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Method article

# Extension of an extraction method for the determination of 305 organic compounds in clay-loam soil to soils of different characteristics

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A B S T R A C T

Soil is one of the environmental compartments most affected by pollution. From this medium, the organic compounds can be emitted to the groundwater, the atmosphere, or the biota. Thus, having adequate methods of analysis of organic pollutants in this matrix is essential. However, the soil is a very complex matrix whose organic and inorganic components can determine the degree to which they are retained. Therefore, the methods must account for the various soil characteristics. In this study, the performance of an extraction method that had been already validated in clay loam soils for more than 300 organic compounds of very different nature including pesticides, PhACs, ARs, and POPs has been evaluated in four additional representative soil types of the agricultural land of the Canary archipelago: sandy loam, sandy clay, clay and loamy sand. For this purpose, recovery experiments have been performed at a single concentration (50 ng $g^{-1}$) in each soil type. When there is a significant difference according to the criteria applied for a given compound, a factor has been calculated to correct the difference in performance in each soil type.

- These results allowed to broaden the range of soils that can be analyzed with the proposed methodology.
- In the worst case, which is the loamy sand soil, the original methodology allows the analysis of 180 organic contaminants with adequate recoveries. For analytes outside the acceptable range in this soil and the other soil type analyzed, correction factors are proposed.

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## Background

Soil is a well-known reservoir of organic pollutants from where they can be released to other environmental compartments such as groundwater, atmosphere, and biota [5,6]. Compounds of very different nature and usage can reach the soil through pest control in agriculture -i.e pesticides, or the sewage system -i.e anticoagulant rodenticides (ARs). In addition, pharmaceutical active compounds (PhACs), which are considered compounds of emerging concern (CECs), can reach soil through irrigation with reclaimed wastewater or the use of manure and sewage sludge as compost. Furthermore, although banned or restricted for decades, persistent organic pollutants (POPs) can end up in the soil through their historical use in agriculture -i.e organochlorine pesticides (OCPs), and industry -i.e.polybrominated diphenyl ethers (PBDEs) and polychlorinated Biphenyls (PCBs), and volatilization and deposition processes -i.e. polycyclic aromatic hydrocarbons (PAHs). Given the diversity of compounds that can end up in the soil, there is a need for methods capable of determining and quantifying them to facilitate soil surveillance in line with monitoring programs such as coordinated multi-annual plan of the European Union [7].

For this purpose, a simple and economic method was optimized and validated to extract and analyze organic compounds of diverse nature in agricultural soils that could be used in routine analysis [1–4]. This method is a modification of the QuEChERS (quick, easy, cheap, effective, rugged and safe) initially introduced for the extraction of pesticides in fruit and vegetable by Anastassiades et al. [8], which has been subjected to several modification to improve the overall technique performance in other matrices and analytes [9–15]. The compounds extracted and analyzed in soil with the QuEChERS-based method constitute a large group of 305 organic compounds. These analytes are classified into 213 pesticides of current and recent use, 43 CECs 7 of which are ARs and 36 are PhACs, and 49 POPs, between PBDEs, PCBs, OCPs and PAHs. The complete list of compounds together with their group, and the technique by which they are analyzed is presented in Table 2.

This method was optimized and validated for a representative agricultural soil of the Canary Islands that can be classified as clay loam. However, soil is a heterogeneous and highly complex matrix whose components may influence the degree of adsorption of the organic compounds. Therefore, not only the chemical properties of the compound may affect the mobility and availability of these compounds in the soil but also the soil characteristics, such as pH, moisture, texture and organic matter content [16]. Generally, they are mostly absorbed in soils rich in organic matter or clay [17].

Consequently, it is necessary to consider soil physicochemical properties to achieve more reliable quantifications with the analytical methods.

This study aims to extend the use of the QuEChERS-based method for the extraction and analysis of the above mentioned 305 organic compounds of different nature and origin to other types of soil frequently used in agriculture.

## Chemicals and reagents

Certified standards stock mix solutions of the pesticides included in the coordinated multi-annual plan of the EU for the investigation of residues in food of vegetable or animal origin during the years 2020, 2021 and 2022 [7] were purchased from CPA Chem (Stara Zagora, Bulgaria) in 10 mixes of compatible pesticides at 10 $\mu$g mL$^{-1}$, each in acetonitrile (ACN). Standard stock solutions of the selected POPs were also supplied by CPA Chem in 5 mixes, each of them at 100 $\mu$g mL$^{-1}$: one for the OCPs (in acetone), one for the PAHs (in dichloromethane), one for the PBDEs (in iso-octane) and two for PCBs (in iso-octane). Individual certified standards of ARs, PhACs and additional pesticides (purity 95.19% to 99.9%) were acquired from Dr. Ehrenstorfer (Augsburg, Germany), Sigma-Aldrich (Augsburg, Germany) and European Pharmacopoeia Reference Standards (Strasbourg, France). Atrazine-d5, Carbendazim-d3, Chlorpyrifos-d10, Coumachlor, Cyromazine-d4, Diazinon-d10, Linuron-d3, Pirimicarb-d6 and PCB 200 (Dr. Ehrenstorfer and Sigma-Aldrich, 99.3-99.9% purity) were used as procedural internal standards (P-IS). These internal standards were added to the samples at the beginning of the procedure to account for various sources of errors throughout all stages in the method [18].

LC-MS grade methanol (MeOH), acetonitrile (ACN), acetone (Ac) and formic acid (FA, HCOOH) were obtained from Honeywell (Morristown, NJ). Ammonium acetate ($NH_4CH_3CO_2$) was purchased from Fisher Scientific (Loughborough, UK). AOAC method QuEChERS salts [19] (6 g of $MgSO_4$ and 1.5 g of $CH_3COONa$) were acquired in commercial premixes from Agilent Technologies (Palo Alto, USA). The ultrapure water was produced in the laboratory using a Gradient A10 Milli-Q System (Millipore, Bedfore, MA, USA).

## Standard solutions and calibration curves

Individual stock standard solutions at a concentration of 1000 µg mL$^{-1}$ were prepared for PhACs, ARs, P-IS and additional pesticides in MeOH for the first group and ACN for the last three. From these, mixed stock solutions were prepared in ACN or MeOH at 10 $\mu$g mL$^{-1}$ for each group. Then, working solutions of PhACs, ARs and P-IS were prepared at 1 µg mL$^{-1}$ in those solvents. A working solution containing all the pesticides at a final concentration of 0.833 µg mL$^{-1}$ was prepared by mixing the ten parts of the European Commission commercial mix and the additional in-house solution containing the rest of the pesticides (10 $\mu$g mL$^{-1}$/each). For the POPs, an intermediate solution containing all the analytes at a final concentration of 20 $\mu$g mL$^{-1}$/each was prepared by mixing the five parts of the commercial mix. Then, a working mix solution was prepared at 1 µg mL$^{-1}$ in Ac.

Matrix-matched calibration curves ranging from 0.195 to 100 ng g$^{-1}$ were prepared with the standard working mix solutions of each compound group in either soil extract for GC-MS/MS or in in a mixture of this extract with ultrapure water (1:1, v/v) for LC-MS/MS.

All standards, working mix solutions and matrix-matched calibrators were stored in glass amber vials at -20 °C and checked periodically for stability.

## Sample selection and pretreatment

The method was originally developed, optimized and validated in an agricultural soil which did not present any of the analytes of interest. This sample, located in the midlands of the Canary Islands, can be considered representative of the most fertile soil of the archipelago and is classified as clay loam according to its characteristics (see soil A in Table 1). To test whether this method could be transferred to other types of cropland on the islands, samples were taken from farms located in different areas of the archipelago that may represent other areas typically used for cultivation. In each sampling plot, a

**Table 1**
Physicochemical properties of the soil samples.

| ID | Classification | pH | CE ($\mu$S cm$^{-1}$) | OOC (%) | Coarse sand (%) | Fine Sand (%) | Thick slit (%) | Fine slit (%) | Clay (%) | Moisture (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil A | Clay loam | 4.88 | 209 | 2.19 | 11.48 | 19.39 | 11.27 | 28.33 | 29.53 | 6% |
| Soil B | Sandy loam | 7.16 | 246 | 6.51 | 52.83 | 10.88 | 7.71 | 19.39 | 9.20 | 6% |
| Soil C | Sandy clay | 8.41 | 1072 | 0.49 | 21.96 | 26.52 | 2.70 | 6.82 | 42.99 | 6% |
| Soil D | Clay | 9.00 | 228 | 0.46 | 18.42 | 11.11 | 0.12 | 0.29 | 70.06 | 8% |
| Soil E | Loamy sand | 9.53 | 328 | 0.29 | 54.92 | 27.41 | 2.49 | 6.27 | 8.91 | 4% |

CE: Conductivity; OOC: Oxidizable Organic Carbon.

composite sample was prepared from subsamples collected in depths between 20 and 30 cm. Then, the soil was homogenized, air-dried at room temperature and sieved (2 mm mesh). Once analyzed, it was observed that these soils could be classified into 5 types according to their characteristics, which have been designated with a letter from A to E.

## Soil characterization

The physicochemical properties of each soil type (texture, oxidizable organic carbon, moisture, conductivity and pH) are shown in Table 1 together with their edaphology classification. The electrical conductivity and pH were measured with suitable electrodes in soil-water suspensions (1:5, w/v). Moisture was calculated as the difference between the air-dried soil weight and the weight after 24 h in an oven at 105°C. Particle distribution was obtained using the hydrometer method [20]. The oxidizable organic carbon to calculate the organic matter content was determined according to the spectrophotometric method in which the absorbance reading is compared with a curve of sucrose solutions with increasing carbon concentration. Once these properties were determined, the soils were classified using the texture diagram of the United States Department of Agriculture (USDA) [21].

## Modified QuEChERS method

First, 10 $\pm$ 0.05 g of dried and sieved soil were weighed into a 50 mL centrifuge tube. Before starting the extraction process, the appropriated volume of the standard mix solutions to achieve the desired concentration (50 ng g$^{-1}$) were added to the recovery samples. Likewise, all samples and blanks were spiked with 50 µL of the P-IS solution, thoroughly shaken, and left to stand for an hour. Then, 10 mL of ACN-2.5%FA were added to each tube and vigorously shaken for 1 min. After that, 6 g of $MgSO_4$ and 1.5 g of $CH_3COONa$ were incorporated to the tubes, energetically shaken for another minute, and sonicated in an ultrasonic bath (50/60 Hz, 120 V VWR, Radnor, Pennsylvania, USA) for 15 min to ensure aggregate breakdown. After that, samples were placed in a rotatory shaker (Ovan, Barcelona, Spain) for 25 min and then centrifuged for 10 min at 4200 rpm (3175.16 $\times$ g) (5804 R, Eppendorf, Hamburg, Germany). Finally, the supernatant was filtered through 0.20 µm Chromafil® PET filters (Macherey-Nagel, Düren, Germany) and either directly analyzed in GC-MS/MS or dissolved in ultrapure water (1:1, v/v) and analyzed in LC-MS/MS.

## Instrumentation

The LC-MSMS analysis was performed using a 1290 Infinity II LC System coupled to a Triple Quad 6460 mass spectrometer (Agilent Technologies, Palo Alto, CA, USA). A Poroshell 120 EC-C18 column (2.1 $\times$ 100 mm, 2.7 µm; Agilent Technologies) equipped with a guard pre-filter with a 0.3 µm SS frit and a pre-column (2.1 $\times$ 5 mm, 1.8 µm; Agilent Technologies) at 50 °C was used for the chromatographic separation. The mobile phases were 2 mM ammonium acetate 0.1% FA in ultrapure water (A) and 2 mM ammonium acetate in MeOH (B). A binary gradient using mobile phases A and B was programmed as follows: 5% B - 0.5 min; 5% B - 1 min; 40% B - 2.5 min; 85% B - 8 min; 100% B - 10 to 14 min; 5% B - 14.01 min. The flow rate was set at 0.4 mL min$^{-1}$, the volume injected was 5 $\mu$L and the total run time was 18 min. MS/MS analyses were performed using the Agilent Jet Stream Electrospray Ionization Source (AJS-ESI), in both positive and negative ionization mode, with dynamic

multiple reaction monitoring (dMRM). The nitrogen supplied by Zefiro 40 nitrogen generator (F-DGSi, Evry, France) was used as desolvation and drying gas. Nitrogen 6.0 (99.9999% purity, Linde, Dublin, Ireland) was used as collision gas. The sheath gas was set at 12 L $min^{-1}$ at 330 °C. The desolvation and nebulizing gas temperature was 190 °C and the flow rate was 11 L $min^{-1}$ with a pressure of 26 psi. The capillary voltages were set at 3900 and 2600 V in positive and negative ionization mode, respectively. The cycle time was 700 ms and dwell time 3-83 ms.

