# Use of Capillary Electrophoresis for Detection of Metsulfuron and Chlorsulfuron in Tap Water

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The present paper describes chlorsulfuron and metsulfuron determination in tap water by capillary electrophoresis (CE). A method for metsulfuron and chlorsulfuron concentration by solid-phase extraction is also presented. Metsulfuron and chlorsulfuron were detected by capillary electrophoresis quantitatively to 0.1 ppb and to 0.01 ppb qualitatively. Particular attention was devoted to instrumental reproducibility of the method and to problems linked to matrix interferences during extraction phases from tap water. Finally, correlation studies between bioassay and CE analysis were effected to confirm the validity of the solid-phase extraction procedure.

## INTRODUCTION

Sulfonylureas are a relatively new class of herbicides known for bioactivity at low doses [10–60 g of active ingredient (ai) $ha^{-1}$] and low mammalian toxicity, comparable to the triazine herbicides from which they are derived (Beyer et al., 1987). In 1982 only chlorsulfuron, the progenitor of the family, was commercially available, whereas 16 sulfonylureas are currently on the market for such diverse crops as autumn–winter cereals, rice, maize, rape, cotton, and soybean and for nonfarm use. Sensitivity to sulfonylureas varies among crop species: wheat is about 1000-fold more tolerant to chlorsulfuron than corn and sugar beet (Sweetser et al., 1982). Because soil concentrations of even 0.1 ppb of active ingredient affect the growth of susceptible plants (Palm et al., 1980; Walker and Brown, 1982), it is necessary to restrict the use of chlorsulfuron and metsulfuron, which have persistent residual activity under conditions of high soil pH, low rainfall and temperatures, and poor microbial activity (Walker and Brown, 1983; Smith, 1986; Walker and Welch, 1989; Vicari et al., 1991).

According to European Economic Community (EEC) directives, a pesticide residue in drinking water must not be present at a concentration greater than 0.1 ppb (EEC, 1980). Therefore, there is a need for a rapid, reliable qualitative and quantitative method for analysis of soil and water residues of sulfonylureas not only in field drainage or runoff water and soil but also in tap water. Available methods, including bioassay (Hsiao and Smith, 1983; Gunther et al., 1989; Gomez de Barreda and Lorenzo, 1991; Sunderland et al., 1991), enzyme immunoassay (Kelley et al., 1985; Nord-Christerson and Bergstrom, 1989), liquid chromatography (HPLC) (Zahnow, 1982; 1985; Peter et al., 1989), and gas chromatography (Ahmad, 1987; Ahmad and Crawford, 1990), detecting from 0.1 to 1 ppb in soil and water, have, however, some disadvantages. The major limit of bioassay is that the test sample must contain only one biologically active compound because plant response is aspecific and any residues of other substances will interfere. The advantages of bioassay are the good relationship between its quantitative results and chemical analysis (Peter et al., 1989; Morishita et al., 1985; Smith and Hsiao, 1985; Iwanzick and Egli, 1989) and its ease of use. Immunoassay reduces sample preparation and analysis time (Hall et al., 1990), and its sensitivity ranges from 0.01 to 0.1 ppb (Nord-Christerson and Bergstrom, 1989); it is not influenced by traces of other herbicides (Peter et al., 1989), but it is expensive and not available yet. Gas chromatography is unfeasible because sulfonylureas are nonvolatile, thermally unstable, and polar. The most effective technique is undoubtedly HPLC with a photoconductivity detector. It is important to note, however, that until now only monoresidue analyses were cited in literature (Zahnow, 1982, 1985; Prince and Guinivan, 1988; Peter et al., 1989).

Capillary electrophoresis (CE), albeit widely used in many fields of chemical and biochemical analysis (Kuhr, 1990), had never been applied to pesticide detection, except for glyphosate and its metabolite in animal serum (Tomita et al., 1991). This paper reports on the results of laboratory experiments designed to work out (i) water extraction and concentration procedures for metsulfuron and chlorsulfuron and (ii) CE separation of these two sulfonylureas in tap water at parts per billion concentrations. Finally, the proposed CE procedure was compared with a bioassay method.

