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Shaughnessy No.: 059101

Date Out of EAB: 20 SEP 1983

To: Jay Ellenberger
Product Manager 12
Registration Division (TS-767)

From: Richard V. Moraski, Head (acting)
Review Section 1
Exposure Assessment Branch
Hazard Evaluation Division (TS-769c)

Attached please find the EFB review of...

Reg./File No.: 464-562

Chemical: Chlorpyrifos

Type Product: Insecticide

Product Name: Dursban TC

Company Name: Dow

Submission Purpose: Additional Use in Plenum Houses

ZBB Code: ?

ACTION CODE: 311

Date In: 6/28/83

EFB # 3440

Date Completed: 9/19/83

TAIS (level II)	Days
63	2.0

Deferrals To:

_____ Ecological Effects Branch

_____ Residue Chemistry Branch

_____ Toxicology Branch

1.0 INTRODUCTION

On June 14, 1983, the registrant (Dow) responded to issues raised in the EAB review of 06/04/83, providing some of the requested information. Evaluation of the submitted data uncovered a significant number of deficiencies. The PM agreed to a revision of the original due date (copy of request appended) to allow time for the registrant to provide copies of all pertinent information. As of 9/19/83, no response from the registrant has been received by EAB.

2.0 STRUCTURE and DIRECTIONS FOR USE

See previous reviews.

3.0 CONCLUSIONS and RECOMMENDATION

EAB now does not have sufficient time between today and the due date of 9/23/83 to properly review any data which the registrant may submit. Therefore, the original package is being returned to RD unreviewed.

Please resubmit this and all other data when available, allowing the usual review time for this kind of data. At that time, EAB will evaluate additional time requirements, if any.

Emil Regelman
Chemist
EAB/HED (TS-769c)
September 19, 1983

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

31 AUG 1983

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

TO: Jay Ellenberger
Product Manager # 12
Registration Division (TS-767)

SUBJECT: RD Due Date for Chlorpyrifos
Reg./File No. 464-562

This is to confirm that the RD due date for Chlorpyrifos has been changed to 9/23/83. The original due date was 9/7/83. This submission was received in HED on 6/27 and the RD action code is 311. Attached is the revised copy of the RD data Review Record.

This due date revision is based on the assumption that the registrant will provide EAB with copies of all requested supportive documentation for the proposed use in plenum houses on or before the original due date.

Thank you for your cooperation in the matter.

Richard V. Moraski

Richard V. Moraski, Ph.D.
Acting Chief
Environmental Chemistry Review Section No. 1.
Exposure Assessment Branch/HED (TS-769)

cc: Joan Moyer, MSS/HED
Mary Ellen Geraghty, RSERS/RD
Emil Regelman, EAB Reviewer/HED

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DOW CHEMICAL U.S.A.

POST OFFICE BOX 1706
MIDLAND, MICHIGAN 48640

9008 Building
June 14, 1983

Mr. Jay S. Ellenberger
Product Manager 12
Registration Division (TS-767C)
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

Subject: DURSBAN* TC Insecticide (EPA Reg. No. 464-562) Additional Claims for Use in Plenum-type Structures

Dear Mr. Ellenberger:

In the response from the Agency to the application dated August 20, 1982, three general comments are made. In response to these the following data are submitted:

1. Method for determining the airborne levels of chlorpyrifos.

   Collection of Chlorpyrifos and Other Pesticides in Air on chemically Bonded Sorbents, Analytical Chemistry, Vol. 50, No. 2, February 1978, R. G. Melcher, W. L. Garner, L. W. Severo and J. R. Vaccaro. This literature contains the procedure for collecting and analyzing airborne chlorpyrifos. The percent recovery obtainable and sample analytical curves are also included. The chromatographic standard was DURSBAN R Insecticide.

2. The collection times for the samples reported in the August 20 submission were all 1.0 to 1.5 hours. The sampling times for the report submitted with the letter of February 22, 1983 (4.5 months after treatment) were also 1 to 1.5 hours. The worker exposure data was obtained throughout the application period which lasted from 1 to 2 hours. To obtain reliable results numerous samples were collected and the averages reported.