The GC-MS/MS analysis was conducted with a GC System 7890B equipped with a 7693 Autosampler and Triple Quad 7010 mass spectrometer (Agilent Technologies, Palo Alto, USA). The chromatographic separations were performed using two fused silica ultra-inert capillary columns Agilent J&WHP-5MS (Crosslinked 5% phenyl-methyl-polysiloxane, Agilent Technologies) 15 m length, 0.25 mm i.d., and 0.25 µm film thickness of 0.25 µm each connected in series by a Purged Ultimate Union (PUU; Agilent Technologies). This configuration allowed the use of the back-flushing technique. Helium (99.999% purity, Linde, Dublin, Ireland) was used as the carrier gas and the flow was adjusted by the retention time lock feature using chlorpyrifos methyl as a reference (retention time = 9.143 min). The column temperature was maintained at 80 °C for 1.8 min, increased to 170 °C at a rate of 40 °C $min^{-1}$, then increased to 310 °C at a rate of 10 °C $min^{-1}$ and held for 3 min. The injection volume was 1.5 µL in splitless mode using a 4 mm Ultra Inert Liner with glass wool (Agilent Technologies) and it was set at 250 °C. Each chromatographic analysis lasted 20.75 min. Post-run backflush was set at -5.8 mL $min^{-1}$ and 315 °C for 5 min. MS/MS analyses were performed using electron impact (EI) ionization source in multiple reaction monitoring (MRM) mode, using 24-time segments. The EI source temperature was set at 280 °C. Nitrogen 6.0 (99,9999% purity, Linde, Dublin, Ireland) was used as the collision gas at a flow of 1.5 mL $min^{-1}$. The transfer line temperature was 280 °C. A solvent delay of 3.7 min was left. The cycle time was in the range of 52-334 ms and the dwell time was between 15-40 ms.

Data analysis was performed using Agilent software MassHunter Quantitative Analysis (for QQQ) version B.07.01 and MassHunter Qualitative Analysis version B.07.00 for both GC-MS/MS and LC-MS/MS.

## Method performance study

### *Recovery studies*

The performance of the method in soils B, C, D, and E in comparison with soil A, the claim loam soil used in the previous validation of the method, was assessed through recovery experiments in quintuplicate at a single concentration of 50 ng $g^{-1}$. This concentration was selected as it is typically fixed as the limit of quantification (LOQ) for residues in soils [18]. Moreover, all the compounds are efficiently extracted at this concentration in clay loam soil, and actually it is the LOQ of two of the analytes: naphthalene and imipenem. In addition, extractions without the addition of analytes (blanks) were performed for each soil type in duplicates.

The mean and the standard deviation of the recoveries in each type of soil (A-E) are summarized in Table 2 (this information is also represented in Figure 1, Panels A-L, in the supplementary material). The range where recoveries are considered acceptable is 70-120% and a Relative Standard Deviation (%RSD) below 20% according to the guidelines followed in the validation [18,22].

### *Correction factors*

From the data obtained in the recovery experiments, the difference in the performance of the compounds in each type of soil was calculated. It was calculated by taking soil A as a reference. Thus, it is the quotient of the recoveries in soils B, C, D, and E by the recoveries in soil A. In those cases where this factor was within the range of 0.8 and 1.2, it has been considered that there is no marked difference between the two types of soil for a given compound, and, therefore, it would not be necessary to apply any correction to the results obtained in the respective soil. The correction factors are intended to correct the concentrations obtained with the proposed method for each analyte in soils of types B to E and can be consulted in Table 3.

**Table 2**
Percentage recoveries and relative standard deviation of the 305 analytes in each soil type at the stablished concentration together with the compound number identification, group, and technique of analysis. This information is represented in figure 1 of the supplementary material.

