and F were higher when compared with recovery from SDC samples. However, laboratory E had lower recoveries from in-house samples compared with SDC samples. For each laboratory, lower recoveries were observed for chlorpyrifos from the SDC samples compared with samples analyzed in-house. Bromacil recovery was <65% at two of the seven laboratories in the study. Bromacil recoveries for the remaining laboratories were >75%. Three laboratories showed no differences in metolachlor recovery; two laboratories had higher recoveries for samples analyzed in-house, and two other laboratories showed higher metolachlor recovery for SDC samples. Laboratory G had a higher recovery in SW for all four compounds compared with DW. Other laboratories that had significant differences in pesticide recovery between the two water sources showed higher recovery in DW than in the SW regardless of the compound. In comparison to earlier work, recovery of these compounds using SPE disks as a temporary storage matrix may be more effective than shipping dried samples in a vial. Problems with analytes such as chlorpyrifos are unavoidable, and it should not be assumed that an extraction procedure using SPE disks will be adequate for all compounds and transferrable across all chromatographic conditions.

**KEYWORDS: Solid-phase extraction (SPE); pesticides; surface water; extraction; interlaboratory analysis; Empore disks**

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## INTRODUCTION

Solid-phase extraction (SPE) disks containing octadecyl ($C_{18}$) bonded silica have provided many analytical laboratories with reproducible extraction from water samples and is becoming a widely used analytical technique. This technique has reduced the volume of potentially hazardous solvents used and their ultimate disposal, decreased sample preparation time and labor needed, and increased extract purity from drinking water samples compared with liquid−liquid extraction (LLE) (*1*−*3*).

In the past several years, researchers have studied the potential use of SPE disks for temporary pesticide storage (*4*, *5*), field extraction of pesticides (*6*), and shipping pesticides from one location to another (*7*, *8*). Temporary storage on $C_{18}$ worked well and enhanced the stability of most compounds compared with storage in water at 4 °C (*4*, *5*). Mattice et al. (*6*) tested a field extraction manifold using $C_{18}$ disks. They found lower recoveries from field extractions compared with sample collection followed by laboratory extraction. However, the difference was small enough that many samples would be required to detect the difference (*6*). Results from a previous southern region collaborative project (S-271) showed that extraction efficiencies of the disks were comparable with or better than the recoveries obtained from shipped water samples (*7*). Also, many problems associated with shipping water samples, such as storage stability, bottle breakage, and high shipping charges were eliminated by using the disk as storage and shipping media (*7*). Further studies reported by Mersie et al. (*8*) demonstrated the capacity of this technique for a wide range of compounds.

In our previous study, an interlaboratory comparison was conducted to examine the feasibility of using $C_{18}$ solid-phase extraction disks (Empore) to simultaneously determine the herbicides atrazine, bromacil, and metolachlor and the insecticide chlorpyrifos in water samples (*7*). A common fortification source and a common sample processing procedure were used to minimize variation in initial concentrations and operator inconsistencies. The protocol consisted of paired laboratories in different locations coordinating their activities and shipping fortified water samples (deionized or local surface water) or Empore disks on which the pesticides had been retained and then quantitating the analytes by a variety of gas chromatographic methods (*7*). Average recoveries from all laboratories were >80% for atrazine, bromacil, and metolachlor and >70% for chlorpyrifos (*7*). Detection of bromacil was unachievable at some locations because of chromatographic problems. Shipping samples between cooperating laboratories did not affect the recovery of atrazine, chlorpyrifos, or metolachlor in either matrix (*7*). Recoveries tended to be higher from disks shipped to cooperating laboratories compared with those from fortified water (*7*). Shipping disks eliminated many problems associated with the shipment of water samples, such as bottle breakage, higher shipping cost, and possible pesticide degradation (*7*). Recoveries of bromacil and metolachlor were lower from fortified surface water samples than from fortified deionized water samples (*7*). This collaborative research demonstrated that pesticides in water samples can be concentrated on solid-phase extraction disks at one location and quantitated under diverse analytical conditions at another location. The extraction efficiencies of the disks were comparable with or better than the recoveries obtained from the shipped water samples, and the problems associated with shipping water samples were eliminated by using the disks (*7*).

