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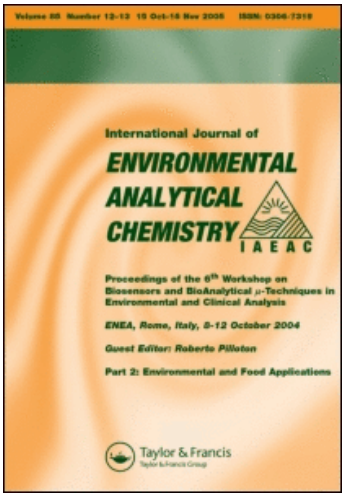

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K. M. Erstfeld[ab]; M. S. Simmons[a]; Y. H. Atallah[ac]
[a] Department of Environmental and Industrial Health, The University of Michigan, Ann Arbor, Michigan, USA [b] Envirosphere Co., Chicago, IL, U.S.A. [c] Environmental Sciences Department, Sandoz Crop Protection Corporation, Des Plaines, IL, U.S.A.

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DETERMINATION OF CHLORDANE IN LABORATORY-GENERATED ENVIRONMENTAL FATE SAMPLES

K. M. ERSTFELD,* M. S. SIMMONS and Y. H. ATALLAH†

Department of Environmental and Industrial Health, The University of Michigan, Ann Arbor, Michigan 48109, USA

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Trans- and *cis*-chlordane have been analyzed in several samples used for aquatic environmental fate studies. These samples include air, water, fish and sediments, The procedures involve extraction with various solvents followed by sulfuric acid clean-up of the extracts. The various solvents used were hexane (air and water), isopropanol/hexane (sediment), and pentane (fish). The fish were further cleaned-up using acetonitrile/pentane partitioning. Extracts were concentrated prior to analysis by GC/ECD. The GC columns used were either 12.5 or 25 m fused silica capillary column coated with OV-17 or a 2 m × 2 mm glass column packed with 3% SE-30 on 80/100 mesh Gas Chrom Q. GC/MS confirmation was conducted on selected samples. Mean recoveries of *trans*- and *cis*-chlordane from the various media ranged from 83.0 to 104.5%, with precisions ranging from 3.0 to 15.6%. The lowest recoveries and precision were found for the fish samples. Limits of detection were 0.01, 0.38 and 0.30 ppb for both *trans*- and *cis*-chlordane in water, sediment and fish, respectively. The detection limit in air was 4.2×10^{-5} ug/L. The analytical methods presented here could easily be implemented for routine analysis of *trans*- and *cis*-chlordane in environmental fate studies.

KEY WORDS: Chlordane, analysis, fish, water, sediment, air.

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INTRODUCTION

In spite of the concern for environmental persistence of certain organochlorine insecticides, their usage continues where there are no economically feasible alternatives. One member of the organochlorine insecticides that falls in this category is chlordane. The use of chlordane has been severely limited during the past seven years. The remaining uses around the world are mostly soil applications under structures for subterranean termite control. In 1987, the use of chlordane in the United States was further limited only to preconstruction soil treatment for termite control.

The fate of chlordane in the environment is not well understood. Several publications addressed the bioaccumulation in the food chain and in certain tissue

*Present address: Envirosphere Co., 111 North Canal Street, Chicago, IL 60606, U.S.A.
†Present address: Environmental Sciences Department, Sandoz Crop Protection Corporation, 1300 East Touhy Avenue, Des Plaines, IL 60018, U.S.A.

of animals.[1] Chlordane residues have been recently reported at several trophic levels in both fresh and salt water[2,3] and at low concentrations in sediment.[4] Chlordane residues of up to nearly 14 ppm have been reported in Harbor Porpoises inhabiting the Bay of Fundy, Canada.[3] Chlorinated hydrocarbons including chlordane have been detected in fish samples taken from large freshwater lakes and streams in Finland, Japan, Iraq, U.S. and Canada.[2,5–11] Residues of chlordane components have been found in seal blubber from the Antarctic. However, the levels of chlordane were low and were 1/100–1/200 of the levels found in mammals off the California coast.

There have been however, questions raised about the validity of the analytical methods used in some of these reports.[1,13] This paper will present verified analytical methods for the analysis of two major chlordane isomers, *trans*- and *cis*-chlordane, in several samples used for aquatic environmental studies (water, sediment, air and fish). These methods can be implemented in a normally equipped residue analysis laboratory.