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 1 | 4,4′-Dichlorobenzophenone | Pesticides, OCPs | GC | 164.0 | 12.4 | 138.7 | 27.3 | 87.0 | 12.2 | 95.3 | 12.3 | 126.8 | 18.0 |
| 2 | 4,4′-Dicofol | Pesticides, OCPs | GC | 52.8 | 14.6 | 37.7 | 17.0 | 32.0 | 10.3 | 33.2 | 15.8 | 14.9 | 13.8 |
| 3 | Abamectine | Pesticides | LC | 67.1 | 8.0 | 31.4 | 9.9 | 60.6 | 13.5 | 58.8 | 0.8 | 57.1 | 5.9 |
| 4 | Acephate | Pesticides | LC | 61.3 | 3.2 | 57.3 | 1.2 | 52.8 | 1.3 | 39.8 | 1.5 | 63.8 | 1.2 |
| 5 | Acetamiprid | Pesticides | LC | 82.3 | 2.7 | 73.7 | 1.9 | 75.6 | 1.0 | 73.9 | 1.1 | 77.6 | 1.5 |
| 6 | Acrinathrin | Pesticides | LC | 83.6 | 8.0 | 39.5 | 25.2 | 76.3 | 6.4 | 77.8 | 5.9 | 72.9 | 2.9 |
| 7 | Aldicarb | Pesticides | LC | 83.7 | 3.8 | 71.6 | 1.4 | 76.9 | 1.2 | 80.0 | 0.6 | 77.6 | 1.3 |
| 8 | Aldicarb sulfone | Pesticides | LC | 85.3 | 2.8 | 77.9 | 2.6 | 78.1 | 1.0 | 77.9 | 2.4 | 75.5 | 1.7 |
| 9 | Atrazine | Pesticides | LC | 86.9 | 3.8 | 62.9 | 2.4 | 82.3 | 2.1 | 92.1 | 0.7 | 84.4 | 0.9 |
| 10 | Azinphos methyl | Pesticides | LC | 84.2 | 1.6 | 73.0 | 2.6 | 75.4 | 2.1 | 79.2 | 2.0 | 75.7 | 2.4 |
| 11 | Azoxystrobin | Pesticides | LC | 85.9 | 3.2 | 76.6 | 2.0 | 75.3 | 3.4 | 77.0 | 1.2 | 71.3 | 2.1 |
| 12 | Benalaxyl | Pesticides | LC | 87.1 | 2.9 | 71.7 | 4.3 | 85.5 | 1.6 | 88.5 | 1.1 | 84.7 | 1.3 |
| 13 | Bendiocarb | Pesticides | LC | 84.3 | 4.3 | 74.0 | 1.2 | 81.4 | 1.0 | 82.3 | 1.2 | 79.5 | 0.8 |
| 14 | Bifenthrin | Pesticides | GC | 129.6 | 11.6 | 112.7 | 21.3 | 80.4 | 7.8 | 86.2 | 5.8 | 74.4 | 9.9 |
| 15 | Bitertanol | Pesticides | LC | 79.5 | 1.9 | 77.4 | 3.0 | 70.4 | 4.1 | 67.8 | 2.8 | 62.5 | 3.9 |
| 16 | Boscalid | Pesticides | GC | 119.6 | 11.7 | 197.3 | 23.2 | 86.2 | 8.1 | 81.9 | 15.5 | 71.6 | 8.0 |
| 17 | Bromopropylate | Pesticides | GC | 128.1 | 15.8 | 113.6 | 15.2 | 90.4 | 5.7 | 93.3 | 4.3 | 83.8 | 9.6 |
| 18 | Bromuconazole | Pesticides | LC | 85.3 | 5.6 | 68.5 | 3.0 | 71.9 | 4.1 | 74.7 | 0.8 | 74.2 | 4.9 |
| 19 | Bupirimate | Pesticides | LC | 74.7 | 8.9 | 58.2 | 2.3 | 75.7 | 2.0 | 73.0 | 2.0 | 77.4 | 2.0 |
| 20 | Cadusafos | Pesticides | LC | 88.9 | 3.1 | 53.5 | 2.5 | 80.0 | 10.9 | 90.5 | 1.9 | 85.7 | 1.7 |
| 21 | Carbaryl | Pesticides | LC | 83.6 | 2.8 | 69.9 | 1.3 | 76.6 | 1.6 | 80.0 | 0.9 | 76.7 | 1.6 |
| 22 | Carbofuran | Pesticides | LC | 97.5 | 6.0 | 74.1 | 1.1 | 89.4 | 1.2 | 94.6 | 1.0 | 72.4 | 2.0 |
| 23 | Carbofuran-3-hydroxy | Pesticides | LC | 84.1 | 2.1 | 75.2 | 2.1 | 78.1 | 0.9 | 79.3 | 2.0 | 77.5 | 1.1 |
| 24 | Chlorantraniliprole | Pesticides | LC | 78.5 | 4.0 | 78.9 | 3.1 | 68.9 | 3.9 | 67.6 | 1.6 | 64.1 | 3.9 |
| 25 | Chlorfenapyr | Pesticides | GC | 111.0 | 4.4 | 96.1 | 29.3 | 76.9 | 13.1 | 73.1 | 13.4 | 57.2 | 3.8 |
| 26 | Chlorobenzilate | Pesticides | GC | 130.6 | 12.0 | 123.1 | 28.6 | 102.5 | 13.2 | 102.0 | 17.3 | 81.7 | 4.2 |
| 27 | Chlorpropham | Pesticides | GC | 152.1 | 19.0 | 143.8 | 31.6 | 106.4 | 16.0 | 100.7 | 9.9 | 96.5 | 8.7 |
| 28 | Chlorpyrifos | Pesticides | GC | 128.9 | 10.1 | 103.5 | 34.8 | 89.2 | 14.4 | 88.8 | 8.8 | 71.6 | 10.9 |
| 29 | Chlorpyrifos methyl | Pesticides | GC | 104.3 | 5.8 | 95.0 | 20.7 | 71.3 | 8.3 | 71.4 | 15.9 | 49.3 | 5.2 |
| 30 | Chlorthal dimethyl | Pesticides | GC | 117.8 | 10.5 | 102.3 | 20.0 | 86.1 | 15.6 | 92.6 | 11.2 | 80.3 | 10.2 |
| 31 | Clofentezine | Pesticides | LC | 90.4 | 3.6 | 54.1 | 3.0 | 87.1 | 2.2 | 96.7 | 1.5 | 91.0 | 1.6 |
| 32 | Clothianidin | Pesticides | LC | 77.5 | 6.0 | 64.5 | 2.9 | 69.8 | 2.5 | 69.9 | 1.2 | 69.2 | 2.9 |
| 33 | Coumachlor | Pesticides | LC | 88.4 | 1.6 | 80.7 | 1.2 | 84.4 | 5.3 | 84.3 | 1.4 | 74.6 | 3.2 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 34 | Coumaphos | Pesticides | LC | 109.1 | 2.4 | 99.2 | 1.5 | 102.3 | 2.8 | 102.3 | 1.9 | 96.2 | 1.5 |
| 35 | Cyazofamid | Pesticides | LC | 77.4 | 3.2 | 61.5 | 1.4 | 77.3 | 1.8 | 92.4 | 3.0 | 56.4 | 4.9 |
| 36 | Cyflufenamid | Pesticides | LC | 89.2 | 2.4 | 58.3 | 3.2 | 81.9 | 3.8 | 86.6 | 0.9 | 79.8 | 2.1 |
| 37 | Cyfluthrin | Pesticides | GC | 128.4 | 11.4 | 106.1 | 35.3 | 80.2 | 13.3 | 77.6 | 14.7 | 60.2 | 8.4 |
| 38 | Cyhalothrin (lambda isomer) | Pesticides | LC | 83.4 | 7.9 | 30.1 | 24.3 | 75.5 | 11.8 | 72.2 | 7.7 | 62.9 | 21.9 |
| 39 | Cymoxanil | Pesticides | LC | 81.5 | 2.3 | 71.2 | 1.3 | 74.4 | 1.7 | 77.2 | 1.2 | 74.1 | 2.0 |
| 40 | Cypermethrin | Pesticides | GC | 133.8 | 15.0 | 128.8 | 31.7 | 80.3 | 13.2 | 70.2 | 21.4 | 58.8 | 10.3 |
| 41 | Cyproconazole | Pesticides | LC | 85.2 | 2.7 | 80.1 | 1.8 | 78.4 | 2.0 | 78.7 | 1.8 | 76.6 | 2.6 |
| 42 | Cyprodinil | Pesticides | GC | 122.8 | 13.8 | 107.9 | 21.9 | 87.0 | 11.3 | 89.3 | 7.9 | 77.9 | 8.9 |
| 43 | Deltamethrin | Pesticides | GC | 98.6 | 10.1 | 99.1 | 38.7 | 63.5 | 11.1 | 51.2 | 26.0 | 39.5 | 11.9 |
| 44 | Demeton-S-methyl | Pesticides | LC | 79.5 | 4.2 | 63.0 | 1.4 | 78.5 | 2.3 | 80.6 | 1.6 | 66.7 | 2.1 |
| 45 | Demeton-S-methyl-sulfone (Dioxydemeton) | Pesticides | LC | 81.7 | 2.1 | 76.2 | 1.9 | 75.6 | 1.3 | 75.1 | 1.5 | 75.0 | 1.8 |
| 46 | Diazinon | Pesticides | GC | 125.3 | 8.8 | 118.3 | 19.9 | 96.4 | 10.7 | 96.1 | 7.0 | 77.9 | 8.9 |
| 47 | Dichlofluanid | Pesticides | GC | 54.6 | 18.4 | 45.3 | 29.4 | 36.1 | 12.5 | 37.0 | 11.2 | 26.5 | 29.2 |
| 48 | Dichloran | Pesticides | GC | 126.0 | 20.3 | 94.4 | 29.5 | 80.2 | 10.5 | 66.0 | 12.6 | 62.5 | 10.4 |
| 49 | Diethathyl ethyl | Pesticides | LC | 94.6 | 3.1 | 75.9 | 3.1 | 88.3 | 2.9 | 93.4 | 1.2 | 88.1 | 2.9 |
| 50 | Diethofencarb | Pesticides | LC | 89.6 | 3.0 | 82.1 | 2.3 | 82.7 | 2.7 | 86.9 | 1.4 | 78.8 | 1.8 |
| 51 | Difenoconazole | Pesticides | LC | 81.6 | 2.7 | 64.5 | 2.4 | 72.9 | 11.0 | 73.0 | 2.5 | 69.8 | 2.1 |
| 52 | Diflubenzuron | Pesticides | LC | 84.4 | 4.7 | 70.5 | 2.5 | 78.1 | 5.0 | 82.3 | 2.5 | 72.6 | 4.2 |
| 53 | Diflufenican | Pesticides | LC | 84.2 | 2.3 | 53.3 | 3.9 | 79.8 | 2.9 | 82.4 | 3.0 | 77.4 | 3.6 |
| 54 | Dimethenamide | Pesticides | LC | 82.1 | 2.2 | 68.7 | 2.4 | 78.3 | 2.7 | 82.6 | 1.4 | 75.2 | 0.5 |
| 55 | Dimethoate | Pesticides | LC | 83.4 | 3.5 | 72.0 | 2.4 | 76.2 | 1.4 | 75.9 | 0.7 | 76.3 | 1.4 |
| 56 | Dimethomorph | Pesticides | LC | 85.1 | 1.5 | 89.8 | 2.1 | 70.7 | 2.1 | 67.9 | 1.6 | 64.3 | 2.5 |
| 57 | Diniconazole-M | Pesticides | LC | 84.4 | 3.2 | 68.3 | 2.7 | 69.3 | 6.1 | 74.3 | 3.7 | 61.5 | 3.2 |
| 58 | Dinocap | Pesticides | LC | 99.1 | 10.0 | 46.0 | 16.1 | 57.0 | 19.1 | 62.6 | 4.9 | 67.1 | 25.4 |
| 59 | Diphenylamine | Pesticides | LC | 77.8 | 7.4 | 65.9 | 3.0 | 71.9 | 6.9 | 63.6 | 6.8 | 69.6 | 4.3 |
| 60 | Endosulfan alfa | Pesticides, OCPs | GC | 110.6 | 10.7 | 89.9 | 29.7 | 73.8 | 5.6 | 76.7 | 10.5 | 66.6 | 5.3 |
| 61 | Endosulfan beta | Pesticides, OCPs | GC | 109.6 | 15.1 | 84.9 | 39.0 | 72.8 | 10.7 | 69.1 | 1.6 | 60.4 | 3.7 |
| 62 | EPN | Pesticides | LC | 83.2 | 7.2 | 48.2 | 3.7 | 74.7 | 1.1 | 80.7 | 2.5 | 77.9 | 4.1 |
| 63 | Epoxiconazole | Pesticides | LC | 76.0 | 4.4 | 64.1 | 3.5 | 73.1 | 5.6 | 76.4 | 1.6 | 53.7 | 6.0 |
| 64 | Esfenvalerate | Pesticides | GC | 100.8 | 10.7 | 87.2 | 37.7 | 58.9 | 8.8 | 55.5 | 13.4 | 41.7 | 10.3 |
| 65 | Ethion | Pesticides | LC | 90.6 | 2.3 | 39.3 | 2.2 | 83.3 | 0.9 | 91.1 | 1.7 | 83.8 | 1.6 |
| 66 | Ethofumesate | Pesticides | GC | 147.1 | 15.6 | 119.4 | 35.5 | 99.8 | 21.0 | 110.3 | 21.6 | 92.4 | 9.8 |
| 67 | Ethoprophos | Pesticides | LC | 80.2 | 1.8 | 60.1 | 2.1 | 75.1 | 2.9 | 79.8 | 2.0 | 71.6 | 2.7 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 68 | Etofenprox | Pesticides | LC | 64.3 | 14.8 | 31.8 | 9.1 | 47.6 | 38.3 | 22.4 | 10.8 | 43.4 | 32.2 |
| 69 | Etoxazole | Pesticides | LC | 81.5 | 2.2 | 32.8 | 6.4 | 74.6 | 4.1 | 78.6 | 0.6 | 78.1 | 2.8 |
| 70 | Fenamidone | Pesticides | LC | 77.9 | 4.6 | 77.9 | 1.9 | 71.5 | 1.6 | 71.9 | 2.5 | 64.3 | 2.0 |
| 71 | Fenamiphos | Pesticides | LC | 92.1 | 2.0 | 76.1 | 2.3 | 86.5 | 2.2 | 86.4 | 1.6 | 77.8 | 2.3 |
| 72 | Fenamiphos sulfone | Pesticides | LC | 81.6 | 2.6 | 82.1 | 1.3 | 70.0 | 3.7 | 66.8 | 1.6 | 67.4 | 1.9 |
| 73 | Fenamiphos sulfoxide | Pesticides | LC | 72.0 | 2.9 | 72.5 | 1.7 | 62.5 | 3.0 | 52.2 | 0.8 | 67.4 | 2.5 |
| 74 | Fenarimol | Pesticides | GC | 120.7 | 10.7 | 110.9 | 24.2 | 79.3 | 10.2 | 76.5 | 5.1 | 73.6 | 8.4 |
| 75 | Fenazaquin | Pesticides | LC | 78.4 | 7.4 | 22.7 | 33.7 | 71.5 | 13.2 | 88.1 | 4.0 | 87.5 | 3.2 |
| 76 | Fenbuconazole | Pesticides | LC | 91.6 | 1.6 | 88.0 | 2.9 | 85.5 | 4.7 | 83.9 | 1.6 | 80.8 | 2.4 |
| 77 | Fenbutatin oxide | Pesticides | LC | 97.6 | 31.8 | 51.7 | 63.6 | 54.1 | 29.7 | 75.3 | 16.8 | 84.6 | 15.9 |
| 78 | Fenitrothion | Pesticides | GC | 110.3 | 16.1 | 103.0 | 32.1 | 79.0 | 18.0 | 72.5 | 18.7 | 51.8 | 9.3 |
| 79 | Fenoxycarb | Pesticides | LC | 92.8 | 2.6 | 70.9 | 2.1 | 86.7 | 2.1 | 89.1 | 1.4 | 82.4 | 2.1 |
| 80 | Fenpropathrin | Pesticides | LC | 85.9 | 4.2 | 28.4 | 14.9 | 72.8 | 3.8 | 74.2 | 1.4 | 68.0 | 1.8 |
| 81 | Fenpropimorph | Pesticides | LC | 65.7 | 2.3 | 68.3 | 1.8 | 57.8 | 2.4 | 37.9 | 2.7 | 78.3 | 2.2 |
| 82 | Fenpyroximate | Pesticides | LC | 84.8 | 4.1 | 26.7 | 36.7 | 78.8 | 1.3 | 78.5 | 0.7 | 73.4 | 2.4 |
| 83 | Fenthion | Pesticides | LC | 91.2 | 5.6 | 64.2 | 4.9 | 80.2 | 5.0 | 79.6 | 4.1 | 75.9 | 4.9 |
| 84 | Fenthion oxon | Pesticides | LC | 89.0 | 2.4 | 74.9 | 1.6 | 83.0 | 1.7 | 87.3 | 1.1 | 83.0 | 0.6 |
| 85 | Fenthion oxon sulfone | Pesticides | LC | 76.8 | 2.4 | 70.7 | 2.0 | 69.0 | 2.8 | 67.6 | 2.1 | 67.9 | 2.7 |
| 86 | Fenthion oxon sulfoxide | Pesticides | LC | 70.5 | 3.1 | 66.0 | 2.8 | 61.4 | 1.6 | 52.4 | 1.5 | 69.5 | 2.3 |
| 87 | Fenthion sulfone | Pesticides | LC | 81.0 | 2.0 | 71.3 | 2.0 | 72.1 | 1.8 | 75.5 | 1.1 | 69.3 | 1.6 |
| 88 | Fenthion sulfoxide | Pesticides | LC | 82.6 | 1.4 | 79.8 | 1.5 | 71.2 | 3.0 | 70.4 | 1.3 | 69.2 | 2.4 |
| 89 | Fenvalerate | Pesticides | GC | 111.6 | 13.1 | 104.5 | 30.2 | 68.8 | 18.3 | 57.4 | 10.6 | 49.6 | 8.1 |
