A Method Scope Extension for the Simultaneous Analysis of POPs, Current-Use and Banned Pesticides, Rodenticides, and Pharmaceuticals in Liver. Application to Food Safety and Biomonitoring

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Abstract: The screening of hundreds of substances belonging to multiple chemical classes in liver is required in areas such as food safety or biomonitoring. We adapted a previous QuEChERS-based method in blood to the liver matrix and applied to these fields of study. The validation of the method allowed the inclusion of 351 contaminants, 80% with a LOQ < 2 ng/g. In the analysis of 42 consumer liver samples, we detected trace levels of 29 different contaminants. The most frequent and concentrated was 4,4’-DDE. POPs accounted for 66% of the compounds detected. In no case was the MRL reached for any of the contaminants detected. We also applied the method to 151 livers of wild birds to perform a biomonitoring pilot study in the Canary Islands. We detected 52 contaminants in 15 bird species. These were also mostly POPs, although high frequencies and concentrations of anticoagulant rodenticides (AR) and some other agricultural pesticides also stand out. POPs and AR contamination levels were significantly higher in terrestrial birds, raptors and particularly in nocturnal birds. Pesticide contamination levels were also higher in terrestrial birds, as well as in non-raptors and diurnal birds. The validated method is simple, robust, and sensitive and performs well in a variety of practical scenarios, where it can be carried out relatively quickly and inexpensively.

Keywords: persistent organic pollutants; agrochemicals; environmental pollution; QuEChERS; LC-MS/MS; GC-MS/MS

1. Introduction

Animal liver is a common matrix for the search of chemical residues for several reasons. On the one hand, the use of veterinary drugs, which is widely accepted in veterinary practice to treat bacterial infections, parasitism (internal or external), inflammation, and other animal diseases or their symptoms in livestock practice [1,2], may condition the appearance of residues of these chemicals in foods of animal origin, especially in the liver [3–5]. This is particularly relevant, since the presence of antimicrobial compounds can induce the spread of drug-resistant pathogenic bacterial strains or produce allergic reactions in humans [3,6]. In addition, pesticide products used in agriculture can leave...
residues in the raw materials used in the preparation of animal food, and these residues can in turn generate the appearance of residues in food products of animal origin [5], that can pose a serious risk to the health of consumers [5]. Therefore, to protect consumers from these undesirable effects, as a food safety measure, maximum residue limits (MRLs) have been established for many veterinary medicinal products and pesticides on a range of commodities of animal origin, including meat and meat products, liver, fish, honey, milk, and eggs [5,7]. According to the European regulations, the liver must be investigated for the presence of several hundred compounds, including pesticides commonly used in agriculture, pesticides already banned but of great environmental persistence, and also residues of veterinary drugs [7]. Although there are differences between countries, if we take as a common reference what is established in the Codex Alimentarius, there are at least maximum residue limits (MRLs) established for some 218 pesticides and for another 75 veterinary drugs (also including combinations of drugs and animal feed) [4,5].

On the other hand, wildlife lives in an environment that is increasingly contaminated with chemical substances (pesticides, industrial pollutants, wastewater, urban solid waste, etc.) and therefore can serve as first line indicators of the levels of pollutants and their possible health impacts [8–10]. Wildlife biomonitoring can provide important information about the bioavailability of contaminants in the environment humans share with these species [11–13], which supports the design of appropriate remediation strategies [14]. These data can result in substantial savings of limited remediation resources while maximizing the preservation of important natural areas and supporting effective site remediation [14]. The action of monitoring wildlife exposure to chemical contaminants is usually known as biomonitoring [10,15,16].

Like pesticides and pharmaceuticals, the annual use of anticoagulant rodenticides (ARs) for rodent control is measured in thousands of tons. This extensive use often leads to unintentional exposure of non-target animals, especially birds of prey, to these poisons, so there is a need for these substances to be included in biomonitoring studies. With regard to food safety, it should be noted that ARs are not authorized for use on edible crops within the EU, so the Codex Alimentarius Commission has not set MRLs for any of them, and residues of ARs are not expected to be present in any plant or animal products [17], and therefore they are not usually routinely investigated in food.

Whether it is for residue research in the context of food safety, or in the context of biomonitoring, the liver is an extremely interesting matrix, as it is one of the organs of that concentrates more quantity of chemicals [18]. The range of substances that it is interesting to investigate in one or another circumstance is quite similar, since the substances that are of concern from the point of view of food safety generally also concern from the point of view of the environmental pollution and their effects on wildlife [11,19–21]. The availability of multi-residue methods that are capable of accurately and simultaneously identifying and quantifying the concentration of any of these substances subjected to MRLs that may be present in the liver tissue is extremely interesting [5]. Therefore, it is necessary to develop multi-residue methods belonging to multiple chemical classes. In the case of biomonitoring, moreover, the challenge of detecting such a variety of potentially harmful substances in a complex matrix such as the liver is compounded by the fact that the amount of sample available is usually small [16].

Although there are numerous published multi-residue/multi-class methods for the determination of chemicals in animal liver, most focus either on the analysis of pesticides [22,23] or on the analysis of certain groups of veterinary drugs [24–26]. However, very few of the published methods address the simultaneous analysis of compounds from both classes [27], and are generally limited to a discrete number of compounds. Therefore, to cover the whole spectrum of compounds of interest in any of the fields (food safety and biomonitoring), it is usually necessary to apply several methods in a complementary manner, which consumes time, economic resources, and sample quantity, which may be limited in the case of wildlife.
The first part of this research consists of a validation of a method scope extension. The original method was developed for whole blood [16,28], and now it has been validated for liver matrix. But more interesting, probably, is the second part of our paper, in which we present and discuss the results of the application of this methodology to the two fields described. On the one hand we analyzed the residues of substances subjected to MRLs in 46 samples of liver intended for human consumption sampled, acquired in markets, supermarkets, and slaughterhouses. On the other hand, we applied the method to the biomonitoring of 151 wildlife specimens from the Canary Islands received in our service from mid-2020 to April 2021.

2. Materials and Methods

2.1. Certified Standards and Reagents

Methanol (MeOH, 99.9% purity), acetonitrile (ACN, 99.9% purity), and formic acid (FA, 98.0% purity), all LC-MS grade, were purchased from Honeywell (Charlotte, NC, USA). LC-Grade water (18.2 MΩ/cm) was purified by a MilliQ A10 Gradient system (Millipore, Molsheim, France). Ammonium acetate Optima LC-MS grade was purchased from Fisher (Fisher Scientific UK, Loughborough, UK). QuEChERS Extract Pouch, AOAC Method (6 g de magnesium sulphate and 1.5 g sodium acetate), were purchased in commercial premixes from Agilent Technologies (Palo Alto, CA, USA).

All certified standards (liquid or solid) of all the individual pollutants and deuterated compounds (P-ISs, procedural internal standards) were obtained from A2S—Analytical Standard Solutions (Staint Jean D’Illac, France), Sigma-Aldrich (Augsburg, Germany), CPA Chem (Stara Zagora, Bulgaria), European Pharmacopoeia Reference Standards (Strasbourg, France), Accustandard (New Haven, CT, USA), and Dr. Ehrenstorfer (Augsburg, Germany). All standards were from the highest purity available (93.1% to 99.8%). Individual 1 mg/mL stock solutions of each pollutant were prepared either dissolving or diluting certified standards in ACN, MeOH, water, or acetone (according to the solubility of substances), and stored no more than a year at −32 °C. The standard solutions were sorted, grouping by pesticide, pharmaceuticals, COPs, or procedural internal standards (pIS) to get four intermediate solutions at 1 µg/mL/each. Matrix-matched calibration and quality control points were fortified independently, to get 11 points between 0.4 to 40 ng/mL, with 4 quality controls at 1, 4, 10, and 20 ng/mL.

2.2. Liver for Method Validation

For the development, optimization, and validation of the analytical technique, we employed liver samples obtained from chickens of an organic farm. All the chickens were born in this facility, were healthy and had never been exposed to chemicals (no farms or agricultural facilities in the nearby, and no pharmacological treatments, according to the standards of the production mode), to avoid drug interference. The livers were obtained directly from the slaughterhouse, when these animals were slaughtered for consumption, and placed in 50 mL propylene tubes. Upon arrival at the laboratory, these samples were immediately stored at −24 °C until use.

2.3. Sample Preparation and Extraction

The QuEChERS method [29] is a matrix dispersion extraction method, which was initially developed for the analysis of pesticides in fruits and vegetables, but has proven to be versatile, allowing the analysis of many other compounds in complex matrices such as blood, milk, meat, eggs, and even soil [30,31]. We applied it to liver samples, for which it is first necessary to homogenize the liver before applying the QuEChERS extraction. For this purpose, one gram of liver sample was weighed into a tube suitable for homogenization with a Precellys Evolution homogenizer (Bertin Technologies, Rockville, Washington D.C., USA), operated at 6500 rpm, 2 × 30 s. After that, when needed, the fortification was performed, either for validation experiments, for calibration curves, or for the preparation of the quality controls (QC). Then, the homogenate was diluted with 4 mL ultrapure
water, and one milliliter of the diluted homogenate was placed in a 5 mL Eppendorf tube to be processed. Ten µL of pIS mix (acenaphthene-d10, atrazine-d5, carbenzadim-d3, chloryrifos-d10, chrysene-d12, cyromazine-d4, diazinon-d10, linuron-d3, PCB 200, phenanthrene-d10, and pirimicarb-d6) was added to all the tubes (either fortified or not) to reach a final concentration of 10 ng/mL. Next, anhydrous magnesium sulfate (480 mg) and sodium acetate (120 mg) were added to each sample tube, followed by 30 s of vortexing and 1 min of vertical manual shaking. Finally, the Eppendorf tubes were centrifuged for 5 min, at 4500 rpm and 2 °C. The supernatant was then filtered through a 0.2 µm Chromafil PET-20/15 syringe filter (polyester, certified for HPLC, Macherey-Nagel, Düren, Germany) into an amber vial directly, for sequential LC and GC-MS/MS analysis.

2.4. Instrumental Analysis

We found that two complementary analyses are required to detect and quantify the 351 compounds that finally could be included in this method. Thus, an analysis by gas chromatography coupled to triple quadrupole mass spectrometry (GC-MS/MS) is needed for the analysis of the most volatile compounds (mainly persistent organic pollutants and some less polar pesticides) and an analysis by liquid chromatography coupled to triple quadrupole mass spectrometry (LC-MS/MS) for the pharmaceuticals, the rodenticides, and the most polar pesticides.

2.4.1. GC-MS/MS

Gas chromatography was employed for the separation of 126 compounds using an Agilent 7890B gas chromatograph (Agilent Technologies, Palo Alto, CA, USA). Two Agilent J&W HP-5MS (5% cross-linked phenyl-methyl-polysiloxane, Agilent Technologies) ultra-inert fused silica capillary columns, with a total length of 30 m (15 + 15), a film thickness of 0.25 µm and 0.25 mm in diameter, were employed for the separations. The columns were joined by means of a purged joint to allow the application of the back-flushing technique that reduces the background noise and extends the column lifetime. An ultra-inert glass wool inlet liner at 250 °C was used at the injection port, and the injection (1.5 µL) was performed in splitless pulsed mode. The gases used were supplied by Linde (Dublin, Ireland), the carrier gas being helium 5.0 (99.999% purity) at a constant flow 1.5 mL/min, and the collision gas being nitrogen 6.0 (99.9999% purity). The initial oven temperature of 80 °C was maintained for 1.8 min, then increased at a rate of 40 °C/min to 170 °C, then increased at a rate of 10 °C/min to 310 °C, and finally maintained for 3 min at 310 °C. The post-run backflush to clean the column was set at 315 °C for 5 min at −5.8 mL/min for the first column, and the final run time at 21.05 min. For the identification and quantification of the compounds, an Agilent 7010 mass spectrometer (Agilent Technologies, Palo Alto, CA, USA) was used. This equipment was operated in the multiple reaction monitoring mode (MRM), with 24-time segments, cycle time between 300 and 600 ms and a dwell time between 15 and 40 ms. The electron impact (EI) and transfer line ionization source temperatures were set at 280 °C, with a solvent delay of 3.7 min.

2.4.2. LC-MS/MS

Liquid chromatography was employed to separate 225 substances using an Agilent 1290 Infinity II UHPLC (Agilent Technologies, Palo Alto, CA, USA). The column was an InfinityLab Poroshell 120 (2.1 mm × 100 mm, 2.7 µm), coupled to an inline filter and an UHPLC guard column with the same characteristics as the analytical column, to protect the column. The gradient of mobile phase A was: 95%—0.5 min; 80%—1 min; 60%—2.5 min; 15%—8 min; 0%—10 to 14 min; 95%—14.01 min. Mobile phase A contained 0.1% FA and 2 mM ammonium acetate in ultrapure water; mobile phase B consisted of 2 mM ammonium acetate in MeOH. 8 µL were injected at a flow rate set at 0.4 mL/min and an oven column temperature of 50 °C. For identification and quantification, an Agilent 6460 mass spectrometer (Agilent Technologies, Palo Alto, CA, USA) was employed. It was operated in the dynamic multiple reaction monitoring mode (dMRM), in both positive
and negative polarities, with a cycle time 800 ms, a dwell time of 8 to 60 ms, and a total run time of 18 min. The Agilent Jet Stream Electrospray Ionization Source (AJS-ESI) was operated under the following conditions: gas temperature 190 °C; nebulizer gas flow and pressure were 11 L/min and 26 psi, respectively; the temperature of the sheath gas and the flow were 330 °C and 12 L/min, respectively; and the positive and negative capillary voltages were 3900 V and 2600 V. The drying and desolvation gas was nitrogen provided by the Zefiro 40 nitrogen generator (F-DGSi, Evry, France). Nitrogen 6.0 (99.9999% purity, Linde, Dublin, Ireland) was used as the collision gas.

2.5. Validation Procedures

Although this is an extension of the analytical scope of a previous method [16]—in this case a change of matrix—it is necessary to undertake a validation process to verify the capacity of the assay to obtain satisfactory results for the analytes in the new matrix. In this research the validation process included the evaluation of linearity, accuracy, precision, calculation of the limit of quantification (LOQ), uncertainty, and the study of the carryover and matrix effect. For most compounds included in this method, there is no specific guide for method validation. For veterinary drugs and considering liver as a food product of animal origin, the requirements for the methods and validation are presented in the UE’s Regulation 808/2021 [32]. Therefore, we decided to follow this regulation, and also the guide of Standard Practices for Method Validation in Forensic Toxicology (SWGTOX) [33], and the EU’s Directorate-General for Health and Food Safety analytical method validation guide (SANTE) [34].

The linearity of the response was studied by injecting blank liver extract samples spiked with all analytes at 11 levels (range 0.4–40 ng/g) and processed with the method described in Section 2.3 of this section, in quintuplicate. To determine accuracy and precision, % recovery (range 70–120% being acceptable, as specified in the guideline used) and % relative standard deviation (%RSD, values ≤ 20% being acceptable) were calculated, respectively. Recovery and RSD experiments were performed with blank liver samples fortified at least in five quintuplicate concentrations within the working range. For the calculation of the LOQ, matrix-matched calibration curves were prepared in quintuplicate (below 20 ng/g). From these, the lowest concentration level of each analyte that met the criteria for identification, accuracy and precision was considered as the LOQ. For confirmation of compound identity and selectivity, 2 MRM transitions were used, one for quantification (Q) and one for confirmation (q). A maximum deviation of ±30% was tolerated for the ion ratio [35]. Similarly, a maximum deviation of ±0.1 min was established for the retention time.

2.6. Samples for the Applicability of the Method

The main objective of this research is to demonstrate the applicability of the validated method in the two fields of application mentioned above: (a) verification of compliance with maximum residue limits in livers intended for human consumption; and (b) biomonitoring of contaminants in wildlife. For this purpose, a set of samples was collected for each of the two independent studies. The samples are described in the following subsections.

2.6.1. Sampling for the Food Safety Study

To verify the applicability of this method for the control of residues subject to MRLs in livers intended for human consumption, 46 liver samples from butcheries, supermarkets, and the general slaughterhouse of Gran Canaria were acquired: 34 samples of beef liver and 12 samples of chicken liver. All the samples, as they were acquired, were transferred to the laboratory and frozen at −20 °C until they were processed.

2.6.2. Sampling for the Biomonitoring Study

The validated method was applied to real samples of wildlife specimens that were received in our laboratory for forensic analyses in the period between September 2020 and
May 2021. Thus, we studied a series of 151 fresh liver samples belonging to 15 different species of birds. All the specimens were sent by environmental agents or by the Tafira Fauna Rehabilitation Centre, within the framework of the Strategy for the Prevention and Control of Poisoning in the Canary Islands [36]. All the birds included in this part of the study died from different classes of trauma, and there was no suspicion that they died of poisoning. The species included in this study were: Accipiter nisus \((n = 5)\); Ardea cinerea \((n = 12)\); Asio otus canariensis \((n = 34)\); Burhinus oedicnemus \((n = 10)\); Buteo buteo insularum \((n = 12)\); Calonectris diomedea \((n = 8)\); Ciconia ciconia \((n = 2)\); Corvus corax canariensis \((n = 16)\); Egretta garzetta \((n = 4)\); Falco eleanorae \((n = 2)\); Falco pelegrinoides \((n = 6)\); Falco tinnunculus canariensis \((n = 14)\); Larus michaellis \((n = 14)\); Turdus merula \((n = 4)\); and Tyto alba \((n = 8)\). The livers, received or extracted at in situ necropsy, were kept at \(-24^\circ\text{C}\) until the moment of their processing for analysis. No animals were sacrificed for the purposes of this work.

2.7. Statistical Analyses

All statistical analyses were performed with GraphPad Prism v9.2 software (GraphPad Software, CA, USA). The distribution of the variables included in this study was evaluated using the Kolmogorov–Smirnov test. The concentration of most of the contaminants detected did not follow a normal distribution, so the results are expressed in terms of median and range. For this same reason nonparametric tests to check for statistical differences between groups were employed, as these evaluate the median rather than the mean, which is appropriate given the relatively high number of undetected values in some groups. Homogeneity of variance (homoscedasticity) was previously tested using Levene’s test. The Kruskal–Wallis and Mann–Whitney U tests were used as nonparametric tests for overall and pairwise comparisons, respectively. However, as an additional check, pairwise comparisons were also performed using Student’s t-test after logarithmic transformation of the data. A P-value of less than 0.05 (two-tailed) was considered statistically significant. The prevalence of exposure to each contaminant for each species was calculated as the percentage of animals with that residue detected in the liver over the total number of individuals of that species studied. For the study of determinants in the series used for biomonitoring, the response variables considered for comparisons were the amount in the liver of (a) the sum of non-persistent pesticides; (b) the sum of persistent organic pollutants; and (c) the sum of rodenticides.

3. Results and Discussion

3.1. Method Scope Extension Optimization

In our previous research we optimized and validated a multi-residue method for the analysis of 360 substances (pharmaceuticals, pesticides, rodenticides, and POPs) in blood for biomonitoring purposes [16,28]. Therefore, this is not an ex-novo methodological development, but an extension of the scope of our previously published method to include a new matrix, the liver. However, for a better method performance in this more complex matrix, we considered optimizing the previously established chromatographic conditions, including recalculation of RTs, as well as optimization of MRM transitions to allow for higher sensitivity, as well as adjusting qualifiers and qualifier ratios, and identifying possible interferences with matrix components. The compounds are shown in alphabetical order in Appendix A along with their retention time, transitions, and their collision energies. As we did with the original method in blood, we decided to directly inject the extracts obtained in acetonitrile for LC-MS/MS and GC-MS/MS analyses, without using evaporation and solvent change, to avoid the loss of the more volatile compounds. Several authors, including our group [30,37,38], have shown that ACN, although not the most commonly used solvent in GC-MS/MS, is an appropriate solvent for this type of analysis.

The final number of validated compounds in this scope extension counts 351 chemicals and metabolites compared to 360 in the previous work. With respect to the original method, there are 18 compounds that met the validation criteria in whole blood, which do not meet the validation criteria when the method is applied to liver samples: acetaminophen, chlor-
fenapyr, corticosterone 21 acetate, phenbutatin oxide, iprodione, isocarbophos, leptophos,
malaoxon, malathion, marbofloxacin, methomyl oxime, N,N-dimethyl-N-tolylsulfamide,
paraaxon ethyl, parathion ethyl, penicillin G, phosphmet oxon, pipracillin, and trichlorfon.
On the other hand, the opposite occurred with 9 compounds. Dichlorvos, doramectin,
metalaxyl, methiocarb-sulfoxide, moxidectin, oxime, phtalimide, pyrimicarb-desmethyl
and spirotetramat met the validation criteria in the presence of liver matrix and could
therefore be included in the method in liver, whereas in blood they did not and had to be
left out.

3.2. Validation Parameters

For confirmation of compound identity and selectivity, 2 MRM transitions were used,
one for quantification (Q) and one for confirmation (q). A maximum deviation of ±30% was tolerated for the ion ratio. Similarly, a maximum deviation of ±0.1 min was established for the retention time.

We first studied the linearity of the response by injecting blank liver extract samples spiked with all analytes at 11 levels (range 0.4–40 ng/g) and processed in quintuplicate with the method described in Section 2.3. The linearity study on the response (R²), indicated that this was higher than 0.98 for all analytes in the range studied.

To determine accuracy and precision, % recovery and % relative standard deviation (RSD) was calculated. A recovery within the range 70–120% and RSD values ≤ 20% was considered acceptable, as specified in the guidelines employed [33,34]. Recovery and RSD experiments were performed with blank liver samples fortified at least in four quintuplicate concentrations within the working range. The results of the recovery experiments are presented in Appendix B. Regarding accuracy and precision, most compounds meet the validation criteria for concentrations between their LOQ and the highest level studied (40 ng g⁻¹). There were some exceptions where recoveries were outside the above range, especially at the lower concentrations. However, these cases are covered, both in the SANTE guideline and in the SWGTOX working document [33,34], which also accepts as a good validation criterion obtaining recoveries between 60% and 140% at some of the concentrations tested, provided that the RSD is less than 15%. Likewise, in some cases, the recoveries were within the established limits with an RSD slightly higher than 15%, a scenario that is also contemplated in the methodological guidelines, provided that the result is reproducible. As a rule, this second exception applies for concentrations equal to or lower than 4 ng g⁻¹. As SANTE analytical guide recommends, the expanded measurement uncertainty (U') was calculated, from precision and bias, and all analytes presented U' < 50%, that complies with the requirement.

For the calculation of the LOQ, matrix-matched calibration curves were prepared in quintuplicate (0.2–20 ng g⁻¹). From these, the lowest concentration level of each analyte that met the criteria for accuracy and precision was considered as the LOQ. As in the original method, the LOQ for the analytes included in this scope extension was calculated from five replicates of fortified blank matrix, within the working range. The lowest non-zero calibrator approximation was used to calculate LOQs. This means that the lowest point on the calibration curve that met the identity, bias, and precision criteria was established as the LOQ for a given compound. The LOQs for the 351 liver analytes are shown in Appendix B. The LOQ was set at 0.4 ng g⁻¹ for 61 compounds, at 0.8 ng g⁻¹ for 82 compounds, at 1.2 ng g⁻¹ for 40 compounds, at 1.6 ng g⁻¹ for 37 compounds, at 2 ng g⁻¹ for 50 compounds, at 4 ng g⁻¹ for 46 compounds, at 8.0 ng g⁻¹ for 24 compounds, at 12 ng g⁻¹ for 5 compounds, at 16 ng g⁻¹ for 4 compounds, and at 20 ng g⁻¹ for 2 compounds. That is, 76.9% of the compounds included in this method can be reliably and accurately quantified at concentrations below 2 ng g⁻¹, making it suitable not only for food safety or poisoning diagnostic studies, but also for biomonitoring studies.

In the original method from which we started it was observed that there was a strong blood matrix effect on about 40% of the analytes. Presumably, a similar situation would occur with the liver matrix. Nevertheless, we decided to include the study of the
matrix effect within the validation strategy of this analytical scope extension to prove it, as recommended in the reference guides. All validation assays involve the addition of known concentrations of analytes to the matrix. For the matrix effect study, we worked with the addition of three known concentrations of all analytes (2 ng g\(^{-1}\), 10 ng g\(^{-1}\), and 20 ng g\(^{-1}\)) on blank liver extract, and the quantification was done against calibration curves prepared in solvent (without matrix). Experiments were performed in quintuplicate for each concentration. One difficulty was that, given the enormous number of substances included in the method, the liver was not completely free of 100% of the chemicals, in particular POPs. Therefore, in these cases, the response of the white matrix sample was subtracted from the calibration standards and QC to calculate the response of the externally added analyte. As we expected, matrix effect (ME) was observed for both, compounds analyzed by LC-MS/MS and GC-MS/MS, especially for compounds analyzed by the latter technique. A strong or medium suppression of the signal was demonstrated for 17.66% of the compounds (\(n = 62\)), and signal enhancement was verified for 36.47% of the compounds (\(n = 128\)). For the remaining 45.87% (\(n = 161\) contaminants), the ME was considered negligible (\(-20% < M < 20\%\)). Since for most of the compounds, significant ME was indeed observed, and it was concluded that matrix-matched calibration had to be used to compensate for these interferences. All detailed ME data for individual compounds in liver are shown in Appendix C.

Finally, we also assessed if carryover occurred after injecting a blank matrix fortified at 80 ng g\(^{-1}\) and processed with this method, before a blank matrix extract. We were not able to find a clear response in that blank matrix, so we concluded that in our working range, we had not any carryover effect in any of the analyzed compounds.

### 3.3. Application to Food Safety

In the study of the 34 beef liver samples, the results indicated the presence of a discrete number and concentration of contaminants, which ranged from 0 to 15 residues per sample, with an average of 3.13 residues. Of the 351 contaminants and metabolites included in the method, only 25 were detected in the total of beef liver samples, and of these 19 belong to the group of persistent or semi-persistent contaminants (4,4’-DDE, 4,4’-DDD, Dieldrin, Hexachlorobenzene, beta-hexachlorocyclohexane, BDE 153, PCB congeners #105, 118,126, 138, 155, 156, 157, 180, 189, naphthalene, phenanthrene, and pyrene). It is noteworthy that none of the concentrations in any of the samples exceeded the MRL, or even the value of half the MRL. In general, the concentrations of the contaminants detected were low, with the highest values being those of 4,4’-DDE, which was detected in 65.2% of the samples and with a median value of 92.2 ng g\(^{-1}\). The relatively high levels of DDT derivatives may seem surprising, as this substance was banned in Spain almost 5 decades ago. However, there is abundant literature that has documented that this pesticide was widely used in the Canary archipelago, and how this translates into the levels of this pesticide detected in food for human consumption produced in this region [39–42].