MATERIALS

Chlordane standards *Cis*- and *trans*-chlordane used in this investigation were analytical reference standards (Minimum Purity: >99% by GC) supplied by Velsicol Chemical Corporation, Chicago, IL.

Reagents and solvents All reagents and solvents used were analytical grade and pesticide-grade quality, respectively. They were obtained either from Burdick and Jackson Company, Muskegon, MI or J. T. Baker Chemical Company, Phillipsburg, NJ.

Water De-ionized water, used throughout the study, was obtained from a Milli-Q water purification system (Millipore Corp., Bedford, MA) equipped with ion-exchange and carbon cartridges to remove dissolved inorganic and organic contaminants.

Sediments Sediments used were obtained from the following sources: Sand, pre-ignited and washed was obtained from Mallinckrodt, Inc., Paris, KY. Kaolin clay, Suprex grade, was obtained from J. M. Huber, Inc., Langley, SC. Muck soil, sieved through a 2 mm mesh screen, was collected at Woodstock, IL.

Fish species and specifications The test fish used were goldfish (*Carassius auratus*) weighing between 1.0 to 5.0 grams obtained from Carolina Biological Supply Co., Burlington, NC.

Glassware All glassware was obtained from either Kontes Corp., Evanston, IL or American Scientific Products, Inc., McGaw Park, IL. They were cleaned and were free from organic contaminants.

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EQUIPMENT AND APPARATUS

Analytical Instruments

Gas chromatograph The gas chromatographs used were Hewlett-Packard (Avondale, PA) Models 5710A and 5880A, equipped with ^{63}Ni electron capture detectors. The columns were 12.5 (0.53 mm ID) and 25 (0.20 mm ID) meter fused-silica capillary coated with OV-17 or a 2 mm × 2.0 m glass column packed with 3% SE-30 on 80/100 mesh Gas Chrom Q.

For injections into the 12.5 m capillary column, the GC conditions were as follows: Hewlett-Packard 5880 with ^{63}Ni electron capture detector with temperature programming of 0.5 min at 100 °C then 30 °C/min to 190 °C and hold for 5 min. Detector and injector temperatures were 300 °C and 250°C respectively. Helium was the carrier gas at a flow rate of 3.0 mL/min with 5% methane/argon as a make up gas at 30 mL/min. Splitless injections were made using helium as inlet purge at 30–50 mL/min with a delay purge of 0.5 min using an attenuation of 256.

For injections into the 25 m capillary column, the oven temperature was programmed as follows: 0.5 min at 150 °C, then 25 °C/min to 210 °C and hold for 9.5 min. All other GC parameters remained identical to the conditions used for injections into the 12.5 m capillary column.

For packed column injections, the GC conditions were as follows: Hewlett-Packard 5710A with ^{63}Ni electron capture detector with an oven temperature of 190 °C. Injector and detector temperatures were 250 °C and 300 °C, respectively. The carrier gas was 5% methane/argon at 30 mL/min, with an attenuation of 16. The column used was a 2.0 m × 2 mm i.d., silane treated pyrex glass, 3% SE-30 on 80/100 mesh Gas Chrom Q.

Gas chromatograph/mass spectrometer Confirmation of the chlordane residues in selected samples were conducted using a Quadrapole Extranuclear Model ELQ-400-2 GC/MS system, operated in the electron impact mode (70 ev). Selective ion monitoring of masses 371, 373 and 375 were scanned for the analysis of *trans*- and *cis*-chlordane. The GC column used was a 30 m × 0.25 mm DB5 Durabond fused-silica column.

Air sampling Chromosorb-102 air sampling tubes, were obtained from SKC Inc., Eight Four, PA. These tubes were connected to air sampling pumps (Gilian, Model HFS 1137, with programmable timer). A mass flowmeter (Kurz Instruments, Inc., Model No. 541, Carmel Valley, CA) was used to calibrate air flow through the sampling tube.

Miscellaneous equipment Polytron (reg.) issue homogenizer, distributed by Brinkman Instruments. Shaker, reciprocating, mechanical, Eberbach Corp., Ann Arbor, MI. Centrifuge, Sorvall model RC5C, GSA rotor, DuPont Co. Wilmington, DE. Vortex mixer, Super-Mixer, Lab-Line Instruments, Inc., Melrose Park, IL.