| 90 | Fipronil | Pesticides | LC | 80.6 | 4.4 | 85.6 | 3.5 | 69.9 | 4.5 | 72.1 | 2.6 | 68.7 | 1.2 |
| 91 | Fipronil sulfide | Pesticides | GC | 147.2 | 7.7 | 145.3 | 13.4 | 150.9 | 18.7 | 150.0 | 15.2 | 112.8 | 2.3 |
| 92 | Fluazinam | Pesticides | LC | 88.1 | 1.4 | 73.6 | 2.9 | 83.8 | 4.4 | 83.3 | 1.9 | 78.7 | 1.8 |
| 93 | Flubendiamide | Pesticides | LC | 82.9 | 2.8 | 69.3 | 2.9 | 79.8 | 1.7 | 80.6 | 3.1 | 75.5 | 1.9 |
| 94 | Flucythrinate | Pesticides | GC | 121.8 | 11.6 | 129.2 | 26.9 | 80.8 | 10.8 | 73.0 | 10.9 | 60.0 | 10.8 |
| 95 | Fludioxonil | Pesticides | LC | 86.0 | 3.0 | 74.7 | 9.0 | 75.2 | 5.4 | 79.7 | 2.6 | 72.5 | 3.9 |
| 96 | Flufenoxuron | Pesticides | LC | 80.5 | 3.8 | 45.3 | 1.4 | 71.7 | 4.1 | 74.6 | 2.3 | 70.9 | 2.1 |
| 97 | Fluopyram | Pesticides | LC | 81.0 | 1.1 | 66.7 | 2.0 | 69.2 | 3.7 | 71.7 | 1.6 | 67.6 | 0.5 |
| 98 | Fluquinconazole | Pesticides | LC | 79.5 | 6.3 | 61.9 | 4.6 | 70.1 | 8.0 | 73.5 | 4.5 | 73.2 | 4.6 |
| 99 | Flusilazole | Pesticides | LC | 90.7 | 4.9 | 72.6 | 3.2 | 82.7 | 2.8 | 85.6 | 4.0 | 73.7 | 2.3 |
| 100 | Flutolanil | Pesticides | LC | 78.4 | 3.5 | 65.2 | 1.4 | 69.7 | 4.1 | 70.5 | 0.9 | 66.6 | 2.1 |
| 101 | Flutriafol | Pesticides | LC | 78.5 | 3.7 | 69.1 | 2.3 | 73.5 | 1.7 | 77.9 | 0.7 | 74.5 | 1.0 |
| 102 | Fluvalinate tau | Pesticides | LC | 76.0 | 6.5 | 40.5 | 16.8 | 66.7 | 7.1 | 59.9 | 4.1 | 54.4 | 4.1 |
| 103 | Fonofos | Pesticides | GC | 126.3 | 9.7 | 117.4 | 24.9 | 93.4 | 11.7 | 97.2 | 8.7 | 79.2 | 6.3 |
| 104 | Fosthiazate | Pesticides | LC | 81.8 | 3.0 | 73.1 | 1.4 | 73.2 | 0.7 | 71.6 | 0.7 | 78.3 | 0.9 |
| 105 | Hexaconazole | Pesticides | LC | 98.4 | 4.8 | 73.0 | 2.0 | 85.5 | 1.5 | 89.5 | 1.9 | 86.1 | 2.4 |
| 106 | Hexaflumuron | Pesticides | LC | 71.5 | 3.4 | 73.9 | 3.9 | 60.6 | 5.6 | 56.7 | 3.1 | 51.3 | 5.8 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 107 | Hexythiazox | Pesticides | LC | 83.3 | 2.2 | 30.9 | 1.2 | 70.6 | 12.1 | 80.3 | 0.9 | 76.2 | 1.9 |
| 108 | Imidacloprid | Pesticides | LC | 84.5 | 2.6 | 77.2 | 4.3 | 77.2 | 3.0 | 74.5 | 2.2 | 72.8 | 2.7 |
| 109 | Indoxacarb | Pesticides | LC | 89.1 | 2.6 | 69.0 | 4.2 | 80.6 | 3.3 | 77.2 | 4.5 | 69.3 | 2.7 |
| 110 | Iprodione | Pesticides | GC | 72.9 | 10.9 | 53.2 | 18.6 | 40.4 | 10.2 | 39.3 | 4.4 | 31.7 | 10.9 |
| 111 | Iprovalicarb | Pesticides | LC | 79.5 | 1.3 | 69.7 | 1.5 | 73.7 | 0.6 | 76.3 | 0.6 | 72.7 | 1.4 |
| 112 | Isocarbophos | Pesticides | GC | 123.7 | 16.3 | 114.9 | 21.0 | 83.4 | 9.7 | 84.9 | 9.6 | 68.1 | 9.1 |
| 113 | Isofenphos methyl | Pesticides | LC | 83.8 | 3.6 | 60.0 | 3.6 | 76.9 | 4.6 | 82.4 | 2.8 | 77.3 | 3.1 |
| 114 | Isoprothiolane | Pesticides | LC | 81.2 | 4.3 | 66.0 | 2.9 | 73.4 | 2.0 | 73.7 | 1.4 | 71.6 | 3.6 |
| 115 | Kresoxim methyl | Pesticides | LC | 88.0 | 4.2 | 59.6 | 1.6 | 79.8 | 3.5 | 84.0 | 3.3 | 79.4 | 3.6 |
| 116 | Linuron | Pesticides | LC | 81.4 | 4.3 | 66.3 | 2.4 | 75.9 | 6.1 | 80.8 | 3.1 | 75.9 | 3.1 |
| 117 | Lufenuron | Pesticides | LC | 67.9 | 4.1 | 39.5 | 15.4 | 73.0 | 3.8 | 79.3 | 2.1 | 72.5 | 3.9 |
| 118 | Malaoxon | Pesticides | LC | 80.0 | 2.3 | 69.3 | 1.4 | 73.7 | 1.0 | 73.5 | 2.1 | 70.5 | 3.1 |
| 119 | Malathion | Pesticides | LC | 81.6 | 3.7 | 68.0 | 2.1 | 71.6 | 2.3 | 73.2 | 2.1 | 69.0 | 2.4 |
| 120 | Mandipropamid | Pesticides | LC | 81.5 | 2.2 | 78.0 | 1.4 | 72.3 | 2.7 | 70.8 | 0.8 | 66.7 | 3.6 |
| 121 | Mefenoxam (metalaxyl-M) | Pesticides | LC | 75.7 | 2.4 | 68.6 | 1.1 | 69.1 | 0.7 | 67.3 | 0.6 | 69.2 | 0.8 |
| 122 | Mepanipyrim | Pesticides | LC | 88.7 | 4.6 | 59.4 | 2.2 | 80.8 | 4.2 | 89.1 | 1.8 | 82.6 | 1.6 |
| 123 | Metaflumizone | Pesticides | LC | 64.4 | 2.4 | 76.8 | 2.8 | 58.7 | 4.0 | 55.6 | 1.5 | 51.6 | 1.8 |
| 124 | Metalaxyl | Pesticides | GC | 124.2 | 9.1 | 108.2 | 32.9 | 82.4 | 11.3 | 86.4 | 14.6 | 76.6 | 7.7 |
| 125 | Metaldehyde | Pesticides | LC | 81.2 | 5.3 | 81.1 | 4.8 | 77.1 | 9.3 | 79.9 | 4.1 | 80.5 | 4.6 |
| 126 | Metconazole | Pesticides | LC | 85.8 | 2.0 | 67.7 | 1.7 | 82.1 | 1.9 | 80.5 | 1.8 | 75.7 | 0.8 |
| 127 | Methamidophos | Pesticides | LC | 44.2 | 6.5 | 47.6 | 1.6 | 52.3 | 0.5 | 41.9 | 1.2 | 46.0 | 2.2 |
| 128 | Methidathion | Pesticides | LC | 89.6 | 3.3 | 73.7 | 1.1 | 80.6 | 2.4 | 81.3 | 2.1 | 78.7 | 2.5 |
| 129 | Methiocarb | Pesticides | LC | 84.5 | 3.8 | 68.1 | 2.8 | 79.6 | 1.5 | 84.7 | 1.2 | 77.3 | 1.7 |
| 130 | Methiocarb sulfone | Pesticides | LC | 81.2 | 2.8 | 71.6 | 2.0 | 71.9 | 2.6 | 73.1 | 3.6 | 74.5 | 3.6 |
| 131 | Methiocarb sulfoxide | Pesticides | LC | 74.1 | 1.7 | 64.7 | 1.6 | 69.3 | 2.2 | 65.0 | 2.7 | 71.2 | 1.6 |
| 132 | Methomyl | Pesticides | LC | 89.5 | 4.1 | 77.0 | 1.5 | 77.7 | 0.9 | 76.7 | 0.8 | 80.0 | 2.2 |
| 133 | Methomyl oxime | Pesticides | LC | 73.2 | 7.8 | 67.4 | 7.1 | 68.5 | 4.3 | 69.6 | 5.8 | 64.9 | 4.6 |
| 134 | Methoxyfenozide | Pesticides | LC | 81.5 | 2.8 | 72.4 | 1.5 | 74.4 | 1.4 | 75.3 | 1.4 | 71.8 | 1.8 |
| 135 | Metrafenone | Pesticides | LC | 89.5 | 6.3 | 67.6 | 7.3 | 83.4 | 16.4 | 98.1 | 2.4 | 86.5 | 2.5 |
| 136 | Mevinphos (phosdrin) | Pesticides | LC | 77.9 | 2.6 | 72.7 | 1.9 | 75.4 | 0.7 | 75.8 | 0.7 | 75.7 | 0.9 |
| 137 | Monocrotophos | Pesticides | LC | 73.0 | 3.2 | 68.3 | 1.7 | 70.1 | 1.1 | 62.5 | 2.4 | 72.3 | 3.0 |
| 138 | Myclobutanil | Pesticides | LC | 87.7 | 7.5 | 88.7 | 4.0 | 70.7 | 3.2 | 71.8 | 1.5 | 67.1 | 1.8 |
| 139 | N,N-Dimethyl-N'-p-tolylsulphamide (DMST) | Pesticides | LC | 94.3 | 3.4 | 84.9 | 0.8 | 84.0 | 4.2 | 86.4 | 2.1 | 77.5 | 2.1 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 140 | N,N-dimethylformamidine (DMF) | Pesticides | LC | 102.4 | 6.1 | 76.6 | 1.7 | 95.3 | 4.0 | 101.4 | 3.7 | 82.7 | 3.8 |
| 141 | Nuarimol | Pesticides | LC | 78.4 | 8.4 | 73.7 | 3.0 | 71.3 | 3.4 | 73.1 | 3.3 | 67.5 | 2.3 |
| 142 | Ofurace | Pesticides | LC | 81.6 | 3.2 | 70.0 | 2.1 | 76.0 | 3.1 | 75.9 | 3.0 | 66.1 | 1.0 |
| 143 | Omethoate | Pesticides | LC | 64.3 | 2.7 | 58.5 | 1.2 | 59.6 | 1.0 | 45.4 | 1.0 | 69.1 | 1.6 |
| 144 | Oxadixyl | Pesticides | LC | 79.3 | 2.1 | 72.7 | 1.1 | 69.6 | 1.1 | 66.7 | 1.0 | 70.8 | 1.6 |
| 145 | Oxamyl | Pesticides | LC | 76.5 | 3.6 | 70.1 | 1.7 | 69.1 | 1.2 | 65.3 | 1.0 | 75.0 | 1.8 |
| 146 | Oxamyl oxime | Pesticides | LC | 82.2 | 3.3 | 70.7 | 1.9 | 78.1 | 0.5 | 79.8 | 0.8 | 68.0 | 1.4 |
| 147 | Oxyfluorfen | Pesticides | GC | 98.0 | 11.2 | 92.1 | 23.3 | 76.7 | 9.1 | 67.5 | 9.3 | 62.0 | 11.7 |
| 148 | Paclobutrazol | Pesticides | LC | 78.5 | 4.8 | 71.2 | 3.2 | 69.7 | 2.1 | 71.0 | 3.5 | 70.3 | 1.5 |
| 149 | Paraoxon methyl | Pesticides | GC | 89.4 | 6.3 | 97.2 | 25.1 | 61.9 | 5.5 | 60.8 | 10.6 | 50.7 | 7.6 |
| 150 | Parathion ethyl | Pesticides | GC | 105.3 | 13.9 | 92.3 | 16.3 | 79.8 | 14.5 | 65.4 | 8.9 | 57.9 | 15.3 |
| 151 | Parathion methyl | Pesticides | GC | 98.1 | 4.7 | 92.5 | 20.5 | 70.3 | 8.7 | 60.3 | 15.5 | 45.0 | 7.8 |
| 152 | Penconazole | Pesticides | LC | 84.6 | 1.7 | 63.6 | 3.5 | 78.0 | 1.5 | 81.5 | 2.8 | 77.7 | 3.5 |
| 153 | Pencycuron | Pesticides | LC | 81.8 | 3.7 | 52.1 | 2.7 | 69.7 | 5.7 | 78.1 | 3.2 | 58.7 | 1.6 |
| 154 | Pendimethalin | Pesticides | LC | 86.6 | 3.3 | 38.4 | 4.4 | 79.3 | 2.6 | 79.5 | 3.4 | 76.7 | 1.5 |
| 155 | Permethrin | Pesticides | GC | 126.3 | 14.2 | 128.8 | 30.4 | 93.0 | 9.2 | 92.7 | 17.4 | 71.2 | 6.2 |
| 156 | Phosalone | Pesticides | LC | 86.3 | 2.0 | 60.6 | 4.1 | 82.2 | 2.0 | 87.4 | 1.0 | 81.1 | 2.6 |
| 157 | Phosmet | Pesticides | LC | 86.9 | 3.6 | 76.5 | 2.7 | 77.1 | 2.9 | 78.3 | 1.7 | 72.6 | 2.6 |
| 158 | Phosmet oxon | Pesticides | LC | 79.0 | 2.8 | 70.5 | 1.5 | 70.0 | 0.7 | 69.3 | 0.9 | 71.4 | 0.9 |
| 159 | Phthalimide (Folpet deg) | Pesticides | GC | 131.6 | 5.2 | 131.3 | 23.3 | 109.7 | 4.2 | 104.9 | 5.3 | 115.0 | 3.6 |
| 160 | Pirimicarb | Pesticides | LC | 56.0 | 8.8 | 61.9 | 1.1 | 67.3 | 1.3 | 59.4 | 1.3 | 71.6 | 0.9 |
| 161 | Pirimiphos ethyl | Pesticides | LC | 81.3 | 3.6 | 37.1 | 2.4 | 79.6 | 1.1 | 86.9 | 0.6 | 81.3 | 1.1 |
| 162 | Pirimiphos methyl | Pesticides | LC | 90.4 | 4.5 | 54.5 | 2.0 | 83.3 | 2.5 | 91.7 | 0.9 | 85.3 | 1.3 |
| 163 | Prochloraz | Pesticides | LC | 95.3 | 4.5 | 69.3 | 3.8 | 90.2 | 1.5 | 88.6 | 1.9 | 86.9 | 4.8 |
| 164 | Procymidone | Pesticides | GC | 139.6 | 19.3 | 108.9 | 19.9 | 98.8 | 10.3 | 104.0 | 12.6 | 87.8 | 4.9 |
| 165 | Profenofos | Pesticides | LC | 87.4 | 3.0 | 44.6 | 1.9 | 77.0 | 2.9 | 81.6 | 2.1 | 73.9 | 1.9 |
| 166 | Propargite | Pesticides | LC | 82.2 | 3.1 | 30.0 | 8.7 | 74.6 | 5.8 | 81.7 | 2.1 | 77.6 | 1.6 |
| 167 | Propoxur | Pesticides | LC | 86.3 | 2.8 | 73.1 | 2.2 | 80.3 | 0.9 | 82.5 | 1.6 | 80.9 | 1.3 |
| 168 | Propyzamide (pronamide) | Pesticides | LC | 83.4 | 6.3 | 62.4 | 0.8 | 73.8 | 1.9 | 79.9 | 3.6 | 75.5 | 4.2 |
| 169 | Proquinazid | Pesticides | LC | 78.6 | 2.6 | 21.0 | 51.3 | 71.9 | 8.5 | 76.7 | 3.4 | 70.6 | 1.0 |
| 170 | Prothioconazole-desthio | Pesticides | LC | 76.3 | 5.3 | 55.9 | 3.1 | 74.0 | 4.6 | 84.6 | 3.5 | 59.4 | 3.3 |
| 171 | Prothiophos | Pesticides | GC | 129.3 | 11.0 | 105.7 | 40.2 | 84.8 | 11.7 | 87.2 | 14.0 | 69.5 | 5.1 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 172 | Pyraclostrobin | Pesticides | LC | 85.4 | 3.0 | 59.2 | 2.4 | 77.2 | 3.0 | 81.0 | 1.9 | 75.1 | 1.2 |
| 173 | Pyrazophos | Pesticides | LC | 84.5 | 2.6 | 76.1 | 2.1 | 75.4 | 2.7 | 72.7 | 1.3 | 67.1 | 3.4 |
| 174 | Pyridaben | Pesticides | LC | 81.6 | 1.9 | 27.5 | 35.1 | 75.2 | 5.9 | 84.2 | 0.9 | 80.8 | 0.9 |
| 175 | Pyridaphenthion | Pesticides | LC | 79.3 | 7.4 | 72.0 | 2.9 | 61.6 | 3.0 | 62.0 | 2.1 | 55.8 | 1.8 |
| 176 | Pyrimethanil | Pesticides | GC | 129.0 | 11.8 | 119.3 | 26.3 | 94.2 | 12.7 | 95.0 | 5.7 | 80.5 | 11.5 |
| 177 | Pyriproxifen | Pesticides | LC | 82.0 | 2.3 | 32.3 | 3.2 | 76.4 | 0.9 | 81.4 | 1.4 | 78.6 | 1.8 |
| 178 | Quinalphos | Pesticides | LC | 91.9 | 2.3 | 74.2 | 3.6 | 89.5 | 1.6 | 93.9 | 3.7 | 89.6 | 1.8 |
| 179 | Quinoxyfen | Pesticides | LC | 78.8 | 7.1 | 22.6 | 2.9 | 70.4 | 8.5 | 88.7 | 2.1 | 88.7 | 2.1 |