## MATERIALS AND METHODS

**Apparatus.** A complete P/ACE 2000 Beckman system was employed for CE analysis. Separations were made with a silica-fused capillary 50 cm long (from injection point to detector), 75 $\mu$m internal diameter (i.d.), using free zone capillary electrophoresis (FZCE) (Gordon et al., 1988). The electrolyte buffer was 20 mM boric acid and 20 mM sodium borate at pH 9.0. The electrophoregrams were obtained by applying a potential difference of 25 kV at 30-$\mu$A intensity. Samples were injected under constant $3.44 \times 10^3$ Pa pressure at the capillary's anode end, and detection at the cathode with a UV detector was at 214 nm. All of the electrophoregrams were made at a thermostat-controlled capillary temperature of 30 ± 0.2 °C. The capillary was washed with a 0.1 M NaOH solution, a 1 M NaOH solution, and with a filtered and bidistilled water for 40, 40, and 60 min, respectively, thereby keeping the standard deviation in retention of consecutive sample runs ($n$ = 10) below 1% and that of the day-to-day retention time accuracies below 8%.

**Reagents.** Reagents for electrolyte buffers and for capillary washing were supplied by Sigma Chemical Co. All solvents, supplied by Bakerbond, were of pesticide grade. Metsulfuron and chlorsulfuron standards were furnished by Lab Service at purities of 99.5% and 99.9%, respectively, while their dry-flowable formulations were supplied by Du Pont Conid S.p.a. and had active ingredient weight percentages of 20% and 75%, respectively.

The sample concentration columns for reversed-phase extraction, consisting of 500 mg of $C_{18}$ (octadecylsilane) resin linked to silica gel with an average 40-$\mu$m particle size, were supplied by Bakerbond.

**Calibration Curves and Sample Preparation.** Two stock solutions at concentrations of 100 ppm were prepared by

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dissolving 10 mg of analytical grade metsulfuron or chlorsulfuron in 100 mL of methanol, from which 50, 100, 200, 300, 400, or 500 μL was taken and brought to 10 mL with methanol to produce standard solutions of 0.5, 1, 2, 3, 4, and 5 ppm.

One milliliter was then taken from each of the two stock solutions and brought to 10 mL with methanol, to yield a 10 ppm solution of metsulfuron or chlorsulfuron; 10 50, 100, 200, 300, 400, or 500 μL was then drawn from this solution and brought to 1 L with bidistilled water to give concentrations of 0.1, 0.5, 1, 2, 3, 4, and 5 ppb for extraction.

Ten milligrams of active ingredient of commercially formulated metsulfuron and chlorsulfuron was dissolved in 1 L of tap water, from which 1-, 5-, 10-, 100-, and 300-μL samples were brought to 1 L with tap water to yield concentrations of 0.01, 0.05, 0.1, 0.5, 1, and 3 ppb. These samples were filtered before concentration through Whatman No. 3 paper to eliminate particulate matter that might later interfere with subsequent concentration procedures. Tap water was drinking water from the municipal water system of Bologna (pH 7.2, hardness 2.5 g/L, residue 0.8 g/L).

The instrumental efficiency was measured by number of theoretical plates ($N$) according to the method of Nishi et al. (1989).

**Sample Extraction and Concentration.** A modified version of Junk and Richard's (1988) method for organic compounds extraction from water was employed, using columns of 500 mg of $C_{18}$ (octadecylsilane). Prior to analysis, the samples were brought to pH 2.5 ± 0.2 with a 0.1 M solution of HCl, and the column was conditioned with 6 mL of ethyl acetate, followed by 2 mL of methanol and 2 mL of HPLC grade water; the solvents were run through by gravity drop. Then 2 mL of HPLC grade water was added and the sample (1 L) aspirated by vacuum pump at a flow rate of 25 mL/min.

The column was subsequently vacuum-dried for 10 min, and, with the vacuum pump off, 0.8 mL of ethyl acetate was eluted by gravity. The concentrated sample was placed in a test tube and dried at room temperature under light nitrogen flow, and the resulting amount was reconstituted with 0.02 mL of methanol for the 0.01 ppb concentration sample and with 0.05 mL of methanol for the sample with the 0.05 ppb concentration. These samples were concentrated 50 000 and 20 000 times, respectively, bringing them to analytical concentrations of 0.5 and 1 ppm.