The next in frequency and concentration were PCB 153 (26.1%; 35.2 ng g\(^{-1}\)) and PCB 138 (21.8%; 24.5 ng g\(^{-1}\)). The other contaminants were detected in frequencies and concentrations much lower than these. Among the non-persistent pesticides detected in this series of consumption livers, very low levels of bifenthrin, fenazaquin, fluquinconazole, flutaloni, flutriafol, and imidacloprid were detected.

If the detection of residues in beef liver was low and of little toxicological relevance, it was even more so in chicken liver. In the 12 samples analyzed, we detected only four contaminants out of the 351 included in the method: fenpropidin, fenpropimorph, levamisole, and 4,4’ DDE. The latter was the more relevant, and it was only detected in three of the 12 livers analyzed and at a much lower concentration than that detected in beef liver (mean = 4.3 ng g\(^{-1}\)).

Although it is not the main objective of this study, we made an estimate of the risk of exposure to these contaminants through liver consumption. The calculations were made according to the standard methodology that has been described previously [43], and in no
case were the tolerable daily intake levels for these contaminants exceeded, mainly due to the low consumption of liver by the Spanish population (only 1 g/day for the total offal consumption) [44].

3.4. Application to Biomonitoring

Regarding biomonitoring of chemical substances, this method was applied to fresh livers obtained from 151 carcasses of 15 species of wild birds whose causes of death were not related to poisoning (mainly trauma). Table 1 shows the results for each of the species, limited to show only the 52 contaminants that were detected in the series. This represents that 15% of the contaminants included in the method were detected.

The mean value of the number of contaminants per sample was 17. The species with the greatest variety of residues detected was Asio otus (n = 41), followed by Falco tinnunculus (n = 27). In contrast, the species with the lowest number of liver contaminants were Turdus merula (n = 5) and Ciconia ciconia (n = 3). Figure 1 shows the LC-MS/MS and GC-MS/MS chromatograms of one of the birds in the series with the highest number of different contaminants (a long-eared owl).

The most frequently detected contaminant was 4,4’-DDE, which was detected in 138 birds (91.4%), followed by PCB 153, detected in 116 animals (76.8%), brodifacoum in 109 animals (72.2%), bromadiolone in 87 animals (57.6%), and dieldrin in 59 animals (39.1%). With respect to concentrations, the highest concentrations corresponded to enrofloxacin, clindamycin and meloxicam (Table 1). However, these values cannot be considered within the biomonitoring study, since they correspond to drugs used during the hospitalization of many of these animals. Therefore, high concentrations of these substances have been marked with an asterisk. However, other veterinary pharmaceuticals detected in some specimens, such as tetraconazole, metronidazole, or sulfathiazole, are not part of the treatment administered and should be considered contaminants. In general terms, the highest concentrations of contaminants corresponded to 4,4’-DDE in all species. Overall, in quantitative terms, the group of organochlorine pesticides was the most abundant (Figure 2), and the group of persistent and semi-persistent organic pollutants accounted for more than 92% of the total concentration of pollutants detected in the livers of wild birds sampled in the Canary Islands very recently (September 2020–May 2021). This reflects, once again, that contamination by these compounds, in particular organochlorine insecticides, is still very prevalent in the Canary Islands, as has been reported for wildlife in this region [28,45–47]. As indicated in the previous section, there is a large literature body documenting the high levels of contamination by organochlorine pesticides in this region [48–50], which also translates into high levels in the biota that inhabit the archipelago. There is a possibility that the high levels detected could also come from the neighboring African continent [51], but in this biomonitoring study this option is ruled out, since all the birds sampled for this pilot study are residents in the archipelago and not migratory birds.

With respect to non-persistent pollutants, several aspects should be highlighted. First, the high prevalence of second-generation anticoagulant rodenticides in wildlife’s liver is noteworthy. It was expected, as it has been described in many parts of the world [52–54] and recently in the Canary Islands [45,55,56]. However, the presence of at least one of these compounds in more than 80% of the birds studied is striking, even in non-predatory birds such as the blackbird (Turdus merula) or the common curlew (Burhinus oedicnemus), which would point to the fact that these compounds penetrate the trophic chain by several routes, probably including invertebrates, as suggested by other authors [57,58].
| Compound            | Accipiter Nisus (n = 5) | Ardea Cinerea (n = 12) | Asio Otus (n = 34) | Burrinus Oedicnemus (n = 10) | Buteo Buteo (n = 12) | Calonectris Diomedea (n = 8) | Ciconia Ciconia (n = 2) | Corvus Corax (n = 16) | Egretta Garzetta (n = 4) | Falco Eleonora (n = 2) | Falco Pellegrinoides (n = 6) | Falco Tinnunculus (n = 14) | Larus Michaelis (n = 14) | Turdus Merula (n = 4) | Tyto Alba (n = 8) |
|---------------------|-------------------------|------------------------|-------------------|-------------------------------|----------------------|-----------------------------|-------------------------|------------------------|--------------------------|----------------------|-----------------------------|-----------------------------|--------------------------|------------------------|------------------------|
| Meloxicam           | 9.9 (40)                | -                      | 47.8 (12)         | 880.3 * (20)                  | -                    | -                           | -                       | 10.3 (13)              | -                        | -                    | 96.1 (29)                    | -                           | -                       | -                      | -                      |
| Tetraconazole       | -                       | -                      | 0.6 (6)           | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Clindamycin         | 1.3 (40)                | -                      | 1423 * (18)       | 1.4 (20)                      | 28.4 (33)            | -                           | -                       | -                      | -                        | -                    | 2.3 (33)                     | -                           | -                       | -                      | -                      |
| Enrofloxacin        | 5300 * (80)            | -                      | 4739 * (29)       | 4638 * (40)                   | 5453 * (67)          | -                           | -                       | 1970 * (13)            | 20.4 (100)              | 5144 * (50)                   | 5531 * (57)                   | 10234 * (7)                    | -                       | 14508 * (50)         | -                      |
| Metronidazole       | -                       | -                      | -                 | 50.8 (10)                     | -                    | -                           | -                       | -                      | -                        | -                    | 603.3 (14)                    | -                           | -                       | -                      | -                      |
| Sulfatiazole        | -                       | -                      | 15.5 (6)          | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| 2-Phenylnaphthol     | -                       | -                      | 16.3 (18)         | 22.7 (20)                     | 2.4 (17)             | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Boscalid (formerly nicobiene) | -               | -                      | -                 | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Fludioxonil         | -                       | -                      | 0.4 (6)           | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Fluquinconazole     | -                       | -                      | -                 | -                             | -                    | 4.3 (17)                    | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Flutriafol          | -                       | -                      | -                 | 10.3 (3)                      | 16.4 (10)            | 7.2 (26)                    | -                       | 94.5 (38)              | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Carbofuran          | -                       | -                      | -                 | 10.3 (3)                      | 16.4 (10)            | 7.2 (26)                    | -                       | 94.5 (38)              | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Carbofuran-3-hydroxy| -                       | -                      | -                 | -                             | -                    | -                           | -                       | 2.1 (38)               | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Fipronil            | -                       | -                      | 1.4 (12)          | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Fipronil sulfide    | -                       | -                      | 3.0 (6)           | -                             | -                    | -                           | -                       | -                      | -                        | -                    | -                           | -                           | -                       | -                      | -                      |
| Permethrin          | -                       | -                      | -                 | 23.4 (13)                     | -                    | -                           | -                       | -                      | -                        | -                    | 12.3 (7)                     | -                           | -                       | -                      | -                      |

Table 1. Median concentrations and frequencies (between parentheses) of organic pollutants detected in wild birds of the Canary Islands. All the results are in ng/g.
Table 1. Cont.

| Compound | Accipiter Nisus (n = 5) | Ardea Cinerea (n = 12) | Asio Otus (n = 34) | Burhinus Oedicnemus (n = 10) | Buteo Buteo (n = 12) | Calonectris Diomedea (n = 8) | Ciconia Ciconia (n = 2) | Corvus Corax (n = 16) | Egrete Garzetta (n = 4) | Falco Eleonora (n = 2) | Falco Pellegrinoides (n = 6) | Falco Tinnunculus (n = 14) | Larus Michaelis (n = 14) | Turdus Merula (n = 4) | Tyto Alba (n = 8) |
|----------|----------------------|----------------------|-------------------|-------------------------------|---------------------|---------------------------|----------------------|---------------------|---------------------|---------------------|---------------------------|--------------------------|------------------------|-------------------|---------------------|
| Acenaphthene | 2.4 (80)            | 1.4 (17)             | 0.8 (18)          | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 2.4 (14)                  | -                        | -                     | -                 | -                   |
| Anthracene         | 1.5 (40)           | -                    | -                 | -                             | -                   | -                         | -                    | -                   | -                   | -                   | -                          | -                        | -                     | -                 | -                   |
| Chrysene           | -                   | -                    | -                 | -                             | -                   | -                         | -                    | -                   | -                   | -                   | -                          | -                        | -                     | -                 | 4.8 (25) |
| Fluoranthene       | 0.4 (40)           | -                    | -                 | -                             | -                   | -                         | -                    | -                   | -                   | -                   | -                          | -                        | -                     | -                 | 1.7 (25) |
| Fluorene           | 5.9 (80)           | -                    | 2.5 (12)          | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 5.3 (7)                    | -                        | -                     | -                 | -                   |
| Naphtalene         | 1.8 (100)          | -                    | 2.2 (6)           | 5.7 (40)                      | 3.9 (26)            | 16.6 (13)                  | -                    | 3.4 (26)            | 0.7 (25)            | -                   | 1.8 (14)                  | -                        | 0.9 (50)               | 8.0 (25)          |
| Phenanthrene       | 13.3 (100)         | -                    | 7.5 (12)          | -                             | 0.4 (13)            | 2.7 (13)                  | -                    | -                   | -                   | -                   | 7.6 (28)                  | -                        | -                     | 851.3 (50)        |
| Pyrene             | 1.8 (40)           | -                    | 0.7 (9)           | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 20.0 (28)                 | -                        | -                     | -                 | -                   |
| 4,4′-Dichlorobenzophenone (metabolite of dicofol) | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| BDE-100            | -                   | 1.9 (33)             | 0.2 (6)           | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 0.4 (33)                  | -                        | -                     | -                 | -                   |
| BDE-153            | -                   | 0.3 (33)             | 4.9 (41)          | -                             | 1.1 (13)            | -                         | -                    | -                   | -                   | -                   | 0.7 (50)                  | 19.0 (67)                | 0.6 (29)               | -                 | 0.8 (25) |
| BDE-154            | -                   | 2.1 (17)             | 2.6 (6)           | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 6.9 (33)                  | -                        | -                     | -                 | -                   |
| BDE-183            | -                   | -                    | 0.9 (12)          | -                             | -                   | -                         | -                    | -                   | -                   | -                   | -                          | -                        | -                     | -                 | -                   |
| BDE-47             | -                   | 1.5 (17)             | 2.4 (6)           | -                             | -                   | -                         | -                    | -                   | -                   | -                   | 0.6 (7)                   | 0.4 (14)                 | -                     | -                 | -                   |
Table 1. Cont.

| Compound                        | Accipiter Nisus (n = 5) | Ardea Cinerea (n = 12) | Asio Otus (n = 34) | Buteo Buteo (n = 12) | Calonectris Diomedea (n = 8) | Ciconia Ciconia (n = 2) | Corvus Corax (n = 16) | Egretta Garzetta (n = 4) | Falco Eleonorae (n = 2) | Falco Pellegrinoides (n = 6) | Falco Tinunculus (n = 14) | Larus Michaelis (n = 14) | Turdus Merula (n = 4) | Tyto Alba (n = 8) |
|---------------------------------|--------------------------|------------------------|--------------------|----------------------|----------------------------|--------------------------|-----------------------|--------------------------|--------------------------|----------------------------|---------------------------|--------------------------|--------------------------|--------------------------|
| BDE-99                          | -                        | -                      | 1.5 (29)           | -                    | -                          | -                        | -                     | -                        | -                        | 3.6 (33)                   | 0.4 (14)                  | 0.6 (14)                 | 7.5 (100)                | 0.4 (13)                 |
| Dichlorodiphenyldichloroethane   |                          |                        |                    |                      |                            |                          |                       |                          |                          |                            |                          |                          |                          |                          |                          |
| (p,p′ DDD)                       | 1.2 (80)                 | -                      | -                  | -                    | -                          | -                        | -                     | -                        | -                        | 2.2 (21)                   | -                         | -                       | -                       | -                       |
| Dichlorodiphenyldichloroethylene |                          |                        |                    |                      |                            |                          |                       |                          |                          |                            |                          |                          |                          |                          |                          |
| (p,p′ DDE)                       | 211.1 (100)              | 21.1 (100)             | 305.6 (100)        | 25.9 (60)            | 5.6 (83.3)                 | 16.6 (100)               | -                     | 6.7 (75)                 | 4.7 (100)                 | 68.4 (100)                 | 318.6 (100)               | 45.3 (100)               | 4.4 (100)                | -                       | 24.1 (100)               |
| Dieldrin                         | 7.8 (80)                 | 3.5 (17)               | 5.6 (41.2)         | 3.0 (10)             | 0.9 (33)                   | 3.1 (13)                 | -                     | 2.1 (100)                | 5.8 (100)                 | 8.5 (100)                  | 11.9 (100)                | 2.3 (7)                  | -                       | 1.2 (75)                |
| Hexachlorobenzene                |                          | 1.4 (34)               | 0.6 (24)           | -                    | -                          | 7.1 (13)                 | -                     | 12.9 (50)                | 0.6 (50)                 | -                          | 1.1 (7)                   | 1.1 (7)                  | -                       | 0.8 (25)                |
| Hexachlorocyclohexane (beta)     |                          |                        |                    |                      |                            |                          |                       |                          |                          |                            |                          |                          |                          |                          |                          |
| Mirex                           |                          |                        |                    |                      |                            |                          |                       |                          |                          |                            |                          |                          |                          |                          |                          |
| PCB 105                          |                          | 1.2 (50)               | 1.3 (35)           | -                    | -                          | 2.0 (26)                 | -                     | 3.6 (25)                 | 0.6 (50)                 | 0.6 (67)                  | 1.1 (14)                  | 0.4 (7)                  | -                       | -                       |
| PCB 118                          |                          | 0.5 (40)               | 5.4 (50)           | 4.8 (35)             | -                          | 0.5 (17)                 | -                     | 45.1 (13)                | 5.0 (100)                | 2.2 (100)                 | 1.3 (28)                  | 1.4 (7)                  | -                       | 1.2 (37)                |
| PCB 138                          |                          | 1.4 (80)               | 7.9 (100)          | 2.9 (76)             | 2.2 (30)                   | 1.3 (34)                 | -                     | 8.8 (75)                 | 3.9 (38)                 | 24.1 (100)                | 15.3 (100)                | 7.7 (100)                | 4.2 (71)                 | 2.7 (72)                | -                       | 5.3 (75)                |
| PCB 153                          |                          | 3.4 (80)               | 15.5 (100)         | 3.6 (94)             | 0.5 (80)                   | 3.3 (34)                 | -                     | 6.8 (100)                | 1.7 (88)                 | 55.3 (100)                | 115.3 (100)               | 16.0 (100)               | 5.7 (71)                 | -                       | -                       | 7.8 (100)               |
| PCB 156                          |                          | 2.0 (50)               | 2.0 (33)           | -                    | -                          | 8.5 (13)                 | -                     | 0.8 (25)                 | 8.8 (50)                 | 0.8 (100)                 | 0.9 (7)                   | -                       | 0.5 (25)                | -                       | -                       | -                       |
### Table 1. Cont.

| Compound           | Accipiter Nisus ($n = 5$) | Ardea Cinerea ($n = 12$) | Asio Otus ($n = 34$) | Burrinus Oedicnemus ($n = 10$) | Buteo Buteo ($n = 12$) | Calonectris Diomedea ($n = 8$) | Ciconia Ciconia ($n = 2$) | Corvus Corax ($n = 16$) | Egretta Garzetta ($n = 4$) | Falco Eleonora ($n = 2$) | Falco Pellegrinoides ($n = 6$) | Falco Tinunculus ($n = 14$) | Larus Michaelis ($n = 14$) | Turdus Merula ($n = 4$) | Tyto Alba ($n = 8$) |
|--------------------|---------------------------|--------------------------|----------------------|-------------------------------|------------------------|-------------------------------|-----------------------------|---------------------------|----------------------------|------------------------|-----------------------------|-----------------------------|---------------------------|--------------------------|-----------------------------|
| PCB 157            | -                         | 0.8 (17)                 | 1.1 (6)              | -                             | 2.1 (13)               | -                             | 9.1 (50)                    | 1.1 (50)                  | -                          | -                      | -                          | 0.4 (7)                     | -                         | -                        | -                          |
| PCB 167            | -                         | 1.7 (50)                 | 1.5 (25)             | -                             | 6.7 (50)               | -                             | 44.6 (100)                  | 6.7 (50)                  | 1.3 (67)                   | 0.8 (14)               | 2.2 (72)                    | -                          | 0.4 (13)                  | -                        | -                          |
| PCB 180            | 3.9 (80)                  | 24.3 (67)                | 3.2 (88)             | -                             | 2.1 (50)               | 3.6 (100)                    | 2.8 (88)                    | -                          | 123.6 (100)                | 19.8 (100)            | 5.2 (71)                    | -                          | -                        | 8.0 (75)                  | -                          |
| PCB 189            | -                         | -                        | 2.1 (6)              | 1.0 (80)                      | -                      | -                             | -                          | -                          | 1.8 (50)                   | -                      | -                          | -                          | -                        | -                        | -                          |
| PCB 28             | -                         | -                        | 5.8 (3)              | -                             | -                      | -                             | -                          | -                          | -                          | -                      | -                          | -                          | -                        | -                        | -                          |
| Bromifacoum        | 1.7 (100)                 | 0.4 (100)                | 32.9 (100)           | 2.3 (80)                      | 0.9 (100)              | -                             | -                          | 27.4 (75)                  | -                          | -                      | 20.4 (100)                  | 8.8 (50)                    | 1.4 (21)                  | -                        | 20.31 (100)               |
| Bromadiolone       | -                         | -                        | 1.3 (100)            | 2.1 (100)                     | 8.5 (100)              | -                             | -                          | 2.25 (38)                  | -                          | 1.1 (50)               | 4.6 (100)                    | 2.5 (75)                    | -                        | 0.34 (25)                | 2.2 (75)                   |
| Difenacoum         | -                         | 0.8 (17)                 | 0.6 (24)             | 1.5 (50)                      | -                      | -                             | 0.9 (13)                    | -                          | 0.9 (33)                   | 1.2 (57)               | -                          | -                        | 3.6 (25)                  | -                        | -                          |
| Difethialone       | -                         | -                        | 18.9 (18)            | -                             | -                      | -                             | 1.9 (29)                    | -                          | -                          | -                      | 1.4 (21)                    | -                        | -                        | -                          |
| Flocoumafen        | -                         | -                        | 0.7 (24)             | 4.1 (17)                      | -                      | -                             | -                          | -                          | -                          | -                      | -                          | 2.2 (7)                     | -                        | -                        | -                          |

* These values cannot be considered as biomonitoring, since these pharmaceuticals were employed during the treatment of the animals at the Wildlife Recovery Centers.
The result for carbofuran is also surprising, given that none of the birds studied had any suspicion of intoxication. However, this potent insecticide, banned in the EU since 2007 [59] was detected in small concentrations in the liver of 10 birds of the series, being higher in the case of canary crows (6/12 positives, median = 94.5 ng/g). In all these crows, the main carbofuran metabolite was also detected. This toxicant has widely affected wildlife worldwide [60–63] and in the Canary Islands its use has been extensive and also has affected wildlife in the past [64]. From the results of this study, it still is today, and it can be concluded that it even penetrates the trophic chain. With respect to the rest of the non-persistent compounds detected in this series, 2-phenylphenol (PHP) stands out. PHP was detected in eleven birds, including five common curlews (Table 1). PHP is a biocide used as a preservative and surface disinfectant on fibers and other materials in homes, hospitals, and elsewhere, and is recognized as a potential endocrine disruptor [65]. Other
authors have also reported that PHP is a highly prevalent contaminant in biota samples, such as river fish of different species, where it is found in up to 100% of samples [66].

Figure 2. Occurrence of environmental pollutants in the liver of a series of 151 wild birds of the Canary Islands.

Since this was an opportunistic study on carcasses obtained from wildlife recovery centers, we did not have too many quality variables to carry out an in-depth study of the determinants of contamination patterns. Even so, we wanted to explore the influence of the variables inherent to the species studied and found a series of statistically significant differences. Thus, when we compared aquatic versus terrestrial birds, we found that the latter presented significantly higher levels of contamination by the three major chemical groups studied (Figure 3).

Figure 3. Study of the determinants of environmental contamination detected in the livers of wild birds in the Canary Islands: Habitat type (terrestrial (TB) vs. aquatic birds (AQB)).
This result was expected with regard to rodenticides, since in a previous study by our group focused on these compounds, we had already discarded the group of waterfowl due to their low incidence in these pollutants [56]. Regarding POPs and non-persistent pesticides, although there is not much literature comparing both types of birds from the same region, the available studies usually indicate results similar to ours, with levels in landbirds usually being higher than in waterbirds [67–69].

Another variable that seems to influence the pattern of contamination is the raptor/predator bird status. The raptors in our study presented higher levels of POPs than non-predatory birds (Figure 4), which is logical given that they feed higher in the trophic chain, and has been described in the literature [68,69]. They also presented higher levels of AR, as we expected from having previously observed it in this region [56], and also described by other authors [70]. However, in the case of agricultural pesticides the statistical significance was the opposite, with non-predatory birds presenting the highest levels. There is not much literature to support this finding, but a recent study using the terrestrial pesticide residue exposure (T-REX) model estimated that the highest risk was presented by insectivorous birds, followed by fruit and seed feeders [71].

![Figure 4. Study of the determinants of environmental contamination detected in the livers of wild birds in the Canary Islands: Diet type (raptors vs. non-raptors).](image)

Finally, we also studied the influence of the diurnal/nocturnal habits of the birds in the study, and found that diurnal species have higher pesticide levels, but lower POPs and ARs than nocturnal species (Figure 5). We believe that the pesticide result has to do with the previous variable, in the sense that, in our study, all insectivorous species, and those that feed on fruits and seeds are diurnal, while the nocturnal birds in our series are both raptors that feed mainly on large and small rodents. For this same reason, and as we had already verified in previous studies, the nocturnal birds of the Canary Islands have higher levels of POPs [72] and AR [45,56].
Figure 5. Study of the determinants of environmental contamination detected in the livers of wild birds in the Canary Islands: Habit type (diurnal vs. nocturnal).

4. Conclusions

The validated method allows the simultaneous analysis in liver of 351 substances (POPs, pesticides including rodenticides and drugs), using only 1 gram of sample. This is important, since in veterinary forensic medicine, especially with small animals, the amount of sample available is very limited. The proposed analytical method can detect trace amounts of all chemicals in the liver of multiple species. Therefore, it can be successfully applied and used as a routine method in environmental chemistry and forensic toxicology laboratories. The method we have developed can also be used in residue control studies in food intended for human consumption and for the purpose of food safety assessment.