Preparation of Chlordane Solutions

To prepare stock aqueous solution, 1–2 mg each of *trans*- and *cis*-chlordane were

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shaken with 3.50 liters reagent grade water in a 3.75 liter amber glass bottle. The solution was allowed to settle for one day and the supernate was carefully collected using a pipet. The concentration of *trans*- and *cis*-chlordane was chromatographically determined. The working aqueous solution was then prepared by the appropriate dilution of the stock solution. The solutions were stored in amber 3.75 liter bottles.

To prepare fortifying and GC standards, one hundred milligrams each of *trans*- and *cis*-chlordane were weighed into separate 100 mL volumetric flasks. For the fortifying standard, acetone was added and the volume brought up to the mark. For the GC standard, hexane was used. The concentration of these stock solutions were 10^{-6} g/uL. Fortifying standards of 10^{-8} and 10^{-9} g/uL containing both *trans*- and *cis*-chlordane were prepared by making the appropriate dilutions. Concentrations of 10^{-10}, 10^{-11}, and 10^{-12} g/uL GC standards were prepared by making serial dilutions of the 10^{-6} g/uL solution.

Extraction of Water

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Water samples were extracted with hexane in graduated Kuderna-Danish (K-D) tubes. The hexane extract was transferred to another K-D tube treated with sodium sulfate to remove traces of water, and concentrated prior to analysis by GC/ECD. The details of this procedure are shown below:

Water samples (8 mL each) were fortified at 1.0 ppb *trans*- and *cis*-chlordane. In addition, triplicate 8 mL aqueous chlordane samples were removed from the amber 3.75 liter bottle with a pipet. The samples were measured into 12 mL graduated K-D tubes.

The samples were then extracted by adding 2 mL of hexane to the water sample and shaking for 30 seconds. One mL of the hexane extract was removed and transferred to a clean graduated K-D tube containing approximately 0.1 g Na_2SO_4. Fortified water samples were also extracted immediately after fortification. Further clean up was not needed. All hexane extracts were then analyzed by GC/ECD. A typical chromatogram of a fortified water sample compared to a standard is given in Figure 1.

Extraction and Cleanup of Sediment

Two sets of sediment samples were fortified at 100 ppb each of *trans*- and *cis*-chlordane. The first set was composed of sediment samples (sand, muck, and clay; 100 g each). Each sediment type was placed into an Erlenmeyer flask and fortified with 1.0 mL acetone containing 10^{-8} g/uL *cis*- and *trans*-chlordane. The samples were mixed on a mechanical shaker for 1 hour. The acetone was evaporated under a stream of nitrogen. These samples served as a source of subsamples used to develop a method for recovery of *trans*- and *cis*-chlordane from sediment. Two gram subsamples were taken for analysis.

The second set was composed of wet sand samples (2 g each; dry weight). The

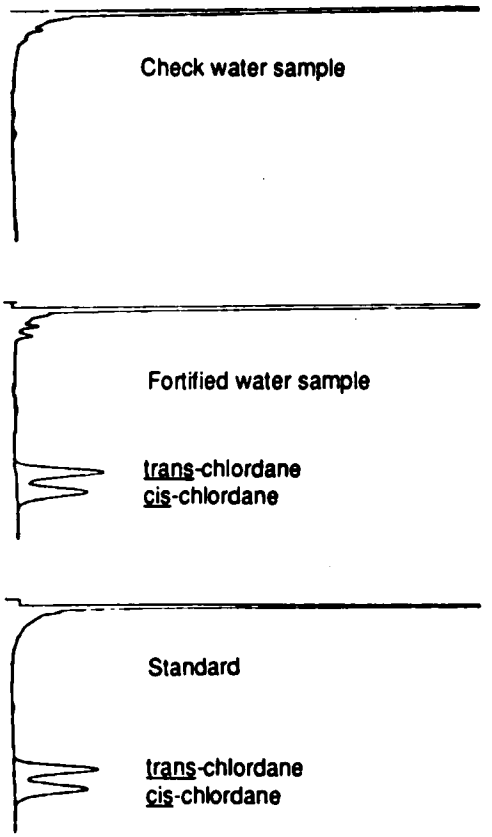

Figure 1 Representative chromatograms of *cis*- and *trans*-chlordane in water extracts. Packed column GC conditions, 5 ul injection. Retention times (min) of chlordane for fortified samples: *trans* (1.55 ppb), 5.415; *cis* (1.45 ppb), 6.041. Retention times (min) for standards: *trans* (0.03 ng on column) 5.414; *cis* (0.03 ng on column), 6.047.