The 0.1 and 0.5 ppb samples were also reconstituted with 0.1 and 0.5 mL of methanol. They were then concentrated 10 000 and 2000 times, respectively, to an analytical concentration of 1 ppm. Lastly, the 1, 2, 3, 4, and 5 ppb samples were reconstituted with 1 mL of methanol and concentrated 1000 times and brought to respective analytical concentrations of 1, 2, 3, 4, and 5 ppm.

**Bioassay Procedure.** Ten milligrams of each formulated herbicide was suspended in 1000 mL of tap water. One milliliter of this solution was brought to 1000 mL, and 0, 5, 10, 20, 40, 80, or 160 mL of this was then brought to 200 mL as a standard solution for the calibration curve. The herbicide concentrations were 0, 0.25, 0.5, 1, 2, 4, and 8 ppb. For each concentration and each chemical, 35 mL of herbicide solution was applied via subirrigation to plastic pots (5-cm diameter and 4-cm height) each containing 75 g of a dry (oven-dry basis) sandy loam soil: 58% sand, 15% silt, 27% clay, 1.3% organic matter, and pH 6.5.

The surface of the potted soil was sprinkled with 205 ± 5 seeds of *Nasturtium officinale* R. Br., and the plots were placed in a growth chamber with a photoperiod of 16 h (26 °C), 390 μE $m^{-2}$ $s^{-1}$, a dark period of 8 h (18 °C) and 80–90% relative humidity. Each pot was put in a covered transparent plastic container to prevent soil moisture evaporation during germination and to promote uniform plant growth. The pots were uncovered after 5 days and subirrigated with a further 10 mL of water; pots were kept covered in the containers during the dark period until harvest.

The whole plants in each pot were harvested after 12 days, soil was cleaned from roots by low-pressure water jet, and fresh weight was determined. The bioassay fresh weight data, expressed as percentage of control, were then subjected to variance and regression analysis and the means separated by the Student–Newman–Keuls (SNK) multiple-range test. The experiment was a randomized-block design (benches being blocks) with three replicates.

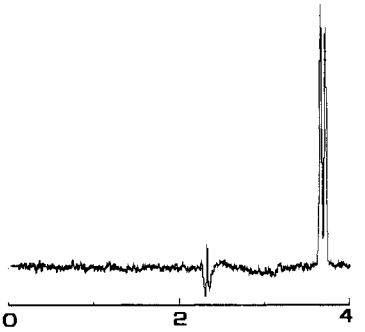


**Figure 1.** Chlorsulfuron (60 pg) and metsulfuron (60 pg) calibration curves electrophoregram. Elution order: methanol, metsulfuron, and chlorsulfuron.

The tap water samples were used after the 1 and 5 ppb chlorsulfuron plus metsulfuron solutions had been run through the column to check the effectiveness of the water extraction method. These samples were brought to pH 7.5 with 1 M NaOH solution before the bioassay.

The 1-L samples containing 0.01, 0.05, and 0.5 ppb of chlorsulfuron were respectively reconstituted, after extraction as described above, with 35, 70, and 100 mL of tap water and then concentrated 33, 14, and 10 times, respectively.

Each solution (35 mL) was supplied via subirrigation to potted soil and bioassayed with *N. officinale* as described above. Each sample was replicated three times except for the 0.01 and 0.05 ppb concentrated samples, which could only be assayed once and twice, respectively.

## RESULTS AND DISCUSSION

**Calibration Curves and Sample Preparation.** Chlorsulfuron and metsulfuron are compounds of very similar structure (Beyer et al., 1987). As with all of the sulfonylureas, the molecule consists of three distinct parts: an aryl group, the sulfonylurea bridge, and a nitrogen-containing heterocycle with different substituents in the ortho position in the phenyl or aryl group. Chlorsulfuron and metsulfuron are weak acids with dissociation constants ($pK_a$) of 3.3 and 3.6, respectively.

Given that both compounds can be ionized, they were separated in an electrical field (Figure 1). The resulting electrophoregram shows good separation: an efficiency of 147 000 theoretical plates for metsulfuron and of 145 000 for chlorsulfuron. Column efficiency was 292 000 N/m; the asymmetry of the metsulfuron peak is 0.94 and chlorsulfuron's 0.92.