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Appendix A
Table A1. Chromatographic and mass spectrometric conditions of the compounds analyzed in liver.

| No. | Compound                                      | Technique | Retention Time (min) | Polarity | Quantification    | Confirmation  | Fragmentor Voltage (V) |
|-----|-----------------------------------------------|-----------|----------------------|----------|-------------------|---------------|------------------------|
| 1   | 2-Phenylphenol                                | GC        | 6.28                 | positive | 169.0 → 115.0     | 169.0 → 141.0 | 15, 70                 |
| 2   | 4,4’-Dichlorobenzophenone (metabolite of dicofol) | GC        | 9.99                 | positive | 250.0 → 139.0     | 250.0 → 215.0 | 5, 70                  |
| 3   | Abamectine                                    | LC        | 10.99                | positive | 890.5 → 567.1     | 895.5 → 751.4 | 45, 160                |
| 4   | Acenaphthene                                  | GC        | 6.15                 | positive | 153.0 → 152.0     | 153.0 → 151.0 | 35, 70                 |
| 5   | Acenaphthylene                                | GC        | 5.94                 | positive | 152.0 → 151.0     | 152.0 → 126.0 | 30, 70                 |
| 6   | Acephate                                      | LC        | 1.64                 | positive | 184.0 → 143.0     | 143.0 → 95.0  | 70                     |
| 7   | Acetamiprid                                   | LC        | 4.43                 | positive | 223.1 → 126.0     | 223.1 → 90.0  | 45, 140                |
| 8   | Acrinathrin                                   | LC        | 10.70                | positive | 559.0 → 208.0     | 559.0 → 181.0 | 30, 70                 |
| 9   | Albendazole                                   | LC        | 7.14                 | positive | 266.1 → 234.1     | 266.1 → 191.0 | 32, 155                |
| 10  | Aldicarb                                      | LC        | 5.11                 | positive | 208.0 → 116.0     | 116.0 → 89.1  | 4, 100                 |
| 11  | Aldicarb-sulfone                              | LC        | 3.21                 | positive | 240.1 → 76.0      | 223.1 → 86.1  | 13, 75                 |
| 12  | Aldicarb-sulfoxide                            | LC        | 2.75                 | positive | 207.1 → 131.9     | 207.1 → 89.1  | 10, 86                 |
| 13  | Aldrin                                        | GC        | 9.90                 | positive | 255.0 → 220.0     | 263.0 → 228.0 | 10, 70                 |
| 14  | Anthracene                                    | GC        | 8.40                 | positive | 178.0 → 176.0     | 178.0 → 152.0 | 30, 70                 |
| 15  | Atrazine                                      | LC        | 6.73                 | positive | 216.0 → 173.9     | 216.0 → 103.8 | 30, 130                |
| 16  | Azinphos-methyl                               | LC        | 7.27                 | positive | 318.0 → 132.1     | 340.0 → 160.0 | 10, 60                 |
| 17  | Azoxyostrobin                                 | LC        | 7.59                 | positive | 404.1 → 372.1     | 404.1 → 344.1 | 24, 110                |
| 18  | BDE-28                                        | GC        | 12.22                | positive | 406.0 → 246.0     | 406.0 → 167.0 | 25, 70                 |
| 19  | BDE-47                                        | GC        | 14.31                | positive | 326.0 → 138.0     | 484.0 → 324.0 | 25, 70                 |
| 20  | BDE-85                                        | GC        | 17.08                | positive | 564.0 → 404.0     | 566.0 → 406.0 | 25, 70                 |
| 21  | BDE-99                                        | GC        | 16.27                | positive | 566.0 → 406.0     | 564.0 → 404.0 | 30, 70                 |
Table A1. Cont.

| No. | Compound                        | Technique | Retention Time (min) | Polarity | Quantification          | Confirmation | Fragmentor Voltage (V) |
|-----|---------------------------------|-----------|----------------------|----------|-------------------------|--------------|------------------------|
| 22  | BDE-100                         | GC        | 15.85                | positive | 566.0 → 406.0           | 25           | 564.0 → 404.0          | 25                   |
| 23  | BDE-153                         | GC        | 18.04                | positive | 644.0 → 484.0           | 25           | 486.0 → 377.0          | 30                   |
| 24  | BDE-154                         | GC        | 17.47                | positive | 644.0 → 484.0           | 25           | 486.0 → 377.0          | 30                   |
| 25  | BDE-183                         | GC        | 20.12                | positive | 561.6 → 454.7           | 40           | 563.6 → 454.7          | 40                   |
| 26  | Benalaxyl                       | LC        | 8.96                 | positive | 326.2 → 148.0           | 20           | 326.2 → 208.0          | 12                   |
| 27  | Beniocarb                       | LC        | 5.88                 | positive | 224.1 → 166.9           | 8            | 224.2 → 108.9          | 15                   |
| 28  | Bendiocarb metabolite (2,2-dimethylbenzo-1,3-dioxol-4-ol) | GC | 4.84 | positive | 166.0 → 151.0 | 10 | 166.0 → 126.0 | 20 | 70 |
| 29  | Benfuracarb                     | LC        | 9.73                 | positive | 411.2 → 190.0           | 13           | 411.2 → 252.0          | 15                   |
| 30  | Benzo[a]anthracene              | GC        | 13.95                | positive | 228.0 → 226.0           | 40           | 228.0 → 202.0          | 35                   |
| 31  | Benzo[a]pyrene                  | GC        | 16.89                | positive | 252.0 → 250.0           | 45           | 252.0 → 248.0          | 60                   |
| 32  | Benzo[b]fluoranthene            | GC        | 16.30                | positive | 252.0 → 248.0           | 60           | 252.0 → 226.0          | 35                   |
| 33  | Benzo[g,h,i]perylene            | GC        | 19.61                | positive | 276.0 → 274.0           | 50           | 276.0 → 272.0          | 60                   |
| 34  | Benzo[k]fluoranthene            | GC        | 16.29                | positive | 252.0 → 250.0           | 45           | 252.0 → 224.0          | 40                   |
| 35  | Bifenthrin                      | GC        | 11.25                | positive | 440.0 → 181.0           | 5            | 440.0 → 165.0          | 60                   |
| 36  | Bitertanol                      | LC        | 9.23                 | positive | 338.2 → 70.0            | 4            | 338.2 → 269.2          | 5                    |
| 37  | Boscalid (formerly nicobifen)    | GC        | 7.84                 | positive | 3434.0 → 272.0          | 30           | 343.0 → 140.0          | 45                   |
| 38  | Brodifacoum                     | LC        | 10.78                | negative | 521.3 → 79.0            | 50           | 523.3 → 135.0          | 45                   |
| 39  | Bromadiolone                    | LC        | 9.75                 | negative | 525.3 → 250.0           | 40           | 527.3 → 250.0          | 40                   |
| 40  | Bromopropylate                  | GC        | 13.87                | positive | 341.0 → 183.0           | 15           | 341.0 → 137.0          | 45                   |
| 41  | Bromuconazole (two isomers)     | GC        | 13.81/14.24          | positive | 295.0 → 173.0           | 10           | 295.0 → 175.0          | 10                   |
| 42  | Bupirimate                      | LC        | 11.78                | positive | 273.0 → 108.0           | 15           | 273.0 → 193.0          | 5                    |
| No. | Compound                  | Technique | Retention Time (min) | Polarity  | Quantification         | Confirmation         | Fragmentor Voltage (V) |
|-----|---------------------------|-----------|----------------------|-----------|------------------------|----------------------|------------------------|
| 43  | Buprofezin                | LC        | 9.83                 | positive  | 306.1 → 201.0          | 306.1 → 116.0        | 12                     |
| 44  | Cadusafos (ebufos)        | LC        | 9.39                 | positive  | 271.1 → 159.0          | 271.1 → 131.0        | 16                     |
| 45  | Carbayl                   | LC        | 6.21                 | positive  | 202.1 → 145.1          | 202.1 → 127.1        | 4                      |
| 46  | Carbendazim (azole)       | LC        | 2.90                 | positive  | 192.1 → 160.1          | 202.1 → 127.1        | 4                      |
| 47  | Carbofuran                | LC        | 5.91                 | positive  | 222.1 → 123.1          | 222.1 → 165.1        | 20                     |
| 48  | Carbofuran-3-hydroxy      | LC        | 4.27                 | positive  | 238.1 → 163.1          | 238.1 → 181.1        | 10                     |
| 49  | Carbosulfan (two isomers) | LC        | 11.03                | positive  | 381.2 → 160.2          | 381.2 → 76.1         | 12                     |
| 50  | Cefuroxime axetil         | LC        | 5.13                 | positive  | 533.0 → 447.0          | 533.0 → 386.0        | 15                     |
| 51  | Chloramphenicol           | LC        | 4.63                 | negative  | 321.0 → 152.1          | 323.0 → 152.1        | 4                      |
| 52  | Chlorantraniliprole       | LC        | 7.32                 | positive  | 483.9 → 452.9          | 483.9 → 285.9        | 8                      |
| 53  | Chlorfenvinphos           | LC        | 9.09                 | positive  | 361.1 → 98.9           | 358.9 → 155.1        | 8                      |
| 54  | Chlorobenzilate           | GC        | 12.14                | positive  | 251.0 → 111.0          | 251.0 → 139.0        | 15                     |
| 55  | Chlorphacpine             | LC        | 8.88                 | negative  | 373.2 → 201.0          | 375.2 → 203.0        | 20                     |
| 56  | Chlorpropham              | GC        | 7.13                 | positive  | 213.0 → 127.0          | 153.0 → 90.0         | 25                     |
| 57  | Chlorpyrifos              | GC        | 9.93                 | positive  | 314.0 → 258.0          | 314.0 → 286.0        | 5                      |
| 58  | Chlorpyrifos methyl       | GC        | 9.12                 | positive  | 286.0 → 93.0           | 286.0 → 271.0        | 15                     |
| 59  | Chlorthal dimethyl        | GC        | 10.02                | positive  | 300.9 → 166.9          | 300.9 → 222.9        | 25                     |
| 60  | Chrysene                  | GC        | 13.86                | positive  | 228.0 → 226.0          | 228.0 → 227.0        | 25                     |
| 61  | Clindamycin               | LC        | 5.33                 | positive  | 425.2 → 126.1          | 425.2 → 377.2        | 20                     |
| 62  | Clofentazine               | LC        | 9.19                 | positive  | 303.1 → 138.0          | 303.1 → 102.0        | 12                     |
| 63  | Clothianidin              | LC        | 3.91                 | positive  | 250.0 → 169.0          | 250.0 → 131.9        | 8                      |
| 64  | Cloxacillin               | LC        | 6.86                 | positive  | 436.1 → 160.0          | 436.1 → 277.0        | 12                     |
| 65  | Coumachlor                | LC        | 8.63                 | positive  | 343.1 → 162.8          | 342.1 → 285.0        | 15                     |
Table A1. Cont.

| No. | Compound                          | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|-----------------------------------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 66  | Coumaphos                         | LC        | 8.98                 | positive | 363.0 → 227.0  | 30           | 363.0 → 306.9          | 15                      | 120                     |
| 67  | Coumatetralyl                     | LC        | 8.31                 | negative | 291.1 → 141.0  | 30           | 291.1 → 247.0          | 20                      | 140                     |
| 68  | Cyazofamid                        | LC        | 8.49                 | positive | 325.0 → 108.0  | 20           | 325.0 → 261.1          | 15                      | 90                      |
| 69  | Cyflufenamid                      | LC        | 9.18                 | positive | 413.1 → 223.1  | 33           | 413.1 → 295.1          | 23                      | 70                      |
| 70  | Cyfluthrin (sum of four isomers)  | GC        | 16.07/16.19/16.25/16.32 | positive | 226.0 → 206.0  | 25           | 198.9 → 170.1          | 25                      | 70                      |
| 71  | Cyhalothrin (lambda isomer)       | GC        | 10.49                | positive | 181.1 → 152.1  | 10           | 181.1 → 127.1          | 46                      | 70                      |
| 72  | Cymoxanil                         | LC        | 4.67                 | positive | 199.1 → 128.0  | 4            | 199.1 → 110.9          | 12                      | 90                      |
| 73  | Cypermethrin (sum of four isomers)| GC        | 16.34/16.44/16.52/16.63 | positive | 163.0 → 109.0  | 20           | 163.0 → 127.0          | 5                       | 70                      |
| 74  | Cyproconazole (two isomers)       | GC        | 11.98                | positive | 222.0 → 125.0  | 20           | 222.0 → 82.0           | 10                      | 70                      |
| 75  | Cyprodinil                        | LC        | 8.46                 | positive | 226.0 → 93.0   | 33           | 226.0 → 108.0          | 25                      | 100                     |
| 76  | Cyromazine                        | LC        | 1.23                 | positive | 167.1 → 85.0   | 16           | 167.1 → 125.0          | 20                      | 120                     |
| 77  | Danofloxacin                      | LC        | 4.04                 | positive | 358.2 → 340.1  | 20           | 358.2 → 82.1           | 50                      | 159                     |
| 78  | Dazomet                           | GC        | 7.80                 | positive | 161.9 → 44.0   | 28           | 161.9 → 89.0           | 5                       | 70                      |
| 79  | Deltamethrin                      | LC        | 10.65                | positive | 523.0 → 281.0  | 10           | 523.0 → 506.0          | 5                       | 100                     |
| 80  | Demeton-S-methyl                  | LC        | 5.97                 | positive | 230.9 → 88.9   | 5            | 230.9 → 61.0           | 30                      | 50                      |
| 81  | Demeton-S-methyl-sulfone (Dioxydemeton) | LC  | 3.31                | positive | 263.0 → 169.0  | 24           | 263.0 → 109.0          | 12                      | 120                     |
| 82  | Dexamethasone                     | LC        | 7.16                 | positive | 393.2 → 373.2  | 2            | 393.2 → 355.2          | 6                       | 103                     |
| 83  | Diazinon                          | GC        | 8.29                 | positive | 137.1 → 54.0   | 20           | 304.0 → 179.0          | 15                      | 70                      |
| 84  | Dibenzo[a,h]anthracene            | GC        | 19.15                | positive | 278.0 → 276.0  | 40           | 278.0 → 250.0          | 60                      | 70                      |
| No. | Compound                                      | Technique | Retention Time (min) | Polarity | Quantification       | Confirmation | Fragmentor Voltage (V) |
|-----|-----------------------------------------------|-----------|----------------------|----------|----------------------|--------------|------------------------|
| 85  | Dichlorodiphenylchloroethane (p,p′ DDD)       | GC        | 12.31                | positive | 235.0 → 165.0        | 20           | 15                     | 70 |
| 86  | Dichlorodiphenylchloroethylene (p,p′ DDE)     | GC        | 11.58                | positive | 318.0 → 176.0        | 60           | 30                     | 70 |
| 87  | Dichlorodiphenyltrichloroethane (p,p′ DDT)    | GC        | 12.84                | positive | 235.0 → 165.0        | 40           | 15                     | 70 |
| 88  | Diclofenac                                    | LC        | 8.73                 | positive | 296.0 → 215.1        | 16           | 16                     | 103|
| 89  | Dicloran                                      | GC        | 7.80                 | positive | 206.0 → 176.0        | 10           | 206.0 → 148.0          | 25 |
| 90  | Dichlorvos                                    | GC        | 4.74                 | positive | 184.9 → 93.0         | 10           | 185.0 → 109.0          | 15 |
| 91  | Dicloxacillin                                 | LC        | 7.24                 | positive | 470.0 → 160.0        | 8            | 470.0 → 310.8          | 10 |
| 92  | Dieldrin                                      | GC        | 11.66                | positive | 263.0 → 228.0        | 15           | 277.0 → 241.0          | 15 |
| 93  | Diethathyl ethyl                              | LC        | 8.71                 | positive | 312.2 → 238.1        | 15           | 312.2 → 162.0          | 30 |
| 94  | Diethofencarb                                 | LC        | 7.57                 | positive | 268.2 → 226.1        | 5            | 268.2 → 152.0          | 20 |
| 95  | Difencacoum                                   | LC        | 10.38                | negative | 443.2 → 135.0        | 40           | 443.2 → 293.0          | 35 |
| 96  | Difenconazole                                  | LC        | 9.41                 | positive | 406.1 → 250.9        | 28           | 406.1 → 337.0          | 16 |
| 97  | Difethialone                                  | LC        | 10.93                | negative | 537.3 → 79.0         | 50           | 537.3 → 151.0          | 45 |
| 98  | Difloxacin                                    | LC        | 3.86                 | positive | 400.2 → 382.1        | 20           | 400.2 → 356.1          | 16 |
| 99  | Diflubenzuron                                  | LC        | 8.63                 | positive | 311.0 → 158.0        | 8            | 311.0 → 141.0          | 32 |
| 100 | Diflufenican                                  | GC        | 13.27                | positive | 394.0 → 266.0        | 10           | 266.0 → 246.0          | 10 |
| 101 | Dimethenamid-P (and its R-isomer)             | LC        | 7.68                 | positive | 276.1 → 244.1        | 10           | 276.1 → 168.1          | 20 |
| 102 | Dimethoate                                    | LC        | 4.21                 | positive | 230.0 → 125.0        | 16           | 230.0 → 198.8          | 20 |
| No.  | Compound                                      | Technique | Retention Time (min) | Polarity | Quantification  | Confirmation | Fragmentor Voltage (V) |
|------|----------------------------------------------|-----------|----------------------|----------|-----------------|--------------|------------------------|
| 103  | Dimethomorph (two isomers)                   | LC        | 7.86                 | positive | 388.1 → 301.1   | 20           | 388.1 → 165.1          | 32                     |
| 104  | Dimethylphenylsulfamide (DMSA, metabolite of dichlofluanid) | LC        | 5.21                 | positive | 201.1 → 92.1    | 15           | 201.1 → 137.1          | 5                      |
| 105  | Diniconazole-M                               | LC        | 9.34                 | positive | 326.1 → 70.0    | 28           | 328.1 → 70.0           | 28                     | 110                   |
| 106  | Dinocap                                      | LC        | 10.51                | negative | 295.4 → 208.9   | 30           | 295.4 → 193.0          | 35                     | 150                   |
| 107  | Diphenylamine                                | GC        | 6.98                 | positive | 168.0 → 167.2   | 15           | 169.0 → 66.0           | 15                     | 70                    |
| 109  | Dodine                                       | LC        | 9.02                 | positive | 228.3 → 43.0    | 40           | 228.3 → 57.0           | 25                     | 150                   |
| 110  | Doramectina                                  | LC        | 11.31                | positive | 921.5 → 777.4   | 55           | 899.5 → 145.1          | 30                     | 220                   |
| 111  | Endosulfan alfa                              | GC        | 11.21                | positive | 241.0 → 206.0   | 15           | 195.0 → 160.0          | 10                     | 70                    |
| 112  | Endosulfan beta                              | GC        | 12.21                | positive | 241.0 → 206.0   | 15           | 195.0 → 159.0          | 15                     | 70                    |
| 113  | Endosulfan sulfate                           | GC        | 12.96                | positive | 270.0 → 235.0   | 15           | 387.0 → 289.0          | 5                      | 70                    |
| 114  | Endrin                                       | GC        | 12.05                | positive | 263.0 → 193.0   | 35           | 245.0 → 173.0          | 25                     | 70                    |
| 115  | Enrofloxacin                                 | LC        | 3.94                 | positive | 360.2 → 316.1   | 16           | 360.2 → 245.1          | 28                     | 144                   |
| 116  | EPN                                          | GC        | 13.90                | positive | 157.0 → 63.0    | 10           | 157.0 → 110.0          | 15                     | 70                    |
| 117  | Epoxiconazole                                | LC        | 8.47                 | positive | 330.0 → 120.9   | 24           | 330.1 → 100.9          | 50                     | 120                   |
| 118  | Eprinomectin                                 | LC        | 10.84                | positive | 878.5 → 186.0   | 15           | 936.5 → 490.4          | 60                     | 160                   |
| 119  | Eritromycin                                  | GC        | 6.74                 | positive | 734.5 → 158.1   | 32           | 734.5 → 576.3          | 16                     | 172                   |
| 120  | Esfenvalerate                                | GC        | 17.56                | positive | 167.1 → 125.1   | 15           | 167.1 → 89.1           | 45                     | 70                    |
| 121  | Ethion (diethion)                            | LC        | 10.03                | positive | 385.0 → 199.0   | 5            | 385.0 → 171.0          | 10                     | 100                   |
| 122  | Ethirimol                                    | LC        | 4.80                 | positive | 210.2 → 140.1   | 20           | 210.2 → 98.1           | 28                     | 160                   |
| 123  | Ethofumesate                                 | GC        | 9.59                 | positive | 286.0 → 207.0   | 5            | 286.0 → 161.0          | 20                     | 70                    |
| 124  | Ethoprophos                                  | LC        | 8.38                 | positive | 243.1 → 97.0    | 30           | 243.1 → 130.9          | 15                     | 90                    |
### Table A1. Cont.

| No. | Compound               | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|------------------------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 125 | Etofenprox             | GC        | 16.75                | positive | 163.0 → 107.0  | 20           | 163.0 → 135.0          | 10 | 70 |
| 126 | Etoxazole              | LC        | 10.34                | positive | 360.1 → 141.0  | 26           | 360.1 → 304.0          | 16 | 160 |
| 127 | Farnoxadone            | LC        | 9.07                 | positive | 392.1 → 330.9  | 5            | 392.1 → 238.1          | 12 | 110 |
| 128 | Fenamidone             | LC        | 9.06                 | positive | 392.1 → 330.9  | 5            | 392.1 → 238.1          | 12 | 110 |
| 129 | Fenamiphos             | LC        | 7.72                 | positive | 304.1 → 217.1  | 20           | 304.1 → 202.0          | 36 | 120 |
| 130 | Fenamiphos sulfone     | LC        | 8.63                 | positive | 336.1 → 188.0  | 31           | 336.1 → 266.0          | 23 | 120 |
| 131 | Fenamiphos sulfoxide   | LC        | 5.93                 | positive | 320.1 → 233.0  | 20           | 320.1 → 108.1          | 44 | 120 |
| 132 | Fenarimol              | GC        | 15.03                | positive | 139.0 → 75.0   | 30           | 139.0 → 111.0          | 15 | 70 |
| 133 | Fenazaquin             | LC        | 10.73                | positive | 207.2 → 57.1   | 25           | 207.2 → 161.1          | 16 | 90 |
| 134 | Fenbendazole           | LC        | 8.04                 | positive | 300.1 → 268.1  | 20           | 300.1 → 159.0          | 36 | 156 |
| 135 | Fenbuconazole          | GC        | 16.17                | positive | 198.0 → 102.0  | 30           | 198.0 → 78.0           | 30 | 70 |
| 136 | Fenhexamid             | LC        | 8.35                 | positive | 302.1 → 97.1   | 20           | 302.1 → 55.1           | 40 | 130 |
| 137 | Fenitrothion           | GC        | 9.57                 | positive | 277.0 → 109.0  | 15           | 277.0 → 125.0          | 15 | 70 |
| 138 | Fenoxythion            | LC        | 8.69                 | positive | 302.1 → 88.0   | 20           | 302.1 → 116.1          | 10 | 110 |
| 139 | Fenpropthrin           | LC        | 10.43                | positive | 367.2 → 125.0  | 16           | 350.1 → 125.0          | 16 | 72 |
| 140 | Fenpropidin            | LC        | 7.13                 | positive | 274.3 → 147.0  | 30           | 274.3 → 86.0           | 25 | 170 |
| 141 | Fenpropimorph          | LC        | 7.37                 | positive | 304.3 → 147.1  | 30           | 304.3 → 130.0          | 25 | 120 |
| 142 | Fenpyroximate          | LC        | 10.49                | positive | 422.2 → 366.2  | 12           | 422.2 → 135.0          | 36 | 160 |
| 143 | Fenthion               | GC        | 8.90                 | positive | 278.0 → 109.0  | 15           | 278.0 → 125.0          | 20 | 70 |
| 144 | Fenthion oxon          | LC        | 7.31                 | positive | 263.1 → 231.2  | 16           | 263.1 → 216.0          | 24 | 120 |
| 145 | Fenthion oxon sulfone  | LC        | 4.50                 | positive | 295.3 → 217.0  | 15           | 295.0 → 104.2          | 24 | 110 |
| 146 | Fenthion oxon sulfoxide| LC        | 4.26                 | positive | 279.0 → 264.2  | 20           | 279.0 → 104.1          | 28 | 110 |
| 147 | Fenthion sulfone       | LC        | 6.39                 | positive | 311.0 → 125.0  | 22           | 311.0 → 109.0          | 28 | 140 |
| 148 | Fenthion sulfoxide     | LC        | 6.16                 | positive | 295.0 → 108.9  | 30           | 295.0 → 280.0          | 18 | 140 |
| 149 | Fenvalerate            | GC        | 17.36                | positive | 167.0 → 125.1  | 22           | 167.0 → 89.0           | 30 | 70 |
Table A1. Cont.

| No. | Compound                        | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|---------------------------------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 150 | Fipronil                        | LC        | 8.68                 | negative | 435.0 → 330.0 | 12           | 435.0 → 249.9          | 26                     | 116                    |
| 151 | Fipronil sulfide                | GC        | 10.49                | positive | 351.0 → 255.0 | 20           | 420.0 → 351.0          | 25                     | 70                     |
| 152 | Flucoumafen                     | LC        | 10.44                | negative | 541.3 → 382.0 | 25           | 541.3 → 161.0          | 40                     | 230                    |
| 153 | Fluazinam                       | LC        | 10.01                | negative | 462.9 → 416.0 | 10           | 462.9 → 398.0          | 9                      | 140                    |
| 154 | Flubendiamide                   | LC        | 8.82                 | positive | 408.0 → 274.0 | 15           | 408.0 → 256.0          | 30                     | 120                    |
| 155 | Flucythrinate (two isomers)     | GC        | 16.67/16.84          | positive | 156.9 → 107.1 | 15           | 199.1 → 107.1          | 25                     | 70                     |
| 156 | Fludioxonil                     | GC        | 11.51                | positive | 248.0 → 127.0 | 30           | 248.1 → 182.1          | 10                     | 70                     |
| 157 | Fluphenoxuron                    | LC        | 10.37                | positive | 489.1 → 158.0 | 20           | 489.1 → 140.9          | 56                     | 110                    |
| 158 | Flumequine                      | LC        | 6.12                 | positive | 262.1 → 244.0 | 16           | 262.1 → 202.0          | 32                     | 116                    |
| 159 | Flunixin                        | LC        | 8.09                 | positive | 297.1 → 279.1 | 24           | 297.1 → 264.1          | 32                     | 141                    |
| 160 | Fluopyram                       | GC        | 10.61                | positive | 173.0 → 95.0  | 35           | 223.0 → 196.0          | 40                     | 70                     |
| 161 | Fluoranthene                    | GC        | 10.66                | positive | 202.0 → 201.0 | 27           | 202.0 → 152.0          | 42                     | 70                     |
| 162 | Fluorene                        | GC        | 6.81                 | positive | 165.0 → 163.0 | 40           | 165.0 → 139.0          | 30                     | 70                     |
| 163 | Fluquinconazole                 | GC        | 15.81                | positive | 340.0 → 298.0 | 15           | 340.0 → 286.0          | 25                     | 70                     |
| 164 | Flusilazole                     | LC        | 8.64                 | positive | 316.1 → 247.1 | 15           | 316.1 → 165.0          | 20                     | 160                    |
| 165 | Flutolanil                      | LC        | 7.93                 | positive | 324.1 → 262.1 | 16           | 324.1 → 242.1          | 24                     | 130                    |
| 166 | Flutriafol                      | GC        | 11.26                | positive | 219.0 → 95.0  | 35           | 219.0 → 123.0          | 15                     | 70                     |
| 167 | Fluvalinate tau                 | GC        | 17.56                | positive | 250.1 → 55.1  | 30           | 252.0 → 200.0          | 20                     | 70                     |
| 168 | Fonofos                         | GC        | 8.24                 | positive | 246.0 → 109.0 | 15           | 246.0 → 237.0          | 5                      | 70                     |
| 169 | Formetanate                     | LC        | 1.76                 | positive | 222.1 → 165.1 | 12           | 222.1 → 46.2           | 28                     | 105                    |
| 170 | Fosthiazate                     | LC        | 6.50                 | positive | 284.0 → 104.0 | 20           | 284.0 → 227.8          | 8                      | 90                     |
| 171 | Heptachlor                      | GC        | 9.31                 | positive | 272.0 → 237.0 | 15           | 274.0 → 239.0          | 15                     | 70                     |
| 172 | Hexachlorobencene               | GC        | 7.77                 | positive | 284.0 → 214.0 | 40           | 284.0 → 249.0          | 25                     | 70                     |
| No. | Compound | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|----------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 173 | Hexachlorocyclohexane (alpha) | GC | 7.64 | positive | 219.0 → 109.0 | 10 | 219.0 → 183.0 | 10 | 70 |
| 174 | Hexachlorocyclohexane (beta) | GC | 8.02 | positive | 219.0 → 109.0 | 40 | 219.0 → 183.0 | 5 | 70 |
| 175 | Hexachlorocyclohexane (delta) | GC | 8.50 | positive | 219.0 → 109.0 | 45 | 219.0 → 183.0 | 5 | 70 |
| 176 | Hexachlorocyclohexane (gamma. lindane) | GC | 8.13 | positive | 291.0 → 109.0 | 40 | 219.0 → 183.0 | 10 | 70 |
| 177 | Hexaconazole (two isomers) | LC | 8.49 | positive | 314.1 → 70.1 | 20 | 316.0 → 70.1 | 20 | 95 |
| 178 | Hexaflumuron | LC | 9.58 | negative | 458.8 → 439.0 | 8 | 458.8 → 175.0 | 30 | 100 |
| 179 | Hexythiazox | LC | 10.18 | positive | 353.1 → 227.9 | 8 | 353.1 → 168.1 | 24 | 120 |
| 180 | Imazalil (enilconazole) | LC | 6.53 | positive | 297.1 → 159.0 | 20 | 297.1 → 69.1 | 18 | 100 |
| 181 | Imidacloprid | LC | 3.93 | positive | 256.0 → 175.0 | 12 | 256.0 → 209.0 | 12 | 110 |
| 182 | Indeno [1,2,3-cd] pyrene | GC | 19.08 | positive | 276.0 → 274.0 | 50 | 276.0 → 272.0 | 60 | 70 |
| 183 | Indoxacarb | LC | 9.49 | positive | 528.1 → 293.1 | 10 | 528.1 → 202.8 | 48 | 140 |
| 184 | Iprovalicarb | LC | 8.18 | positive | 321.2 → 119.0 | 15 | 321.2 → 202.9 | 20 | 110 |
| 185 | Isofenphos methyl | GC | 10.38 | positive | 199.0 → 121.0 | 10 | 241.0 → 121.0 | 25 | 70 |
| 186 | Isoprotiolane | LC | 7.94 | positive | 291.1 → 189.0 | 30 | 291.1 → 145.0 | 36 | 100 |
| 187 | Ivermectin B1a | LC | 11.52 | positive | 897.5 → 733.5 | 50 | 897.5 → 329.3 | 60 | 160 |
| 188 | Josamycin | LC | 7.40 | positive | 860.5 → 173.9 | 40 | 860.5 → 108.9 | 40 | 200 |
| 189 | Ketoprofen | LC | 7.34 | positive | 255.1 → 209.1 | 8 | 255.1 → 77.1 | 48 | 123 |
| 190 | Kresoxim methyl | GC | 11.78 | positive | 116.0 → 89.0 | 15 | 206.0 → 131.0 | 10 | 70 |
| 191 | Levamisole | LC | 3.12 | positive | 205.1 → 178.1 | 20 | 205.1 → 123.0 | 32 | 141 |
| 192 | Lincomycin | LC | 3.50 | positive | 407.2 → 126.1 | 24 | 407.2 → 359.2 | 16 | 150 |
| 193 | Linuron | LC | 7.54 | positive | 249.0 → 160.1 | 20 | 249.0 → 182.3 | 8 | 120 |
Table A1. Cont.