| 180 | Rotenone | Pesticides | LC | 89.4 | 2.6 | 79.7 | 4.1 | 78.7 | 7.1 | 80.7 | 1.0 | 66.9 | 4.5 |
| 181 | Simazine | Pesticides | LC | 78.4 | 5.2 | 56.7 | 2.7 | 75.9 | 2.9 | 76.0 | 2.4 | 76.0 | 2.0 |
| 182 | Spirodiclofen | Pesticides | LC | 86.9 | 4.3 | 27.0 | 39.3 | 79.8 | 2.3 | 87.0 | 1.1 | 79.0 | 1.4 |
| 183 | Spiromesifen | Pesticides | LC | 82.8 | 4.5 | 31.5 | 3.3 | 71.2 | 6.1 | 73.6 | 3.8 | 68.0 | 1.3 |
| 184 | Spirotetramat | Pesticides | LC | 78.8 | 4.2 | 106.1 | 3.2 | 71.5 | 4.4 | 66.0 | 3.2 | 61.8 | 3.1 |
| 185 | Spirotetramat-enol | Pesticides | LC | 77.7 | 5.7 | 109.3 | 1.0 | 70.6 | 3.6 | 67.2 | 3.6 | 61.9 | 6.4 |
| 186 | Spiroxamine | Pesticides | GC | 62.7 | 12.0 | 105.0 | 19.1 | 37.9 | 8.5 | 17.7 | 5.4 | 86.3 | 4.9 |
| 187 | Tebuconazole | Pesticides | LC | 81.1 | 3.1 | 63.3 | 1.4 | 77.3 | 2.5 | 78.6 | 6.2 | 77.0 | 5.4 |
| 188 | Tebufenocide | Pesticides | LC | 80.7 | 2.6 | 65.4 | 1.2 | 73.5 | 2.9 | 74.6 | 2.3 | 70.4 | 2.6 |
| 189 | Tebufenpyrad | Pesticides | LC | 101.5 | 2.6 | 48.0 | 2.1 | 91.9 | 2.3 | 97.1 | 3.1 | 88.9 | 2.3 |
| 190 | Teflubenzuron (artifact 3) | Pesticides | GC | 153.4 | 8.7 | 131.7 | 21.4 | 103.5 | 10.5 | 93.9 | 9.9 | 96.6 | 7.7 |
| 191 | Tefluthrin | Pesticides | GC | 137.2 | 19.3 | 138.0 | 31.2 | 102.0 | 13.2 | 98.8 | 12.6 | 86.7 | 4.4 |
| 192 | Telodrin | Pesticides | GC | 130.4 | 13.9 | 101.1 | 24.7 | 83.1 | 16.7 | 85.5 | 15.2 | 76.0 | 12.5 |
| 193 | Terbufos | Pesticides | GC | 135.6 | 7.0 | 125.2 | 25.5 | 96.8 | 6.5 | 100.2 | 11.1 | 79.3 | 3.5 |
| 194 | Terbuthylazine | Pesticides | LC | 81.3 | 2.6 | 55.8 | 1.9 | 72.6 | 2.6 | 78.6 | 1.1 | 71.8 | 2.1 |
| 195 | Tetrachlorvinphos | Pesticides | LC | 100.8 | 3.4 | 80.2 | 2.6 | 96.6 | 1.2 | 100.6 | 2.2 | 95.6 | 4.1 |
| 196 | Tetraconazole | Pesticides | LC | 78.6 | 3.6 | 63.5 | 5.8 | 70.3 | 2.4 | 72.5 | 2.3 | 67.8 | 3.6 |
| 197 | Tetradifon | Pesticides | GC | 135.8 | 12.0 | 116.3 | 26.5 | 82.3 | 8.0 | 84.9 | 14.3 | 68.9 | 3.6 |
| 198 | Tetramethrin | Pesticides | GC | 113.5 | 14.6 | 102.7 | 28.5 | 57.9 | 7.2 | 60.0 | 5.4 | 48.9 | 10.6 |
| 199 | Thiacloprid | Pesticides | LC | 78.3 | 3.1 | 71.6 | 2.0 | 69.2 | 0.6 | 67.5 | 0.2 | 73.5 | 1.4 |
| 200 | Thiamethoxam | Pesticides | LC | 76.1 | 3.7 | 62.1 | 3.6 | 66.0 | 3.2 | 62.6 | 1.6 | 61.3 | 2.3 |
| 201 | Thiodicarb | Pesticides | LC | 72.0 | 3.0 | 74.0 | 1.9 | 61.8 | 1.3 | 58.6 | 0.6 | 71.9 | 1.6 |
| 202 | Tolclofos methyl | Pesticides | GC | 129.7 | 9.0 | 115.7 | 28.1 | 90.7 | 13.7 | 97.1 | 16.7 | 68.3 | 5.0 |
| 203 | Tolylfluanid | Pesticides | GC | 70.1 | 13.7 | 56.9 | 33.9 | 42.9 | 12.8 | 40.9 | 10.5 | 31.2 | 24.3 |
| 204 | Triadimefon | Pesticides | LC | 84.1 | 2.4 | 84.4 | 1.9 | 77.7 | 0.9 | 75.5 | 3.1 | 71.4 | 4.9 |
| 205 | Triadimenol | Pesticides | LC | 81.0 | 1.1 | 99.6 | 1.8 | 78.6 | 2.0 | 76.0 | 4.3 | 72.5 | 2.8 |
| 206 | Triazophos (hostathion) | Pesticides | LC | 81.4 | 3.1 | 70.8 | 1.7 | 74.1 | 0.5 | 77.9 | 1.9 | 74.0 | 1.5 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 207 | Trichlorfon | Pesticides | LC | 79.6 | 3.5 | 65.6 | 2.4 | 70.6 | 2.5 | 71.4 | 3.0 | 68.9 | 3.0 |
| 208 | Trifloxystrobin | Pesticides | LC | 83.5 | 3.2 | 49.5 | 3.1 | 77.6 | 1.7 | 82.9 | 0.7 | 77.5 | 1.8 |
| 209 | Triflumizole | Pesticides | LC | 77.2 | 6.0 | 45.8 | 2.2 | 78.3 | 1.7 | 81.7 | 2.9 | 82.1 | 1.3 |
| 210 | Triflumuron | Pesticides | LC | 93.7 | 3.2 | 63.2 | 2.2 | 84.5 | 2.3 | 85.6 | 3.3 | 81.7 | 5.0 |
| 211 | Trifluralin | Pesticides | GC | 114.2 | 28.5 | 134.2 | 33.4 | 100.5 | 8.9 | 80.7 | 5.1 | 78.3 | 12.0 |
| 212 | Triticonazole | Pesticides | LC | 82.9 | 2.5 | 82.3 | 3.9 | 76.9 | 1.8 | 73.3 | 2.8 | 72.2 | 3.8 |
| 213 | Vinclozolin | Pesticides | GC | 150.3 | 5.8 | 130.5 | 27.2 | 99.3 | 13.8 | 105.7 | 22.1 | 89.3 | 6.2 |
| 214 | Aldrin | OCPs | GC | 115.2 | 10.2 | 100.7 | 18.0 | 83.0 | 9.0 | 91.6 | 10.9 | 78.6 | 8.2 |
| 215 | Dichlorodiphenyldichloroethane (p,p' DDD) | OCPs | GC | 113.5 | 17.7 | 88.4 | 36.9 | 83.1 | 20.7 | 73.9 | 9.7 | 112.3 | 4.2 |
| 216 | Dichlorodiphenyldichloroethylene (p,p' DDE) | OCPs | GC | 121.2 | 9.6 | 96.8 | 30.6 | 116.0 | 36.2 | 90.6 | 16.0 | 110.7 | 30.6 |
| 217 | Dieldrin | OCPs | GC | 102.7 | 9.7 | 80.7 | 25.1 | 127.1 | 26.1 | 75.5 | 13.9 | 53.7 | 12.1 |
| 218 | Endrin | OCPs | GC | 90.8 | 13.1 | 73.4 | 31.1 | 66.5 | 13.9 | 63.6 | 10.4 | 61.6 | 7.6 |
| 219 | Heptachlor | OCPs | GC | 98.1 | 12.1 | 71.0 | 41.6 | 57.2 | 13.5 | 65.6 | 16.3 | 54.8 | 6.4 |
| 220 | Hexachlorobenzene | OCPs | GC | 140.9 | 8.2 | 112.9 | 27.0 | 103.0 | 9.7 | 110.9 | 12.3 | 97.1 | 5.2 |
| 221 | Hexachlorocyclohexane (alpha) | OCPs | GC | 127.2 | 11.4 | 103.5 | 39.5 | 80.9 | 15.2 | 79.0 | 10.2 | 67.3 | 5.2 |
| 222 | Hexachlorocyclohexane (gamma, lindane) | OCPs | GC | 103.9 | 11.8 | 72.1 | 36.8 | 57.5 | 7.6 | 57.7 | 18.5 | 62.6 | 1.9 |
| 223 | Hexachlorocyclohexano (beta) | OCPs | GC | 125.2 | 11.7 | 86.9 | 34.4 | 61.5 | 12.5 | 69.4 | 12.3 | 14.9 | 39.0 |
| 224 | Hexaclorociclohexano (delta) | OCPs | GC | 89.6 | 12.4 | 78.7 | 31.6 | 62.2 | 18.8 | 62.6 | 11.5 | 44.7 | 3.1 |
| 225 | Mirex | OCPs | GC | 91.7 | 15.7 | 69.0 | 31.9 | 56.8 | 8.9 | 60.7 | 5.5 | 59.6 | 9.4 |
| 226 | PCB 28 | PCBs | GC | 136.1 | 7.2 | 127.3 | 30.7 | 99.7 | 9.3 | 111.0 | 14.4 | 93.5 | 4.8 |
| 227 | PCB 52 | PCBs | GC | 130.1 | 13.0 | 108.4 | 27.4 | 100.1 | 19.0 | 109.9 | 17.3 | 94.7 | 6.7 |
| 228 | PCB 77 | PCBs | GC | 123.5 | 12.0 | 98.2 | 28.4 | 92.7 | 10.7 | 99.7 | 13.0 | 86.8 | 4.2 |
| 229 | PCB 81 | PCBs | GC | 117.1 | 8.3 | 95.8 | 30.0 | 85.1 | 5.4 | 94.5 | 12.4 | 79.3 | 10.5 |
| 230 | PCB 101 | PCBs | GC | 127.6 | 10.9 | 99.9 | 31.4 | 90.0 | 8.3 | 93.6 | 4.1 | 86.5 | 6.9 |
| 231 | PCB 105 | PCBs | GC | 132.6 | 9.3 | 109.0 | 29.4 | 104.8 | 8.0 | 110.1 | 13.2 | 95.2 | 8.8 |
| 232 | PCB 114 | PCBs | GC | 120.9 | 12.5 | 101.2 | 28.8 | 95.3 | 14.3 | 104.8 | 17.5 | 83.2 | 9.2 |
| 233 | PCB 118 | PCBs | GC | 106.4 | 16.3 | 88.2 | 25.1 | 90.6 | 13.9 | 89.8 | 4.4 | 77.2 | 5.2 |
| 234 | PCB 123 | PCBs | GC | 129.7 | 10.1 | 119.9 | 25.8 | 114.0 | 11.7 | 119.9 | 17.3 | 103.9 | 4.3 |
| 235 | PCB 126 | PCBs | GC | 104.7 | 17.3 | 77.9 | 14.8 | 75.7 | 5.2 | 85.7 | 11.8 | 73.9 | 6.6 |
| 236 | PCB 138 | PCBs | GC | 96.6 | 19.7 | 78.8 | 16.9 | 74.1 | 14.5 | 80.3 | 11.5 | 74.3 | 9.6 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 237 | PCB 153 | PCBs | GC | 117.7 | 16.2 | 95.3 | 25.0 | 96.2 | 9.1 | 100.2 | 18.1 | 81.1 | 6.5 |
| 238 | PCB 156 | PCBs | GC | 119.3 | 12.6 | 95.5 | 17.7 | 82.9 | 6.4 | 90.7 | 7.8 | 86.9 | 11.1 |
| 239 | PCB 157 | PCBs | GC | 127.0 | 12.4 | 92.5 | 20.4 | 89.2 | 9.9 | 95.2 | 9.2 | 85.1 | 10.2 |
| 240 | PCB 167 | PCBs | GC | 107.1 | 12.6 | 80.5 | 17.1 | 81.9 | 13.0 | 89.5 | 7.4 | 80.4 | 8.1 |
| 241 | PCB 169 | PCBs | GC | 109.1 | 15.4 | 90.2 | 20.0 | 84.7 | 12.1 | 89.8 | 7.5 | 75.4 | 8.3 |
| 242 | PCB 180 | PCBs | GC | 114.5 | 12.8 | 98.9 | 21.6 | 86.4 | 11.0 | 93.4 | 14.2 | 78.0 | 4.5 |
| 243 | PCB 189 | PCBs | GC | 110.7 | 14.5 | 88.6 | 15.3 | 89.5 | 11.1 | 88.2 | 8.8 | 83.1 | 9.9 |
| 244 | PBDE 28 | PBDEs | GC | 100.7 | 6.0 | 93.9 | 16.0 | 94.5 | 5.7 | 96.6 | 3.9 | 85.8 | 1.4 |
| 245 | PBDE 47 | PBDEs | GC | 96.7 | 10.0 | 89.6 | 13.2 | 84.2 | 3.6 | 86.8 | 6.5 | 75.5 | 3.7 |
| 246 | PBDE 85 | PBDEs | GC | 98.8 | 4.7 | 97.4 | 9.8 | 98.4 | 8.2 | 85.5 | 5.4 | 76.8 | 5.7 |
| 247 | PBDE 99 | PBDEs | GC | 102.5 | 5.9 | 89.3 | 19.9 | 91.1 | 8.9 | 95.9 | 5.3 | 85.2 | 7.8 |
| 248 | PBDE 100 | PBDEs | GC | 97.6 | 6.8 | 88.3 | 12.4 | 88.0 | 2.6 | 94.7 | 6.7 | 83.9 | 4.3 |
| 249 | PBDE 153 | PBDEs | GC | 90.4 | 6.4 | 91.9 | 11.2 | 89.4 | 3.5 | 88.2 | 6.2 | 75.5 | 2.7 |
| 250 | PBDE 154 | PBDEs | GC | 97.1 | 2.8 | 95.9 | 11.6 | 95.6 | 8.0 | 93.7 | 11.3 | 82.4 | 2.6 |
| 251 | PBDE 183 | PBDEs | GC | 91.8 | 6.4 | 76.1 | 10.8 | 81.8 | 4.9 | 71.7 | 7.5 | 64.3 | 6.5 |
| 252 | Acenaphthene | PAHs | GC | 123.3 | 20.1 | 116.6 | 37.1 | 79.7 | 6.1 | 78.8 | 10.2 | 76.1 | 5.5 |
| 253 | Acenaphthylene | PAHs | GC | 138.7 | 16.0 | 126.4 | 33.4 | 92.6 | 5.4 | 89.8 | 7.1 | 93.9 | 9.3 |
| 254 | Anthracene | PAHs | GC | 117.1 | 19.1 | 115.0 | 36.3 | 87.2 | 15.5 | 87.0 | 8.9 | 76.3 | 4.1 |
| 255 | Benzo[a]anthracene | PAHs | GC | 127.8 | 16.9 | 101.1 | 34.2 | 69.3 | 8.9 | 75.1 | 7.1 | 66.7 | 10.9 |
| 256 | Benzo[b]fluoranthene | PAHs | GC | 136.4 | 44.4 | 113.9 | 43.8 | 78.8 | 11.2 | 66.3 | 9.0 | 50.5 | 9.7 |
| 257 | Chrysene | PAHs | GC | 149.5 | 32.7 | 99.8 | 43.2 | 70.7 | 8.0 | 76.1 | 4.2 | 66.5 | 10.4 |
| 258 | Fluoranthene | PAHs | GC | 132.1 | 21.7 | 107.7 | 51.2 | 76.6 | 17.9 | 73.3 | 12.6 | 63.7 | 7.6 |
| 259 | Fluorene | PAHs | GC | 144.5 | 17.4 | 139.6 | 39.5 | 97.8 | 14.8 | 90.2 | 5.1 | 88.9 | 9.5 |
| 260 | Naphthalene | PAHs | GC | 125.7 | 16.1 | 132.0 | 33.1 | 93.4 | 10.9 | 89.1 | 9.1 | 88.6 | 6.5 |
| 261 | Phenanthrene | PAHs | GC | 128.5 | 11.6 | 147.1 | 37.3 | 95.7 | 16.2 | 94.6 | 5.4 | 81.7 | 14.1 |
| 262 | Pyrene | PAHs | GC | 132.9 | 17.5 | 106.8 | 48.5 | 74.0 | 12.4 | 76.5 | 5.7 | 67.4 | 7.8 |
| 263 | Brodifacoum | ARs | LC | 66.6 | 6.7 | 24.0 | 21.1 | 75.2 | 3.1 | 84.1 | 3.4 | 78.0 | 2.1 |
| 264 | Bromadiolone | ARs | LC | 69.5 | 2.4 | 43.1 | 4.5 | 68.5 | 2.7 | 73.1 | 3.2 | 63.5 | 3.7 |
| 265 | Coumatetralyl | ARs | LC | 69.4 | 1.9 | 57.5 | 4.6 | 69.2 | 3.7 | 75.0 | 3.5 | 69.4 | 2.5 |
| 266 | Difenacoum | ARs | LC | 79.6 | 3.3 | 31.6 | 5.8 | 87.8 | 2.0 | 91.5 | 1.2 | 94.9 | 0.8 |
| 267 | Difetihalone | ARs | LC | 68.6 | 9.1 | 27.7 | 8.4 | 70.2 | 4.6 | 85.8 | 2.1 | 76.9 | 3.5 |
| 268 | Flocoumafen | ARs | LC | 71.4 | 3.5 | 33.6 | 4.0 | 74.0 | 6.1 | 79.4 | 0.6 | 75.4 | 2.6 |
| 269 | Warfarin | ARs | LC | 80.4 | 5.1 | 81.6 | 5.7 | 74.9 | 3.8 | 77.2 | 3.9 | 75.1 | 3.7 |
| 270 | Albendazole | PhACs | LC | 56.6 | 6.5 | 46.9 | 1.0 | 67.6 | 1.6 | 71.6 | 1.2 | 65.4 | 1.6 |
| 271 | Cefuroxima axetil | PhACs | LC | 72.2 | 2.3 | 57.5 | 3.1 | 63.7 | 4.2 | 65.2 | 1.4 | 64.8 | 0.8 |