The calibration curves for quantitative analysis of metsulfuron and chlorsulfuron show a linear instrumental response of the capillary-injected ai in the 30–300-pg range, which matches the 10-s (=60 nL) injection of standard with concentration in the 0.5–5 ppm range. The chlorsulfuron regression equation is $y = (7.96 \times 10^{-3}) + (5.34 \times 10^{-4})x$ ($r^2 = 0.991$) and metsulfuron's $y = (7.93 \times 10^{-3}) + (5.29 \times 10^{-4})x$ ($r^2 = 0.994$), where $y$ is the peak area and $x$ is the herbicide amount in picograms. The detection limit at 214 nm for CE with a UV detector was 10 pg. Since response at that amount was nonlinear, the instrument's detection threshold was set at 30 pg. The values of mean migration time and of their coefficients of variation (CV) for the runs made on different days indicate good reproducibility (Table I). The area mean values of the peaks corresponding to injection of 60 pg at different days and the respective CV (Table I) suggest good quantitative accuracy for CE.

**Sample Extraction and Concentration.** The method tested for the extraction and concentration of the two

**Table I. Retention Times and Peak Areas of Standard Samples**

| | chlorsulfuron | metsulfuron |
|---|---|---|
| no. of replicates | 11 | 11 |
| mean migration time, min | 3.71 ± 0.08 | 3.64 ± 0.07 |
| CV, % | 2.13 | 1.93 |
| mean area[a] | 0.041 ± 0.002 | 0.039 ± 0.002 |
| CV, % | 4.87 | 5.35 |

[a] Injection of 60 pg.

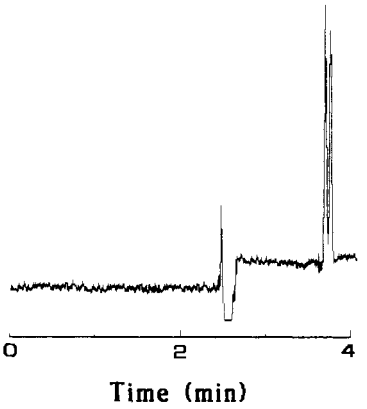


**Figure 2.** Electrophoregrams of metsulfuron and chlorsulfuron (0.1 ppb) in bidistilled water: 10000-fold concentration. Elution order was as in Figure 1.

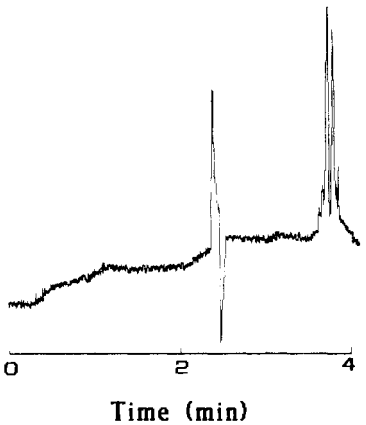


**Figure 3.** Electrophoregrams of metsulfuron and chlorsulfuron (0.1 ppb) in tap water: 10000-fold concentration. Elution order was as in Figure 1.

**Table II. Recovery and Mean Migration Time of Concentrated Samples**

| | chlorsulfuron | metsulfuron |
|---|---|---|
| no. replicates | 11 | 11 |
| mean recovery,[a] % | 92.25 ± 9.52 | 96.32 ± 15.58 |
| CV, % | 8.79 | 15.01 |
| mean migration time,[a] min | 3.69 ± 0.07 | 3.63 ± 0.07 |
| CV, % | 1.99 | 1.94 |

[a] Data are from tap water and distilled water tests (range 0.1–5 ppb).

sulfonylureas studied proved to be valuable. The electrophoregrams of the extracts from the 0.1 ppb samples in distilled water and in tap water (Figures 2 and 3) evidence the effectiveness of multiresidue analysis. The average overall recovery, in the 0.1–5 ppb range, was 96.32 ± 15.58% for metsulfuron and 92.25 ± 9.52% for chlorsulfuron (Table II). The CV of migration time was under 2% for both, confirming instrument reproducibility and showing that the sample concentration technique does not alter physicochemical traits of the two sulfonylureas and is a valid method for multiresidue analysis in CE.