| No. | Compound                        | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|---------------------------------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 194 | Lufenuron                       | LC        | 10.05                | negative | 509.0 → 339.0 | 5            | 509.0 → 326.1          | 15 | 90 |
| 195 | Mandipropamid                   | LC        | 7.90                 | positive | 412.1 → 328.1 | 8            | 412.1 → 356.1          | 4  | 130 |
| 196 | Mebendazole                     | LC        | 6.68                 | positive | 296.1 → 264.1 | 20           | 296.1 → 77.0           | 48 | 151 |
| 197 | Mefenamic acid                  | LC        | 9.52                 | positive | 242.1 → 209.1 | 28           | 242.1 → 180.1          | 0  | 108 |
| 198 | Mefenoxam (metalaxyl-M)         | LC        | 6.95                 | positive | 280.0 → 220.0 | 10           | 280.0 → 192.0          | 15 | 110 |
| 199 | Meloxicam                       | LC        | 7.17                 | positive | 352.5 → 114.8 | 20           | 352.5 → 140.8          | 20 | 130 |
| 200 | Mepanipyrim                     | GC        | 11.13                | positive | 222.0 → 221.0 | 15           | 222.0 → 207.0          | 15 | 70  |
| 201 | Meptiqual                      | LC        | 0.64                 | positive | 114.0 → 98.0  | 36           | 114.0 → 70.0           | 45 | 100 |
| 202 | Metaflumizone                   | LC        | 9.94                 | negative | 505.0 → 302.0 | 14           | 541.0 → 302.0          | 20 | 90  |
| 203 | Metalaxyl                       | GC        | 9.31                 | positive | 234.0 → 146.1 | 20           | 249.0 → 146.0          | 20 | 70  |
| 204 | Metaldehyde                     | LC        | 3.87                 | positive | 194.1 → 61.9  | 5            | 194.1 → 106.0          | 5  | 50  |
| 205 | Metconazole                     | LC        | 9.17                 | positive | 320.1 → 70.2  | 33           | 322.1 → 70.2           | 24 | 250 |
| 206 | Methamidophos (two isomers)     | LC        | 1.18                 | positive | 142.0 → 94.0  | 12           | 142.0 → 125.0          | 12 | 85  |
| 207 | Methidathion                    | LC        | 7.12                 | positive | 320.1 → 144.8 | 8            | 320.1 → 85.0           | 30 | 84  |
| 208 | Methiocarb                      | LC        | 7.67                 | positive | 226.1 → 169.0 | 4            | 226.1 → 121.1          | 12 | 90  |
| 209 | Methiocarb-sufone               | LC        | 4.52                 | positive | 258.1 → 201.1 | 8            | 258.1 → 122.1          | 22 | 100 |
| 210 | Methiocarb-sulfoxide            | LC        | 4.03                 | positive | 242.0 → 185.0 | 22           | 242.0 → 122.0          | 28 | 90  |
| 211 | Methomyl                        | LC        | 3.23                 | positive | 163.1 → 88.0  | 5            | 163.0 → 106.0          | 8  | 80  |
| 212 | Methoxyfenozide                 | LC        | 8.00                 | positive | 369.2 → 149.0 | 10           | 369.2 → 313.1          | 15 | 85  |
| 213 | Metoxychlor                     | GC        | 13.98                | positive | 227.0 → 141.0 | 20           | 227.0 → 169.0          | 15 | 70  |
| 214 | Metrafenone                     | LC        | 9.27                 | positive | 409.1 → 209.1 | 8            | 411.1 → 209.1          | 12 | 108 |
| 215 | Metronidazole                   | LC        | 2.63                 | positive | 172.1 → 128.0 | 12           | 172.1 → 82.1           | 24 | 98  |
| 216 | Mevinphos (phosdrin)            | LC        | 4.38                 | positive | 225.0 → 193.1 | 15           | 225.0 → 127.0          | 12 | 65  |
| No. | Compound                                                                 | Technique | Retention Time (min) | Polarity | Quantification         | Confirmation          | Fragmentor Voltage (V) |
|-----|---------------------------------------------------------------------------|-----------|----------------------|----------|------------------------|-----------------------|------------------------|
| 217 | Mirex                                                                     | GC        | 5.66                 | positive | 237.0 → 143.0          | 30                    | 274.0 → 237.0          | 10                     | 70                     |
| 218 | Monocrotophos                                                             | LC        | 3.31                 | positive | 224.1 → 126.8          | 12                    | 224.1 → 98.1           | 15                     | 100                    |
| 219 | Moxidectin                                                                | LC        | 11.24                | positive | 641.4 → 529.2          | 5                     | 641.4 → 499.2          | 5                      | 100                    |
| 220 | Myclobutanil                                                              | LC        | 8.10                 | positive | 289.1 → 70.1           | 16                    | 289.1 → 125.1          | 32                     | 110                    |
| 221 | N-(2,4-dimethylphenyl)-N′-methylformamidine (DMPF, metabolite of amitraz) | LC        | 3.35                 | positive | 163.1 → 122.1          | 15                    | 163.1 → 107.1          | 15                     | 100                    |
| 222 | N,N-Dimethyl-N′-p-tolysulphamide (DMST, metabolite of tolyfluanid)        | LC        | 6.06                 | positive | 215.1 → 106.1          | 10                    | 215.1 → 151.1          | 4                      | 90                     |
| 223 | Nafcilin                                                                  | LC        | 7.33                 | positive | 415.0 → 199.1          | 8                     | 415.0 → 171.0          | 36                     | 103                    |
| 224 | Naphtalene                                                                | GC        | 4.45                 | positive | 128.0 → 127.0          | 15                    | 128.0 → 102.0          | 25                     | 70                     |
| 225 | Naproxen                                                                  | LC        | 7.59                 | positive | 231.0 → 185.0          | 10                    | 231.0 → 169.9          | 21                     | 120                    |
| 226 | Nitenpyram                                                                | LC        | 3.30                 | positive | 271.1 → 56.1           | 36                    | 271.1 → 224.9          | 12                     | 100                    |
| 227 | Novobiocin                                                                | LC        | 9.69                 | positive | 613.2 → 218.1          | 10                    | 613.2 → 396.1          | 10                     | 150                    |
| 228 | Nuarimol                                                                  | GC        | 13.27                | positive | 235.0 → 139.0          | 15                    | 235.0 → 111.0          | 40                     | 70                     |
| 229 | Ofurace                                                                   | LC        | 5.97                 | positive | 282.0 → 159.9          | 20                    | 282.0 → 147.9          | 30                     | 100                    |
| 230 | Omethoate                                                                 | LC        | 2.80                 | positive | 214.1 → 124.8          | 22                    | 214.1 → 183.0          | 5                      | 100                    |
| 231 | Oxadixyl                                                                  | LC        | 5.43                 | positive | 279.1 → 219.2          | 5                     | 279.1 → 132.2          | 32                     | 110                    |
| 232 | Oxamyl                                                                    | LC        | 2.87                 | positive | 237.1 → 72.0           | 12                    | 237.1 → 90.0           | 5                      | 70                     |
| 233 | Oxamyl-oxime                                                              | LC        | 2.46                 | positive | 163.3 → 115.2          | 10                    | 163.3 → 72.1           | 10                     | 70                     |
| 234 | Oxendazole                                                                | LC        | 5.61                 | positive | 316.1 → 159.0          | 32                    | 316.1 → 191.1          | 16                     | 166                    |
| 235 | Oxolinic acid                                                             | LC        | 5.04                 | positive | 262.1 → 216.0          | 32                    | 262.1 → 160.0          | 36                     | 110                    |
| 236 | Oxydemeton methyl                                                         | LC        | 3.01                 | positive | 247.0 → 169.0          | 12                    | 247.0 → 109.0          | 24                     | 100                    |
| 237 | Oxyfluorfen                                                               | GC        | 11.68                | positive | 252.0 → 146.0          | 40                    | 300.0 → 223.0          | 15                     | 70                     |
Table A1. Cont.

| No. | Compound          | Technique | Retention Time (min) | Polarity | Quantification          | Confirmation          | Fragmentor Voltage (V) |
|-----|-------------------|-----------|----------------------|----------|-------------------------|-----------------------|------------------------|
| 238 | Paclobutrazol     | LC        | 7.89                 | positive | 294.1 → 70.1            | 294.1 → 125.2         | 16                     |
| 239 | Parathion methyl  | GC        | 9.12                 | positive | 263.0 → 109.0           | 263.0 → 79.0          | 15                     |
| 240 | PCB 28            | GC        | 9.58                 | positive | 256.0 → 186.0           | 256.0 → 151.0         | 25                     |
| 241 | PCB 52            | GC        | 11.73                | positive | 292.0 → 220.0           | 292.0 → 222.0         | 25                     |
| 242 | PCB 77            | GC        | 11.56                | positive | 292.0 → 220.0           | 292.0 → 222.0         | 25                     |
| 243 | PCB 81            | GC        | 11.56                | positive | 292.0 → 220.0           | 292.0 → 222.0         | 25                     |
| 244 | PCB 101           | GC        | 11.08                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 245 | PCB 105           | GC        | 12.66                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 246 | PCB 114           | GC        | 12.38                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 247 | PCB 118           | GC        | 12.18                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 248 | PCB 123           | GC        | 12.10                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 249 | PCB 126           | GC        | 13.23                | positive | 326.0 → 256.0           | 328.0 → 256.0         | 30                     |
| 250 | PCB 138           | GC        | 13.07                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 251 | PCB 153           | GC        | 12.57                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 252 | PCB 156           | GC        | 13.96                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 253 | PCB 157           | GC        | 14.07                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 254 | PCB 167           | GC        | 13.55                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 255 | PCB 169           | GC        | 14.61                | positive | 360.0 → 290.0           | 360.0 → 288.0         | 25                     |
| 256 | PCB 180           | GC        | 14.25                | positive | 394.0 → 324.0           | 394.0 → 322.0         | 30                     |
| 257 | PCB 189           | GC        | 15.25                | positive | 394.0 → 324.0           | 394.0 → 322.0         | 30                     |
| 258 | Penconazole       | GC        | 10.52                | positive | 248.0 → 157.0           | 248.0 → 192.0         | 15                     |
| 259 | Pencycuron        | LC        | 9.33                 | positive | 329.1 → 125.1           | 329.1 → 127.9         | 12                     |
| 260 | Pendimethalin     | GC        | 10.49                | positive | 252.0 → 162.0           | 252.0 → 191.0         | 5                      |
| 261 | Penicillin V      | LC        | 6.47                 | positive | 383.2 → 159.9           | 383.2 → 113.9         | 40                     |
| No. | Compound                  | Technique | Retention Time (min) | Polarity | Quantification       | Confirmation         | Fragmentor Voltage (V) |
|-----|--------------------------|-----------|----------------------|----------|----------------------|----------------------|------------------------|
| 262 | Permethrin               | GC        | 15.69                | positive | 183.0 → 128.0        | 15                   | 183.1 → 153.1          | 15                     | 70                     |
| 263 | Phenanthrene             | GC        | 8.40                 | positive | 178.0 → 176.0        | 35                   | 178.0 → 152.0          | 28                     | 70                     |
| 264 | Phenylbutazone           | LC        | 8.25                 | positive | 309.2 → 160.2        | 20                   | 309.2 → 77.1           | 55                     | 140                    |
| 265 | Phosalone                | LC        | 9.20                 | positive | 385.1 → 182.0        | 20                   | 385.1 → 110.9          | 55                     | 80                     |
| 266 | Phosmet                  | LC        | 7.34                 | positive | 318.0 → 159.9        | 16                   | 318.0 → 133.0          | 40                     | 90                     |
| 267 | Pthalamide (Folpet deg)  | GC        | 5.94                 | positive | 104.0 → 50.0         | 25                   | 147.0 → 76.0           | 25                     | 70                     |
| 268 | Pirimicarb               | LC        | 5.11                 | positive | 239.1 → 72.1         | 20                   | 239.1 → 182.1          | 12                     | 100                    |
| 269 | Pirimicarb-desmethyl     | LC        | 3.71                 | positive | 225.1 → 168.1        | 8                    | 225.1 → 72.1           | 20                     | 100                    |
| 270 | Pirimiphos ethyl         | GC        | 10.26                | positive | 318.0 → 166.0        | 15                   | 318.0 → 182.0          | 15                     | 70                     |
| 271 | Pirimiphos methyl        | LC        | 9.13                 | positive | 306.1 → 164.0        | 20                   | 306.1 → 108.1          | 32                     | 100                    |
| 272 | Prochloraz               | LC        | 9.08                 | positive | 376.0 → 308.0        | 10                   | 376.0 → 70.1           | 20                     | 100                    |
| 273 | Procymidone              | GC        | 10.80                | positive | 283.0 → 67.0         | 40                   | 283.0 → 68.0           | 25                     | 70                     |
| 274 | Profenofos               | LC        | 9.75                 | positive | 375.0 → 305.0        | 20                   | 373.0 → 303.0          | 20                     | 100                    |
| 275 | Propamocarb              | LC        | 2.85                 | positive | 189.2 → 102.0        | 12                   | 189.2 → 144.0          | 8                      | 110                    |
| 276 | Propargite               | LC        | 10.37                | positive | 368.2 → 231.1        | 4                    | 368.2 → 175.0          | 12                     | 88                     |
| 277 | Propiconazole            | LC        | 9.01                 | positive | 342.0 → 69.0         | 21                   | 342.0 → 159.0          | 39                     | 90                     |
| 278 | Propoxur                 | LC        | 5.83                 | positive | 210.1 → 168.1        | 35                   | 210.1 → 65.1           | 40                     | 70                     |
| 279 | Propyzamide (pronamide)  | LC        | 7.92                 | positive | 256.1 → 190.0        | 16                   | 256.1 → 173.0          | 25                     | 90                     |
| 280 | Proquinazid              | GC        | 13.32                | positive | 288.0 → 245.0        | 15                   | 288.0 → 217.0          | 30                     | 70                     |
| 281 | Prothioconazol           | GC        | 11.85                | positive | 186.0 → 49.0         | 20                   | 186.0 → 53.0           | 25                     | 70                     |
| 282 | Prothiophos              | GC        | 11.45                | positive | 266.9 → 221.0        | 35                   | 162.0 → 63.1           | 30                     | 70                     |
| 283 | Pymetrozine              | LC        | 2.74                 | positive | 218.1 → 105.0        | 20                   | 218.1 → 78.0           | 52                     | 120                    |
| 284 | Pyraclostrobin           | LC        | 9.15                 | positive | 388.1 → 193.8        | 8                    | 388.1 → 163.1          | 28                     | 120                    |
Table A1. Cont.

| No. | Compound            | Technique | Retention Time (min) | Polarity | Quantification | Confirmation | Fragmentor Voltage (V) |
|-----|---------------------|-----------|----------------------|----------|----------------|--------------|------------------------|
| 285 | Pyrazophos          | LC        | 9.22                 | positive | 374.1 → 222.1 | 23           | 374.1 → 194.0          | 100                    |
| 286 | Pyrene              | GC        | 11.13                | positive | 202.0 → 201.0 | 27           | 202.0 → 200.0          | 70                     |
| 287 | Pyridaben           | LC        | 10.75                | positive | 365.2 → 309.0 | 8            | 309.1 → 147.0          | 168                    |
| 288 | Pyridaphenthion     | LC        | 8.11                 | positive | 341.0 → 189.0 | 22           | 341.0 → 205.0          | 100                    |
| 289 | Pyrimethanil        | GC        | 8.27                 | positive | 198.0 → 118.0 | 40           | 198.0 → 158.0          | 70                     |
| 290 | Pyriproxifen        | LC        | 10.07                | positive | 322.2 → 96.0  | 12           | 322.2 → 184.9          | 80                     |
| 291 | Quinalfos           | LC        | 8.72                 | positive | 299.1 → 96.9  | 30           | 299.1 → 147.1          | 130                    |
| 292 | Quinoxyfen          | LC        | 10.13                | positive | 308.0 → 197.0 | 32           | 308.2 → 161.8          | 120                    |
| 293 | Rifampicin          | LC        | 7.89                 | positive | 823.5 → 791.4 | 15           | 823.5 → 399.1          | 160                    |
| 294 | Rotenone            | LC        | 8.64                 | positive | 395.1 → 213.1 | 20           | 395.1 → 192.1          | 150                    |
| 295 | Roxithromycin       | LC        | 7.67                 | positive | 838.5 → 158.1 | 40           | 838.5 → 116.1          | 200                    |
| 296 | Sarafloxacin        | LC        | 4.16                 | positive | 386.1 → 342.1 | 16           | 386.1 → 299.1          | 144                    |
| 297 | Simazine            | LC        | 5.81                 | positive | 202.4 → 68.1  | 30           | 202.4 → 68.1           | 120                    |
| 298 | Spinosad (two isomers) | LC   | 9.10/9.43            | positive | 732.4 → 142.0 | 22           | 732.4 → 98.0           | 130                    |
| 299 | Spiramycin (two isomers) | LC | 4.58/4.90        | positive | 439.1 → 101.1 | 20           | 439.1 → 88.0           | 70                     |
| 300 | Spirodiclofen       | LC        | 10.50                | positive | 411.1 → 71.2  | 15           | 411.1 → 313.0          | 110                    |
| 301 | Spiromesifen        | LC        | 10.27                | positive | 388.0 → 273.1 | 25           | 273.0 → 187.0          | 110                    |
| 302 | Spirotetramat       | LC        | 8.23                 | positive | 374.2 → 302.2 | 12           | 374.2 → 216.1          | 150                    |
| 303 | Spiroxamine         | LC        | 7.55                 | positive | 298.3 → 144.1 | 16           | 298.3 → 100.1          | 120                    |
| 304 | Strychnine          | LC        | 3.00/3.61            | positive | 335.1 → 184.0 | 45           | 335.1 → 156.0          | 105                    |
| 305 | Sulfacetamide       | LC        | 2.13                 | positive | 215.3 → 155.9 | 10           | 215.3 → 92.0           | 90                     |
| 306 | Sulfachloropiridacine | LC    | 3.77                 | positive | 285.0 → 156.0 | 12           | 285.0 → 92.1           | 101                    |
| 307 | Sulfadiacline       | LC        | 2.80                 | positive | 251.0 → 92.0  | 28           | 251.0 → 156.0          | 111                    |
| 308 | Sulfadimethoxine    | LC        | 4.81                 | positive | 311.0 → 92.0  | 32           | 311.0 → 156.0          | 139                    |
| No. | Compound               | Technique | Retention Time (min) | Polarity | Quantification       | Confirmation    | Fragmentor Voltage (V) |
|-----|------------------------|-----------|----------------------|----------|----------------------|-----------------|------------------------|
| 309 | Sulfadoxine            | LC        | 4.12                 | positive | 311.1 → 92.0         | 32              | 311.1 → 156.0          | 16 | 126               |
| 310 | Sulfameracine          | LC        | 3.26                 | positive | 265.0 → 92.0         | 28              | 265.0 → 156.0          | 12 | 126               |
| 311 | Sulfametacine          | LC        | 3.44                 | positive | 279.1 → 186.0        | 12              | 279.1 → 92.0           | 32 | 134               |
| 312 | Sulfametizole          | LC        | 3.37                 | positive | 271.0 → 92.0         | 28              | 271.0 → 155.9          | 8  | 103               |
| 313 | Sulfamethoxazole       | LC        | 3.93                 | positive | 254.0 → 92.0         | 28              | 254.0 → 156.0          | 12 | 111               |
| 314 | Sulfametoxipiridacine  | LC        | 3.45                 | positive | 281.0 → 155.9        | 12              | 281.0 → 92.1           | 28 | 121               |
| 315 | Sulfamonomethoxine     | LC        | 4.11                 | positive | 281.1 → 156.0        | 14              | 281.1 → 92.1           | 32 | 120               |
| 316 | Sulfapyridine          | LC        | 2.82                 | positive | 250.0 → 156.0        | 12              | 250.0 → 92.0           | 28 | 126               |
| 317 | Sulfatoxine            | LC        | 4.99                 | positive | 301.0 → 156.0        | 12              | 301.0 → 92.1           | 32 | 159               |
| 318 | Sulfamefoxazone        | LC        | 2.98                 | positive | 268.0 → 156.0        | 8               | 268.0 → 92.1           | 24 | 106               |
| 319 | Sulfadiazole           | LC        | 4.12                 | positive | 308.2 → 70.2         | 22              | 308.2 → 125.1          | 53 | 120               |
| 320 | Sulfathiazole          | LC        | 8.92                 | positive | 353.1 → 132.9        | 22              | 353.1 → 297.1          | 20 | 90                |
| 321 | Sulfanilamide          | GC        | 8.66                 | positive | 334.2 → 117.0        | 47              | 334.2 → 145.0          | 37 | 180               |
| 322 | Tebufenpyrad           | GC        | 9.88                 | positive | 339.0 → 339.0        | 15              | 379.0 → 196.0          | 25 | 100               |
| 323 | Tebufenpyrid           | GC        | 10.01                | negative | 270.0 → 127.0        | 15              | 177.0 → 87.0           | 15 | 70                |
| 324 | Telodrin (isobenzan)   | GC        | 10.14                | positive | 310.8 → 240.8        | 25              | 310.8 → 274.8          | 5  | 70                |
| 325 | Telodrin               | GC        | 8.15                 | positive | 231.0 → 97.0         | 20              | 231.0 → 129.0          | 15 | 70                |
| 326 | Tebufos                | GC        | 8.12                 | positive | 214.0 → 104.0        | 20              | 214.0 → 132.0          | 10 | 70                |
| 327 | Tetrahydrolazine       | GC        | 8.72                 | positive | 367.0 → 127.0        | 16              | 365.0 → 127.0          | 16 | 110               |
| 328 | Tetrachlorvinphos      | GC        | 10.04                | positive | 336.0 → 204.0        | 35              | 336.0 → 218.0          | 20 | 70                |
| 329 | Tetrachlorphos         | GC        | 14.36                | positive | 158.9 → 111.0        | 20              | 354.0 → 159.0          | 10 | 70                |
| 330 | Tetrachlorvinphos      | GC        | 13.87                | positive | 164.0 → 77.0         | 30              | 164.0 → 107.0          | 15 | 70                |
| 332 | Thiabendazole          | GC        | 5.94                 | positive | 201.0 → 174.0        | 15              | 201.0 → 130.0          | 30 | 70                |
Table A1. Cont.