3. During the initial sampling the forced air, air-conditioning systems were on in house #1, 4926 Florodora; house #3, 5088 Weathermaker; and house #4, 5067 E Home Street. During the 4.5 after treatment sampling the forced air heating systems were on in each structure.

The results from the testing indicates that there is no difference in airborne levels of chlorpyrifos associated with or without furnace operation.

A timely response to this additional data is anticipated.

Sincerely,

D. H. Naffziger

David H. Naffziger
Product Registration Manager
Agricultural Products Department

nbe
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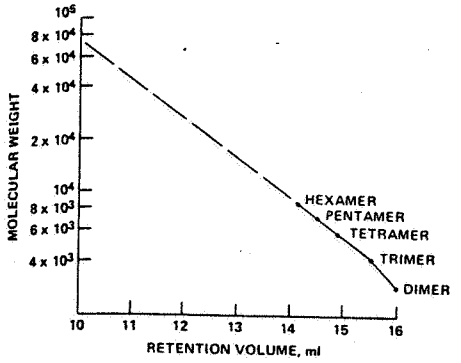

Figure 6. Calibration curve obtained with triazine fluoroether polymer for SE 60/SE 100/SE 500 columns

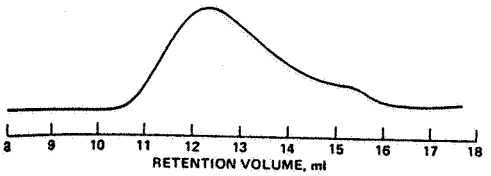

Figure 7. Chromatogram of high molecular weight 1,2,4-oxadiazole fluoroether polymer with $m + n = 6$. SE 60, SE 100, and SE 500 columns, refractive index detector, 1 mL/min flow rate 2.3 mg sample injected in 50 μL Freon 113. Maximum decrease in refractive index was 6×10^{-6} RI

series with either the DuPont SE 100 column or the Waters μ Styragel 300 column. The chromatogram of a higher molecular weight oxadiazole polymer is shown in Figure 5. Similar chromatograms were obtained for several high molecular weight oxadiazole and triazine polymers. All of these chromatograms showed a rapid increase in UV absorbance and a rapid change in refractive index, indicating elution of polymer beginning at 6.17 mL. The position of this exclusion limit is shown in Figure 4 and corresponds to a molecular weight of approximately 32 000.

To extend molecular weight measurements to higher molecular weights, the DuPont SE 500 column was placed in series with other columns with lower molecular weight ranges. Using three DuPont columns, SE 60, SE 100, and SE 500, resolution of the fluoroether oligomers was reduced, and only distinct peaks up to the hexamer could be observed in chromatograms of low molecular weight triazine fluoroethers. The calibration curve obtained for these three columns is shown in Figure 6. The oxadiazole polymer used to obtain the chromatogram of Figure 5 was injected into this three-column set, and the resulting chromatogram is shown in Figure 7. The calibration curve of Figure 6 can be applied to the chromatogram in Figure 7 to compute the weight and number average molecular weights of the oxadiazole polymer (*3*). Thus $\bar{M}_w$ equals 19 500 and $\bar{M}_n$ equals 13 200. In this way, it is possible to calculate the various molecular weight averages of fluoroether polymers.

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RECEIVED for review August 25, 1977. accepted November. 7, 1977.

Collection of Chlorpyrifos and Other Pesticides in Air on Chemically Bonded Sorbents

Richard G. Melcher,* Warren L. Garner, Loren W. Severs, and James R. Vaccaro

Analytical and Industrial Hygiene Laboratories, Dow Chemical U.S.A., Midland, Michigan 48640

A procedure is described for the collection and determination of chlorpyrifos [*O,O*-diethyl *O*-(3,5,6-trichloro-2-pyridyl)-phosphorothioate] in air at the low parts per trillion level for sampling periods of 10 min up to 16 h. Worker breathing zone or area samples are collected by pumping air through a small tube containing a chemically bonded sorbent, GC Durapak–Carbowax 400/Porasil F. Samples are analyzed by desorption in diethyl ether and injection into a gas chromatograph equipped with a Poly I-110 column and an electron capture detector. Preliminary results for ronnel, lindane, carbaryl and diazinon indicate applicability of this technique to a wide range of pesticides.