(*continued on next page*)

**Table 2** (*continued*)

| N° | Compound | Group | Technique | Soil A | | Soil B | | Soil C | | Soil D | | Soil E | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD | %REC | %RSD |
| 272 | Chloramphenicol | PhACs | LC | 88.0 | 7.3 | 82.2 | 9.3 | 76.9 | 11.6 | 84.2 | 14.1 | 77.6 | 8.4 |
| 273 | Cloxacillin | PhACs | LC | 58.3 | 17.6 | 87.8 | 9.8 | 36.9 | 5.0 | 33.6 | 12.0 | 62.3 | 2.8 |
| 274 | Cortiscosterone 21 acetate | PhACs | LC | 74.9 | 2.1 | 62.7 | 5.2 | 66.3 | 10.0 | 66.9 | 2.3 | 63.9 | 4.4 |
| 275 | Dexamethasone | PhACs | LC | 54.5 | 9.4 | 66.0 | 3.4 | 60.4 | 1.7 | 46.1 | 4.4 | 56.3 | 3.1 |
| 276 | Diclofenac | PhACs | LC | 60.0 | 19.2 | 69.9 | 2.8 | 61.0 | 7.2 | 66.3 | 3.1 | 64.2 | 6.1 |
| 277 | Eprinomectin | PhACs | LC | 71.1 | 8.1 | 46.0 | 28.2 | 69.4 | 4.8 | 60.5 | 2.6 | 57.6 | 5.0 |
| 278 | Fenbendazole | PhACs | LC | 65.6 | 9.2 | 46.7 | 3.3 | 62.8 | 2.8 | 65.3 | 1.4 | 59.4 | 0.8 |
| 279 | Flunixin | PhACs | LC | 47.0 | 2.6 | 30.4 | 3.1 | 53.1 | 2.9 | 44.9 | 2.7 | 65.6 | 1.6 |
| 280 | Imipenem | PhACs | LC | 102.4 | 6.4 | 76.7 | 16.0 | 73.7 | 17.7 | 83.6 | 11.7 | 83.7 | 10.3 |
| 281 | Josamycin | PhACs | LC | 52.7 | 3.7 | 63.7 | 3.6 | 37.2 | 3.8 | 20.9 | 4.5 | 58.4 | 2.0 |
| 282 | Ketoprofen | PhACs | LC | 69.4 | 3.8 | 80.3 | 4.2 | 66.6 | 1.8 | 66.4 | 1.2 | 65.0 | 3.4 |
| 283 | Mebendazole | PhACs | LC | 61.5 | 3.6 | 65.5 | 2.6 | 69.1 | 2.3 | 69.6 | 1.2 | 65.6 | 2.1 |
| 284 | Mefenamic acid | PhACs | LC | 48.3 | 20.9 | 66.3 | 5.0 | 41.5 | 13.0 | 57.5 | 8.1 | 45.1 | 8.3 |
| 285 | Metronidazole | PhACs | LC | 69.8 | 8.5 | 45.6 | 5.0 | 65.8 | 2.8 | 64.9 | 1.8 | 52.8 | 2.2 |
| 286 | Moxidectin | PhACs | LC | 67.5 | 8.5 | 26.1 | 15.1 | 54.3 | 9.1 | 74.4 | 4.6 | 71.3 | 2.5 |
| 287 | Naproxen | PhACs | LC | 68.1 | 5.0 | 73.5 | 4.3 | 68.7 | 4.9 | 69.2 | 5.8 | 63.5 | 5.2 |
| 288 | Oxfendazole | PhACs | LC | 46.9 | 3.8 | 54.3 | 2.7 | 64.3 | 2.3 | 57.2 | 0.4 | 63.1 | 2.7 |
| 289 | Penicilina V | PhACs | LC | 35.6 | 13.5 | 59.8 | 5.7 | 27.1 | 7.7 | 22.8 | 3.6 | 51.5 | 5.1 |
| 290 | Sulfacetamide | PhACs | LC | 60.3 | 7.8 | 51.4 | 4.0 | 63.9 | 0.7 | 67.0 | 3.1 | 47.0 | 2.0 |
| 291 | Sulfacloropiridacine | PhACs | LC | 64.2 | 4.7 | 56.4 | 5.5 | 69.5 | 3.6 | 77.3 | 1.8 | 54.4 | 3.2 |
| 292 | Sulfadiacine | PhACs | LC | 58.8 | 4.8 | 54.1 | 3.8 | 62.8 | 2.4 | 63.8 | 1.5 | 51.9 | 3.0 |
| 293 | Sulfadimetoxine | PhACs | LC | 69.6 | 3.0 | 73.8 | 4.8 | 66.5 | 1.5 | 69.7 | 1.1 | 62.5 | 1.6 |
| 294 | Sulfadoxine | PhACs | LC | 68.2 | 1.6 | 70.1 | 2.3 | 69.3 | 1.4 | 72.6 | 1.3 | 64.9 | 1.1 |
| 295 | Sulfameracine | PhACs | LC | 61.8 | 3.3 | 57.2 | 4.8 | 64.7 | 2.4 | 65.5 | 1.2 | 54.7 | 1.6 |
| 296 | Sulfametacine | PhACs | LC | 66.0 | 3.6 | 61.0 | 4.6 | 68.1 | 2.5 | 70.7 | 1.3 | 61.3 | 3.8 |
| 297 | Sulfametizole | PhACs | LC | 45.3 | 11.6 | 39.9 | 3.4 | 65.3 | 2.4 | 71.8 | 2.3 | 52.4 | 3.2 |
| 298 | Sulfametoxazole | PhACs | LC | 77.9 | 3.3 | 70.7 | 4.8 | 77.2 | 2.4 | 79.9 | 2.0 | 62.9 | 2.6 |
| 299 | Sulfametoxipiridacine | PhACs | LC | 57.7 | 4.4 | 53.4 | 5.8 | 67.7 | 1.7 | 69.6 | 1.8 | 57.9 | 3.1 |
| 300 | Sulfamonomethoxine | PhACs | LC | 60.5 | 2.8 | 55.5 | 3.8 | 63.9 | 2.4 | 70.6 | 2.1 | 58.0 | 1.5 |
| 301 | Sulfanilamide | PhACs | LC | 59.4 | 23.7 | 20.1 | 3.6 | 58.1 | 8.3 | 81.3 | 6.1 | 22.3 | 6.5 |
| 302 | Sulfapiridine | PhACs | LC | 57.2 | 4.6 | 50.4 | 3.7 | 68.0 | 2.2 | 70.0 | 3.2 | 57.9 | 3.9 |
| 303 | Sulfaquinoxaline | PhACs | LC | 61.8 | 3.0 | 63.1 | 4.7 | 66.4 | 1.5 | 68.4 | 2.4 | 60.9 | 0.5 |
| 304 | Sulfisoxazole | PhACs | LC | 40.4 | 38.1 | 66.4 | 3.3 | 54.7 | 3.8 | 70.3 | 1.4 | 38.8 | 8.1 |
| 305 | Tolfenamic acid | PhACs | LC | 79.4 | 4.9 | 60.6 | 2.8 | 77.9 | 9.0 | 90.1 | 6.6 | 85.5 | 6.2 |

[a]PBDE – Polybrominated diphenyl ethers, OCP – Organochlorine pesticides, PAH – Polycyclic aromatic hydrocarbon, PCB – Polychlorinated biphenyl, PhACs – Pharmaceuticals Active Compounds, ARs – Anticoagulant Rodenticides, P-IS – Procedural Internal Standard.

[b]Gas chromatography (GC) or liquid chromatography (LC), both coupled with tandem triple quadrupole mass spectrometry.

**Table 3**
Multiplying factors for the correction of the concentration found after the application of the analytical method to sandy loam soil (B), sandy clay soil (C), clay soil (D), and loamy sand soil (E).