| No. | Compound                  | Technique | Retention Time (min) | Polarity  | Quantification          | Confirmation      | Fragmentor Voltage (V) |
|-----|---------------------------|-----------|----------------------|-----------|-------------------------|-------------------|------------------------|
| 333 | Thiacloprid               | LC        | 4.80                 | positive  | 253.0 → 126.0           | 16                | 253.0 → 90.0           | 40 | 140 |
| 334 | Thiamethoxam              | LC        | 3.59                 | positive  | 292.0 → 211.1           | 8                 | 292.0 → 132.0          | 22 | 80  |
| 335 | Thiophanate methyl        | LC        | 5.87                 | positive  | 343.0 → 151.0           | 20                | 343.0 → 93.0           | 46 | 90  |
| 336 | Tolclofos methyl          | GC        | 9.21                 | positive  | 265.0 → 93.0            | 30                | 265.0 → 220.0          | 25 | 70  |
| 337 | Tolfenamic acid           | LC        | 9.80                 | negative  | 260.0 → 216.1           | 8                 | 260.0 → 35.1           | 20 | 108 |
| 338 | Triadimefon               | LC        | 8.03                 | positive  | 294.1 → 69.3            | 20                | 294.1 → 197.2          | 15 | 100 |
| 339 | Triadimenol               | LC        | 8.22                 | positive  | 296.1 → 70.0            | 10                | 298.1 → 70.0           | 10 | 80  |
| 340 | Triazophos (hostathion)   | LC        | 8.18                 | positive  | 314.1 → 162.0           | 19                | 314.1 → 118.9          | 35 | 100 |
| 341 | Trifloxystrobin           | LC        | 9.50                 | positive  | 409.1 → 186.0           | 12                | 409.1 → 145.0          | 52 | 110 |
| 342 | Triflumizole              | LC        | 9.53                 | positive  | 346.1 → 278.0           | 4                 | 345.9 → 73.0           | 15 | 80  |
| 343 | Triflumuron               | LC        | 9.19                 | positive  | 359.0 → 156.0           | 8                 | 359.0 → 139.0          | 32 | 120 |
| 344 | Trifluralin               | GC        | 7.27                 | positive  | 264.0 → 160.0           | 15                | 306.0 → 264.0          | 5  | 70  |
| 345 | Trime thoprim             | LC        | 3.45                 | positive  | 291.2 → 123.0           | 24                | 291.2 → 230.1          | 20 | 162 |
| 346 | Triflucinatox              | LC        | 8.38                 | positive  | 318.1 → 70.1            | 33                | 320.1 → 70.1           | 16 | 110 |
| 347 | Tylicosin                 | LC        | 5.52                 | positive  | 869.6 → 174.1           | 48                | 869.6 → 696.4          | 44 | 294 |
| 348 | Tylosin                   | LC        | 6.76                 | positive  | 916.5 → 174.1           | 40                | 916.5 → 772.4          | 28 | 210 |
| 349 | Vinclolizolin             | GC        | 9.10                 | positive  | 212.0 → 145.0           | 25                | 212.0 → 109.0          | 50 | 70  |
| 350 | Warfarin                  | LC        | 7.86                 | negative  | 307.1 → 161.1           | 20                | 307.1 → 250.1          | 20 | 140 |
| 351 | Zoxamide                  | LC        | 9.03                 | positive  | 336.0 → 187.1           | 25                | 187.1 → 88.9           | 40 | 200 |
## Appendix B

### Table A2. Method validation results: Limits of quantification (LOQ), percentage recoveries and relative standard deviation obtained from intraday and interday studies.

| No. | Compound | LOQ (ng/mL) | Rec (%) | Precision (RSD. %) | Rec (%) | Precision (RSD. %) | Rec (%) | Precision (RSD. %) | Rec (%) | Precision (RSD. %) | Rec (%) | Precision (RSD. %) |
|-----|----------|-------------|---------|-------------------|---------|-------------------|---------|-------------------|---------|-------------------|---------|-------------------|
| 1   | 2-Phenylphenol | 0.4 | 101.43 | 19.69 | 18.85 | 107.17 | 20.66 | 19.28 | 106.47 | 18.24 | 17.13 | 96.41 | 13.81 | 14.32 |
| 2   | 4,4'-Dichlorobenzophenone (metabolite of dicofol) | 4,4' | 91.36 | 13.23 | 14.48 | 86.99 | 13.79 | 15.85 | 116.77 | 11.76 | 10.07 | 98.37 | 15.59 | 15.85 |
| 3   | Abamectine | 4 | 106.43 | 14.53 | 13.65 | 93.89 | 5.21 | 5.55 | 99.43 | 14.50 | 14.58 |
| 4   | Acenaphthene | 1.2 | 110.88 | 19.39 | 19.29 | 94.91 | 6.78 | 7.14 | 96.45 | 12.08 | 12.52 | 95.23 | 4.52 | 4.75 |
| 5   | Acenaphthylene | 2 | 107.06 | 18.90 | 21.39 | 95.74 | 12.53 | 13.09 | 100.50 | 12.26 | 12.20 | 98.40 | 2.93 | 2.98 |
| 6   | Acephate | 8 | 90.75 | 20.15 | 22.20 | 93.24 | 14.21 | 14.03 | 104.42 | 4.70 | 4.50 | 104.71 | 4.55 | 2.98 |
| 7   | Acetamiprid | 2 | 122.13 | 8.03 | 6.57 | 101.26 | 14.21 | 14.03 | 104.42 | 4.37 | 4.50 | 104.71 | 4.55 | 4.55 |
| 8   | Acrinathrin | 4 | 116.08 | 16.73 | 14.41 | 103.11 | 22.19 | 21.52 | 104.55 | 5.45 | 5.21 | 107.89 | 5.21 | 5.21 |
| 9   | Albenzazole | 0.4 | 121.03 | 13.9 | 9.41 | 92.56 | 6.35 | 6.86 | 86.92 | 12.48 | 14.36 | 99.53 | 3.66 | 3.68 |
| 10  | Aldicarb | 0.8 | 119.20 | 13.25 | 10.26 | 97.22 | 7.43 | 7.64 | 89.92 | 14.70 | 16.35 | 100.15 | 4.24 | 4.23 |
| 11  | Aldicarb-sulfone | 2 | 121.84 | 18.51 | 15.19 | 94.84 | 16.90 | 19.91 | 85.77 | 12.91 | 15.05 | 97.26 | 10.13 | 10.42 |
| 12  | Aldicarb- sulfoxide | 4 | 104.82 | 18.42 | 17.11 | 81.91 | 5.97 | 7.29 | 105.26 | 12.19 | 11.58 |
| 13  | Aldrin | 2 | 123.66 | 19.11 | 15.45 | 91.59 | 15.23 | 16.65 | 106.54 | 5.58 | 5.24 | 97.33 | 12.12 | 12.45 |
| 14  | Anthracene | 1.6 | 99.67 | 15.35 | 15.40 | 91.47 | 15.47 | 16.91 | 105.34 | 7.32 | 6.95 | 94.68 | 6.50 | 6.87 |
| 15  | Atrazine | 0.8 | 124.53 | 4.21 | 3.38 | 93.04 | 7.01 | 7.53 | 87.60 | 13.23 | 15.10 | 102.49 | 5.46 | 5.33 |
| 16  | Azinphos-methyl | 2 | 126.85 | 18.74 | 14.77 | 103.98 | 13.75 | 13.22 | 105.19 | 3.50 | 3.33 | 102.89 | 5.44 | 5.29 |
| 17  | Azoxyostrobin | 0.4 | 116.28 | 7.94 | 6.83 | 95.41 | 3.96 | 4.15 | 92.31 | 6.03 | 6.53 | 102.95 | 4.52 | 4.39 |
| 18  | BDE-28 | 1.2 | 100.53 | 14.19 | 14.12 | 81.65 | 9.78 | 11.98 | 104.45 | 6.20 | 5.94 | 96.62 | 11.51 | 11.91 |
| 19  | BDE-47 | 0.8 | 98.71 | 20.31 | 20.58 | 109.45 | 21.87 | 19.98 | 89.88 | 8.16 | 9.08 | 110.24 | 8.74 | 7.93 | 95.96 | 8.89 | 9.26 |
### Table A2. Cont.

| No. | Compound | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday |
|-----|----------|-------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|
| 20  | BDE-85   | 0.8         | 100.77  | 15.79    | 15.59    | 103.10  | 16.65    | 16.15    | 85.61   | 12.99    | 15.17    | 108.23  | 16.09    | 14.87    | 98.51   | 17.91   | 13.96   |
| 21  | BDE-99   | 0.4         | 107.54  | 18.55    | 19.69    | 103.74  | 15.02    | 14.48    | 89.65   | 12.66    | 14.12    | 114.05  | 18.00    | 15.78    | 90.97   | 11.97   | 13.16   |
| 22  | BDE-100  | 0.8         | 98.15   | 21.94    | 22.35    | 106.72  | 17.54    | 21.81    | 93.71   | 10.60    | 11.31    | 109.98  | 12.80    | 11.64    | 94.05   | 19.80   | 21.05   |
| 23  | BDE-153  | 0.8         | 93.58   | 16.95    | 19.48    | 108.66  | 17.04    | 19.88    | 93.65   | 15.26    | 16.29    | 111.40  | 15.44    | 13.86    | 96.11   | 17.16   | 17.85   |
| 24  | BDE-154  | 0.4         | 96.19   | 15.79    | 16.42    | 113.57  | 19.84    | 17.47    | 92.43   | 10.71    | 11.59    | 118.15  | 16.34    | 13.83    | 93.42   | 13.93   | 15.62   |
| 25  | BDE-183  | 4           |         |          |          |         |          |          | 78.46   | 5.92     | 7.55     | 104.68  | 12.90    | 12.32    | 95.99   | 8.73    | 9.09    |
| 26  | Benalaxyl | 0.4         | 116.67  | 15.06    | 12.91    | 94.56   | 3.53     | 3.73     | 97.64   | 6.31     | 6.46     | 101.10  | 3.37     | 3.33     | 101.39  | 3.01    | 2.97    |
| 27  | Bendiocarb| 0.8         | 123.80  | 9.46     | 7.29     | 90.11   | 14.65    | 16.26    | 93.23   | 14.26    | 15.30    | 101.72  | 3.10     | 3.05     | 110.76  | 8.93    | 8.06    |
| 28  | Bendiocarb metabolite (2,2-dimethylbenzo-1,3-dioxol-4-ol) | 4   |         |          |          |         | 86.86    | 16.14    | 21.61    | 102.82   | 16.72    | 16.26    | 95.65    | 3.05    | 3.19    |
| 29  | Benfuracarb | 0.8      | 121.43  | 6.42     | 5.24     | 88.09   | 12.05    | 13.68    | 85.50   | 9.48     | 11.09    | 99.93   | 4.45     | 4.45     | 102.76  | 5.96    | 5.80    |
| 30  | Benzo[a]anthracene | 0.8   | 98.90   | 19.38    | 19.71    | 97.89   | 8.00     | 8.17     | 85.04   | 4.75     | 5.59     | 109.73  | 14.44    | 13.16    | 101.08  | 11.10   | 10.98   |
| 31  | Benzo[a]pyrene | 0.8  | 118.01  | 19.78    | 20.66    | 94.10   | 10.74    | 11.41    | 90.35   | 9.53     | 10.55    | 106.01  | 11.75    | 11.08    | 98.16   | 14.17   | 14.44   |
| 32  | Benzo[b]fluoranthene | 1.2 | 101.28  | 11.89    | 11.61    | 91.62   | 10.11    | 11.03    | 105.11  | 8.02     | 7.63     | 93.83   | 8.80     | 9.38     | 17.32   | 17.42   |
| 33  | Benzo[g,h,i]perylene | 0.8 | 80.16   | 19.32    | 20.41    | 100.35  | 15.43    | 15.38    | 93.47   | 9.37     | 10.02    | 114.39  | 6.35     | 5.55     | 95.50   | 10.80   | 11.31   |
| 34  | Benzo[k]fluoranthene | 1.2 | 102.66  | 13.24    | 12.90    | 88.84   | 14.63    | 16.47    | 101.90  | 7.87     | 7.72     | 99.44   | 7.72     | 9.94     | 17.32   | 17.42   |
| 35  | Bifenthrin | 2       | 118.63  | 18.93    | 19.10    | 86.17   | 14.08    | 16.34    | 113.53  | 16.83    | 14.82    | 98.99   | 10.04    | 10.14    | 10.04   | 10.14   |
| 36  | Bitertanol | 0.4    | 121.41  | 23.34    | 18.61    | 84.35   | 14.31    | 16.97    | 80.69   | 10.74    | 13.31    | 99.71   | 4.76     | 4.77     | 105.11  | 2.05    | 1.95    |
| 37  | Boscalid (formerly nicoctin) | 0.8 | 74.96   | 22.43    | 22.81    | 85.76   | 5.16     | 6.02     | 85.67   | 9.78     | 11.42    | 112.15  | 6.66     | 5.94     | 89.33   | 8.33    | 9.32    |
| 38  | Brodifacoum | 0.4     | 102.44  | 23.57    | 23.01    | 88.99   | 13.67    | 15.36    | 85.84   | 12.99    | 15.13    | 90.41   | 5.61     | 6.21     | 107.51  | 6.87    | 6.39    |
| 39  | Bromadiolone | 0.4   | 118.13  | 11.13    | 19.82    | 97.11   | 20.55    | 19.19    | 89.46   | 13.13    | 14.68    | 97.66   | 8.85     | 9.06     | 101.30  | 8.87    | 8.76    |
| No. | Compound                        | LOQ (ng/mL) | Rec. (%) Intraday | Rec. (%) Interday | Rec. (%) Intraday | Rec. (%) Interday | Rec. (%) Intraday | Rec. (%) Interday | Rec. (%) Intraday | Rec. (%) Interday | Rec. (%) Intraday | Rec. (%) Interday |
|-----|--------------------------------|-------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| 40  | Bromopropylate                 | 0.4         | 110.83           | 21.15            | 22.86            | 96.37            | 19.85            | 20.60            | 93.29            | 8.21             | 8.80             | 107.25           | 10.02            | 9.34             | 107.25           | 10.02            | 8.80             | 107.25           | 10.02            | 11.05           |
| 41  | Bromuconazole (two isomers)    | 1.6         | 106.70           | 16.63            | 14.96            | 89.48            | 17.19            | 19.21            | 112.75           | 13.08            | 11.60            | 94.54            | 8.05             | 8.51             |
| 42  | Bupirimate                     | 1.2         | 86.77            | 10.74            | 12.38            | 93.94            | 5.74             | 6.11             | 98.53            | 6.03             | 6.12             | 102.43           | 4.99             | 4.87             |
| 43  | Buprofezin                     | 0.4         | 115.89           | 11.18            | 9.65             | 93.22            | 1.92             | 2.06             | 88.57            | 10.64            | 12.01            | 101.63           | 4.50             | 4.43             | 102.68           | 3.54             | 3.45             |
| 44  | Cadusafos (ebufos)             | 0.4         | 126.81           | 3.48             | 2.74             | 94.26            | 4.04             | 4.29             | 96.81            | 5.74             | 5.93             | 101.63           | 5.00             | 4.92             | 100.58           | 3.04             | 3.02             |
| 45  | Carbaryl                       | 0.8         | 118.84           | 13.56            | 10.52            | 96.23            | 6.27             | 6.52             | 87.81            | 11.26            | 12.82            | 100.43           | 4.37             | 4.35             | 108.80           | 4.34             | 3.99             |
| 46  | Carbendazim (azole)            | 2           | 120.17           | 8.97             | 6.40             | 98.20            | 9.83             | 10.01            | 100.39           | 2.72             | 2.71             | 103.56           | 3.81             | 3.68             |
| 47  | Carbofuran                     | 0.4         | 123.88           | 10.74            | 8.67             | 94.30            | 6.42             | 6.81             | 88.14            | 13.54            | 15.36            | 104.41           | 6.50             | 6.23             | 105.57           | 3.33             | 3.15             |
| 48  | Carbofuran-3-hydroxy           | 0.8         | 120.98           | 10.76            | 8.89             | 89.04            | 8.10             | 9.10             | 94.46            | 15.41            | 16.31            | 100.86           | 3.61             | 3.58             | 107.55           | 4.88             | 4.54             |
| 49  | Carbosulfan                    | 1.2         | 109.16           | 14.87            | 19.19            | 89.29            | 21.22            | 20.62            | 92.19            | 22.12            | 23.99            | 96.57            | 21.02            | 21.77           |
| 50  | Cefuroxime axetil (two isomers)| 4           | 132.33           | 13.51            | 10.21            | 119.80           | 8.73             | 7.29             | 127.08           | 13.98            | 11.00           |
| 51  | Chloramphenicol                | 16          | 98.91            | 7.74             | 7.83             |
| 52  | Chloroantraniliprole           | 16          | 106.00           | 2.93             | 2.76             |
| 53  | Chlorfenviphos                 | 0.8         | 114.28           | 16.10            | 14.09            | 97.14            | 14.61            | 15.04            | 91.56            | 15.96            | 17.43            | 104.40           | 2.64             | 2.53             | 106.32           | 3.78             | 3.56             |
| 54  | Chlorfenzinilzate              | 1.6         | 110.47           | 17.93            | 16.23            | 89.89            | 4.22             | 4.69             | 114.60           | 12.66            | 11.05            | 90.71            | 2.94             | 3.24             |
| 55  | Chloropahcione                 | 8           | 98.40            | 21.84            | 22.56            | 99.28            | 5.40             | 5.40             | 100.11           | 21.56            | 21.56            |
| 56  | Chlorpropham                   | 2           | 119.39           | 17.61            | 13.13            | 93.11            | 14.39            | 15.45            | 112.93           | 6.10             | 5.40             | 100.11           | 21.56            | 21.56            |
| 57  | Chlorpyrifos                   | 1.6         | 74.81            | 17.17            | 19.32            | 92.21            | 1.74             | 1.89             | 119.54           | 9.95             | 8.32             | 90.50            | 6.87             | 7.59             |
| 58  | Chlorpyrifos methyl            | 2           | 115.24           | 17.63            | 14.98            | 88.08            | 15.56            | 19.02            | 90.85            | 9.97             | 10.97            | 106.30           | 6.98             | 6.57             |
| No. | Compound                  | LOQ (ng/mL) | 0.4 ng/mL | 1 ng/mL | 4 ng/mL | 20 ng/mL | 40 ng/mL |
|-----|---------------------------|-------------|-----------|---------|---------|----------|----------|
|     |                           |             | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) |
|     |                           |             | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday | Intraday  | Interday |
| 59  | Chlorthal dimethyl         | 0.8         | 104.38    | 17.25    | 19.43    | 96.72    | 14.55    | 15.04    | 91.37    | 6.53     | 7.15     | 113.90   | 9.82     | 8.62     | 90.34    | 3.66     | 4.05     |
| 60  | Chrysene                  | 1.6         | 109.91    | 12.42    | 11.30    | 91.83    | 7.03     | 7.66     | 106.10   | 17.06    | 16.08    | 100.25   | 11.34    | 11.31    |
| 61  | Clindamycin               | 4           | 129.25    | 10.53    | 8.15     | 109.20   | 7.44     | 6.81     | 107.36   | 6.37     | 5.93     |
| 62  | Clofentezine               | 0.8         | 116.80    | 20.59    | 17.63    | 94.99    | 7.99     | 8.41     | 83.66    | 11.18    | 13.36    | 96.74    | 7.45     | 7.70     | 105.68   | 4.56     | 4.31     |
| 63  | Clothinidin                | 12          | 105.52    | 8.63     | 8.18     | 97.88    | 11.44    | 11.69    |
| 64  | Cloxacillin                | 8           | 99.33     | 18.27    | 18.39    | 107.79   | 10.04    | 9.31     |
| 65  | Coumachlor                | 0.8         | 120.85    | 16.85    | 20.84    | 99.90    | 12.14    | 12.15    | 85.48    | 16.06    | 18.79    | 101.57   | 4.20     | 4.14     | 102.89   | 2.20     | 2.14     |
| 66  | Coumphos                  | 0.8         | 112.59    | 14.03    | 12.46    | 86.24    | 15.34    | 17.79    | 86.49    | 10.71    | 12.38    | 105.36   | 4.20     | 3.99     | 103.38   | 4.36     | 4.22     |
| 67  | Coumatetralyl             | 1.6         | 116.12    | 22.62    | 19.48    | 97.65    | 9.98     | 10.22    | 96.87    | 4.29     | 4.43     | 102.71   | 4.39     | 4.27     |
| 68  | Cyazofamid                | 2           | 112.26    | 13.66    | 12.17    | 100.29   | 12.06    | 12.03    | 103.98   | 7.08     | 6.81     | 97.87    | 5.04     | 5.15     |
| 69  | Cyflufenamid              | 1.6         | 116.75    | 17.97    | 23.96    | 96.02    | 19.44    | 20.25    | 98.16    | 6.89     | 7.02     | 105.14   | 7.86     | 7.48     |
| 70  | Cyfluthrin (sum of four isomers) | 8 | 128.33 | 19.82 | 15.44 | 87.94 | 19.51 | 21.60 |
| 71  | Cyhalothrin (lambda isomer) | 4 | 118.45 | 17.58 | 14.84 | 110.36 | 18.54 | 16.80 | 102.15 | 14.52 | 14.21 |
| 72  | Cymoxanil                 | 2           | 127.98    | 23.20    | 18.13    | 112.78   | 13.14    | 11.65    | 111.18   | 3.17     | 2.85     | 107.24   | 7.19     | 6.70     |
| 73  | Cypermethrin (sum of four isomers) | 20 | 125.42 | 15.64 | 12.47 | 74.31 | 10.18 | 13.70 |
| 74  | Cyproconazole (two isomers) | 4 | 120.66 | 19.73 | 20.01 | 89.98 | 15.51 | 17.24 | 110.68 | 5.93 | 5.36 | 93.41 | 7.33 | 7.85 |
| 75  | Cyprodinil                | 1.2         | 110.91    | 13.69    | 12.34    | 89.72    | 15.25    | 17.00    | 97.14    | 8.22     | 8.46     | 104.22   | 7.41     | 7.11     |
| 76  | Cyromazine                | 8           | 94.58     | 11.29    | 11.94    | 96.74    | 11.32    | 11.70    |
| 77  | Danofloxacin              | 8           | 113.55    | 18.30    | 16.12    | 89.76    | 10.39    | 11.58    |
| No. | Compound                        | LOQ (ng/mL) | 0.4 ng/mL | 1 ng/mL | 4 ng/mL | 20 ng/mL | 40 ng/mL |
|-----|---------------------------------|-------------|-----------|---------|---------|---------|---------|
|     |                                 |             | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) |
| 78  | Dazomet                         | 4           | 71.53     | 17.55   | 21.50   | 83.03   | 13.84   | 16.67   | 90.82 | 16.95 | 18.66 |
| 79  | Deltamethrin                    | 4           | 95.89     | 17.74   | 19.36   | 107.28  | 18.61   | 17.24   | 100.61 | 12.28 | 12.21 |
| 80  | Demeton-S-methyl                | 0.8         | 123.35    | 9.93    | 7.74    | 95.67   | 3.32    | 3.47    | 87.38 | 12.64 | 14.47 | 101.00 | 5.18 | 5.13 | 108.47 | 6.18 | 5.70 |
| 81  | Demeton-S-methyl-sulphone       | 12          | 80.24     | 4.83    | 6.02    | 96.53   | 6.34    | 6.57    |       |       |       |
| 82  | Dexamethasone                   | 2           | 123.79    | 17.67   | 12.29   | 104.61  | 13.93   | 13.32   | 106.53 | 14.48 | 13.59 | 102.96 | 4.32 | 4.20 |
| 83  | Diazinon                        | 1.2         | 101.78    | 19.05   | 18.68   | 97.76   | 8.04    | 8.22    | 110.63 | 15.03 | 13.59 | 92.16   | 7.39 | 8.02 |
| 84  | Dibenzo[a]anthracene            | 0.8         | 70.39     | 18.68   | 16.54   | 112.50  | 16.62   | 14.77   | 93.06 | 11.73 | 12.60 | 111.29 | 4.83 | 4.34 | 84.41 | 6.54 | 7.75 |
| 85  | Dichlorodiphenyl dichloromethane (p,p' DDD) | 0.8 | 88.27     | 19.42   | 17.67   | 112.25  | 6.36    | 5.67    | 92.57 | 11.03 | 11.92 | 101.42 | 6.07 | 5.99 | 93.46 | 4.77 | 5.10 |
| 86  | Dichlorodiphenyl dichloromethane (p,p' DDE) | 0.8 | 99.71     | 18.39   | 18.44   | 110.56  | 17.85   | 18.67   | 97.62 | 7.46   | 7.64  | 104.29 | 0.72  | 0.69 | 98.40 | 8.58 | 8.72 |
| 87  | Dichlorodiphenyl trichloromethane (p,p' DDT) | 2 | 101.87    | 15.98   | 14.95   | 104.65  | 20.63   | 19.71   | 110.43 | 6.32   | 5.72  | 92.27   | 6.86  | 7.43 |
| 88  | Diclofenac                      | 4           | 111.25    | 21.71   | 19.51   | 91.03   | 12.48   | 13.71   | 94.44  | 12.42 | 13.15 |
| 89  | Dicloran                        | 4           | 95.68     | 14.38   | 15.03   | 113.17  | 15.89   | 14.04   | 92.28  | 5.09   | 5.52 |
| 90  | Diclorvos                       | 8           | 113.48    | 10.38   | 9.15    | 98.10   | 2.98    | 3.04    |       |       |       |
| 91  | Dicloxacillin                   | 12          | 77.54     | 12.35   | 15.93   | 103.35  | 10.63   | 10.29   |       |       |       |
| 92  | Dieldrin                       | 8            | 80.70     | 18.40   | 18.43   | 95.08   | 5.85    | 6.15    |       |       |       |
| 93  | Diethanoyl ethyl                | 0.4         | 113.26    | 12.83   | 20.16   | 96.15   | 13.17   | 13.70   | 89.84  | 15.24  | 16.96 | 97.95   | 6.16  | 6.29 | 99.30 | 7.45 | 7.50 |
| 94  | Diethofencarb                   | 0.4         | 113.98    | 6.38    | 5.60    | 90.65   | 3.94    | 4.35    | 90.80  | 10.90  | 12.00 | 101.24  | 1.48  | 1.46 | 104.26 | 1.97 | 1.89 |
| 95  | Difenacoum                      | 0.8         | 117.66    | 13.84   | 21.51   | 88.27   | 10.36   | 11.74   | 85.40  | 7.72   | 9.04  | 92.19   | 6.14  | 6.66 | 104.37 | 3.77 | 3.61 |
| 96  | Difenconazole                   | 0.8         | 119.78    | 17.98   | 13.85   | 94.45   | 8.83    | 9.35    | 84.08  | 7.72   | 9.18  | 97.08   | 6.45  | 6.64 | 106.47 | 4.12 | 3.87 |
| 97  | Difethialone                    | 1.6         | 112.44    | 10.79   | 9.60    | 88.10   | 18.22   | 20.68   | 95.86  | 6.45   | 6.73  | 103.22  | 8.66  | 8.39 |       |       |
Table A2. Cont.