A wide variety of sampling techniques have been applied to the determination of pesticides in air, and are described in a recent review (*1*). Most of the earlier methods for sampling pesticide vapors utilized solvent absorbent impingers; however, recently the general emphasis is toward the use of solid sorbents. Solid sorbents are now being used successfully for the collection of a variety of solvent vapors in work atmospheres. A small tube containing a solid sorbent is convenient to use, provides concentration of the trace pollutants, and can be attached to the worker for determination of actual breathing zone exposures for periods up to eight hours.

Some work has been reported in the literature (*2, 3*) using solid adsorbents such as silica gel and activated charcoal for the sampling of some pesticides in air. However, when these adsorbents were tried for the collection of chlorpyrifos [*O,O*-diethyl *O*-(3,5,6-trichloro-2-pyridyl)phosphorothioate] at the trace amounts which may be found in work atmospheres, very low recoveries were obtained.

A highly sensitive method has been developed by Russell (*4*) for chlorpyrifos and ronnel [*O,O*-dimethyl *O*-(2,4,5-trichlorophenyl)phosphorothioate] using a sampling tube filled with 20% DC 200 chromatographic packing, and analyzed by thermal desorption directly into a gas chromatograph. This

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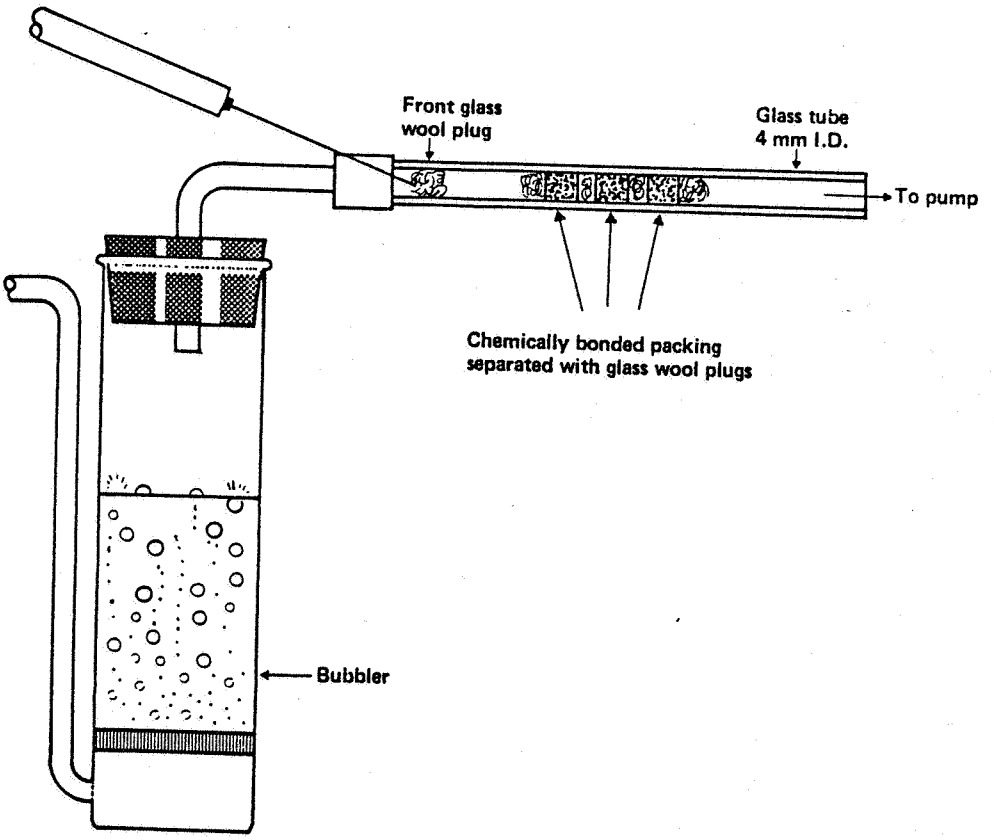

Figure 1. Experimental apparatus for determining breakthrough and recovery

technique can be adapted to personnel sampling but the analysis requires a somewhat specialized technique and the information obtainable is limited due to the "one shot" nature of the analysis.