On the basis of this earlier work, substantial interlaboratory variation existed in the extraction of atrazine, metolachlor, bromacil, and chlorpyrifos regardless of whether they were stored on $C_{18}$ disks and eluted later or whether they were analyzed in-house using conventional $C_{18}$ filtration (*7*). This is consistent with results from other interlaboratory studies (*9*). However, the robustness of the method was conclusively demonstrated across 11 laboratories (*7*). In the previous study, each laboratory was paired with another and samples were exchanged. We have decided that it would be important to measure the variability among laboratories by comparing the results for each laboratory to the results of one laboratory rather than using the previous method of paired comparisons. Therefore, the objectives of this interlaboratory study were (1) to assess solid-phase extraction using Empore disks to extract atrazine, bromacil, metolachlor, and chlorpyrifos from various water sources accompanied by different sample shipping and quantitative techniques and (2) to compare quantitative results of each individual laboratory with results of one common laboratory.

## MATERIALS AND METHODS

The premise of this study distinguishing it from that done by Mueller et al. (*7*) is that samples from a common batch of surface water were sent to all laboratories and a common extraction protocol was followed for fortifying, extracting, and shipping samples that had been evaporated in vials rather than on Empore disks. Part of each sample was sent to a central laboratory for analysis. These results were compared with the results from each laboratory's in-house analysis. For the sake of anonymity the cooperating laboratories have been identified as letters A−G. Although substantial collaboration in the form of information exchange occurred, each laboratory independently determined the concentration of each pesticide for the in-house samples. The conditions that each laboratory used to analyze samples for in-house analysis and at the central laboratory and the analytical equipment used are listed in **Table 1**.

Materials required included octadecyl ($C_{18}$) Empore extraction disks, 47-mm diameter (3M Co., St. Paul, MN); 47-mm filters to remove particulates (GF/B Whatman glass fiber filter and 0.45-$\mu$m Gelman nylon membrane); an extraction manifold suitable for 47-mm disks; ethyl acetate and methanol (all solvents of GC or HPLC grade); and anhydrous sodium sulfate.

**Surface Water Collection and Fortification Procedure.** The surface water was collected from the Tennessee River outside Knoxville, TN, in 2-L bottles. The bottles were capped and brought back to the University of Tennessee laboratory. In preliminary analysis of the water, none of the four analytes were present at detectable levels in the water samples prior to shipping. Four bottles each were sent to participating laboratories by mail. Once the water samples arrived at each participating laboratory, subsamples were created and appropriate blanks and fortified samples were prepared. Deionized water blanks were extracted along with unfortified surface water (matrix blanks) to ensure that no contamination was introduced into the samples from the laboratory and that the surface water samples were clear of contamination. To establish a uniform initial pesticide concentration, a single location prepared a fortification solution and shipped it in duplicate to each participating laboratory.

Solutions containing atrazine, bromacil, chlorpyrifos, and metolachlor at 200 $\mu$g/mL were prepared in methanol and shipped in two 4-mL borosilicate glass vials sealed with Teflon-lined caps externally sealed with Parafilm. The total volume of fortification solution shipped to each laboratory was 8 mL. To reduce the chance of contamination due

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**Table 1.** Chromatographic Conditions Used at Various Laboratories To Determine Simultaneously Atrazine, Bromacil, Chlorpyrifos, and Metolachlor during In-House Analysis