| No. | Compound                                | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) |
|-----|-----------------------------------------|-------------|---------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 98  | Difloxacin                              | 4           |         | 123.36   | 19.34    | 15.68    | 102.87   | 8.83     | 8.58     | 99.17    | 8.54     | 8.61     |
| 99  | Diflubenzuron                           | 1.6         |         | 121.03   | 16.61    | 18.98    | 99.73    | 16.54    | 16.58    | 102.86   | 17.01    | 16.54    | 105.63   | 4.13     | 3.91     |
| 100 | Diflufenican                            | 0.4         |         | 114.39   | 18.24    | 19.49    | 101.56   | 9.38     | 9.24     | 95.89    | 9.59     | 10.00    | 115.49   | 8.35     | 7.23     | 95.62    | 9.05     | 9.46     |
| 101 | Dimethenamid-P (and its R-isomer)       | 0.4         |         | 112.73   | 15.63    | 12.74    | 87.82    | 4.74     | 5.40     | 90.21    | 8.85     | 9.81     | 103.64   | 3.95     | 3.81     | 102.49   | 5.02     | 4.90     |
| 102 | Dimethoate                              | 0.8         |         | 121.83   | 20.22    | 15.57    | 93.11    | 5.47     | 5.87     | 87.05    | 11.89    | 13.66    | 100.07   | 10.40    | 10.39    | 108.51   | 7.39     | 6.81     |
| 103 | Dimethomorph (two isomers)              | 0.4         |         | 123.37   | 18.68    | 15.14    | 87.72    | 6.32     | 7.20     | 87.24    | 11.29    | 12.94    | 104.93   | 4.63     | 4.41     | 105.80   | 5.87     | 5.55     |
| 104 | Dimethylphenylsulfamide (DMSA, metabolite of dichlofluanid) | 4 |         | 114.05   | 11.55    | 10.13    | 109.55   | 9.65     | 8.81     | 102.37   | 4.33     | 4.23     |          |          |          |          |          |          |
| 105 | Diniconazole-M                           | 1.2         |         | 97.80    | 16.52    | 16.89    | 88.78    | 6.85     | 7.72     | 102.75   | 14.54    | 14.15    | 100.66   | 5.86     | 5.82     |          |          |          |
| 106 | Dinocap                                 | 4           |         | 115.19   | 16.40    | 14.24    | 105.54   | 2.90     | 2.75     | 105.88   | 5.85     | 5.53     |          |          |          |          |          |          |
| 107 | Diphacinone                             | 8           |         | 96.74    | 18.11    | 18.72    | 103.66   | 19.59    | 18.90    |          |          |          |          |          |          |          |          |          |
| 108 | Diphenylamine                           | 1.6         |         | 92.81    | 20.64    | 13.01    | 96.18    | 5.32     | 5.53     | 106.11   | 10.45    | 9.85     | 93.91    | 2.44     | 2.60     |          |          |          |
| 109 | Dodine                                  | 0.8         |         | 107.19   | 12.63    | 11.78    | 97.64    | 8.18     | 8.38     | 87.26    | 10.47    | 12.00    | 102.27   | 5.32     | 5.20     | 102.46   | 4.26     | 4.16     |
| 110 | Doramectina                             | 8           |         | 101.04   | 19.63    | 19.43    | 99.13    | 9.03     | 9.11     |          |          |          |          |          |          |          |          |          |
| 111 | Endosulfan alfa                         | 2           |         | 112.45   | 11.73    | 10.43    | 84.22    | 19.47    | 23.12    | 122.57   | 20.34    | 20.75    | 96.35    | 10.24    | 10.63    |          |          |          |
| 112 | Endosulfan beta                         | 4           |         | 85.36    | 23.81    | 19.45    | 115.99   | 3.80     | 3.28     | 88.35    | 9.64     | 10.91    |          |          |          |          |          |          |
| 113 | Endosulfan sulfate                      | 4           |         | 70.48    | 20.30    | 18.80    | 116.21   | 19.54    | 20.32    | 89.14    | 17.93    | 20.11    |          |          |          |          |          |          |
| 114 | Endrin                                  | 4           |         | 88.96    | 17.68    | 22.54    | 107.57   | 12.66    | 11.77    | 92.69    | 12.73    | 13.73    |          |          |          |          |          |          |
| 115 | Enrofloxacin                            | 4           |         | 118.69   | 21.02    | 19.54    | 98.46    | 7.06     | 7.17     | 104.38   | 11.64    | 11.15    |          |          |          |          |          |          |
| 116 | EPN                                     | 2           |         | 81.34    | 17.89    | 19.10    | 87.12    | 22.90    | 19.18    | 112.30   | 12.97    | 11.55    | 95.56    | 5.50     | 5.76     |          |          |          |
| 117 | Epoxiconazole                           | 0.8         |         | 122.98   | 16.59    | 13.49    | 89.44    | 11.43    | 13.96    | 97.21    | 19.34    | 17.51    | 106.20   | 12.49    | 11.76    | 106.31   | 5.68     | 5.34     |
Table A2. Cont.

| No. | Compound (diethion) | LOQ (ng/mL) | Rec. (%) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) |
|-----|---------------------|-------------|----------|-------------------|-------------------|-------------------|-------------------|-------------------|
| 118 | Eprinomectin        | 1.2         | 100.03   | 21.36             | 21.35             | 89.96             | 19.07             | 21.20             | 95.57             | 10.18             | 10.65             | 102.43            | 8.65              | 8.44              |
| 119 | Eritromicin         | 0.8         | 122.85   | 10.10             | 7.96              | 101.21            | 19.49             | 17.83             | 101.51            | 4.20              | 4.14              | 90.24             | 7.45              | 8.26              |
| 120 | Esfenvalerate       | 2           | 127.54   | 21.58             | 16.92             | 105.91            | 19.49             | 17.83             | 101.51            | 4.20              | 4.14              | 90.24             | 7.45              | 8.26              |
| 121 | Ethion (diethion)   | 0.8         | 109.21   | 7.32              | 6.70              | 92.19             | 7.99              | 8.67              | 89.52             | 8.27              | 9.24              | 98.50             | 2.63              | 2.67              | 100.66            | 3.50              | 3.48              |
| 122 | Ethirimol           | 1.2         | 103.80   | 13.91             | 13.40             | 84.85             | 8.49              | 10.01             | 98.86             | 6.54              | 6.62              | 109.68            | 5.38              | 4.91              |
| 123 | Ethofumesate        | 2           | 126.97   | 15.41             | 16.48             | 90.72             | 15.48             | 21.11             | 103.99            | 13.91             | 13.38             | 96.26             | 8.98              | 9.33              |
| 124 | Ethoprophos         | 0.8         | 121.58   | 17.90             | 22.95             | 85.95             | 23.63             | 17.49             | 96.79             | 13.42             | 13.87             | 103.33            | 4.56              | 4.41              | 108.44            | 8.96              | 8.26              |
| 125 | Etofenprox          | 1.2         | 103.05   | 20.35             | 19.75             | 88.00             | 12.88             | 14.64             | 87.54             | 4.70              | 5.37              | 107.43            | 8.42              | 7.84              |
| 126 | Etoxazole           | 0.4         | 116.44   | 7.26              | 6.23              | 95.39             | 5.73              | 6.01              | 88.69             | 5.00              | 5.64              | 98.79             | 1.96              | 1.98              | 99.61             | 4.04              | 4.06              |
| 127 | Farnoxadone         | 1.2         | 105.50   | 21.91             | 19.80             | 84.93             | 8.09              | 9.53              | 100.06            | 8.50              | 8.49              | 105.60            | 8.62              | 8.16              |
| 128 | Fenamidone          | 0.4         | 120.97   | 11.07             | 9.15              | 96.95             | 7.86              | 8.11              | 90.00             | 12.52             | 13.91             | 102.36            | 3.51              | 3.43              | 105.44            | 5.42              | 5.14              |
| 129 | Fenamiphos          | 0.4         | 108.30   | 19.35             | 26.33             | 96.23             | 10.37             | 10.78             | 91.06             | 5.88              | 6.46              | 102.96            | 6.58              | 6.39              | 104.25            | 4.70              | 4.51              |
| 130 | Fenamiphos sulfone  | 1.2         | 94.25    | 18.82             | 19.97             | 93.80             | 9.16              | 9.77              | 102.18            | 10.58             | 10.35             | 112.34            | 3.88              | 3.45              |
| 131 | Fenamiphos sulfoxide| 1.2         | 89.34    | 10.67             | 11.94             | 98.26             | 10.61             | 10.80             | 99.74             | 5.76              | 5.78              | 112.66            | 2.19              | 1.94              |
| 132 | Fenarimol           | 1.6         | 100.34   | 12.81             | 12.77             | 99.76             | 7.71              | 7.73              | 109.44            | 6.21              | 5.67              | 94.11             | 1.40              | 1.49              |
| 133 | Fenazaquin          | 0.4         | 120.20   | 17.57             | 14.62             | 91.49             | 3.55              | 3.88              | 82.11             | 9.35              | 11.39             | 100.13            | 2.89              | 2.89              | 101.08            | 3.40              | 3.36              |
| 134 | Fenbendazole        | 0.4         | 120.77   | 22.32             | 17.07             | 98.07             | 9.36              | 9.54              | 84.88             | 12.32             | 14.51             | 99.37             | 8.88              | 8.94              | 105.25            | 5.15              | 4.89              |
| 135 | Fenbuconazole       | 0.8         | 117.33   | 16.47             | 17.97             | 80.70             | 15.65             | 19.39             | 92.40             | 23.56             | 15.50             | 101.06            | 9.40              | 9.30              | 104.91            | 5.73              | 5.46              |
| 136 | Fenhexamid          | 4           | 113.78   | 16.34             | 17.33             | 102.52            | 7.16              | 6.98              | 96.72             | 9.77              | 9.77              | 102.94            | 6.86              | 6.84              | 102.94            | 2.05              | 1.99              |
| 137 | Fenitrothion        | 4           | 92.58    | 17.86             | 21.77             | 91.31             | 11.66             | 12.77             | 97.21             | 6.00              | 6.17              |
| 138 | Fenoxycarb          | 0.4         | 105.61   | 17.40             | 16.48             | 94.00             | 12.18             | 12.96             | 87.03             | 6.88              | 7.91              | 101.05            | 3.54              | 3.50              | 99.09             | 6.54              | 6.60              |
| 139 | Fenpropathrin       | 0.8         | 121.06   | 18.95             | 23.91             | 81.90             | 11.15             | 13.61             | 85.72             | 10.16             | 11.85             | 96.22             | 3.83              | 3.98              | 103.49            | 5.20              | 5.02              |
| 140 | Fenpropidin         | 0.4         | 118.57   | 11.79             | 9.94              | 88.81             | 8.30              | 9.35              | 90.98             | 3.24              | 3.56              | 100.27            | 6.86              | 6.84              | 102.94            | 2.05              | 1.99              |
Table A2. Cont.

| No. | Compound                  | LOQ (ng/mL) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) | Precision (RSD. %) |
|-----|---------------------------|-------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| 141 | Fenpropimorph             | 0.8         | 118.79             | 13.41              | 11.29              | 85.42              | 10.61              | 12.42              |
| 142 | Fenpyroximate             | 0.4         | 112.81             | 6.87               | 6.09               | 94.83              | 3.34               | 3.52               |
| 143 | Fenthion                  | 0.8         | 97.17              | 14.25              | 26.12              | 109.28             | 12.00              | 10.98              |
| 144 | Fenthion oxon             | 0.4         | 112.27             | 10.39              | 9.25               | 93.67              | 4.79               | 5.11               |
| 145 | Fenthion oxon sulfoxide   | 8           |                    |                    |                    |                    |                    |                    |
| 146 | Fenthion oxon sulfoxide   | 1.2         |                    |                    |                    |                    |                    |                    |
| 147 | Fenthion sulfoxide        | 1.6         |                    |                    |                    |                    |                    |                    |
| 148 | Fenthion sulfoxide        | 1.6         |                    |                    |                    |                    |                    |                    |
| 149 | Fenvalerate               | 2           |                    |                    |                    |                    |                    |                    |
| 150 | Fipronil                  | 0.8         | 102.09             | 11.72              | 11.48              | 94.23              | 19.32              | 19.56              |
| 151 | Fipronil sulfoxide        | 8           |                    |                    |                    |                    |                    |                    |
| 152 | Flocoumafen               | 0.4         | 106.82             | 17.50              | 16.38              | 94.93              | 7.63               | 8.04               |
| 153 | Fluazinam                 | 2           |                    |                    |                    |                    |                    |                    |
| 154 | Flubendiamide             | 1.6         |                    |                    |                    |                    |                    |                    |
| 155 | Flucythrinate (two isomers) | 2          |                    |                    |                    |                    |                    |                    |
| 156 | Fludioxonil               | 2           |                    |                    |                    |                    |                    |                    |
| 157 | Flufenoxuron              | 0.8         | 118.74             | 20.77              | 17.49              | 90.49              | 6.24               | 6.90               |
| 158 | Flumequine                | 0.8         | 127.04             | 7.72               | 5.63               | 91.69              | 7.33               | 7.99               |
| 159 | Flunixin                  | 0.8         | 123.19             | 8.48               | 6.88               | 99.80              | 8.40               | 18.44              |
| 160 | Fluopyram                 | 0.8         | 123.13             | 22.83              | 18.54              | 93.37              | 13.69              | 15.37              |
| 161 | Fluoranthene              | 2           |                    |                    |                    |                    |                    |                    |
| 162 | Fluorene                  | 1.2         |                    |                    |                    |                    |                    |                    |
Table A2. Cont.

| No. | Compound | LOQ (ng/mL) | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday |
|-----|----------|-------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 163 | Fluquinconazole | 1.2 | 110.55 | 16.66 | 18.74 | 86.38 | 6.85 | 7.93 | 100.01 | 12.08 | 12.08 | 81.40 | 7.90 | 9.71 |
| 164 | Flusilazole | 0.8 | 108.78 | 10.98 | 10.09 | 99.53 | 10.49 | 10.54 | 88.08 | 8.54 | 9.70 | 104.22 | 12.29 | 11.79 | 102.06 | 4.15 | 4.07 |
| 165 | Flutolanil | 0.8 | 117.13 | 16.40 | 14.00 | 99.03 | 10.07 | 10.17 | 90.65 | 12.51 | 13.80 | 100.14 | 3.12 | 3.12 | 102.55 | 7.51 | 7.32 |
| 166 | Flutriafol | 1.2 | 102.18 | 9.65 | 9.44 | 91.70 | 13.15 | 14.34 | 103.28 | 8.34 | 8.08 | 106.17 | 4.42 | 4.16 |
| 167 | Fluvalinate tau | 4 | 78.58 | 18.54 | 23.59 | 99.65 | 18.40 | 18.46 | 105.68 | 17.54 | 16.60 |
| 168 | Fonofos | 1.6 | 96.73 | 20.46 | 22.45 | 82.54 | 10.97 | 13.29 | 115.98 | 10.45 | 9.01 | 90.58 | 4.37 | 4.82 |
| 169 | Formetanate | 1.2 | 105.80 | 11.33 | 10.71 | 91.02 | 5.72 | 6.28 | 90.92 | 7.25 | 7.97 | 102.25 | 6.83 | 6.68 |
| 170 | Fosthiazate | 0.4 | 117.53 | 12.76 | 10.86 | 91.81 | 5.80 | 6.32 | 89.96 | 11.00 | 12.23 | 101.32 | 5.03 | 4.96 | 103.76 | 2.06 | 1.99 |
| 171 | Heptachlor | 1.2 | 105.63 | 13.94 | 13.20 | 95.68 | 11.66 | 12.19 | 109.43 | 7.97 | 7.28 | 91.44 | 3.65 | 3.99 |
| 172 | Hexachlorobenzene | 0.8 | 98.45 | 17.43 | 18.64 | 107.10 | 20.87 | 20.35 | 87.60 | 4.82 | 4.50 | 101.47 | 9.94 | 9.80 | 90.60 | 2.38 | 2.63 |
| 173 | Hexachlorocyclohexane (alpha) | 2 | 117.54 | 12.12 | 8.21 | 88.98 | 5.41 | 6.08 | 103.02 | 9.89 | 9.60 | 92.16 | 8.25 | 8.95 |
| 174 | Hexachlorocyclohexane (beta) | 2 | 126.04 | 17.47 | 13.86 | 97.43 | 3.53 | 3.62 | 108.18 | 8.42 | 7.78 | 93.18 | 8.51 | 9.13 |
| 175 | Hexachlorocyclohexane (delta) | 4 | 90.15 | 19.06 | 20.45 | 104.74 | 16.22 | 15.49 | 95.22 | 11.94 | 12.54 |
| 176 | Hexachlorocyclohexane (gamma, lindane) | 4 | 93.75 | 14.15 | 23.24 | 107.48 | 6.80 | 6.33 | 95.82 | 12.95 | 13.51 |
| 177 | Hexaconazole (two isomers) | 1.6 | 95.19 | 15.08 | 15.84 | 91.80 | 7.62 | 8.30 | 102.65 | 5.70 | 5.55 | 103.19 | 2.94 | 2.85 |
| 178 | Hexaflumuron | 1.2 | 99.41 | 17.91 | 18.02 | 80.35 | 5.75 | 7.16 | 98.14 | 4.07 | 4.15 | 98.75 | 11.10 | 11.24 |
| No. | Compound                      | LOQ (ng/mL) | Precision (RSD. %) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday |
|-----|-------------------------------|-------------|--------------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|
| 179 | Hexythiazox                   | 0.4         | 118.55             | 17.88   | 18.46    | 82.22    | 10.37   | 9.39     | 10.72    | 98.62   | 2.88     | 2.92     | 104.62  | 8.24     | 7.88     | 106.55  | 10.65   | 10.00   |
| 180 | Imazalil (enilconazole)       | 0.8         | 130.92             | 15.49   | 17.18    | 95.75    | 7.74    | 8.08     | 9.74     | 10.87   | 100.82   | 4.01     | 3.98    | 104.51  | 5.19    | 4.97    |
| 181 | Imidacloprid                  | 4           |                    |         |          |          |         |          |          |         |          |          |         |          |          |         |          |          |         |          |
| 182 | Indeno [1,2,3-cd] pyrene      | 1.6         | 117.89             | 15.48   | 13.13    | 116.35   | 14.87   | 12.78    | 116.45   | 17.25   | 14.81    | 100.18   | 19.54   | 19.50   |          |         |          |          |         |          |
| 183 | Indoxacarb                    | 0.8         | 124.61             | 14.87   | 18.70    | 99.01    | 21.65   | 21.87    | 88.98    | 17.78   | 19.98    | 93.09    | 6.87    | 7.38    | 109.78  | 5.44    | 4.96    |
| 184 | Iprovalicarb                  | 0.8         | 115.20             | 10.97   | 9.52     | 93.14    | 7.55    | 8.11     | 99.11    | 5.49    | 5.29     | 102.29   | 3.92    | 3.83    |          |         |          |          |         |          |
| 185 | Isofenphos methyl             | 2           | 126.11             | 13.75   | 10.90    | 96.55    | 9.27    | 9.60     | 112.12   | 5.55    | 4.95     | 96.12    | 9.68    | 10.07   |          |         |          |          |         |          |
| 186 | Isoprotiolane                 | 0.4         | 106.89             | 3.33    | 3.12     | 96.32    | 18.05   | 18.74    | 92.86    | 12.46   | 13.42    | 101.23   | 2.17    | 2.14    | 102.11  | 4.86    | 4.76    |
| 187 | Ivermectin B1a                | 1.6         | 113.41             | 16.89   | 14.89    | 98.11    | 8.63    | 8.80     | 91.74    | 11.91   | 12.98    | 101.63   | 10.59   | 10.42   |          |         |          |          |         |          |
| 188 | Josamycin                     | 1.6         | 127.21             | 18.85   | 14.82    | 103.07   | 5.89    | 5.71     | 94.94    | 7.07    | 7.45     | 108.51   | 2.46    | 2.27    |          |         |          |          |         |          |
| 189 | Ketoprofen                    | 1.6         | 93.43              | 17.84   | 19.09    | 106.39   | 15.59   | 14.05    | 95.64    | 10.20   | 10.66    | 102.80   | 3.50    | 3.40    |          |         |          |          |         |          |
| 190 | Kresoxim methyl               | 2           | 119.34             | 19.83   | 18.72    | 98.02    | 18.09   | 18.66    | 110.88   | 14.03   | 12.65    | 96.62    | 12.85   | 13.30   |          |         |          |          |         |          |
| 191 | Levamisole                    | 1.6         | 114.26             | 22.07   | 19.32    | 92.62    | 8.40    | 9.07     | 86.67    | 7.08    | 8.17     | 101.93   | 6.16    | 6.04    |          |         |          |          |         |          |
| 192 | Lincomycin                    | 4           | 120.69             | 19.64   | 16.27    | 112.23   | 7.04    | 6.27     | 96.55    | 6.39    | 6.62     |          |         |         |          |         |          |
| 193 | Linuron                       | 1.6         | 125.64             | 15.59   | 18.33    | 89.87    | 13.77   | 15.32    | 98.09    | 5.13    | 5.23     | 107.80   | 5.05    | 4.68    |          |         |          |          |         |          |
| 194 | Lufenuron                     | 0.8         | 112.88             | 16.42   | 14.55    | 97.32    | 13.82   | 14.48    | 78.06    | 11.50   | 14.73    | 102.81   | 10.91   | 10.61   | 102.87  | 4.15    | 4.03    |
| 195 | Mandipropamid                 | 0.4         | 110.19             | 14.68   | 13.32    | 91.62    | 7.61    | 8.31     | 91.88    | 8.21    | 8.94     | 103.64   | 4.69    | 4.53    | 104.43  | 3.29    | 3.15    |
| 196 | Mebendazole                   | 0.4         | 128.41             | 10.28   | 8.01     | 92.65    | 6.20    | 6.69     | 86.90    | 10.23   | 11.77    | 95.39    | 7.83    | 8.21    | 104.16  | 2.75    | 2.64    |
| 197 | Mefenamic acid                | 1.6         | 123.97             | 17.95   | 14.48    | 102.51   | 18.77   | 18.31    | 92.91    | 13.78   | 14.83    | 102.92   | 3.83    | 3.72    |          |         |          |          |         |          |
| 198 | Mefenoxam (metalaxyl-M)       | 0.4         | 119.22             | 10.17   | 8.53     | 93.63    | 6.26    | 6.69     | 91.63    | 10.08   | 11.00    | 101.70   | 6.18    | 6.08    | 103.38  | 2.08    | 2.01    |
| 199 | Meloxicam                     | 1.2         | 94.27              | 17.55   | 18.66    | 89.51    | 17.89   | 16.54    | 90.45    | 7.81    | 8.63     | 106.55   | 10.65   | 10.00   |          |         |          |          |         |          |
Table A2. Cont.

| No. | Compound            | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday |
|-----|---------------------|-------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|
| 200 | Mepanipyrim        | 2           | 114.71  | 20.02    | 17.45    | 92.36   | 6.47     | 7.01     | 105.72  | 8.41     | 7.95     | 102.76  | 2.90     | 2.82     |
| 201 | Mepiquat            | 0.4         | 88.75   | 15.72    | 18.98    | 96.69   | 19.96    | 20.64    | 84.95   | 17.75    | 20.89    | 92.24   | 7.48     | 8.11     | 101.87  | 2.68    | 2.63     |
| 202 | Metaflumizone       | 0.4         | 120.69  | 6.98     | 5.78     | 90.72   | 10.11    | 11.14    | 87.97   | 8.18     | 9.30     | 100.90  | 6.18     | 6.12     | 101.69  | 3.74    | 3.68     |
| 203 | Metalaxyl           | 1.6         | 102.24  | 8.98     | 8.78     | 87.83   | 6.49     | 7.39     | 114.56  | 16.92    | 14.77    | 88.61   | 5.54     | 6.25     |
| 204 | Metaldehyde         | 4           | 117.18  | 16.45    | 16.52    | 82.85   | 10.48    | 12.65    | 100.36  | 8.69     | 8.66     |
| 205 | Metconazole         | 0.8         | 126.92  | 8.05     | 6.34     | 91.62   | 13.26    | 14.47    | 85.87   | 13.93    | 16.22    | 98.86   | 2.29     | 2.32     | 104.83  | 4.73    | 4.51     |
| 206 | Methamidophos       |             | 90.08   | 12.33    | 13.69    | 95.62   | 7.09     | 7.41     |
| 207 | Methidathion        | 0.4         | 121.63  | 11.90    | 9.78     | 94.81   | 6.86     | 7.24     | 92.10   | 11.60    | 12.60    | 101.70  | 2.15     | 2.11     | 105.46  | 7.13    | 6.76     |
| 208 | Methiocarb          | 0.4         | 126.57  | 12.25    | 9.68     | 82.56   | 7.76     | 9.40     | 96.74   | 10.42    | 10.77    | 106.92  | 5.78     | 5.41     | 109.28  | 7.12    | 6.52     |
| 209 | Methiocarb-sulfone  | 2           | 124.44  | 19.05    | 20.07    | 110.41  | 9.03     | 8.18     | 106.35  | 7.79     | 7.31     | 105.17  | 9.43     | 8.97     |
| 210 | Methiocarb-sulfoxide| 1.2         | 97.90   | 11.90    | 12.16    | 97.45   | 19.13    | 19.63    | 102.57  | 5.80     | 5.65     | 107.53  | 5.47     | 5.09     |
| 211 | Methomyl            | 1.2         | 105.76  | 12.28    | 11.61    | 116.29  | 18.70    | 16.08    | 114.17  | 3.66     | 3.21     | 106.03  | 7.48     | 7.05     |
| 212 | Methoxyfenozide     | 0.4         | 86.63   | 21.88    | 23.34    | 102.29  | 15.53    | 15.18    | 97.40   | 9.54     | 9.79     |
| 213 | Metoxycarbene       | 4           | 116.96  | 8.59     | 7.34     | 88.75   | 7.33     | 8.26     | 90.85   | 3.70     | 4.07     | 102.21  | 3.91     | 3.83     | 102.30  | 3.36    | 3.28     |
| 214 | Metrafenone         | 0.4         | 126.22  | 20.21    | 16.01    | 98.22   | 13.26    | 13.50    | 85.59   | 7.19     | 8.40     | 95.31   | 6.55     | 6.87     | 107.70  | 5.47    | 5.08     |
| 215 | Metronidazole       | 12          |         |          |          |         |          |          |         |          |          |         |          |          |          |          |          |          |          |
| 216 | Mevinphos (phosdrin)| 1.2         | 107.14  | 19.43    | 18.14    | 87.21   | 9.70     | 11.12    | 99.51   | 6.93     | 6.96     | 112.13  | 4.49     | 4.00     |
| 217 | Mirex               | 4           | 87.30   | 15.87    | 16.63    | 99.75   | 22.33    | 22.39    | 104.43  | 12.55    | 12.02    |
| 218 | Monocrotophos       | 4           | 113.28  | 8.74     | 7.72     | 99.08   | 3.05     | 3.08     | 103.67  | 4.46     | 4.30     |
Table A2. Cont.