The use of liquid-coated cloth screens and supports has been suggested (*5*, *6*); however, this suffers the disadvantage of coextraction of the liquid phase, which may lead to analytical problems. This problem can be avoided by the use of porous polymers or chemically bonded packings. Thomas and Seiber (*7*) have reported the use of Chromosorb 102 for a series of pesticides including chlorpyrifos. The procedure involved the collection on 4–5 g of Chromosorb 102 at the sampling rate of 12 to 13 L/min. The compounds were analyzed by gas chromatography following a 1-h Soxhlet extraction and a vacuum evaporation concentration step. Similar methods using XAD-2 resin (*8*) and polyurethane foam (*9*) for the collection of various pesticides have been reported. Aue and Teli (*10*) reported a brief experiment showing the collection of several chlorinated hydrocarbon insecticides on a support-bonded silicone, $(C_{18}H_{32}SiO_{3/2})_n$, prepared by them. Collection was on a large cartridge with a flowrate of 10 to 20 L/min. The cartridge was extracted by the circulation of 200 mL of pentane which was subsequently concentrated by evaporation and analyzed by gas chromatography. Although all these techniques are highly sensitive for pesticides in ambient air, they are not applicable to personnel breathing zone sampling, and the analytical procedures are lengthy.

This paper describes the development of a procedure for the collection and determination of chlorpyrifos in air at the low parts per trillion level for sampling periods of 10 min up to 16 h. Preliminary testing on a number of other pesticides has also been successful. Samples are collected on a small tube containing a commercially available chemically bonded sorbent and are subsequently analyzed by desorption in a small amount of solvent and direct injection into a gas chromatograph. The technique has been tested in a number of plant and field applications. The solvent desorption system described in this paper permits duplicate analyses by different techniques, treatment to eliminate, separate, and identify interferences, and also permits automatic injection by available liquid handling systems.

EXPERIMENTAL

Apparatus. The laboratory experimental setup is shown in Figure 1. The 8-cm glass tube (4-mm i.d.) contained three 5-mm sections of the sorbent separated by small glass wool plugs. The humidity was controlled by attaching a bubbler containing a saturated solution of either potassium carbonate (45% relative humidity) or ammonium sulfate (80% relative humidity).

A Hewlett-Packard Model 5710A gas chromatograph equipped with a hydrogen flame ionization detector or an electron capture ^{63}Ni detector was used to perform analyses. Chromatographic separations were accomplished on one of two phases, either a 1.8-m glass column packed with 3% SE-30/3% QF-1 or a 0.9-m glass column packed with 3% Poly I-110, at a column temperature of 200 °C, injection port 200 °C, detector 250 °C, and a carrier flow of 35 mL/min. The collection sorbents used in this study were: GC Durapak–Carbowax 400/Porasil F, 100–120 mesh; GC Durapak–*N*–Octane/Porasil C, 100–120 mesh; GC Durapak–Phenylisocyanate/Porasil C, 80–100 mesh; and GC Durapak–OPN/Porasil C, 80–100 mesh, all obtained from Supelco Inc., Bellefonte, Pa. These sorbents were used with no prior treatment and showed no interferences for the pesticides tested. New batches of the packings should be tested before use and rinsed with diethyl ether if the background level is too high. The glass wool and glass tubes (8 cm × 4 mm i.d.) were rinsed with diethyl ether. The portable pump used for personnel sampling was either a Sipin Model SP-1 or an SKC personnel pump.

Collection and Recovery Studies. Solutions of the various pesticides were prepared in a volatile solvent, diethyl ether or methanol, at concentrations of 0.1 to 1.0 mg/mL. Two to 20 μL of the solution was injected into a separate glass wool plug in the front end of the collection tube, while air was being pumped through the system. For the humidity studies, the bubbler was immediately attached to the inlet of the collection tube after

Table I. Laboratory Collection and Recovery Experiments for Selected Pesticides on Bonded Packings