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samples were placed in 2 oz bottles and fortified with 20 uL of 10^{-8} g/uL *trans*- and *cis*-chlordane. The samples were then shaken to ensure homogeneity and purged with nitrogen for 5 minutes. The sand was then wetted with 5 mL Milli-Q water. These samples were used to determine the accuracy of the analytical method.

Extraction and cleanup of the sediments is described below: Triplicate 2.0 g samples of the fortified sediment were weighed into 2 oz screw cap bottles. Five mL of water were added and the mixture was swirled manually for approximately 10 seconds. Five mL of isopropanol were added and the sample was shaken vigorously. Ten mL of hexane was added and the samples were placed on a shaker for 1 hour. The samples were centrifuged for 10 minutes at 568 G and the extracts were decanted through glass wool into 60 mL separatory funnels. The glass wool was rinsed with hexane to ensure quantitative transfer of the sample.

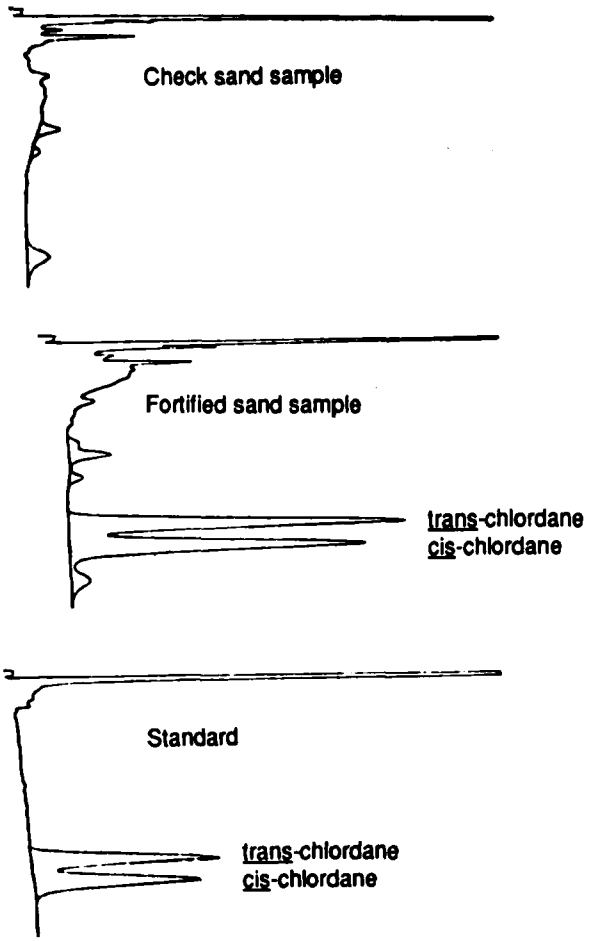

Figure 2 Representative chromatograms of *cis*- and *trans*-chlordane in sediment (sand) extracts. Retention times (min) for sand samples: *trans* (86.6 ppb), 5.939; *cis* (88.1 ppb), 6.621. Retentions times (*min*) for standards in sediments: *trans* (0.06 ng on column), 5.946; *cis* (0.06 ng on column), 6.623.

The extracts were partitioned twice, with successive 25 mL of a 2% sodium sulfate solution. The hexane layer was transferred to 12 mL graduated receivers containing about 0.1 g Na_2SO_4.

Cleanup (when necessary) of the hexane extract was as follows: The hexane extract was concentrated to 2.0 mL and 0.5 mL concentrated sulfuric acid was added. The mixture was placed for 30 seconds on a Vortex mixer. The phases were allowed to separate and 1.0 mL of the hexane layer was removed with a Pasteur pipet into a vial containing about 0.1 g of 9:1 Na_2SO_4: Na_2CO_3. All sample extracts, after appropriate concentration or dilution (if needed) were analyzed by GC/ECD. Chromatograms of a sediment fortified sample compared to a standard is given in Figure 2. Chromatograms of muck and clay extracts are given in Figure 3.