| No. | Compound                                      | LOQ (ng/mL) | Rec (%) | Intraday Rec. (%) | Interday Rec. (%) | Intraday Precision (RSD. %) | Interday Precision (RSD. %) | Rec (%) | Intraday Rec. (%) | Interday Rec. (%) | Intraday Precision (RSD. %) | Interday Precision (RSD. %) | 0.4 ng/mL | 1 ng/mL | 4 ng/mL | 20 ng/mL | 40 ng/mL |
|-----|-----------------------------------------------|-------------|---------|------------------|------------------|----------------------------|----------------------------|---------|------------------|------------------|----------------------------|----------------------------|-----------|---------|---------|---------|---------|
| 219 | Moxidectin                                    | 4           | 97.05   | 17.46            | 19.81            | 97.15                      | 19.05                      | 19.61   | 101.68           | 7.42             | 7.30                       | 45.49                   | 0.4       | 1       | 4       | 20      | 40      |
| 220 | Myclobutanil                                  | 0.8         | 104.83  | 19.53            | 19.31            | 99.69                      | 13.23                      | 13.27   | 102.16           | 9.43             | 9.23                       | 104.53                 | 5.61      | 5.32    | 3.52    | 3.37    | 3.37    |
| 221 | N-(2,4-dimethylphenyl)-N'-methylformamidine   | 4           | 112.03  | 15.22            | 13.59            | 98.34                      | 7.94                       | 8.07    | 102.65           | 5.61             | 5.47                       | 110.23                 | 4.44      | 4.03    | 2.50    | 2.35    | 2.35    |
| 222 | N.N-Dimethyl-N'-p-tolylsulphamide (DMST, metabolite of tolyfluanid) | 4           | 115.30  | 9.04             | 7.84             | 106.19                     | 4.77                       | 4.49    | 106.16           | 3.58             | 3.37                       | 110.23                 | 4.44      | 4.03    | 2.50    | 2.35    | 2.35    |
| 223 | Nafcillin                                     | 4           | 106.61  | 19.05            | 17.87            | 95.48                      | 11.52                      | 12.07   | 98.70            | 4.02             | 4.07                       | 42.87                  | 0.4       | 1       | 4       | 20      | 40      |
| 224 | Naphtalene                                    | 1.6         | 82.87   | 15.67            | 17.47            | 114.46                     | 17.98                      | 14.45   | 106.51           | 18.61            | 17.47                       | 96.49                  | 6.52      | 6.76    | 6.52    | 6.76    | 6.76    |
| 225 | Naproxen                                      | 2           | 128.28  | 18.41            | 16.21            | 112.22                     | 2.15                       | 18.76   | 113.75           | 19.60            | 17.23                       | 102.62                 | 6.86      | 6.68    | 6.86    | 6.68    | 6.68    |
| 226 | Nitrapyrin                                    | 8           | 109.62  | 19.55            | 17.83            | 100.70                     | 19.55                      | 17.83   | 100.70           | 5.77             | 5.73                       | 95.39                  | 7.93      | 8.31    | 7.93    | 8.31    | 8.31    |
| 227 | Novobiocin                                    | 1.2         | 96.87   | 20.40            | 19.19            | 85.98                      | 18.66                      | 21.70   | 89.43            | 13.67            | 15.29                       | 95.39                  | 7.93      | 8.31    | 7.93    | 8.31    | 8.31    |
| 228 | Nuarimol                                      | 1.2         | 106.98  | 17.30            | 15.52            | 92.46                      | 7.78                       | 8.41    | 122.94           | 10.78            | 8.77                       | 89.25                  | 5.47      | 6.13    | 5.47    | 6.13    | 6.13    |
| 229 | Ofurace                                       | 0.8         | 118.87  | 20.45            | 6.62             | 93.50                      | 18.17                      | 19.43   | 88.50            | 9.60             | 10.85                       | 92.05                  | 4.06      | 4.27    | 4.06    | 4.27    | 4.06    |
| 230 | Omethoate                                     | 2           | 106.78  | 14.85            | 13.91            | 97.86                      | 17.25                      | 17.63   | 87.65            | 6.69             | 7.63                       | 105.07                 | 13.17     | 12.53   | 13.17   | 12.53   | 12.53   |
| 231 | Oxadiethyl                                    | 0.8         | 123.61  | 18.91            | 15.30            | 97.19                      | 4.29                       | 4.41    | 85.77            | 7.12             | 8.30                       | 98.57                  | 5.69      | 5.77    | 5.69    | 5.77    | 5.77    |
| 232 | Oxamyl                                        | 8           | 107.50  | 18.14            | 16.87            | 110.16                     | 18.14                      | 16.87   | 110.16           | 21.87            | 19.85                       | 105.18                 | 2.02      | 1.92    | 2.02    | 1.92    | 1.92    |
| 233 | Oxamyl-oxime                                  | 8           | 99.19   | 10.92            | 11.01            | 80.81                      | 9.57                       | 11.84   | 93.86            | 10.65            | 11.35                       | 105.18                 | 2.02      | 1.92    | 2.02    | 1.92    | 1.92    |
| 234 | Oxfendazole                                   | 0.8         | 132.57  | 17.25            | 13.01            | 100.86                     | 3.40                       | 3.37    | 90.01            | 9.30             | 9.72                       | 105.18                 | 2.02      | 1.92    | 2.02    | 1.92    | 1.92    |
Table A2. Cont.

| No. | Compound           | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday |
|-----|--------------------|-------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|
| 235 | Oxolinic acid      | 0.8         |         | 110.62   | 10.56    | 9.55    | 93.32    | 6.53     | 7.00    | 101.82   | 5.02     | 4.93    |
| 236 | Oxydemeton methyl  | 4           | 125.26  | 11.57    | 8.95     | 103.93  | 6.92     | 6.66     | 90.21   | 15.13    | 16.77    | 94.20   | 6.15     | 6.53     | 103.58  | 4.77     | 4.61    |
| 237 | Oxyfluorfen        | 4           |         | 87.69    | 18.03    | 21.96   | 104.71   | 11.43    | 10.92   | 97.01    | 14.18    | 14.62   |
| 238 | Paclobutrazol      | 1.6         | 115.25  | 5.87     | 5.09     | 97.98   | 10.85    | 11.07    | 103.31  | 5.22     | 5.05     | 103.50  | 2.43     | 2.35     |
| 239 | Parathion methyl   | 8           |         | 109.49   | 18.40    | 16.81   | 109.79   | 10.36    | 10.92   | 101.59   | 7.75     | 7.60    |
| 240 | PCB 28             | 0.4         | 109.04  | 17.35    | 21.35    | 109.79  | 10.36    | 9.44     | 89.67   | 5.77     | 6.43     | 103.24  | 6.02     | 5.83     | 94.17   | 7.16     | 7.60    |
| 241 | PCB 52             | 0.8         | 109.29  | 10.86    | 9.94     | 108.98  | 14.57    | 22.55    | 83.40   | 13.14    | 15.76    | 104.75  | 10.45    | 9.98     | 100.99  | 8.14     | 8.06    |
| 242 | PCB 77             | 0.8         | 93.46   | 20.09    | 15.79    | 99.72   | 20.85    | 20.91    | 99.96   | 10.57    | 10.57    | 103.80  | 7.39     | 7.12     | 96.95   | 10.24    | 10.56   |
| 243 | PCB 81             | 0.8         | 89.48   | 15.17    | 16.95    | 106.42  | 16.55    | 16.19    | 87.13   | 11.29    | 12.96    | 104.54  | 2.16     | 2.07     | 99.16   | 8.64     | 8.71    |
| 244 | PCB 101            | 0.4         | 111.65  | 12.54    | 19.14    | 117.34  | 14.18    | 12.08    | 95.12   | 2.94     | 3.09     | 101.23  | 5.71     | 5.64     | 101.01  | 10.12    | 10.02   |
| 245 | PCB 105            | 0.4         | 102.23  | 14.66    | 13.90    | 126.39  | 14.98    | 19.76    | 88.39   | 7.67     | 8.68     | 105.83  | 8.19     | 7.74     | 93.21   | 5.05     | 5.42    |
| 246 | PCB 114            | 0.8         | 96.49   | 20.69    | 19.42    | 103.46  | 13.87    | 13.41    | 94.88   | 11.04    | 11.64    | 104.47  | 5.35     | 5.12     | 98.79   | 7.12     | 7.21    |
| 247 | PCB 118            | 1.2         | 102.19  | 11.53    | 11.28    | 94.03   | 8.10     | 8.61     | 98.74   | 4.78     | 4.84     | 98.20   | 8.76     | 8.92     |
| 248 | PCB 123            | 1.2         | 113.43  | 22.05    | 19.44    | 90.08   | 7.66     | 8.50     | 102.24  | 8.39     | 8.21     | 96.45   | 5.29     | 5.48     |
| 249 | PCB 126            | 0.8         | 87.42   | 18.29    | 19.43    | 111.04  | 14.57    | 13.12    | 91.66   | 7.70     | 8.40     | 107.90  | 9.85     | 9.13     | 96.39   | 7.94     | 8.24    |
| 250 | PCB 138            | 0.4         | 118.82  | 20.45    | 18.79    | 105.03  | 11.58    | 11.03    | 102.64  | 10.09    | 9.83     | 105.83  | 6.52     | 6.16     | 96.84   | 6.40     | 6.61    |
| 251 | PCB 153            | 0.8         | 105.67  | 17.51    | 20.11    | 110.87  | 8.22     | 7.41     | 96.92   | 7.41     | 7.65     | 101.59  | 4.87     | 4.79     | 97.13   | 3.45     | 3.55    |
| 252 | PCB 156            | 0.8         | 89.94   | 17.49    | 19.45    | 115.25  | 23.33    | 20.24    | 90.96   | 7.99     | 8.78     | 106.25  | 7.73     | 7.28     | 98.31   | 9.18     | 9.34    |
| 253 | PCB 157            | 0.8         | 82.22   | 19.43    | 17.63    | 116.24  | 20.04    | 17.24    | 88.00   | 7.66     | 8.70     | 107.18  | 9.20     | 8.58     | 101.20  | 10.33    | 10.21   |
| 254 | PCB 167            | 0.8         | 87.27   | 18.74    | 21.47    | 111.11  | 16.53    | 23.88    | 95.41   | 9.50     | 9.96     | 105.12  | 7.64     | 7.27     | 97.88   | 10.59    | 10.82   |
| 255 | PCB 169            | 1.2         | 99.05   | 18.89    | 17.17    | 98.13   | 9.84     | 10.03    | 103.47  | 10.12    | 9.78     | 96.44   | 8.72     | 9.04     |
| 256 | PCB 180            | 0.4         | 106.04  | 19.00    | 20.25    | 108.14  | 15.01    | 12.37    | 94.39   | 9.82     | 10.40    | 109.04  | 9.09     | 8.34     | 97.26   | 7.54     | 7.75    |
| No. | Compound | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday |
|-----|----------|-------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|
| 257 | PCB 189  | 0.8         | 79.22   | 18.60    | 10.45    | 97.14   | 19.02    | 19.58    | 86.39   | 10.81    | 12.51    | 108.00  | 7.22     | 6.69     | 92.10   | 4.18     | 4.54     |
| 258 | Penconazole | 0.8       | 119.47  | 10.66    | 18.97    | 96.80   | 18.50    | 19.11    | 81.78   | 13.65    | 16.69    | 94.22   | 7.10     | 7.54     | 103.41  | 8.49     | 8.21     |
| 259 | Pencycuron | 0.4       | 110.82  | 13.26    | 11.97    | 90.36   | 11.63    | 12.87    | 86.17   | 10.68    | 12.39    | 98.98   | 3.84     | 3.88     | 101.16  | 6.82     | 6.74     |
| 260 | Pendimethalin | 2       | 102.88  | 19.86    | 19.30    | 75.06   | 16.21    | 21.60    | 104.56  | 5.38     | 5.15     | 94.40   | 11.99    | 12.70    |
| 261 | Penicillin V | 8       | 105.22  | 18.10    | 17.20    | 108.53  | 6.81     | 6.27     |
| 262 | Permethrin | 4        | 113.34  | 17.58    | 15.51    | 115.67  | 16.46    | 14.23    | 98.54   | 12.87    |
| 263 | Phenanthrene | 1.6     | 133.74  | 10.56    | 5.48     | 94.00   | 13.65    | 14.52    | 99.67   | 16.68    | 16.74    | 93.74   | 4.35     | 4.64     |
| 264 | Phenylbutazone | 16     | 110.32  | 17.68    | 21.84    |
| 265 | Phosalone | 0.4      | 110.50  | 17.38    | 15.73    | 98.28   | 8.36     | 8.51     | 89.91   | 5.27     | 5.86     | 101.98  | 2.80     | 2.75     | 102.70  | 2.35     | 2.29     |
| 266 | Phosmet  | 1.2      | 100.60  | 6.02     | 5.98     | 92.49   | 12.42    | 13.43    | 101.79  | 5.92     | 5.82     | 104.63  | 5.42     | 5.18     |
| 267 | Phthalamide (Folpet deg) | 8     | 103.79  | 11.71    | 11.28    | 100.45  | 6.35     | 6.32     |
| 268 | Pirimicarb | 0.4      | 120.22  | 8.00     | 6.65     | 95.32   | 4.74     | 4.97     | 93.09   | 11.13    | 11.96    | 102.45  | 4.63     | 4.52     | 103.60  | 2.50     | 2.41     |
| 269 | Pirimicarb-desmethyl | 2     | 110.38  | 7.61     | 6.89     | 127.79  | 12.92    | 10.11    | 114.99  | 8.23     | 7.16     | 123.05  | 4.57     | 3.71     |
| 270 | Pirimiphos ethyl | 0.8   | 82.75   | 17.50    | 21.15    | 92.12   | 9.50     | 10.31    | 98.11   | 12.10    | 12.33    | 120.39  | 11.92    | 9.90     | 101.28  | 6.04     | 7.51     |
| 271 | Pirimiphos methyl | 0.4   | 111.79  | 15.98    | 14.29    | 95.45   | 16.02    | 16.78    | 88.67   | 10.14    | 11.44    | 102.62  | 4.30     | 4.19     | 101.90  | 6.74     | 6.61     |
| 272 | Prochloraz | 0.4      | 104.39  | 17.43    | 16.70    | 91.03   | 5.00     | 5.49     | 89.27   | 11.46    | 12.84    | 100.96  | 5.85     | 5.79     | 103.41  | 5.28     | 5.11     |
| 273 | Propamidine | 8       | 92.02   | 15.09    | 16.40    | 106.35  | 16.63    | 15.04    |
| 274 | Profenofos | 0.8      | 115.10  | 18.63    | 17.91    | 93.40   | 17.40    | 18.63    | 88.46   | 19.43    | 21.96    | 84.42   | 5.94     | 7.04     | 108.50  | 7.24     | 6.67     |
| 275 | Propamocarb | 2       | 114.94  | 19.15    | 16.66    | 93.17   | 2.45     | 2.63     | 99.37   | 9.65     | 9.71     | 103.21  | 7.96     | 7.71     |
| 276 | Propargite | 0.4      | 113.51  | 7.83     | 6.90     | 97.28   | 4.47     | 4.59     | 90.04   | 6.01     | 6.67     | 99.27   | 1.99     | 2.00     | 100.59  | 5.36     | 5.33     |
| 277 | Propiconazole | 2      | 103.21  | 16.19    | 15.38    | 98.49   | 10.53    | 10.69    | 103.63  | 3.30     | 3.18     |
Table A2. Cont.

| No. | Compound                | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday | Rec (%) | Intraday | Interday |
|-----|-------------------------|-------------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|---------|----------|----------|
| 278 | Propoxur                | 0.8         | 124.75  | 20.23    | 15.08    | 92.34   | 8.00     | 8.66     | 93.26   | 18.90    | 20.27    | 101.62  | 8.07     | 7.94     | 111.03  | 3.68     | 3.31     |
| 279 | Propyzamide (pronamide) | 0.8         | 104.03  | 19.42    | 19.74    | 89.58   | 16.14    | 19.18    | 93.02   | 13.39    | 14.39    | 97.32   | 5.91     | 6.07     | 102.92  | 7.64     | 7.42     |
| 280 | Proquinazid             | 1.6         | 102.99  | 15.84    | 15.38    | 92.93   | 3.72     | 4.00     | 116.31  | 13.68    | 11.76    | 95.04   | 8.78     | 9.24     |
| 281 | Prothioconazole         | 1.6         | 118.94  | 17.99    | 15.13    | 86.94   | 10.77    | 12.39    | 101.86  | 7.03     | 6.90     | 103.49  | 5.02     | 4.85     |
| 282 | Prothiophos             | 2           | 127.35  | 17.52    | 15.03    | 91.22   | 11.43    | 12.53    | 110.73  | 16.36    | 14.77    | 90.19   | 7.42     | 8.23     |
| 283 | Pyretrizone             | 8           |         |          |          |         |          |          |         |          |          |         |          |          |          |          |          |          |          |
| 284 | Pyraclostrobin          | 0.4         | 118.10  | 14.33    | 12.13    | 88.90   | 5.97     | 6.72     | 90.62   | 2.37     | 2.62     | 97.96   | 3.11     | 3.17     | 101.93  | 4.86     | 4.77     |
| 285 | Pyrazophos              | 0.4         | 116.34  | 17.02    | 14.63    | 87.65   | 5.37     | 6.13     | 95.15   | 10.11    | 10.63    | 103.82  | 7.88     | 7.59     | 104.45  | 3.19     | 3.05     |
| 286 | Pyrene                  | 2           | 122.26  | 19.33    | 15.12    | 95.97   | 15.49    | 16.14    | 99.99   | 7.05     | 7.05     | 106.97  | 13.20    | 12.34    |
| 287 | Pyridaben               | 0.4         | 111.80  | 8.50     | 7.60     | 96.96   | 11.34    | 11.70    | 89.29   | 7.37     | 8.25     | 97.16   | 3.54     | 3.64     | 101.94  | 5.96     | 5.85     |
| 288 | Pyridaphenthion         | 0.4         | 125.33  | 10.87    | 8.67     | 88.86   | 12.18    | 13.71    | 90.69   | 9.17     | 10.11    | 103.36  | 5.75     | 5.56     | 104.97  | 2.29     | 2.18     |
| 289 | Pyrimethnilin           | 1.2         | 107.01  | 6.57     | 6.14     | 83.42   | 6.56     | 7.86     | 99.98   | 4.42     | 4.42     | 106.14  | 3.93     | 3.70     |
| 290 | Pyriproxifen            | 0.4         | 108.80  | 6.84     | 6.29     | 93.68   | 5.81     | 6.20     | 84.68   | 4.33     | 5.11     | 99.26   | 4.88     | 4.92     | 97.21   | 10.36    | 10.66    |
| 291 | Quinaldil               | 1.6         | 90.14   | 15.91    | 17.65    | 81.56   | 14.48    | 17.75    | 98.78   | 15.64    | 15.83    | 99.05   | 6.28     | 6.34     |
| 292 | Quinoloxyn              | 0.8         | 106.79  | 20.65    | 20.64    | 102.29  | 14.68    | 14.27    | 92.06   | 12.98    | 14.10    | 113.55  | 3.75     | 3.30     | 88.93   | 8.36     | 9.40     |
| 293 | Rifampicin              | 1.2         | 103.00  | 18.59    | 17.76    | 89.34   | 13.65    | 15.28    | 99.63   | 7.95     | 7.98     | 111.87  | 15.95    | 14.26    |
| 294 | Rotenone                | 0.8         | 120.70  | 16.46    | 13.64    | 89.14   | 17.53    | 20.88    | 81.92   | 14.34    | 17.50    | 101.34  | 12.36    | 12.20    | 105.21  | 3.73     | 3.55     |
| 295 | Roxithromycin           | 1.2         | 116.45  | 19.37    | 16.63    | 88.89   | 12.89    | 14.50    | 88.85   | 16.67    | 18.76    | 102.53  | 3.02     | 2.95     |
| 296 | Sarafloxacin            | 20          |         |          |          |         |          |          |         |          |          |         |          |          |          |          |          |          |          |
| 297 | Simazine                | 0.8         | 127.26  | 22.73    | 17.86    | 89.60   | 13.25    | 14.79    | 90.53   | 13.38    | 14.78    | 101.60  | 7.91     | 7.79     | 108.74  | 8.42     | 7.74     |
| 298 | Spinosad (two isomers)  | 1.6         | 111.72  | 19.81    | 17.73    | 90.14   | 14.70    | 16.31    | 89.32   | 9.26     | 10.37    | 99.29   | 5.46     | 5.50     | 100.87  | 7.97     | 7.90     |
Table A2. Cont.

| No. | Compound | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) |
|-----|----------|-------------|---------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 299 | Spiramycin (two isomers) | 12 | 105.21 | 2.31 | 2.20 | 98.92 | 6.55 | 6.62 |
| 300 | Spirodiclofen | 0.8 | 114.30 | 10.24 | 8.96 | 98.97 | 9.73 | 9.83 | 86.32 | 3.99 | 4.62 | 96.72 | 4.66 | 4.82 | 96.10 | 2.65 | 2.76 |
| 301 | Spiromesifen | 0.4 | 111.87 | 16.94 | 15.14 | 96.88 | 10.62 | 10.96 | 81.93 | 9.64 | 11.77 | 96.25 | 5.78 | 6.01 | 102.02 | 6.81 | 6.68 |
| 302 | Spirotetramat | 0.8 | 136.54 | 14.99 | 18.30 | 91.58 | 15.29 | 17.62 | 87.65 | 16.87 | 19.25 | 99.19 | 10.60 | 10.69 | 109.75 | 7.10 | 6.47 |
| 303 | Spiroxamine | 0.4 | 119.58 | 5.12 | 4.42 | 92.27 | 5.97 | 6.47 | 93.01 | 4.62 | 4.97 | 100.04 | 5.41 | 5.41 | 101.94 | 2.10 | 2.06 |
| 304 | Strychnine | 2 | 121.82 | 15.59 | 16.26 | 84.19 | 20.34 | 20.16 | 100.68 | 5.59 | 5.55 | 102.95 | 5.88 | 5.71 |
| 305 | Sulfacetamide | 16 | 94.80 | 9.96 | 10.51 |
| 306 | Sulfachloropiridacine | 4 | 109.37 | 18.24 | 16.68 | 98.92 | 7.84 | 7.93 | 102.01 | 6.91 | 6.77 |
| 307 | Sulfadiazine | 8 | 91.59 | 5.81 | 4.72 | 97.40 | 5.43 | 5.57 | 97.95 | 5.03 | 5.14 |
| 308 | Sulfadimethoxine | 2 | 126.54 | 15.11 | 10.31 | 109.89 | 11.08 | 10.08 | 103.69 | 8.01 | 7.72 | 103.97 | 3.74 | 3.60 |
| 309 | Sulfadoxine | 2 | 123.17 | 11.68 | 8.48 | 107.05 | 13.06 | 12.20 | 100.75 | 7.53 | 7.47 | 106.39 | 7.08 | 6.65 |
| 310 | Sulfameracine | 4 | 118.06 | 8.76 | 7.42 | 95.45 | 3.06 | 3.21 | 102.43 | 3.58 | 3.50 |
| 311 | Sulfametacine | 2 | 112.28 | 8.28 | 105.33 | 12.84 | 12.19 | 102.14 | 7.00 | 6.85 | 104.35 | 4.92 | 4.71 |
| 312 | Sulfametizole | 4 | 108.41 | 7.69 | 7.09 | 97.15 | 4.81 | 4.95 | 101.75 | 12.24 | 12.03 |
| 313 | Sulfametoxazole | 2 | 117.53 | 19.33 | 21.76 | 99.36 | 6.59 | 6.63 | 100.10 | 5.77 | 5.76 | 104.08 | 10.50 | 10.09 |
| 314 | Sulfamethoxypyridazine | 1.6 | 122.96 | 10.02 | 7.23 | 93.76 | 15.04 | 16.04 | 95.82 | 4.12 | 4.30 | 106.04 | 6.02 | 5.68 |
| 315 | Sulfamonomethoxine | 4 | 115.82 | 19.10 | 16.49 | 109.03 | 9.00 | 8.25 | 102.71 | 5.89 | 5.73 |
| 316 | Sulfapyridine | 4 | 123.12 | 5.81 | 4.72 | 97.40 | 5.43 | 5.57 | 97.95 | 5.03 | 5.14 |
| 317 | Sultaquinonoxaline | 2 | 137.13 | 15.86 | 11.57 | 107.09 | 16.46 | 15.37 | 103.03 | 8.49 | 8.24 | 105.72 | 5.77 | 5.46 |
| 318 | Sulfatiazole | 4 | 114.09 | 12.17 | 10.67 | 89.17 | 3.00 | 3.36 | 96.75 | 9.66 | 9.98 |
| 319 | Sulfisoxazole | 4 | 123.17 | 15.55 | 12.62 | 109.15 | 6.85 | 6.28 | 102.56 | 9.83 | 9.58 |
| Compound               | 0.4 ng/mL | 1 ng/mL | 4 ng/mL | 20 ng/mL | 40 ng/mL |
|-----------------------|-----------|---------|---------|----------|----------|
| **Precision (RSD. %)** |           |         |         |          |          |
| Tebuconazole          | 2         | 124.33  | 10.86   | 8.73     | 102.25   |
| Tebufenocide          | 0.8       | 113.40  | 19.30   | 17.02    | 94.01    |
| Tebufenpyrad          | 0.8       | 125.31  | 18.01   | 14.37    | 106.17   |
| Teflubenzuron         | 1.6       | 108.57  | 19.06   | 17.56    | 89.65    |
| Tefluthrin            | 0.4       | 124.36  | 21.64   | 16.23    | 105.00   |
| Tebucin (isobenzan)   | 2         | 121.92  | 12.85   | 21.31    | 88.03    |
| Terbufos              | 0.8       | 104.09  | 14.21   | 13.26    | 88.68    |
| Terbutylazine         | 0.8       | 120.91  | 9.86    | 8.15     | 93.77    |
| Tetrachlorvinphos     | 2         | 126.81  | 17.78   | 13.00    | 100.05   |
| Tetrachlorvinphos     | 0.8       | 116.66  | 14.53   | 20.60    | 90.40    |
| Telodrin (isobenzan)  | 2         | 120.30  | 14.89   | 12.38    | 86.00    |
| Terbuthylazine        | 0.8       | 106.92  | 12.99   | 19.50    | 84.62    |
| Tetradifon            | 1.6       | 106.92  | 12.99   | 19.50    | 91.97    |
| Tetradifon            | 2         | 120.30  | 14.89   | 12.38    | 113.61   |
| Thiabendazole         | 1.2       | 96.72   | 17.21   | 16.26    | 86.00    |
| Thiacloprid           | 0.8       | 121.34  | 7.44    | 5.66     | 90.02    |
| Thiamethoxam          | 8         | 121.06  | 6.35    | 5.25     | 92.87    |
| Thiophanate methyl    | 2         | 124.15  | 8.09    | 6.03     | 101.95   |
| Tolclofos methyl      | 1.6       | 123.59  | 15.07   | 12.19    | 84.62    |
| Tolfenamic acid       | 1.6       | 105.31  | 15.54   | 20.30    | 104.76   |
| Triadimefon           | 1.2       | 94.15   | 15.09   | 16.33    | 95.62    |
| Triadimenol           | 0.8       | 114.21  | 11.55   | 9.26     | 106.40   |
| Triazophos (hostathion)| 0.4      | 112.50  | 15.63   | 12.76    | 91.77    |