Pesticide	Sorbent[a]	Amount Added (μg)	Amount Found (μg) Front Section	Back Section	% Recovery
Ronnel [O,O-dimethyl O-(2,4,5-trichlorophenyl) phosphorothioate]	1	54.5	47.3	0.5	88
	2	51.3	41.1	2.6	85
	3	50.5	35.7	5.3	81
	4	39.0	25.4	1.9	70
Lindane (1,2,3,4,5,6-hexachlorocyclohexane)	1	5.93	5.68	0.002	96
	2	5.21	4.56	0.05	88
	3	5.97	5.74	0.004	96
	4	5.90	5.53	0.02	94
Carbaryl (naphthyl N-methyl carbamic acid)	1	6.4	6.0	Not detected	94
	2	11.8	3.6	" "	31
	3	7.5	4.8	" "	72
	4	9.6	2.4	" "	25
Diazinon (diethyl 6-isopropyl-2-methyl-4 pyrimidinyl phosphorothionate)	1	1.2	1.0	" "	83

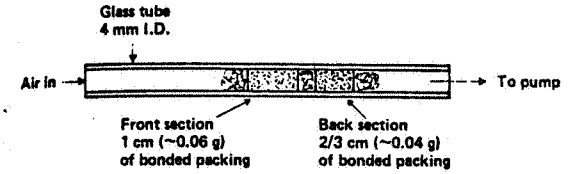

Figure 2. Sampling tube

injection, as shown in Figure 1.

A collection tube for plant, field, and personnel samples was designed and is shown in Figure 2. A collection and storage study was conducted using this size collection tube with a front glass wool plug added for injection of the concentrated solution. Samples for the storage study were collected for 8 h at the concentration of $^1/_{10}$ the TLV (threshold limit value) and stored on the bench top for periods of 1 to 7 days.

Recovery experiments for ronnel, lindane, carbaryl, and diazinon were carried out using the two section tubes. All pesticide loadings were at or below $^1/_2$ the TLV level. In each experiment, air of 45% relative himidity was drawn through the tube at 0.1 L/min for 4 h, and the front glass wool plug and sorbent sections were analyzed separately.

Sample Analysis. After the sampling period, the separate front glass wool plug and each section was analyzed for chlorpyrifos separately by shaking with 2 mL of diethyl ether for 15 min. In the storage study, the front glass wool plug was analyzed the day of collection. Three microliters of the extract was injected into a gas chromatograph equipped with an electron capture detector and either the Poly I-110 or the SE-30/QF-1 column. Some of the field samples were analyzed using a flame photometric detector to obtain additional specificity. The extracts of ronnel and lindane were chromatographed using the Poly I-110 column with an electron capture detector and extracts of carbaryl and diazinon were chromatographed using the Poly I-110 column and a hydrogen flame ionization detector.

Concentrations of compounds in air were calculated from the equation:

$$\text{ppb (vol/vol in air)} = \frac{\mu\text{g found} \times 24.45 \times 10^3}{\text{liters of air collected} \times \text{molecular weight}}$$

The factor 24.45 is the volume occupied by 1 g molecular weight of gas at 25 °C and 760 mm of mercury pressure.

Personnel Monitoring. Personnel working in the plant and applying pesticide formulations were monitored for chlorpyrifos exposures by attaching a collection tube to the worker. The collection tube was connected with flexible tubing to a small battery operated pump calibrated to pump 100 mL of air per minute. The pump was placed in the workers breast or back pocket.

Table II. Laboratory Collection and Recovery Experiment for Chlorpyrifos on Durapak–Carbowax 400/Porasil F

Amount Added (ng)	Flow Rate Liters/Min.	Time, Hrs	% Rel. Hum.	% Recovery[a]	Concentration (ppb v/v)
8.3	1.0	0.2	45	88.6	0.04
986	1.0	0.5	45	92.6	2.1
1060	0.5	1	45	94.5	2.4
758	0.5	1	80	98.9	1.7
160	0.5	1	45	97.3	0.43
195	0.5	2	45	100	0.21
188	0.1	16	45	91.4	0.14

DISCUSSION AND RESULTS

The low recoveries of polar compounds of low volatility, particularly the organophosphate pesticides, from the commonly used solid adsorbents promoted the search for a suitable solid adsorbent for air sampling. The chemically bonded packings, developed for gas chromatography, were shown to have excellent collection and recovery properties. These packings, commonly called "brush" packings (*11*) because of the orientation of the organic moiety on the surface of the support, have been reported (*12*) to retain the organic compounds through the dominant mechanism of adsorption on the brush. The high liquid desorption recoveries of the pesticides can be attributed to the deactivated solid support and to the thin layer and low amount of bonded liquid phase.