| | | | column | | temperature program | | |
|---|---|---|---|---|---|---|---|
| location | GC model | detector | stationary phase | dimensions | detector (°C) | injector (°C) | column |
| A | Varian 3400 | MS | DB-5 | 30 m × 0.25 mm × 0.25 μm film | 240 | 260 | 80 °C for 1 min, increase at 12 °C/min to 290 °C, hold for 1 min |
| B | Hewlett-Packard 5890 | NPD | DB-5 | 30 m × 0.25 mm × 0.25 μm film | 320 | 250 | 150 °C initial, increase at 15 °C/min to 190 °C, hold for 2 min, increase at 2 °C/min to 210 °C, increase at 20 °C/min to 260 °C |
| C | Hewlett-Packard 5890 | ECD | DB-5 | 28 m × 0.25 mm × 0.25 μm film | 320 | 250 | 150 °C for 2 min, increase at 15 °C/min to 190 °C, hold for 1 min, increase at 2 °C/min to 260 °C, hold for 2 min |
| D | Hewlett-Packard 5890 | FID | Ultra 2 | 25 m × 0.2 mm × 0.33 μm film | 300 | 250 | 190 °C for 10 min, increase at 1 °C/min to 200 °C, increase at 40 °C/min to 300 °C, hold for 4 min |
| E[a] | Tracor 540 | ECD | 1.5% SP2250 1.95% SP2401 | 1.83 m × 6.4 mm | 235 | 350 | 195 °C isothermal |
| E[b] | Shimadzu 14A | ECD | RTX 35 | 30 m × 0.53 mm × 0.5 μm film | 275 | 300 | 150 °C, hold for 20 min, increase at 1 °C/min to 175 °C, hold for 2 min, increase at 5 °C/min to 290 °C, hold for 5 min |
| F | Hewlett-Packard 6890 | MS | DB5 | 30 m × 0.25 mm × 0.25 μm film | 280 | 225 | 80 °C for 10 min, increase at 5 °C/min to 210 °C, increase at 50 °C/min to 280 °C, hold for 3.6 min |
| G | Tracor 540 | ECD | DB210 | 30 m × 0.53 mm × 1.0 μm film | 350 | 240 | 170 °C isothermal, 15 min |
| H | Hewlett-Packard 5890 | NPD | Econocap SE-30 | 30 m × 0.56 mm × 1.2 μm film | 230 | 250 | 110 °C for 1 min, increase at 15 °C/m to 190 °C, hold for 2 min, increase at 2 °C/min to 210 °C and at 20 °C/min to 235 °C |

[a] Instrument and set of conditions apply to atrazine analysis only. [b] Instrument and set of conditions apply to chlorpyrifos, metolachlor, and bromacil.

to spillage, the vials were enclosed in a disposable infant diaper. Fortification solutions were shipped to all cooperating laboratories via an overnight carrier. To ensure sample integrity, each solution was carefully weighed before shipping and then after receipt at each location. Losses were negligible (<1%). Each laboratory used 50 μL of this stock solution to fortify 1-L samples, resulting in 10 μg/L of each pesticide. The specified aliquot was added to the water sample and slowly shaken by hand prior to filtration through Empore disks. Methanol (4 mL) was added to the sample before extraction to keep $C_{18}$ disks conditioned during the extraction process.

**Disk Extraction.** Each laboratory used a vacuum extraction manifold that would accommodate 47-mm Empore $C_{18}$ extraction disks. Filtration apparatus design and manufacturer varied among locations. Therefore, the following procedure was used. Water samples were filtered through a Whatman GF/B glass fiber filter and a 0.45-μm Gelman nylon membrane filter to remove particulates. An Empore $C_{18}$ disk was placed in an extraction filter holder, and a reservoir was clamped to the holder (all glass and Teflon). Vacuum was applied, and the disk was cleaned by pulling ethyl acetate through the disk and then drawing air through the disk for 2 min.

Great care was taken to ensure that the disks remained moist with solvent during the next steps prior to water filtration. Methanol (10 mL) was added to the reservoir and drawn through until a thin film of methanol covered the disk, at which point vacuum was removed. Deionized water (10 mL) was then added and drawn through until a thin film of water remained. The vacuum valve was again closed. The water sample (1000 mL) was added and drawn through the Empore disk. After the entire sample passed through the disk, vacuum was pulled for 5 min to partially dry the disk. The filtered water was discarded. The disks were removed from the filtration apparatus and placed in a desiccator overnight.