Table A2. Cont.
Table A2. Cont.

| No. | Compound      | LOQ (ng/mL) | Rec (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) | Intraday | Interday | Rec. (%) |
|-----|---------------|-------------|---------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 341 | Trifloxystrobin | 0.4         | 112.87  | 13.12    | 11.62    | 92.56    | 5.89     | 6.36     | 93.35    | 4.78     | 5.12     | 100.81   | 2.86     | 2.84     | 102.51   | 4.76     | 4.64     | 104.02   | 4.76     | 4.64     | 104.02   | 4.76     | 4.64     | 104.02   | 4.76     | 4.64     | 104.02   | 4.76     | 4.64     |
| 342 | Triflumizole   | 0.4         | 110.08  | 9.26     | 8.41     | 94.01    | 7.01     | 7.46     | 93.26    | 7.62     | 8.17     | 99.91    | 2.69     | 2.69     | 101.62   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     |
| 343 | Triflumuron    | 1.2         | 118.66  | 19.24    | 16.21    | 86.93    | 7.78     | 8.95     | 96.65    | 3.90     | 4.04     | 106.18   | 6.37     | 6.00     | 101.62   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     | 103.18   | 3.67     | 3.61     |
| 344 | Trifluralin    | 1.2         | 93.29   | 17.78    | 19.06    | 92.40    | 6.37     | 6.89     | 105.89   | 9.83     | 9.28     | 93.54    | 4.17     | 4.46     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     |
| 345 | Trimethoprim   | 2           | 116.02  | 16.54    | 12.16    | 109.21   | 15.05    | 13.78    | 94.20    | 12.51    | 13.28    | 97.92    | 8.70     | 8.88     | 101.84   | 8.67     | 8.60     | 104.04   | 8.67     | 8.60     | 104.04   | 8.67     | 8.60     | 104.04   | 8.67     | 8.60     |
| 346 | Triticonazole  | 1.2         | 99.19   | 8.60     | 8.67     | 89.71    | 12.71    | 14.17    | 100.84   | 8.67     | 8.60     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     | 104.04   | 4.36     | 4.19     |
| 347 | Tylmicosin     | 4           | 107.14  | 16.70    | 15.99    | 93.58    | 6.97     | 7.45     | 101.18   | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    | 11.19    | 11.06    |
| 348 | Tylosin        | 8           | 106.35  | 4.46     | 4.19     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     | 104.60   | 2.79     | 2.67     |
| 349 | Vinclozolin    | 1.6         | 116.52  | 23.46    | 17.30    | 88.94    | 12.82    | 14.41    | 105.60   | 12.42    | 11.76    | 92.86    | 4.92     | 5.30     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     |
| 350 | Warfarin       | 0.8         | 100.47  | 17.51    | 19.89    | 95.53    | 9.12     | 9.55     | 85.61    | 11.19    | 14.75    | 93.09    | 6.28     | 6.75     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     | 103.65   | 4.31     | 4.16     |
| 351 | Zoxamide       | 0.8         | 121.96  | 19.52    | 18.47    | 88.50    | 11.13    | 13.88    | 90.57    | 19.33    | 20.34    | 106.93   | 5.18     | 4.84     | 105.40   | 6.44     | 6.11     | 105.40   | 6.44     | 6.11     | 105.40   | 6.44     | 6.11     | 105.40   | 6.44     | 6.11     | 105.40   | 6.44     | 6.11     |
### Appendix C

**Table A3.** Matrix Effect, expressed as percentage, calculated for all of the analytes. The range form −20% to 20% represent the tolerance range in which it is considered that no significant matrix effect exists.

| No. | Compound                        | Matriz effect | No. | Compound                        | Matriz effect | No. | Compound                        | Matriz effect | N°  | Compound                        | Matriz Effect |
|-----|---------------------------------|---------------|-----|---------------------------------|---------------|-----|---------------------------------|---------------|------|---------------------------------|---------------|
| 1   | 2-Phenylphenol                  | −103.4        | 118 | Eprinometin                     | −6.3          | 235 | Oxolinic acid                   | 0.5           |
| 2   | 4,4′-Dichlorobenzophenone       | 96.6          | 119 | Eritromicin                     | 45.4          | 236 | Oxydemeton methyl               | 3.3           |
|     | (metabolite of dicofol)         |               |     |                                 |               |     |                                 |               |
| 3   | Abamectine                      | −70.6         | 120 | Esfenvalerate                   | 23.4          | 237 | Oxyfluorfen                     | 42.4          |
| 4   | Acenaphthene                    | 14.3          | 121 | Ethion (diethion)               | −13.7         | 238 | Paclobutrazol                   | 3.0           |
| 5   | Acenaphtylene                   | 17.0          | 122 | Ethirimol                       | 13.9          | 239 | Parathion methyl                | 12.8          |
| 6   | Acephate                        | −90.4         | 123 | Ethofumesate                    | 30.5          | 240 | PCB 28                         | 32.5          |
| 7   | Acetamiprid                     | 7.0           | 124 | Ethoprofos                      | −20.5         | 241 | PCB 52                         | 32.7          |
| 8   | Acrinathrin                     | 16.2          | 125 | Etofenprox                      | 43.0          | 242 | PCB 77                         | 52.5          |
| 9   | Albinathrin                     | 17.7          | 126 | Etoxazole                       | −29.6         | 243 | PCB 81                         | 43.7          |
| 10  | Aldicarb                        | 5.8           | 127 | Famoxadone                      | 27.9          | 244 | PCB 101                        | 8.2           |
| 11  | Aldicarb-sulfone                | −4.5          | 128 | Fenamidone                      | 12.7          | 245 | PCB 105                        | 54.6          |
| 12  | Aldicarb-sulfoxide              | −2.9          | 129 | Fenamiphos                      | 14.0          | 246 | PCB 114                        | 46.6          |
| 13  | Aldrin                          | 43.7          | 130 | Fenamiphos sulfoxide            | 10.4          | 247 | PCB 118                        | 45.0          |
| 14  | Anthracene                      | 23.9          | 131 | Fenamiphos sulfoxide            | 0.0           | 248 | PCB 123                        | 45.7          |
| 15  | Atrazine                        | 7.8           | 132 | Fenarimol                       | 55.3          | 249 | PCB 126                        | 52.1          |
| 16  | Azinphos-methyl                 | 14.3          | 133 | Fenazaquin                      | −108.8        | 250 | PCB 138                        | 54.9          |
| 17  | Azoxyostrobin                   | −6.9          | 134 | Fenbendazole                    | −7.9          | 251 | PCB 153                        | 48.2          |
| 18  | BDE-28                          | 47.0          | 135 | Fenbuconazole                   | 67.7          | 252 | PCB 156                        | 59.3          |
Table A3. Cont.

| No. | Compound                        | Matriz effect | No.  | Compound                        | Matriz effect |
|-----|---------------------------------|---------------|------|---------------------------------|---------------|
| 19  | BDE-47                          | 47.8          | 136  | Fenhexamid                      | 5.9           |
| 20  | BDE-85                          | 43.7          | 137  | Fenitrothion                     | 54.6          |
| 21  | BDE-99                          | 57.3          | 138  | Fenoxy carb                     | 16.4          |
| 22  | BDE-100                         | 51.3          | 139  | Fenpropathrin                   | −2.2          |
| 23  | BDE-153                         | 35.9          | 140  | Fenpropidin                     | 22.3          |
| 24  | BDE-154                         | 57.8          | 141  | Fenpropimorph                   | 1.7           |
| 25  | BDE-183                         | −14.1         | 142  | Fenpyroximate                   | 19.8          |
| 26  | Benalaxyl                       | −111.5        | 143  | Fenthion                        | −33.9         |
| 27  | Bendiocarb                      | −5.4          | 144  | Fenthion oxon                   | 27.1          |
| 28  | Bendiocarb metabolite           | 39.7          | 145  | Fenthion oxon sulfone           | 5.3           |
|     | (2,2-dimethylbenzo-1,3-dioxol-4-ol) |         | 29   | Benfuracarb                     | 24.0          |
| 30  | Benzo[a]anthracene              | 58.5          | 146  | Fenthion oxon sulfoxide         | 3.7           |
| 31  | Benzo[a]pyrene                  | 45.0          | 147  | Fenthion sulfoxide              | 6.9           |
| 32  | Benzo[b]fluoranthene            | 59.8          | 148  | Fenthion sulfoxide              | 11.9          |
| 33  | Benzo[ghi]perylene              | 24.4          | 149  | Fenvalerate                     | −39.6         |
| 34  | Benzo[k]fluoranthene            | 38.1          | 150  | Fipronil                        | 21.0          |
| 35  | Bifenthrin                      | 80.4          | 151  | Fipronil sulfide                | 676.5         |
| 36  | Bitertanol                      | −0.7          | 152  | Flocoumafene                    | −34.5         |
| 37  | Boscalid (formerly nicobifen)    | 73.5          | 153  | Fluazinam                       | −1.5          |
|     |                                 |               | 154  | Flubendiamide                   | 62.7          |
|     |                                 |               | 253  | PCB 157                         | 51.6          |
|     |                                 |               | 254  | PCB 167                         | 60.1          |
|     |                                 |               | 255  | PCB 169                         | 60.6          |
|     |                                 |               | 256  | PCB 180                         | 52.5          |
|     |                                 |               | 257  | PCB 189                         | 54.8          |
|     |                                 |               | 258  | Penconazole                     | 50.4          |
|     |                                 |               | 259  | Pencycuron                      | −37.4         |
|     |                                 |               | 260  | Pendimethalin                   | 29.8          |
|     |                                 |               | 261  | Penicillin V                    | −8.1          |
|     |                                 |               | 262  | Permethrin                      | 71.5          |
|     |                                 |               | 263  | Phenanthrene                    | 29.6          |
|     |                                 |               | 264  | Phenylbutazone                  | −4.9          |
|     |                                 |               | 265  | Phosalone                       | −4.0          |
|     |                                 |               | 266  | Phosmet                         | 5.8           |
|     |                                 |               | 267  | Phthalamide (Folpet deg)        | 59.3          |
|     |                                 |               | 268  | Pirimicarb                      | 1.7           |
|     |                                 |               | 269  | Pirimicarb-desmethyl            | 57.7          |
|     |                                 |               | 270  | Pirimiphos ethyl                | 60.5          |
|     |                                 |               | 271  | Pirimiphos methyl               | −14.7         |
Table A3. Cont.

| No. | Compound                  | Matriz effect | No. | Compound                  | Matriz effect | Nº  | Compound                  | Matriz Effect |
|-----|---------------------------|---------------|-----|---------------------------|---------------|-----|---------------------------|---------------|
| 38  | Brodifacoum               | −31.7         | 155 | Flucythrinate (two isomers) | 4.0           | 272 | Prochloraz               | −37.2         |
| 39  | Bromadiolone              | −2.1          | 156 | Fludioxonil               | 61.2          | 273 | Procyymidone              | 52.9          |
| 40  | Bromopropylate            | 94.0          | 157 | Flufenoxuron              | 1.1           | 274 | Profenofos                | −15.0         |
| 41  | Bromuconazole (two isomers) | 39.7        | 158 | Flumequine                | −0.8          | 275 | Propamocarb               | 16.2          |
| 42  | Bupirimite                | 57.0          | 159 | Flunixin                  | −9.4          | 276 | Propargite                | −29.3         |
| 43  | Buprofezin                | −0.4          | 160 | Fluopyram                 | 45.4          | 277 | Propiconazole             | −85.8         |
| 44  | Cadusafos (ebufos)        | −54.8         | 161 | Floranthene               | 36.0          | 278 | Propoxur                  | −7.1          |
| 45  | Carbaryl                  | 15.8          | 162 | Fluorene                  | 17.2          | 279 | Propyzamide (pronamide)   | −2.0          |
| 46  | Carbendazim (azole)       | 16.8          | 163 | Fluquinconazole           | 55.3          | 280 | Proquinazid               | 72.7          |
| 47  | Carbofuran                | −9.0          | 164 | Flusilazole               | 3.8           | 281 | Prothioconazol            | 51.5          |
| 48  | Carbofuran-3-hydroxy      | 5.5           | 165 | Flutolanil                | −10.2         | 282 | Prothiophos               | 49.5          |
| 49  | Carbosulfan               | −107.2        | 166 | Flutriafol                | 54.5          | 283 | Pymetrozine               | 16.2          |
| 50  | Cefuroxima axetil (two isomers) | 24.0   | 167 | Fluvalinate tau            | −31.7         | 284 | Pyraclostrobin            | −18.0         |
| 51  | Chloramphenicol           | 24.4          | 168 | Fonofos                   | 28.9          | 285 | Pyrazophos                | 20.6          |
| 52  | Chlorantraniliprole       | 4.5           | 169 | Formetanate               | −38.7         | 286 | Pyrene                    | 45.1          |
| 53  | Chlorfenvinphos           | −31.3         | 170 | Fothiazate                | 2.9           | 287 | Pyridaben                 | −83.6         |
| 54  | Chlorbenzilate            | 95.2          | 171 | Heptachlor                | −110.9        | 288 | Pyridaphenthion           | 14.3          |
| 55  | Chlorphacineone           | 38.1          | 172 | Hexachlorobencene         | 16.9          | 289 | Pyrimethanil              | 30.6          |
| 56  | Chlorpropham              | 24.2          | 173 | Hexachlorocyclohexane (alpha) | −58.1   | 290 | Pyriproxifen              | −24.8         |
| No. | Compound                     | Matriz effect | No. | Compound                     | Matriz effect | No. | Compound                     | Matriz Effect |
|-----|------------------------------|---------------|-----|------------------------------|---------------|-----|------------------------------|---------------|
| 57  | Chlorpyrifos                | 38.0          | 174 | Hexachlorocyclohexane (beta) | −97.4         | 291 | Quinalfos                    | 3.7           |
| 58  | Chlorpyrifos methyl         | 9.6           | 175 | Hexachlorocyclohexane (delta)| −89.1         | 292 | Quinoxyfen                   | −68.5         |
| 59  | Chlorthal dimethyl          | 36.6          | 176 | Hexachlorocyclohexane (gamma, lindane) | −109.8 | 293 | Rifampicin                   | −68.9         |
| 60  | Chrysene                    | 38.1          | 177 | Hexaconazole (two isomers)  | −4.6          | 294 | Rotenone                     | 22.0          |
| 61  | Clindamycin                 | −7.5          | 178 | Hexaflumuron                 | −1.3          | 295 | Roxithromycin                | 0.0           |
| 62  | Clofentezine                | 13.2          | 179 | Hexythiazox                 | −27.6         | 296 | Sarafloxacin                 | 114.8         |
| 63  | Clothianidin                | 17.7          | 180 | Imazalil (eniconazole)      | 11.5          | 297 | Simazine                     | −19.7         |
| 64  | Cloxacillin                 | 4.9           | 181 | Imidaclorpid                | 4.1           | 298 | Spinosad (two isomers)       | −84.2         |
| 65  | Coumachlor                  | 46.2          | 182 | Indeno [1,2,3-cd] pyrene     | −132.0        | 299 | Spiramycin (two isomers)     | 16.8          |
| 66  | Coumaphos                   | −105.9        | 183 | Indoxacarb                  | 2.6           | 300 | Spirodiclofen                | −12.7         |
| 67  | Coumatetralyl               | −25.6         | 184 | Iprovalicarb                | −4.9          | 301 | Spiromesifen                 | −23.6         |
| 68  | Cyazofamid                  | 12.7          | 185 | Isofenphos methyl           | 42.0          | 302 | Spirotetramat                | −34.1         |
| 69  | Cyflufenamid                | −3.0          | 186 | Isoprothiolane              | −13.5         | 303 | Spiroxamine                  | 9.4           |
| 70  | Cyfluthrin (sum of four isomers) | −6.1       | 187 | Ivermectin B1a             | −82.6         | 304 | Strychnine                   | 11.9          |
| 71  | Cyhalothrin (lambda isomer) | −22.4         | 188 | Josamycin                   | −0.4          | 305 | Sulfacetamide                | −27.9         |
| 72  | Cymoxanil                   | 13.6          | 189 | Ketoprofen                  | 10.0          | 306 | Sulfachloropiridacine        | 61.1          |
| 73  | Cypermethrin (sum of four isomers) | 20.9      | 190 | Kresoxim methyl            | 36.3          | 307 | Sulfadiazine                 | −15.0         |
Table A3. Cont.

| No. | Compound (two isomers) | Matriz effect | No. | Compound | Matriz effect | No. | Compound | Matriz Effect |
|-----|------------------------|---------------|-----|----------|---------------|-----|----------|---------------|
| 74  | Cyproconazole          | 8.2           | 191 | Levamisole| 10.0          | 308 | Sulfadimetoxine| 18.2          |
| 75  | Cyprodinil             | −5.7          | 192 | Lincomycin| 135.3         | 309 | Sulfadoxine | 2.2           |
| 76  | Cyromazine             | −106.0        | 193 | Linuron   | 0.0           | 310 | Sulfameracine| 83.7          |
| 77  | Danofloxacin           | 172.1         | 194 | Lufenuron | 7.3           | 311 | Sulfametacine| 166.8         |
| 78  | Dazomet                | 33.1          | 195 | Mandipropamid| 20.6       | 312 | Sulfametizole| 170.5         |
| 79  | Deltamethrin           | −33.7         | 196 | Mefenazol | 11.5          | 313 | Sulfametoxazole| 26.0         |
| 80  | Demeton-S-methyl       | −27.1         | 197 | Mefenamic acid| 33.5       | 314 | Sulfametoxipiridacine| 107.7 |
| 81  | Demeton-S-methyl-sulfone (Dioxydemeton) | 7.5 | 198 | Mefenoxam (metalaxyl-M) | −5.9 | 315 | Sulfamonomethoxine | 1.7 |
| 82  | Dexamethasone          | 5.4           | 199 | Meloxicam | 34.8          | 316 | Sulfapyridine | 24.6          |
| 83  | Diazinon               | 22.3          | 200 | Mepanipyrm| 64.2          | 317 | Sulfadimethoxine | 2.5 |
| 84  | Dibenz[a,hanthracene  | 35.2          | 201 | Mepiquat  | −57.2         | 318 | Sulfadimethoxine | 26.0 |
| 85  | Dichlorodiphenyldichloroethane (p.p’ DDD) | 14.9 | 202 | Metaflumizone | 5.4 | 319 | Sulfisoxazole | 11.6 |
| 86  | Dichlorodiphenyldichloroethylene (p.p’ DDE) | 44.5 | 203 | Metalaxyl | −47.0         | 320 | Tebuconazole | −72.1 |
| 87  | Dichlorodiphenyltrichloroethane (p.p’ DDT) | −124.9 | 204 | Metaldehyde| −25.7       | 321 | Tebufenocid | −7.8 |
| 88  | Diclofenac             | 8.6           | 205 | Metconazole| −26.1         | 322 | Tebufenpyrad | 7.9           |
| 89  | Dicloran               | −0.4          | 206 | Methamidophos (two isomers) | −16.6 | 323 | Teflubenzuron | −31.5 |
| 90  | Diclorvos              | −42.8         | 207 | Methidathion| 7.1          | 324 | Tefluthrin | 33.9          |
| 91  | Dicloxacillin          | −4.4          | 208 | Methiocarb | 6.2           | 325 | Telodrin (isobenzan) | −22.0 |
| 92  | Dieldrin               | 19.7          | 209 | Methiocarb-sulfone | 3.0 | 326 | Terbufos | 24.0          |
Table A3. Cont.

| No. | Compound                                    | Matriz effect | No. | Compound                                    | Matriz effect | Nº  | Compound                                    | Matriz Effect |
|-----|---------------------------------------------|---------------|-----|---------------------------------------------|---------------|-----|---------------------------------------------|---------------|
| 93  | Diethathyl ethyl                            | −15.6         | 210 | Methiocarb-sulfoxide                        | 3.0           | 327 | Terbutylazine                               | 29.2          |
| 94  | Diethofencarb                               | 7.5           | 211 | Methomyl                                    | 1.8           | 328 | Tetrachlorvinphos                           | 1.1           |
| 95  | Difenacoum                                  | 7.8           | 212 | Methoxyfenozide                            | −7.0          | 329 | Tetracronazole                              | 48.4          |
| 96  | Difenoconazole                              | −21.3         | 213 | Metoxychlor                                 | −124.6        | 330 | Tetradiifon                                 | 41.7          |
| 97  | Difethialone                                | 16.8          | 214 | Metrafenone                                | 6.1           | 331 | Tetramethrin                                | 154.7         |
| 98  | Difloxacin                                  | 99.9          | 215 | Metronidazole                              | −13.3         | 332 | Thiabendazole                               | 67.7          |
| 99  | Diflubenzuron                               | 38.5          | 216 | Mevinphos (phosdrin)                        | −3.3          | 333 | Thiacloprid                                 | 12.3          |
| 100 | Diflufenican                                | 8.1           | 217 | Mirex                                      | −82.6         | 334 | Thiamethoxam                                | 19.7          |
| 101 | Dimethamid-P (and its R-isomer)             | 8.7           | 218 | Monocrotophos                              | −1.2          | 335 | Thiophanate methyl                         | −4.2          |
| 102 | Dimethoate                                  | 4.8           | 219 | Moxidectin                                 | 20.7          | 336 | Tolclofos methyl                           | 21.0          |
| 103 | Dimethomorph (two isomers)                  | 24.2          | 220 | Myclobutanil                               | 24.7          | 337 | Tolfenamic acid                            | 136.0         |
| 104 | Dimethylphenylsulfamide (DMSA, metabolite of dichlofluanid) | 4.9 | 221 | N-(2,4-dimethylphenyl)-N'-methylformamidine (DMPE, metabolite of amitraz) | 9.6 | 338 | Triadimefon                                | 20.6          |
| 105 | Diniconazole-M                              | −11.4         | 222 | N,N-Dimethyl-N'-p-tolylsulphamid (DMST, metabolite of tolyfluanid) | −8.2 | 339 | Triadimenoix                               | 27.9          |
| 106 | Dinocap                                     | −11.9         | 223 | Nafcillin                                  | −8.2          | 340 | Triazophos (hostathion)                     | −6.3          |
| 107 | Diphacinone                                 | 14.1          | 224 | Naphtalene                                | 12.9          | 341 | Trifloxystrobin                            | −55.8         |
Table A3. Cont.

| No. | Compound              | Matriz effect | No. | Compound   | Matriz effect | Nº  | Compound       | Matriz Effect |
|-----|-----------------------|---------------|-----|------------|---------------|-----|----------------|---------------|
| 108 | Diphenylamine         | −102.4        | 225 | Naproxen   | 16.5          | 342 | Triflumizole   | −14.3         |
| 109 | Dodine                | −92.0         | 226 | Nitenpyram | −4.5          | 343 | Triflumuron    | 3.6           |
| 110 | Doramectina           | −16.2         | 227 | Novobiocin | 2.9           | 344 | Trifluralin    | −1.7          |
| 111 | Endosulfan alfa       | −2.5          | 228 | Nuarimol   | 74.1          | 345 | Trimethoprim   | 25.2          |
| 112 | Endosulfan beta       | −52.9         | 229 | Ofurace    | −27.2         | 346 | Triticonazole  | 13.7          |
| 113 | Endosulfan sulfate    | −85.8         | 230 | Omethoate  | −0.4          | 347 | Tylmicosin     | 140.3         |
| 114 | Endrin                | −59.9         | 231 | Oxadixyl   | 6.5           | 348 | Tylosin        | −1.7          |
| 115 | Enrofloxacin          | 141.8         | 232 | Oxamyl     | 2.6           | 349 | Vinclozolin    | 33.9          |
| 116 | EPN                   | 32.6          | 233 | Oxamyl-oxime| 30.1         | 350 | Warfarin       | 0.0           |
| 117 | Epoxiconazole         | 10.0          | 234 | Oxfendazole| 7.0           | 351 | Zoxamide       | −83.0         |
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