Initial experiments were performed using a specialized chemically bonded packing which had been prepared using a procedure developed by Nestrick and Stehl (*13*). Additional experiments used the commercially available bonded packing, Durapak–Carbowax 400/Porasil F, and similar collection and recovery properties were observed. Three other available bonded packings were tested on several types of pesticides, Table I. The Durapak–Carbowax 400/Porasil F gave the best recoveries of the commercial sorbents and was used for the in-depth study of chlorpyrifos in air. An additional bonded packing, Durapak–Carbowax 400/Porasil C is available, which contains 7.86% bonded phase compared to 1.41% on the Porasil F (*12*). This packing was not tested because the extraction blank was high and the material would require prior treatment before use.

The experimental data for chlorpyrifos are summarized in Tables II and III. Even under extreme conditions of high humidity, high concentrations, high solvent vapor concentrations and storage periods of 7 days, the recovery was 95

Table III. Collection of Chlorpyrifos on Bonded Durapak-Carbowax 400/Porasil F Storage Study

Storage Time (days)	% Relative Humidity	Amount[a] Collected (ng)	% Recovery	Concentration (ppb v/v)
1	30	888	99.4	2.0
1	30	892	98.5	2.0
3	30	911	95.0	2.1
3	30	872	96.3	2.0
7	30	851	96.1	2.0
7	30	908	98.8	2.1
7	80	784	91.9	1.8

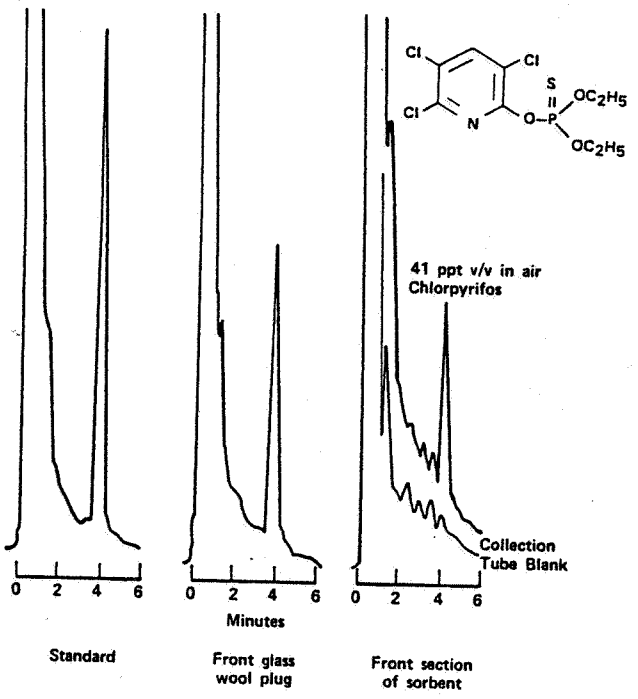

Figure 3. Laboratory experimental run. Collection: 10 min at 1.0 L/min. Gas chromatographic conditions: 0.9 m × 3 mm, glass column, 3% Poly I-110, 200 °C, Carrier gas 35 mL/min argon + 5% methane, electron capture detector attenuation, ×8

± 7% at the 95% confidence level, with only a small amount (less than 5%) found in the back-up section. Recoveries were calculated with the assumption that the amount of pesticide not found on the front glass wool plug had been carried as the vapor into the adsorbent sections. The amount found on the front glass wool plug was usually less than 10% of the total amount injected except for short term samples as shown in Figure 3. Desorption of the bonded packing was rapid and simple. Complete desorption took place in 15 min with gentle shaking.

In a plant study, two areas were surveyed using isooctane impingers and chemically bonded sorbent collection tubes. Two impingers were required to cover the total sampling time in each area while one bonded sorbent tube was used. The two impingers for each area were combined and analyzed. The time weighted average concentration of chlorpyrifos was 2.9 and 9.4 ppb v/v in air compared to the bonded sorbent values of 3.0 and 9.3 ppb, respectively. A few examples of field and plant applications are summarized in Table IV.