Disks were transferred to a 25-mL culture tube with a Teflon-lined screw cap. Ethyl acetate (10 mL) was then added. Tubes were shaken on a flat bed or end-to-end shaker for 15 min. Ethyl acetate was transferred to a tube containing ~3 g of anhydrous $Na_2SO_4$. An additional 5 mL of ethyl acetate was added to the tube containing the disk and shaken for an additional 5 min. The first fraction was transferred to a graduated tube. The $Na_2SO_4$ was rinsed with the second 5-mL fraction of ethyl acetate, and the fractions were combined in the graduated tube. The sample was evaporated to 5 mL using a stream of dry $N_2$ without heat. A 1-mL aliquot was then transferred to a 2-mL GC vial and evaporated to dryness using $N_2$. Vials were shipped dry by overnight mail to the predesignated central laboratory facility at laboratory H **(Table 1)**. Once samples arrived at laboratory H, the vials were placed in a freezer at −20 °C for ~48 h until reconstituted in solvent for analysis. Samples shipped dry (SD) and sent to the central (C) laboratory will be designated from here on as SDC samples. Compounds were redissolved in the original vials with 1 mL of ethyl acetate and analyzed by gas chromatography. Chromatographic analysis of all samples analyzed in-house was done with an aliquot from the remaining 4 mL of the sample. Gas chromatography with an external standard technique was used for all analyses. However, individual conditions varied among laboratories (**Table 1**).

The eight samples analyzed independently at each laboratory and at the central laboratory included three fortified surface water (SW) samples, three fortified deionized water (DW) samples, one unfortified SW sample, and one unfortified DW sample.

Samples were quantified using a four-point calibration curve external standard technique with vial concentrations of 0.1, 0.5, 1.0, and 5.0 μg/mL. These standards were created using dilutions of the fortifying solution that was sent to each laboratory. This calibration curve was linear for all compounds at each of the laboratories. The method limit of quantitation ranged from 0.1 to 0.5 μg/L depending on the compound and laboratory instrumentation.

**Statistical Analysis.** Results were sent to the individual responsible for data statistics and analysis. To elucidate significant factors and interactions, data were subjected to analysis of variance within the General Linear Models (GLM) procedure of the Statistical Analysis Systems software (*11*). Analysis of variance was used to determine significant factors and interactions. The study was analyzed as a completely randomized design arranged in a three-factor factorial. The factors included (1) laboratory (seven participants and one central

**Table 2.** Sources of Variation and Associated Statistical Significance Levels for Percentage Recovery of Pesticide Analytes in Water Samples Using Solid-Phase Extraction Disks

| | | *P*[a] | | | |
|---|---|---|---|---|---|
| source | df[b] | atrazine | bromacil | chlorpyrifos | metalochlor |
| replication | 2 | ns[c] | 0.0270 | ns | ns |
| laboratory | 6 | <0.0001 | <0.0001 | <0.0001 | <0.0001 |
| analysis type[d] | 1 | 0.0434 | <0.0001 | <0.0001 | ns |
| water source[e] | 1 | ns | ns | ns | ns |
| laboratory × analysis type | 6 | 0.0107 | 0.0054 | <0.0001 | 0.0012 |
| laboratory × water source | 6 | 0.0399 | 0.0259 | <0.0001 | 0.0372 |
| analysis type × water source | 1 | ns | ns | ns | ns |
| mean square error | | 154.26 | 185.96 | 68.35 | 235.99 |
| coefficient of variation | | 15.79 | 117.21 | 13.40 | 18.52 |

[a] Results for which the reported *P* values were <0.05 indicate statistical significance at the 5% level. [b] df, degrees of freedom. [c] ns, reported *P* value was >0.05, therefore, not significant at the 5% level of significance. [d] Analysis type, represents either (1) in-house samples that were analyzed by chromatography at the preparing laboratory or (2) samples extracted, evaporated in a 2-mL glass vial, shipped to the central laboratory, resuspended with 1 mL of ethyl acetate, and analyzed. [e] Water source, represents either (1) deionized water or (2) surface water collected by personnel at the central laboratory.

**Table 3.** Mean Percentage Recovery of Atrazine, Chlorpyrifos, Metolachlor, and Bromacil from Solid-Phase Extraction Disks, Demonstrating a Relationship to Analysis Type as a Function of the Laboratory Responsible for Sample Extraction