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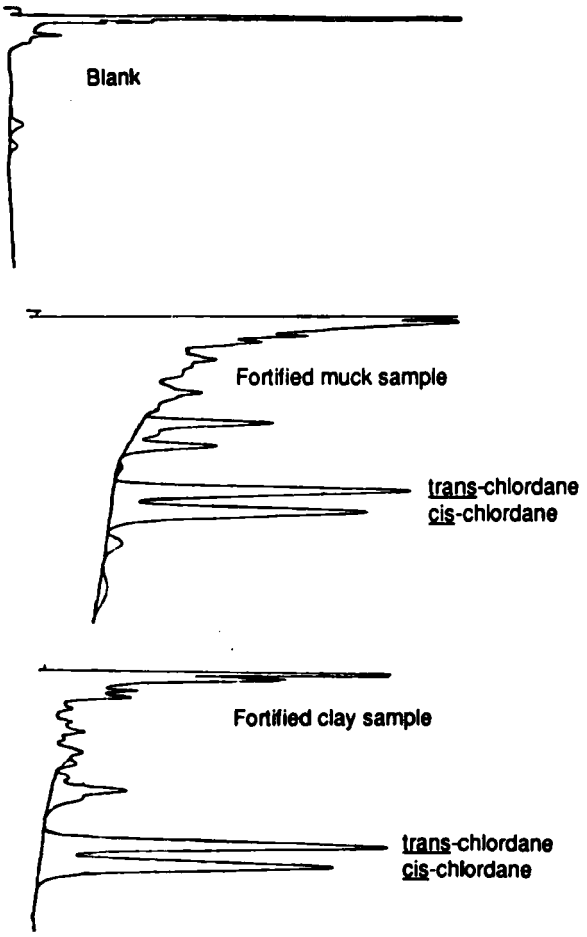

Figure 3 Representative chromatograms of *cis*- and *trans*-chlordane in sediment (muck and clay) extracts. Packed column GC conditions, 2 g sample extracted with a final volume of 1.0 ml, 5.0 ul injection. Retention times (min) for muck samples: *trans* (81.0 ppb), 5.536; *cis* (82.0 ppb), 6.617. Retention times (min) for clay samples: *trans* (93.1 ppb), 5.934; *cis* (90.2 ppb), 6.813.

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Extraction and Cleanup of Fish

Two sets of whole fish samples, ranging in mass from 1.0 to 5.0 g, were respectively fortified with 200 uL of 10^{-8} or 10^{-9} g/uL containing both *cis*- and *trans*-chlordane. Fifty mL of pentane were added to each sample. The samples were homogenized using a Polytron (Reg.) tissue homogenizer equipped with a 10 cm blade. The samples were then filtered through a funnel containing sodium sulfate, into a 125 mL Kuderna-Danish concentrator. The samples were reduced to 10 mL. Five mL of the sample extract was then transferred to a 60 mL separatory funnel containing 10 mL of acetonitrile saturated with pentane. The sample was extracted