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 1 | 4,4′-Dichlorobenzophenone | Pesticides, OCPs | GC | | 1.9 | 1.7 | 1.3 |
| 2 | 4,4′-Dicofol | Pesticides, OCPs | GC | 1.4 | 1.6 | 1.6 | 3.5 |
| 3 | Abamectine | Pesticides | LC | 2.1 | | | |
| 4 | Acephate | Pesticides | LC | | | 1.5 | |
| 5 | Acetamiprid | Pesticides | LC | | | | |
| 6 | Acrinathrin | Pesticides | LC | 2.1 | | | |
| 7 | Aldicarb | Pesticides | LC | | | | |
| 8 | Aldicarb sulfone | Pesticides | LC | | | | |
| 9 | Atrazine | Pesticides | LC | 1.4 | | | |
| 10 | Azinphos methyl | Pesticides | LC | | | | |
| 11 | Azoxystrobin | Pesticides | LC | | | | 1.3 |
| 12 | Benalaxyl | Pesticides | LC | 1.3 | | | |
| 13 | Bendiocarb | Pesticides | LC | | | | |
| 14 | Bifenthrin | Pesticides | GC | | 1.6 | 1.5 | 1.7 |
| 15 | Bitertanol | Pesticides | LC | | | | 1.3 |
| 16 | Boscalid | Pesticides | GC | 0.6 | 1.4 | 1.5 | 1.7 |
| 17 | Bromopropylate | Pesticides | GC | | 1.4 | 1.4 | 1.5 |
| 18 | Bromuconazole | Pesticides | LC | 1.3 | | | |
| 19 | Bupirimate | Pesticides | LC | 1.3 | | | |
| 20 | Cadusafos | Pesticides | LC | 1.7 | | | |
| 21 | Carbaryl | Pesticides | LC | | | | |
| 22 | Carbofuran | Pesticides | LC | 1.3 | | | 1.3 |
| 23 | Carbofuran-3-hydroxy | Pesticides | LC | | | | |
| 24 | Chlorantraniliprole | Pesticides | LC | | | | 1.3 |
| 25 | Chlorfenapyr | Pesticides | GC | | 1.4 | 1.5 | 1.9 |
| 26 | Chlorobenzilate | Pesticides | GC | | 1.3 | 1.3 | 1.6 |
| 27 | Chlorpropham | Pesticides | GC | | 1.4 | 1.5 | 1.6 |
| 28 | Chlorpyrifos | Pesticides | GC | 1.3 | 1.4 | 1.5 | 1.8 |
| 29 | Chlorpyrifos methyl | Pesticides | GC | | 1.5 | 1.5 | 2.1 |
| 30 | Chlorthal dimethyl | Pesticides | GC | | 1.4 | 1.3 | 1.5 |
| 31 | Clofentezine | Pesticides | LC | 1.7 | | | |
| 32 | Clothianidin | Pesticides | LC | 1.3 | | | |
| 33 | Coumachlor | Pesticides | LC | | | | |
| 34 | Coumaphos | Pesticides | LC | | | | |
| 35 | Cyazofamid | Pesticides | LC | 1.3 | | | 1.4 |
| 36 | Cyflufenamid | Pesticides | LC | 1.5 | | | |
| 37 | Cyfluthrin | Pesticides | GC | 1.3 | 1.6 | 1.7 | 2.1 |
| 38 | Cyhalothrin (lambda isomer) | Pesticides | LC | 2.8 | | | 1.3 |
| 39 | Cymoxanil | Pesticides | LC | | | | |
| 40 | Cypermethrin | Pesticides | GC | | 1.7 | 1.9 | 2.3 |
| 41 | Cyproconazole | Pesticides | LC | | | | |
| 42 | Cyprodinil | Pesticides | GC | | 1.4 | 1.4 | 1.6 |
| 43 | Deltamethrin | Pesticides | GC | | 1.6 | 1.9 | 2.5 |
| 44 | Demeton-S-methyl | Pesticides | LC | 1.3 | | | |
| 45 | Demeton-S-methyl-sulfone (Dioxydemeton) | Pesticides | LC | | | | |
| 46 | Diazinon | Pesticides | GC | | 1.3 | 1.3 | 1.6 |
| 47 | Dichlofluanid | Pesticides | GC | 1.3 | 1.5 | 1.5 | 2.1 |
| 48 | Dichloran | Pesticides | GC | 1.3 | 1.6 | 1.9 | 2.0 |
| 49 | Diethathyl ethyl | Pesticides | LC | 1.3 | | | |
| 50 | Diethofencarb | Pesticides | LC | | | | |
| 51 | Difenoconazole | Pesticides | LC | 1.3 | | | |
| 52 | Diflubenzuron | Pesticides | LC | | | | |
| 53 | Diflufenican | Pesticides | LC | 1.6 | | | |
| 54 | Dimethenamide | Pesticides | LC | | | | |
| 55 | Dimethoate | Pesticides | LC | | | | |

(*continued on next page*)

**Table 3** (*continued*)

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 56 | Dimethomorph | Pesticides | LC | | 1.3 | 1.3 | 1.3 |
| 57 | Diniconazole-M | Pesticides | LC | 1.3 | 1.3 | | 1.4 |
| 58 | Dinocap | Pesticides | LC | 2.2 | 1.7 | 1.6 | 1.5 |
| 59 | Diphenylamine | Pesticides | LC | | | 1.3 | |
| 60 | Endosulfan alfa | Pesticides, OCPs | GC | 1.3 | 1.5 | 1.4 | 1.7 |
| 61 | Endosulfan beta | Pesticides, OCPs | GC | 1.3 | 1.5 | 1.6 | 1.8 |
| 62 | EPN | Pesticides | LC | 1.7 | | | |
| 63 | Epoxiconazole | Pesticides | LC | | | | 1.4 |
| 64 | Esfenvalerate | Pesticides | GC | | 1.7 | 1.8 | 2.4 |
| 65 | Ethion | Pesticides | LC | 2.3 | | | |
| 66 | Ethofumesate | Pesticides | GC | 1.3 | 1.5 | 1.3 | 1.6 |
| 67 | Ethoprophos | Pesticides | LC | 1.3 | | | |
| 68 | Etofenprox | Pesticides | LC | 2.0 | 1.4 | 2.9 | 1.5 |
| 69 | Etoxazole | Pesticides | LC | 2.5 | | | |
| 70 | Fenamidone | Pesticides | LC | | | | 1.3 |
| 71 | Fenamiphos | Pesticides | LC | 1.3 | | | |
| 72 | Fenamiphos sulfone | Pesticides | LC | | | 1.3 | 1.3 |
| 73 | Fenamiphos sulfoxide | Pesticides | LC | | | 1.4 | |
| 74 | Fenarimol | Pesticides | GC | | 1.5 | 1.6 | 1.6 |
| 75 | Fenazaquin | Pesticides | LC | 3.5 | | | |
| 76 | Fenbuconazole | Pesticides | LC | | | | |
| 77 | Fenbutatin oxide | Pesticides | LC | 1.9 | 1.8 | 1.3 | |
| 78 | Fenitrothion | Pesticides | GC | | 1.4 | 1.5 | 2.1 |
| 79 | Fenoxycarb | Pesticides | LC | 1.3 | | | |
| 80 | Fenpropathrin | Pesticides | LC | 3.0 | | | 1.3 |
| 81 | Fenpropimorph | Pesticides | LC | | | 1.7 | |
| 82 | Fenpyroximate | Pesticides | LC | 3.2 | | | |
| 83 | Fenthion | Pesticides | LC | 1.4 | | | 1.3 |
| 84 | Fenthion oxon | Pesticides | LC | | | | |
| 85 | Fenthion oxon sulfone | Pesticides | LC | | | | |
| 86 | Fenthion oxon sulfoxide | Pesticides | LC | | | 1.3 | |
| 87 | Fenthion sulfone | Pesticides | LC | | | | |
| 88 | Fenthion sulfoxide | Pesticides | LC | | | | |
| 89 | Fenvalerate | Pesticides | GC | | 1.6 | 1.9 | 2.2 |
| 90 | Fipronil | Pesticides | LC | | | | |
| 91 | Fipronil sulfide | Pesticides | GC | | | | 1.3 |
| 92 | Fluazinam | Pesticides | LC | | | | |
| 93 | Flubendiamide | Pesticides | LC | | | | |
| 94 | Flucythrinate | Pesticides | GC | | 1.5 | 1.7 | 2.0 |
| 95 | Fludioxonil | Pesticides | LC | | | | |
| 96 | Flufenoxuron | Pesticides | LC | 1.8 | | | |
| 97 | Fluopyram | Pesticides | LC | 1.3 | | | |
| 98 | Fluquinconazole | Pesticides | LC | 1.3 | | | |
| 99 | Flusilazole | Pesticides | LC | 1.3 | | | 1.3 |
| 100 | Flutolanil | Pesticides | LC | 1.3 | | | |
| 101 | Flutriafol | Pesticides | LC | | | | |
| 102 | Fluvalinate tau | Pesticides | LC | 1.9 | | 1.3 | 1.4 |
| 103 | Fonofos | Pesticides | GC | | 1.4 | 1.3 | 1.6 |
| 104 | Fosthiazate | Pesticides | LC | | | | |
| 105 | Hexaconazole | Pesticides | LC | 1.3 | | | |
| 106 | Hexaflumuron | Pesticides | LC | | | 1.3 | 1.4 |
| 107 | Hexythiazox | Pesticides | LC | 2.7 | | | |
| 108 | Imidacloprid | Pesticides | LC | | | | |
| 109 | Indoxacarb | Pesticides | LC | 1.3 | | | 1.3 |
| 110 | Iprodione | Pesticides | GC | 1.4 | 1.8 | 1.9 | 2.3 |
| 111 | Iprovalicarb | Pesticides | LC | | | | |
| 112 | Isocarbophos | Pesticides | GC | | 1.5 | 1.5 | 1.8 |

(*continued on next page*)

**Table 3** (*continued*)

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 113 | Isofenphos methyl | Pesticides | LC | 1.4 | | | |
| 114 | Isoprothiolane | Pesticides | LC | 1.3 | | | |
| 115 | Kresoxim methyl | Pesticides | LC | 1.5 | | | |
| 116 | Linuron | Pesticides | LC | 1.3 | | | |
| 117 | Lufenuron | Pesticides | LC | 1.7 | | | |
| 118 | Malaoxon | Pesticides | LC | | | | |
| 119 | Malathion | Pesticides | LC | 1.3 | | | |
| 120 | Mandipropamid | Pesticides | LC | | | | 1.3 |
| 121 | Mefenoxam (metalaxyl-M) | Pesticides | LC | | | | |
| 122 | Mepanipyrim | Pesticides | LC | 1.5 | | | |
| 123 | Metaflumizone | Pesticides | LC | | | | 1.3 |
| 124 | Metalaxyl | Pesticides | GC | | 1.5 | 1.4 | 1.6 |
| 125 | Metaldehyde | Pesticides | LC | | | | |
| 126 | Metconazole | Pesticides | LC | 1.3 | | | |
| 127 | Methamidophos | Pesticides | LC | | | | |
| 128 | Methidathion | Pesticides | LC | 1.3 | | | |
| 129 | Methiocarb | Pesticides | LC | 1.3 | | | |
| 130 | Methiocarb sulfone | Pesticides | LC | | | | |
| 131 | Methiocarb sulfoxide | Pesticides | LC | | | | |
| 132 | Methomyl | Pesticides | LC | | | | |
| 133 | Methomyl oxime | Pesticides | LC | | | | |
| 134 | Methoxyfenozide | Pesticides | LC | | | | |
| 135 | Metrafenone | Pesticides | LC | 1.3 | | | |
| 136 | Mevinphos (phosdrin) | Pesticides | LC | | | | |
| 137 | Monocrotophos | Pesticides | LC | | | | |
| 138 | Myclobutanil | Pesticides | LC | | 1.3 | 1.3 | 1.3 |
| 139 | N,N-Dimethyl-N'-p-tolylsulphamide (DMST) | Pesticides | LC | | | | 1.3 |
| 140 | N,N-dimethylformamidine (DMF) | Pesticides | LC | 1.3 | | | 1.3 |
| 141 | Nuarimol | Pesticides | LC | | | | |
| 142 | Ofurace | Pesticides | LC | | | | 1.3 |
| 143 | Omethoate | Pesticides | LC | | | 1.4 | |
| 144 | Oxadixyl | Pesticides | LC | | | | |
| 145 | Oxamyl | Pesticides | LC | | | | |
| 146 | Oxamyl oxime | Pesticides | LC | | | | 1.3 |
| 147 | Oxyfluorfen | Pesticides | GC | | 1.3 | 1.5 | 1.6 |
| 148 | Paclobutrazol | Pesticides | LC | | | | |
| 149 | Paraoxon methyl | Pesticides | GC | | 1.4 | 1.5 | 1.8 |
| 150 | Parathion ethyl | Pesticides | GC | | 1.3 | 1.6 | 1.8 |
| 151 | Parathion methyl | Pesticides | GC | | 1.4 | 1.6 | 2.2 |
| 152 | Penconazole | Pesticides | LC | 1.3 | | | |
| 153 | Pencycuron | Pesticides | LC | 1.6 | | | 1.4 |
| 154 | Pendimethalin | Pesticides | LC | 2.3 | | | |
| 155 | Permethrin | Pesticides | GC | | 1.4 | 1.4 | 1.8 |
| 156 | Phosalone | Pesticides | LC | 1.4 | | | |
| 157 | Phosmet | Pesticides | LC | | | | |
| 158 | Phosmet oxon | Pesticides | LC | | | | |
| 159 | Phthalimide (Folpet deg) | Pesticides | GC | | | 1.3 | |
| 160 | Pirimicarb | Pesticides | LC | | | | 0.7 |
| 161 | Pirimiphos ethyl | Pesticides | LC | 2.2 | | | |
| 162 | Pirimiphos methyl | Pesticides | LC | 1.7 | | | |
| 163 | Prochloraz | Pesticides | LC | 1.4 | | | |
| 164 | Procymidone | Pesticides | GC | 1.3 | 1.4 | 1.3 | 1.6 |
| 165 | Profenofos | Pesticides | LC | 2.0 | | | |
| 166 | Propargite | Pesticides | LC | 2.7 | | | |
| 167 | Propoxur | Pesticides | LC | | | | |
| 168 | Propyzamide (pronamide) | Pesticides | LC | 1.3 | | | |