| analysis | | % recovery | | | |
|---|---|---|---|---|---|
| type[a] | lab | atrazine | bromacil | chlorpyrifos | metolachlor |
| sent | A | 80.4 | 80.5 | 54.0 | 91.3 |
| | B | 89.0 | 82.5 | 67.8 | 94.8 |
| | C | 63.1 | 77.8 | 26.8 | 50.6 |
| | D | 87.0 | 100.8 | 47.8 | 92.4 |
| | E | 93.4 | 83.2 | 49.3 | 110.3 |
| | F | 38.8 | 14.3 | 15.5 | 41.4 |
| | G | 78.1 | 46.8 | 37.1 | 88.0 |
| in-house | A | 90.7 | 100.1 | 75.5 | 94.0 |
| | B | 96.1 | 100.4 | 121.9 | 115.4 |
| | C | 80.3 | 91.2 | 79.2 | 78.2 |
| | D | 91.2 | 112.1 | 103.4 | 72.3 |
| | E | 72.7 | 91.8 | 79.7 | 87.2 |
| | F | 59.6 | 62.0 | 34.2 | 54.7 |
| | G | 76.2 | 55.9 | 62.3 | 88.4 |
| | $LSD_{0.05}$[b] | 14.6 | 9.7 | 18.0 | 16.0 |

[a] Analysis type, represents either (1) in-house samples that were analyzed by chromatography at the preparing laboratory or (2) samples extracted, evaporated in a 2-mL glass vial, shipped to the central laboratory, resuspended with 1 mL of ethyl acetate, and analyzed. All means have been averaged across water sources. [b] LSD, least significant difference calculated at 0.05 significance level.

laboratory), (2) analysis type (in-house or SDC sample), and (3) water source (DW or SW). Means for percentage recovery were separated using Fisher's protected least significant difference (LDS) test at the 5% probability level.

## RESULTS AND DISCUSSION

**Quality Control Samples.** No detectable contamination from the four analytes was apparent within the extraction system, laboratory glassware, and SW or DW samples before water sample fortification at any of the locations.

**Interaction of Laboratory and Analysis Type.** The laboratory × analysis type was significant for all four compounds (**Tables 2** and **3**). In general, higher recoveries were found in samples that were analyzed in-house than from SDC. No differences in atrazine recoveries were noted from in-house samples analyzed by laboratories A, B, D, and G compared with the atrazine recovery from SDC samples. In-house atrazine recoveries from laboratories C and F were higher when compared with SDC samples (**Table 3**). However, laboratory E had lower recoveries from in-house samples compared with SDC samples.

A higher recovery of bromacil was found for in-house samples at each laboratory compared with SDC samples except laboratory F (**Table 3**). Greater than 75% recovery of bromacil was obtained for all laboratories except for laboratories F and G. SDC samples had only 14% bromacil recovery from laboratory F, where the in-house samples had 62% recovery (**Table 3**). For laboratory G, bromacil recovery was approximately 56% for in-house samples and 47% for SDC samples (**Table 3**).

Chlorpyrifos results differed between laboratories and were consistent with our previous work (*7*) (**Table 3**). For each laboratory, lower recoveries were observed for chlorpyrifos from the SDC samples compared with in-house samples (**Table 3**). Recovery of chlorpyrifos was >50% for all cooperating laboratories in our earlier work in which the compounds were stored on $C_{18}$ disks and sent to cooperating laboratories (*7*). In this study, the samples sent to the central laboratory were evaporated in a vial after elution, sent, and then redissolved. In five of the laboratories, this resulted in <50% recovery of chlorpyrifos for the SDC samples (**Table 3**). Laboratory F had only 16% recovery of chlorpyrifos in these samples (**Table 3**).

No differences were noted for metolachlor recovery whether samples were analyzed in-house or SDC for laboratories A, F, or G (**Table 3**). However, laboratories B and C showed higher metolachlor recoveries for samples that were analyzed in-house (**Table 3**). Conversely, laboratories D and E showed higher recoveries for SDC samples compared with those samples that were analyzed in-house. Laboratory F had the lowest recoveries of metolachlor (55% in-house and 41% for SDC).