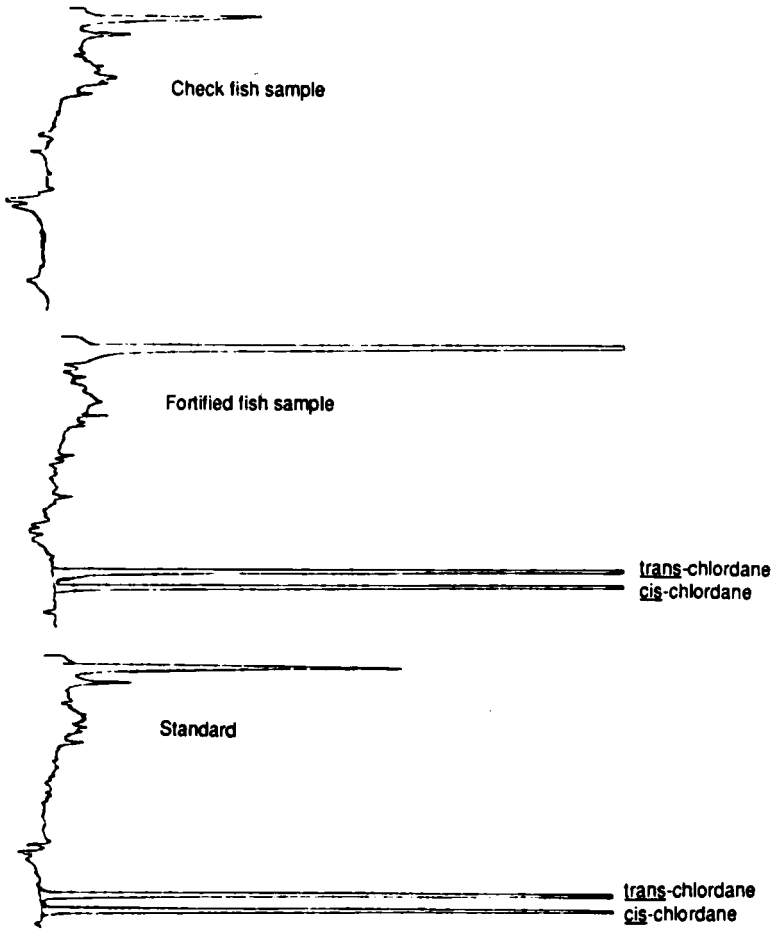

Figure 4 Representative chromatograms of *cis*- and *trans*-chlordane in fish extracts. Capillary column (12.5 m) GC conditions, 2 ul injection. Retention times (min) of chlordane: *trans* (662.9 ppb), 6.531; *cis* (654.5 ppb), 6.963. Retention times (min) of chlordane standards: *trans* (0.08 ng on column), 6.534; *cis* (0.08 ng column), 6.966.

two more times, each with 5 mL of acetonitrile saturated with pentane. The combined acetonitrile layers were drained into a 500 mL separatory funnel containing 190 mL water, 10 mL of saturated sodium chloride solution and 25 mL of pentane. The mixture was shaken vigorously and the aqueous phase was extracted with 10 mL pentane then discarded. The pentane extracts were combined and passed through a funnel containing Na_2SO_4 into a 125 mL Kuderna-Danish apparatus containing about 2 mL of hexane. The sample extract was then concentrated to about 2 mL. Cleanup of the extract (when necessary) was as that described above for the sediment cleanup. A typical chromatogram for fortified fish sample compared to a standard is given in Figure 4.

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Extraction of Air Samples

Two sets of Chromosorb-102 air sampling tubes were prepared to determine both the adsorption and desorption efficiency for *trans*- and *cis*-chlordane. The desorption efficiency was determined at two levels by fortifying Chromosorb 102 (which was removed from the tubes and placed in 2 oz bottles) with 50 mg or 1.0 ug of *trans*- and *cis*-chlordane. Triplicate samples were fortified at each level. A check sample, which was treated in the same manner except that no chlordane was added, was simultaneously analyzed.

To determine the adsorption efficiency of *trans*- and *cis*-chlordane, the front section of three Chromosorb-102 air sampling tubes were fortified with 100 ul of acetone containing 1.0 ug of each compound. An untreated sample was simultaneously run throughout the course of the experiment. The exit ends of the tubes were connected to vacuum pumps which were calibrated using a mass flow meter. The flow rates through the air sampling tubes were adjusted to 0.75 liters/min and were periodically checked after 10–15 minutes and after 3.50 hours. The adsorption efficiency experiment was performed in an environmental chamber where the temperature was maintained at $25 \pm 0.5\,^{\circ}C$, the relative humidity was 70% and the barometric pressure was 1015 mbars. Air was drawn through the air sampling tubes for 4 hours at which times the tubes were removed from the pumps and the ends of the tube were capped until extraction.

The samples were extracted as follows: the adsorbent was dislodged from the tubes and transferred to 2 oz bottles. The samples were shaken with 20 mL of hexane for 30 minutes on a reciprocating shaker and analyzed by GC/ECD. Chromatograms of the Chromosorb-102 air extracts are contrasted with a standard sample in Figure 5.

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Results

Summarized in Tables 1 and 2 are information on the verified analytical methods for the determination of *trans*- and *cis*-chlordane, respectively, from water, sediment, air, and fish. These tables include the average recovery (accuracy of the analytical method), standard deviation (precision of the analytical method), detection limits, and the level of fortification for each of the above mentioned media.

Recoveries of *trans*- and *cis*-chlordane from fortified water samples averaged 103.3 and 104.5%, respectively. The method precision was 5.7% for *trans*-chlordane and 4.9% for *cis*-chlordane.