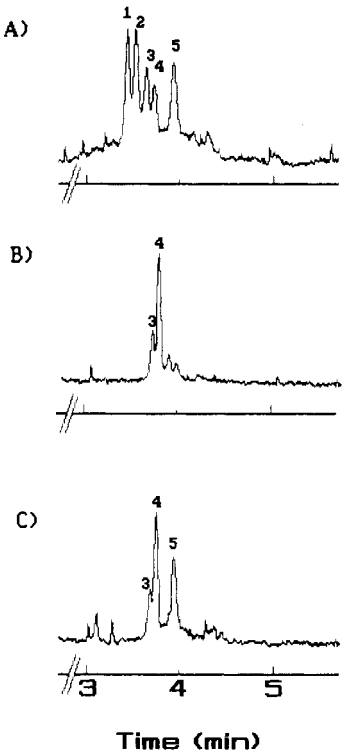


**Figure 4.** (A) Electrophoregram of chlorsulfuron and metsulfuron (0.01 ppb) in tap water: 50000-fold concentration. (B) Electrophoregram of bidistilled water: 50000-fold concentration. (C) Electrophoregram of tap water: 50000-fold concentration. Elution order: (1) metsulfuron; (2) chlorsulfuron; (3, 4) compounds released by $C_{18}$ column; (5) tap water impurity.

The mean recovery of the two sulfonylureas was slightly higher in bidistilled water samples (102.3%) than in tap water (90%), but the difference was not significant. The calibration curve was prepared with analytical grade standards, and the tap water samples were prepared with commercial products. The mean recovery of the two sulfonylureas, given all the tests made with samples from 0.1 to 5 ppb, was 94.28%.

In the tap water tests, although metsulfuron and chlorsulfuron at 0.05 (data not shown) and 0.01 ppb concentrations were detected (Figure 4A), it was impossible to determine the dose and it was necessary to concentrate the samples up to 50 000 times before a fit was achieved on the calibration curve. Concentration procedures probably concentrated other compounds that led to a rise of the baseline and the poorer separation of the two active ingredients, thereby making dose determination impossible. The origin of these other compounds was checked by applying the concentration method to a control sample of bidistilled and filtered water, to assure high analytical grade, and to a tap water sample. Both samples were concentrated 50 000 times and analyzed. Comparison among electrophoregrams (Figure 4) shows that peaks 3 and 4 with retention times equal to 3.72 and 3.75 min, respectively, are probably products released by the $C_{18}$ column used for the sample concentration. In fact, the same peaks are present even in the bidistilled water sample (Figure 4B). Peak 5 (Figure 4A), with retention time of 3.92 min, is a matrix interference. In fact, the electrophoregram of the concentrated tap water sample presents, besides peaks 3 and 4, even peak 5. These data are in agreement with the results of Coquart and Hennion (1991). These authors reported that during preconcentration of natural water by a solid-phase extraction (SPE) procedure with a $C_{18}$ column many interfering compounds may also be concentrated. In HPLC such matrix interferences are

**Table III. Whole Plant Fresh Weight of *N. officinale* R. Br. Grown in Pots Subirrigated with Herbicide Solutions**

| herbicide rate in water, ppb | fresh weight[a] chlorsulfuron | fresh weight[a] metsulfuron |
|---|---|---|
| 0 | 100.0a[b] | 100.0a[b] |
| 0.25 | 72.5b | 59.5b |
| 0.5 | 57.5c | 43.5c |
| 1 | 45.9d | 35.5d |
| 2 | 37.8e | 25.2e |
| 4 | 27.9f | 22.4f |
| 8 | 22.8g | 18.7g |
| CV, % | 7.22 | 6.18 |

regression equations (excluding control)
chlorsulfuron: $y = 46.3 - 36.1(\log x) + 11.1(\log x^2)$ $r^2 = 0.998$
metsulfuron: $y = 34.2 - 31.2(\log x) + 16.2(\log x^2)$ $r^2 = 0.994$

[a] Percent of the fresh weight of plants grown with untreated water. Fresh weight of whole plants of untreated control: 2.33 ± 0.09 g. [b] Means within each column followed by the same letter are not significantly different at $P < 0.05$ according to SNK multiple-range test.