**Interaction of Laboratory and Water Source.** A significant interaction between laboratory and water source was found for all of the compounds tested **(Table 2)**. Laboratories A and G had significantly different atrazine recoveries between deionized water (DW) and surface water (SW) (**Table 4**). Laboratory A showed a higher atrazine recovery from DW than from SW. In contrast, laboratory G showed a higher recovery from SW than from DW. Laboratory G demonstrated this trend for bromacil, chlorpyrifos, and metolachlor recoveries, when SW had statistically higher recoveries than DW (**Table 4**). These results are opposite of what we expected because SW typically contains adverse matrix effects such as organic matter and/or sediment that might reduce recovery. Other laboratories that showed differences in recovery for a given compound always had higher pesticide recoveries for DW than for SW. Laboratory F

**Table 4.** Mean Percentage Recoveries of Pesticides from Solid-Phase Extraction Disks, Demonstrating a Relationship to Water Source as a Function of the Laboratory Responsible for the Extraction

| | | % recovery | | | |
|---|---|---|---|---|---|
| water source[a] | lab | atrazine | bromacil | chlorpyrifos | metolachlor |
| deionized | A | 93.8 | 87.7 | 79.9 | 96.5 |
| | B | 93.5 | 90.6 | 95.7 | 106.8 |
| | C | 72.3 | 87.2 | 55.0 | 65.9 |
| | D | 91.0 | 108.6 | 79.5 | 85.8 |
| | E | 86.8 | 90.4 | 65.3 | 107.1 |
| | F | 51.3 | 55.3 | 28.2 | 53.3 |
| | G | 67.4 | 45.1 | 33.8 | 75.8 |
| surface | A | 77.4 | 93.0 | 49.6 | 88.8 |
| | B | 91.6 | 92.3 | 94.0 | 103.4 |
| | C | 71.1 | 81.8 | 51.0 | 62.9 |
| | D | 87.1 | 104.3 | 71.6 | 79.0 |
| | E | 76.4 | 84.9 | 66.5 | 86.3 |
| | F | 48.7 | 22.3 | 22.7 | 43.1 |
| | G | 86.9 | 57.6 | 65.6 | 100.5 |
| | $LSD_{0.05}$[b] | 14.6 | 9.7 | 18.0 | 16.0 |

[a] Water source: deionized, samples were collected from deionized laboratory water sources at each laboratory; surface, samples were collected from a surface water source near the central laboratory and shipped in 2-L plastic bottles to each participating laboratory. Once at the laboratory, the samples were fortified, extracted, and analyzed. All means have been averaged across analysis types. [b] LSD, least significant difference calculated at 0.05 significance level.

(bromacil), laboratory A (chlorpyrifos), and laboratory E (metolachlor) all showed this trend and contrasted with results obtained from laboratory G (**Table 4**).

**Interaction of Analysis Type and Water Source.** The *P* values for analysis type × water source (**Table 2**) were >0.05 for all four compounds, suggesting that the water source did not affect pesticide recovery whether samples were analyzed in-house or sent to a central laboratory.

**Conclusions.** Due to the varying analytical detection techniques, columns, and temperature programs, the variation in analytical results between laboratories is not surprising. Assuming U.S. EPA standard values for recovery based on EPA Method 525.2 (*10*) of >50% and <200%, some laboratories were out of compliance compared with our earlier work (*7*). Chlorpyrifos was the most problematic compound, considering acceptable recoveries from the water samples, particularly in the sent samples. Difficulties with chlorpyrifos analyses were also noted in our earlier work (*7*). Chlorpyrifos is an organophosphate insecticide that has a relatively high $K_{oc}$ value ranging from 995 to 10450 mL/g and a moderate range vapor pressure of $1.87 \times 10^{-5}$ mmHg (*12*). It is possible that chlorpyrifos adsorbed relatively tightly to the nonpolar matrix of the Empore disks and was not able to be eluted due to its strong attraction to the $C_{18}$ or that some of the material was lost during the evaporation procedure through volatility.

Some laboratories may have lost some analytes in SDC samples during the evaporation step after extraction, when samples were in transit from one laboratory to the central laboratory or could not be effectively redissolved at the central laboratory. Certainly, the large losses of pesticide in this process would render the data unacceptable if this type of methodology were brought under the scrutiny of EPA regulations. In comparison, recovery of these same compounds using an SPE disk as a temporary storage matrix (*7*) seemed to work better than SDC samples. Problems with analytes such as chlorpyrifos are unavoidable, and it should not be assumed that an extraction procedure using SPE disks will be adequate for all compounds and transferrable across all gas chromatographic conditions. At lower water concentrations, we would expect even more variability and potential analytical problems with these analytes with SDC samples. The potential application for this technique should be emphasized as it fits the basic needs of the laboratory's specific analytical goals.

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