During the initial method development work, based on a 8.0 mL sample, 2.0 mL final volume of extract, and a 5.0 uL injection, the limit of detection was 0.23 ppb. However, to attain a lower limit of detection, a sample volume of a 90 mL water sample, a final volume of extract of 1.0 mL and a 2.0 uL injection yields a limit of detection of 0.010 ppb.

Recoveries of *trans*- and *cis*-chlordane from fortified wet sand samples averaged 87.4 and 89.0%, respectively, with less than 1% of the chlordane being recovered in

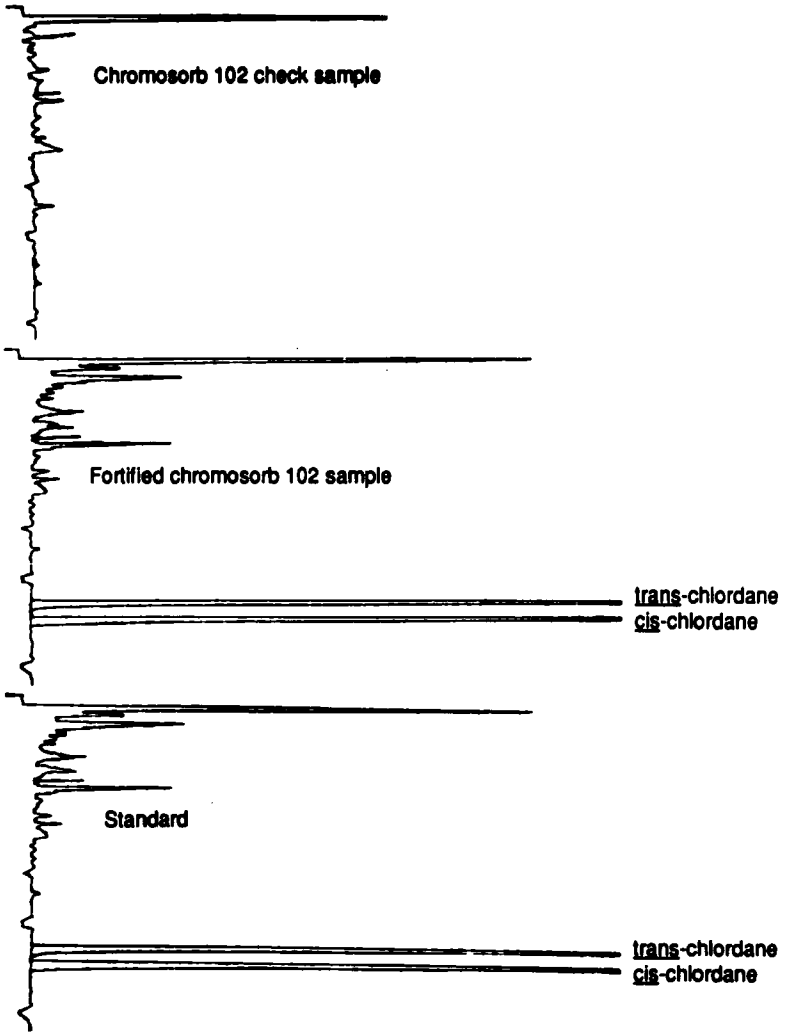

Figure 5 Representative chromatograms of *cis*- and *trans*-chlordane in air extracts. Capillary column (12.5 m) GC conditions, 2 ul injection. Retention times (min) of chlordane: *trans* (5.17×10^{-3} ng/m^3), 6.462; *cis* (5.0×10^{-3} ng/m^3), 6.845. Retention times (min) of chlordane standards: *trans* (0.1 ng on column), 6.465; *cis* (0.1 ng on column), 6.887.

the second and third extracts. Excellent recoveries were also found for the other sediment types.

Recoveries range from 89.4 (muck sediment) to 97.4% (clay sediment) for *trans*-chlordane, with an average precision of $\pm 4.0\%$. Recoveries of *cis*-chlordane range from 89.1 (muck sediment) to 97.6% (clay sediment), with an analytical precision averaging 6.2%. Based on a 2 gram sample, a final extract volume of 1.0 mL, 2.0 uL injection, and an instrument sensitivity of 1.5 pg, the limit of detection is 0.38 ppb.