The chromatogram in Figure 4 shows the typical sensitivity obtained for field samples using an electron capture detector. This sample was collected for 4 h at 75 mL/min during the spraying of formulations containing chlorpyrifos. The peak represents 10.9 ng in 2 mL of diethyl ether and 40 ppt v/v as the time weighted average concentration in air. The limit of detection was 2 to 4 ng per sample at a signal-to-noise ratio

Table IV. Determination of Chlorpyrifos in Field Samples Collected on Durapak-Carbowax 400/Porasil F

Sample	Vol. (Liters)	Amount Found (μg) Front Section	Back Section	% In Back	Concentration (ppb v/v)
Moved around following a sprayer in a confined area 2.5 hr	30	5.83	0.15	2.5	6.4
Breathing zone personnel monitor spraying four lawns 55 minutes	11	0.272	0.004	1.5	1.7
Breathing zone personnel monitor spraying five buildings	15.2	0.123	0.002	1.6	0.57
Near Plant equipment (under cover)	4.8	8.11	0.14	1.7	122
Control panel	2.7	0.293	0.004	1.4	7.7
100 meters downwind	9.5	– None detected at sensitivity of 0.02 ppb –			

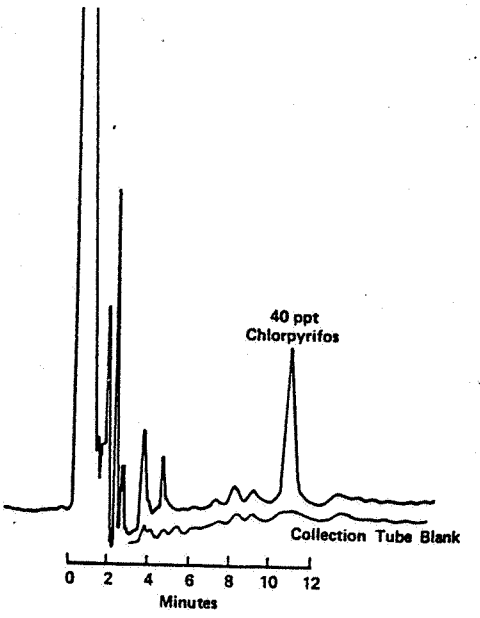

Figure 4. Four-hour breathing zone sample. Gas chromatographic conditions: 1.8 m × 3 mm glass column. 3% SE-30/3% QF-1, carrier gas 35 mL/min argon + 5% methane, electron capture detector attenuation, ×8

of 3:1. No interferences were observed in the field samples even in the presence of high concentrations of solvents present in the formulations.

The technique allowed breathing zone personnel samples for long periods. No other technique for this type of sample has previously been successful for chlorpyrifos. By using collection rates of 1 L/min, short sampling periods may be used to monitor peak exposures during operations which take place for only a few minutes. Another advantage of the bonded packing collection system is its low retention of water and solvent vapors. Solvent vapors, from the formulations, readily pass through the bonded sorbent without increasing breakthrough of the chlorpyrifos. This reduces interferences and will allow solvent vapors to be collected on activated charcoal in a section following the bonded sorbent.

The results for the collection of several other types of pesticides indicate that this technique may be applied to a wide range of compounds. Further development of various bonded packings for the collection of compounds which are difficult to determine in air is under way.

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Selective Separation of Metal Ions by a Chelating Agent-Loaded Anion Exchanger

Kil Sang Lee, Won Lee, and Dai Woon Lee*

Department of Chemistry, Yonsei University, Seoul, Korea

A conventional anion exchanger loaded with sulfonated chelating agents, such as 5-sulfosalicylic (SSA), chromotropic (CTA), and 7-iodo-8-hydroxyquinoline-5-sulfonic (IHQS) acid has been applied to the selective and quantitative separation of Fe(III), Cu(II), Cr(VI), and Pb(II) from aqueous solutions. The optimum conditions were determined with respect to the pH, shaking time, and the effect of anion concentration in the medium. The total absorption capacities of the loaded resins were; 1.28 mmol Fe and 1.07 mmol Cu per gram of SSA-loaded resin, 0.65 mmol Fe and 1.14 mmol Cr per gram of CTA-loaded resin, and 0.73 mmol Cu and 0.70 mmol Pb per gram of IHQS-loaded resin. A few examples of the separation from the synthetic mixtures of the metal ions were examined by the elution method.