**Table IV. Effect of Different Sample Treatments on Whole Plant Fresh Weight of *N. officinale* R. Br. To Confirm Validity of Solid-Phase Extraction Procedure**

| | fresh weight[a] |
|---|---|
| untreated | 100.0a[b] |
| 5 ppb of chlorsulfuron plus 5 ppb of metsulfuron solution after extraction | 108.2a |
| 1 ppb of chlorsulfuron plus 1 ppb of metsulfuron solution after extraction | 105.6a |
| 0.5 ppb of chlorsulfuron solution, concentrated 10 times | 18.8b |
| 0.05 ppb of chlorsulfuron solution, concentrated 14 times | 21.7[c] |
| 0.01 ppb of chlorsulfuron solution, concentrated 33 times | 33.5[c] |

[a] Percent of the fresh weight of plants grown with untreated water. Fresh weight of whole plants of untreated control: 2.41 ± 0.12 g. [b] Means within each column followed by the same letter are not significantly different at $P < 0.05$ according to SNK multiple-range test. [c] Data were not included in the analysis of variance as the number of replicates was not sufficient.

detected with samples at a concentration of 0.1 ppb; in CE the matrix interferences are detected for samples at a concentration of 0.01 ppb.

**Bioassay.** The bioassay findings shown in Table III reveal typical dose–response relationships for chlorsulfuron and metsulfuron. While metsulfuron was more phytotoxic than chlorsulfuron to *N. officinale* R. Br., both herbicides affected the fresh weight of the test species at water concentrations of 0.25 ppb and the effect increased up to 8 ppb. The equation that best related chlorsulfuron or metsulfuron concentration (expressed as log ppb) to plant fresh weight as percentage of control was a second-order polynomial (Table III), with a very high determination coefficient for both herbicides. It is thus possible to correlate the results of the bioassay standard to water samples having unknown concentrations of chlorsulfuron or metsulfuron.

After the extraction by column, samples at 1 and 5 ppb showed no toxicity to the test plant. This confirmed the validity of the SPE procedure (Table IV). The estimated recovery values (94.28% average of both herbicides) are probably underestimated. In fact, if only 5% of herbicide was present in the water after extraction, this would have affected the plant growth. Thus, it is reasonable to presume that 100% of the sulfonylureas is retained on the SPE column. It is also likely that the 0.8 mL of ethyl acetate does not elute all of the sulfonylureas from the column, and indeed a small fraction of the sulfonylureas may be irreversibily retained on the column.

The bioassay findings in Table IV for the water samples containing 0.01, 0.05, and 0.5 ppb of chlorsulfuron, respectively concentrated 33, 14, and 10 times, suggest that the product can even be detected at the lowest concentration. It should, however, be pointed out that, once these doses are concentrated, their quantitative determination is overestimated. Thus, in reference to the standard curve in Table III, the phytotoxicity evidenced by the respective 0.01, 0.05, and 0.5 ppb samples matches concentrations that are markedly higher than the 0.33, 0.70, and 5 ppb postconcentration theoretical ones. The procedure of SPE concentration permitted a lower qualitative detection limit of the bioassay at the parts per trillion level; however, the procedure itself did not permit, at those levels, an adequate quantitative detection.

**Conclusion.** The results reported herein demonstrated that reversed-phase extraction with $C_{18}$ columns of metsulfuron and chlorsulfuron from tap water is simple, effective, and suitable for subsequent residue analysis by CE. It demonstrated also to be rapid, and 12–24 samples can be run in 2–3 h. In addition, CE analysis enabled multiresidue detection and quantification of metsulfuron and chlorsulfuron in tap water at 0.1 ppb concentrations; the active ingredient of the two compounds was also detected, though not quantitatively analyzed, at concentrations as low as 0.01 ppb. The recovery values of 96.32% for metsulfuron and 92.25% for chlorsulfuron indicated satisfactory accuracy of the detection. Bioassay confirmed the validity of the SPE procedure and aspecifically quantified the two herbicides in water at concentrations between 0.25 and 8 ppb. At higher concentrations samples must be diluted to fit the calibration curve; at rates below 0.25 ppb the sample must be concentrated, although quantitative detection was not adequate.

However, further studies are necessary to make the presented method feasible to drainage and runoff water analysis, to soil samples, or, more generally, to samples containing different organic compounds at concentrations higher than that of the sulfonylureas.

ACKNOWLEDGMENT

This work was financed by the Ministry of Agriculture and Forestry within the Lotta biologica ed integrata—Piante infestanti project. We thank Prof. Guido Galletti, Prof. Robert L. Zimdahl, and Dr. Alessandra Bonetti for useful suggestions.

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Received for review October 14, 1992. Accepted January 22, 1993.