Recoveries of *trans*- and *cis*-chlordane from fish fortified between 41.3 and

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Table 1 Summary information on the verified analytical methods for the analysis of *trans*-chlordane from water, sediment, air and fish

Parameter	*Water*	*Sediment*	*Air*	*Fish*
Limit of detection (ppb)	0.010	0.38	4.2×10^{-5} ug/L	0.30
Level of fortification	1.0 ppb	10C _ pb	1.0 ug[a] 50 ng and 1.0 ug[b]	0.59–1.82 ppm
C.V.[c] (precision)	5.7%	4.8% (sand) 4.3% (muck) 3.0% (clay)	3.0%[a] 5.2%[b]	15.6%
Recovery (accuracy)	103.3%	90.7% (sand) 89.4% (muck) 97.4% (clay)	88%[a] 88.7%[b] (50 ng) 83.0[b] (1 ug)	89.7%

[a]Adsorption efficiency.
[b]Desorption efficiency.
[c]Coefficient of variation.

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Table 2 Summary information on the verified analytical methods for the analysis of *cis*-chlordane from water, sediment, air and fish

Parameter	*Water*	*Sediment*	*Air*	*Fish*
Limit of detection (ppb)	0.010	0.38	4.2×10^{-5} ug/L	0.30
Level of fortification	1.0 ppb	100 ppb	1.0 ug[a] 50 ng and 1.0 ug[b]	0.59–1.82 ppm
C.V.[c] (precision)	4.9%	6.7% (sand) 4.2% (muck) 7.7% (clay)	3.9%[a] 5.4%[b]	12.5%
Recovery (accuracy)	104.5%	92.6% (sand) 89.1% (muck) 97.6% (clay)	89%[a] 92%[b] (50 ng) 85.7%[b] (1 ug)	83.4%

[a]Adsorption efficiency.
[b]Desorption efficiency.
[c]Coefficient of variation.

100.5 ppb averaged 96.0 and 82.0%, respectively, with coefficients of variation of 20.0 and 17.6%. Recoveries from fish fortified between 0.59 and 1.82 ppm were $85.0 \pm 8.6\%$ and $84.3 \pm 8.8\%$ for *trans*- and *cis*-chlordane, respectively. The verified analytical method for fish has a recovery of $89.7 \pm 15.6\%$ for *trans*-chlordane and a recovery of $83.4 \pm 12.5\%$ for *cis*-chlordane. Based on 5.08 mg equivalents injected on column in the check sample and an instrument sensitivity of 1.5 pg, the limit of detection is 0.30 ppb.

Recoveries of *trans*- and *cis*-chlordane from Chromosorb-102 tubes (desorption efficiency) averaged 88.7 and 92.0%, respectively for fortification at the 50 ng level.

Recoveries of *trans*- and *cis*-chlordane from Chromosorb-102 tubes fortified at 1.0 ug averaged 83.0 and 85.7% respectively. Thus, the accuracy of the analytical method is approximately 86% for *trans*-chlordane and 89% for *cis*-chlordane. The coefficient of variation for the analytical method is 5.4%.

The adsorption efficiency (recovery) of *trans*- and *cis*-chlordane onto Chromosorb-102 tubes averaged 88 and 89% respectively for the subject compounds, thus indicating that Chromosorb-102 is effective in trapping chlordane.

DISCUSSION

The mean recoveries of *trans*- and *cis*-chlordane from the various media ranged from 83.0 to 104.5%. These recoveries are within the acceptable range for analysis of pesticides in environmental samples.[14–16] Generally, the lowest recoveries and precisions were found for the fish samples.

A detection limit of 0.30 ppb for fish is on the lower end for values reported in the literature for detection of *trans*- and *cis*-chlordane. These values range from approximately 0.10 ppb to 50 ppb.[6,17]

The limit of detection (LOD) for sediment was 0.38 ppb while that for water was 0.01 ppb. Since the subject compound (chlordane) tends to adsorb to sediment and partition in fish, thus leaving very low concentrations in water, a rather low LOD for chlordane in water was needed for environmental fate assessment.

In the analysis of environmental samples, additional cleanup of fish and sediment samples using florisil or gel-permeation chromatography for removal of remaining co-extractives and other interfering organochlorine compounds may be necessary. However, the verified analytical methods presented here could be easily implemented for routine analysis of chlordane in samples used for environmental fate studies.

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