(*continued on next page*)

**Table 3** (*continued*)

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 169 | Proquinazid | Pesticides | LC | 3.7 | | | |
| 170 | Prothioconazole-desthio | Pesticides | LC | 1.4 | | | 1.3 |
| 171 | Prothiophos | Pesticides | GC | 1.3 | 1.5 | 1.5 | 1.9 |
| 172 | Pyraclostrobin | Pesticides | LC | 1.4 | | | |
| 173 | Pyrazophos | Pesticides | LC | | | | 1.3 |
| 174 | Pyridaben | Pesticides | LC | 3.0 | | | |
| 175 | Pyridaphenthion | Pesticides | LC | | 1.3 | 1.3 | 1.4 |
| 176 | Pyrimethanil | Pesticides | GC | | 1.4 | 1.4 | 1.6 |
| 177 | Pyriproxifen | Pesticides | LC | 2.5 | | | |
| 178 | Quinalphos | Pesticides | LC | 1.3 | | | |
| 179 | Quinoxyfen | Pesticides | LC | 3.5 | | | |
| 180 | Rotenone | Pesticides | LC | | | | 1.3 |
| 181 | Simazine | Pesticides | LC | 1.4 | | | |
| 182 | Spirodiclofen | Pesticides | LC | 3.2 | | | |
| 183 | Spiromesifen | Pesticides | LC | 2.6 | | | 1.3 |
| 184 | Spirotetramat | Pesticides | LC | 0.7 | | | 1.3 |
| 185 | Spirotetramat-enol | Pesticides | LC | 0.7 | | | 1.3 |
| 186 | Spiroxamine | Pesticides | GC | 0.6 | 1.7 | 3.6 | 0.7 |
| 187 | Tebuconazole | Pesticides | LC | 1.3 | | | |
| 188 | Tebufenocide | Pesticides | LC | 1.3 | | | |
| 189 | Tebufenpyrad | Pesticides | LC | 2.1 | | | |
| 190 | Teflubenzuron (artifact 3) | Pesticides | GC | | 1.5 | 1.6 | 1.6 |
| 191 | Tefluthrin | Pesticides | GC | | 1.3 | 1.4 | 1.6 |
| 192 | Telodrin | Pesticides | GC | 1.3 | 1.6 | 1.5 | 1.7 |
| 193 | Terbufos | Pesticides | GC | | 1.4 | 1.4 | 1.7 |
| 194 | Terbuthylazine | Pesticides | LC | 1.5 | | | |
| 195 | Tetrachlorvinphos | Pesticides | LC | 1.3 | | | |
| 196 | Tetraconazole | Pesticides | LC | 1.3 | | | |
| 197 | Tetradifon | Pesticides | GC | | 1.6 | 1.6 | 2.0 |
| 198 | Tetramethrin | Pesticides | GC | | 2.0 | 1.9 | 2.3 |
| 199 | Thiacloprid | Pesticides | LC | | | | |
| 200 | Thiamethoxam | Pesticides | LC | 1.3 | | 1.3 | 1.3 |
| 201 | Thiodicarb | Pesticides | LC | | | 1.3 | |
| 202 | Tolclofos methyl | Pesticides | GC | | 1.4 | 1.3 | 1.9 |
| 203 | Tolylfluanid | Pesticides | GC | 1.3 | 1.6 | 1.7 | 2.2 |
| 204 | Triadimefon | Pesticides | LC | | | | |
| 205 | Triadimenol | Pesticides | LC | | | | |
| 206 | Triazophos (hostathion) | Pesticides | LC | | | | |
| 207 | Trichlorfon | Pesticides | LC | 1.3 | | | |
| 208 | Trifloxystrobin | Pesticides | LC | 1.7 | | | |
| 209 | Triflumizole | Pesticides | LC | 1.7 | | | |
| 210 | Triflumuron | Pesticides | LC | 1.5 | | | |
| 211 | Trifluralin | Pesticides | GC | | | 1.4 | 1.5 |
| 212 | Triticonazole | Pesticides | LC | | | | |
| 213 | Vinclozolin | Pesticides | GC | | 1.5 | 1.4 | 1.7 |
| 214 | Aldrin | OCPs | GC | | 1.4 | 1.3 | 1.5 |
| 215 | Dichlorodiphenyldichloroethane (p,p' DDD) | OCPs | GC | 1.3 | 1.4 | 1.5 | |
| 216 | Dichlorodiphenyldichloroethylene (p,p' DDE) | OCPs | GC | 1.3 | | 1.3 | |
| 217 | Dieldrin | OCPs | GC | 1.3 | | 1.4 | 1.9 |
| 218 | Endrin | OCPs | GC | 1.3 | 1.4 | 1.4 | 1.5 |
| 219 | Heptachlor | OCPs | GC | 1.4 | 1.7 | 1.5 | 1.8 |
| 220 | Hexachlorobenzene | OCPs | GC | 1.3 | 1.4 | 1.3 | 1.5 |
| 221 | Hexachlorocyclohexane (alpha) | OCPs | GC | 1.3 | 1.6 | 1.6 | 1.9 |
| 222 | Hexachlorocyclohexane (gamma, lindane) | OCPs | GC | 1.4 | 1.8 | 1.8 | 1.7 |
| 223 | Hexachlorocyclohexano (beta) | OCPs | GC | 1.4 | 2.0 | 1.8 | 8.4 |

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**Table 3** (*continued*)

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 224 | Hexaclorociclohexano (delta) | OCPs | GC | | 1.4 | 1.4 | 2.0 |
| 225 | Mirex | OCPs | GC | 1.3 | 1.6 | 1.5 | 1.5 |
| 226 | PCB 28 | PCBs | GC | | 1.4 | 1.3 | 1.5 |
| 227 | PCB 52 | PCBs | GC | | 1.3 | | 1.4 |
| 228 | PCB 77 | PCBs | GC | 1.3 | 1.3 | 1.3 | 1.4 |
| 229 | PCB 81 | PCBs | GC | 1.3 | 1.4 | 1.3 | 1.5 |
| 230 | PCB 101 | PCBs | GC | 1.3 | 1.4 | 1.4 | 1.5 |
| 231 | PCB 105 | PCBs | GC | 1.3 | 1.3 | 1.3 | 1.4 |
| 232 | PCB 114 | PCBs | GC | | 1.3 | | 1.5 |
| 233 | PCB 118 | PCBs | GC | 1.3 | | | 1.4 |
| 234 | PCB 123 | PCBs | GC | | | | 1.3 |
| 235 | PCB 126 | PCBs | GC | 1.3 | 1.4 | 1.3 | 1.4 |
| 236 | PCB 138 | PCBs | GC | 1.3 | 1.3 | 1.3 | 1.3 |
| 237 | PCB 153 | PCBs | GC | 1.3 | 1.3 | | 1.5 |
| 238 | PCB 156 | PCBs | GC | 1.3 | 1.4 | 1.3 | 1.4 |
| 239 | PCB 157 | PCBs | GC | 1.4 | 1.4 | 1.3 | 1.5 |
| 240 | PCB 167 | PCBs | GC | 1.3 | 1.3 | | 1.3 |
| 241 | PCB 169 | PCBs | GC | 1.3 | 1.3 | 1.3 | 1.4 |
| 242 | PCB 180 | PCBs | GC | | 1.3 | 1.3 | 1.5 |
| 243 | PCB 189 | PCBs | GC | 1.3 | 1.3 | 1.3 | 1.3 |
| 244 | PBDE 28 | PBDEs | GC | | | | |
| 245 | PBDE 47 | PBDEs | GC | | | | 1.3 |
| 246 | PBDE 85 | PBDEs | GC | | | | 1.3 |
| 247 | PBDE 99 | PBDEs | GC | | | | 1.3 |
| 248 | PBDE 100 | PBDEs | GC | | | | |
| 249 | PBDE 153 | PBDEs | GC | | | | |
| 250 | PBDE 154 | PBDEs | GC | | | | |
| 251 | PBDE 183 | PBDEs | GC | 1.3 | | 1.3 | 1.4 |
| 252 | Acenaphthene | PAHs | GC | | 1.5 | 1.6 | 1.6 |
| 253 | Acenaphthylene | PAHs | GC | | 1.5 | 1.5 | 1.5 |
| 254 | Anthracene | PAHs | GC | | 1.3 | 1.3 | 1.5 |
| 255 | Benzo[a]anthracene | PAHs | GC | 1.3 | 1.8 | 1.7 | 1.9 |
| 256 | Benzo[b]fluoranthene | PAHs | GC | | 1.7 | 2.1 | 2.7 |
| 257 | Chrysene | PAHs | GC | 1.5 | 2.1 | 2.0 | 2.2 |
| 258 | Fluoranthene | PAHs | GC | 1.3 | 1.7 | 1.8 | 2.1 |
| 259 | Fluorene | PAHs | GC | | 1.5 | 1.6 | 1.6 |
| 260 | Naphthalene | PAHs | GC | | 1.3 | 1.4 | 1.4 |
| 261 | Phenanthrene | PAHs | GC | | 1.3 | 1.4 | 1.6 |
| 262 | Pyrene | PAHs | GC | 1.3 | 1.8 | 1.7 | 2.0 |
| 263 | Brodifacoum | ARs | LC | 2.8 | | 0.7 | |
| 264 | Bromadiolone | ARs | LC | 1.6 | | | |
| 265 | Coumatetralyl | ARs | LC | 1.3 | | | |
| 266 | Difenacoum | ARs | LC | 2.5 | | | |
| 267 | Difetihalone | ARs | LC | 2.5 | | 0.7 | |
| 268 | Flocoumafen | ARs | LC | 2.1 | | | |
| 269 | Warfarin | ARs | LC | | | | |
| 270 | Albendazole | PhACs | LC | 1.3 | | 0.7 | |
| 271 | Cefuroxima axetil | PhACs | LC | 1.3 | | | |
| 272 | Chloramphenicol | PhACs | LC | | | | |
| 273 | Cloxacillin | PhACs | LC | 0.7 | 1.6 | 1.7 | |
| 274 | Cortiscosterone 21 acetate | PhACs | LC | | | | |
| 275 | Dexamethasone | PhACs | LC | | | | |
| 276 | Diclofenac | PhACs | LC | | | | |
| 277 | Eprinomectin | PhACs | LC | 1.5 | | | 1.3 |
| 278 | Fenbendazole | PhACs | LC | 1.4 | | | |
| 279 | Flunixin | PhACs | LC | 1.5 | | | 0.7 |
| 280 | Imipenem | PhACs | LC | 1.3 | 1.4 | 1.3 | 1.3 |
| 281 | Josamycin | PhACs | LC | | 1.4 | 2.5 | |

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**Table 3** (*continued*)

| N° | Compound | Group | Technique | Factor B | Factor C | Factor D | Factor E |
|---|---|---|---|---|---|---|---|
| 282 | Ketoprofen | PhACs | LC | | | | |
| 283 | Mebendazole | PhACs | LC | | | | |
| 284 | Mefenamic acid | PhACs | LC | 0.7 | | | |
| 285 | Metronidazole | PhACs | LC | 1.5 | | | 1.3 |
| 286 | Moxidectin | PhACs | LC | 2.6 | 1.3 | | |
| 287 | Naproxen | PhACs | LC | | | | |
| 288 | Oxfendazole | PhACs | LC | | 0.7 | | 0.7 |
| 289 | Penicilina V | PhACs | LC | 0.6 | 1.3 | 1.6 | 0.7 |
| 290 | Sulfacetamide | PhACs | LC | | | | 1.3 |
| 291 | Sulfacloropiridacine | PhACs | LC | | | | |
| 292 | Sulfadiacine | PhACs | LC | | | | |
| 293 | Sulfadimetoxine | PhACs | LC | | | | |
| 294 | Sulfadoxine | PhACs | LC | | | | |
| 295 | Sulfameracine | PhACs | LC | | | | |
| 296 | Sulfametacine | PhACs | LC | | | | |
| 297 | Sulfametizole | PhACs | LC | | 0.7 | 0.6 | |
| 298 | Sulfametoxazole | PhACs | LC | | | | 1.3 |
| 299 | Sulfametoxipiridacine | PhACs | LC | | | | |
| 300 | Sulfamonomethoxine | PhACs | LC | | | | |
| 301 | Sulfanilamide | PhACs | LC | 3.0 | | 0.7 | 2.7 |
| 302 | Sulfapiridine | PhACs | LC | | | | |
| 303 | Sulfaquinoxaline | PhACs | LC | | | | |
| 304 | Sulfisoxazole | PhACs | LC | 0.6 | 0.7 | 0.6 | |
| 305 | Tolfenamic acid | PhACs | LC | 1.3 | | | |

Empty cell: when the calculated factor is between 0.8 and 1.2, it is considered that there is not enough difference, and no correction should be applied.

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## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mex.2021.101476.

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