The selective and quantitative separation of metal ions from aqueous solutions, which is closely related to water pollution problems, has been extensively investigated with a number of materials. The materials studied are conventional (*1–4*) and chelating ion-exchange resins including inorganic ion exchangers (*5–11*), solvent-impregnated resins (*12*, *13*), controlled pore glass (*14*), foamed plastics (*15–17*), and so on. It is well known that the chelating resins were recognized as being potentially useful for the selective separation of the specific metal ions. On the other hand, the use of chelating resins is limited by difficulties in their synthesis and high cost.

The purpose of the present work is to examine the application of the sulfonated chelating agent-loaded anion exchangers for the selective and quantitative separation of some metal ions from the aqueous solutions. The chelating agents studied here are 5-sulfosalicylic (SSA), chromotropic (CTA), and 7-iodo-8-hydroxyquinoline-5-sulfonic (IHQS) acid which are held strongly by the anion exchanger. The chelating agents loaded into the exchanger react to form colored stable chelates in the resin with the metal ions existing in the solutions.

It might be applicable to separate the specific metal ions and to remove them from the aqueous solutions for industrial use as well as for laboratory works. In this paper, all experiments were carried out with only the synthetic solutions of metal ions. There are two main advantages: The first is that the preparation of the chelating agent-loaded resins is quite simple, and the second is that the absorption of metal ions on the loaded resins is visible.

EXPERIMENTAL

Apparatus and Materials. A Cary 14 Recording Spectrophotometer, Beckman Infrared Spectrophotometer Model IR-12, and Perkin-Elmer Atomic Absorption Spectrophotometer Model 303 with hollow cathode lamps were used in this work.

The chloride form of a strongly basic anion exchanger, Dowex 1-X8 (50–100 mesh) was used for the preparation of the chelating agent-loaded resins. The chelating agents, 5-sulfosalicylic, chromotropic, and 7-iodo-8-hydroxyquinoline-5-sulfonic acid, and all other chemicals used were of analytical reagent grade.

For the elution method, the chelating agent-loaded resins were placed in a borosilicate glass column of 1.0 × 50 cm furnished with Teflon (Du Pont) fittings and porous Teflon bottom. The column was washed thoroughly with deionized water using a Serva Elutionpumpe, and then the resin bed was adjusted to the required height.

Preparation of Chelating Agent-Loaded Resins. The chelating agent-loaded resins were prepared by simply passing the solutions of 0.05 M sodium salt solutions of SSA, CTA, and IHQS, respectively, through the columns (3.0 × 50 cm) packed with about 50 g of purified Dowex 1-X8, chloride form. The loaded resin beds were washed with deionized water to remove the excess reagent and then were dried at 60–70 °C for a day.

Total Capacity of the Loaded Resin. The aqueous solution containing 100 ppm of metal ion, adjusted previously to the optimum pH value, was passed through the column packed with 1 g of the chelating agent-loaded resin. The flow rate was adjusted to 1.0 ± 0.1 mL per minute. The collected effluents were analyzed by atomic absorption spectrophotometry to obtain the chromatogram of frontal analysis from which the total capacity of the loaded resin was calculated. The value of the total capacity was expressed in mmol of metal ion per gram of dry resin.

Procedure for the Absorption of Metal Ions. In the batch method, aliquots (20–25 mL) of aqueous solution containing metal ion (ca. 100 μg) were shaken with weighed amounts of the chelating agent-loaded resin, ranging from 0.5 to 1.0 g, for about 1 h by a mechanical shaker. The amount of each metal ion absorbed by the resin was measured by atomic absorption spectrophotometry by comparing the concentration of equal aliquot portions of the solution before and after absorption.

The elution method was carried out using glass columns (1.0 × 40 cm) packed with a certain amount of the chelating agent-loaded resins.

All experiments were carried out at room temperature.

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