Analytical Methods for Pesticides and Herbicides

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ABSTRACT: A review of literature published in 2010 on analytical methods for pesticides and herbicides is presented here in eight sections, including extraction methods, chromatographic or mass spectrometric techniques, electrochemical techniques, spectrophotometric techniques, chemiluminescence and fluorescence methods, sensors, biochemical assays and immunoassays, and miscellaneous techniques. Because many of the research studies in the cited publications utilized more than one analytical method, the different sections should not be thought of as being mutually exclusive.

KEYWORDS: analytical methods, pesticides, herbicides, insecticides, fungicides, organochlorine, organophosphate, organophosphorus, GC, LC, MS, analytical chemistry, sensors, immunoassays, chemiluminescence, fluorescence

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Extraction Techniques

Successful extraction techniques are crucial in many applications to enable the proper analyses of pesticides and herbicides in aquatic, soil, and other media. The extraction techniques can be broadly classified as solid– or liquid–phase extraction or dispersion, with much overlap and other sub–categories, such as microextraction techniques. In solid–phase extraction, Carpinteiro et al. (2010) developed a procedure to extract, purify, and analyze for the fungicides metalaxyl–M, azoxystrobin, myclobutanil, flusilazole, penconazole, tebuconazole, propiconazole, diniconazole and difenoconazole in wine samples using mixed–mode, anion exchange, and reversed–phase, OASIS MAX solid–phase extraction (SPE) cartridges for purification and extraction and analyses using liquid chromatography coupled to tandem electrospray ionization mass spectrometry (LC–ESI–MS/MS). Several commercial wines from Spain were analyzed, and metalaxyl–M and azoxystrobin were found at concentrations up to several µg/L in most samples, with the analytical technique exhibiting a limit of quantification (LOQ) in the range of 0.01–0.79 µg/L for the analytes. Rodrigues et al. (2010) analyzed pesticides in onions using matrix solid–phase dispersion (MSPD) and liquid chromatography tandem electrospray ionization mass

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spectrometry (LC–ESI–MS/MS). The best results were obtained using 0.5 g of sample, 1.0 g reused C18 bonded silica, an interaction time of 1 h and dispersion time of 5 min, and with acetonitrile as the elution solvent, and the average recoveries ranged from 78 to 120% and the detection and quantification limits ranged from 0.003 to 0.03 mg/kg and 0.01 to 0.1 mg/kg, respectively.

Garcia–Rodriguez, Carro–Diaz, Cela, et al. (2010) developed a microwave–assisted extraction method followed by clean–up with solid–phase extraction (SPE) using gas chromatography–tandem mass spectrometry (LVI–GC–MS/MS) to analyze for 17 pesticides in seaweed. The most effective extraction conditions were achieved at 125 °C and 12 min using 24 mL of hexane/ethyl acetate (80:20), with SPE cleanup of the extracts with graphitized carbon and Florisil to remove matrix interferences. The analytical recoveries were close to 100% for the analytes, and the limits of detection and limits of quantification ranged from 0.3 to 23.1 pg/g and 2.3 and 76.9 pg/g, respectively. A method for analyzing 20 azole fungicides in vegetables using offline disperse solid phase extraction and gas chromatography–positive chemical ionization mass spectrometry (DSPE–GC–MS/PCI) was developed (Shen et al., 2010). The recoveries of all pesticides were in the range from 71.2% to 102.2% at three spiked level, 0.010, 0.020 and 0.040 mg/kg, and the linearity of the method was good from 50 to 1000 μg/L, and the limits of detection were all 10 μg/kg or less.

Yu et al. (2010) developed a solid phase extraction (SPE) rapid resolution liquid chromatography method coupled with UV detection and electrospray ionization mass spectrometry to analyze for the herbicide mefenacet (MN) and its three photolysis degradation products hydroxylbenzothiazole (HBT), N–methylaniline (N–MA), and 2–benzothiazoloxyacetic acid (2–BAA) in water. The mean recoveries were between 85.6 and 101.4%, and the limits of detection were 0.02 μg/L for MN and 2–BAA and 0.04 μg/L for HBT and N–MA. An analytical method using solid phase extraction (SPE) and analyses by gas chromatography/mass spectrometry (GC–MS) was developed to analyze trace levels of 41 agricultural pesticides and transformation products (Momplaisir et al., 2010). Solid phase extraction using the polymeric resin Abselut Nexus was optimized to extract the analytes from water samples, and recoveries from water with 100 pg/L and 1 ng/L concentrations of the pesticides were generally greater than 75%, although dimethoate, disulfoton, and phorate were not recovered. Covaci et al. (2010) studied the use of matrix solid–phase dispersion and gas chromatography–mass spectrometry for the analyses of 36 pesticides in agricultural products. In the study, homogenized 0.5 g samples , 0.5 g of C8–modified silica, and 1 g of Na_2SO_4 were mixed and transferred to a cartridge containing 0.5 g activated silica, and the best recoveries (e.g., > 60%) were obtained using dichloromethane–ethyl acetate (4: 1 v/v) for elution.

Because interferences from calcium are often observed during enrichment and cleanup when analyzing for glyphosate, aminomethylphosphonic acid, and glufosinate, Kusters and Gerhartz (2010) developed a cleanup and enrichment procedure for the determination of these phosphonic acid herbicides in drinking water using a cation–exchange resin. Limits of detection of 12, 14, and 12 ng/L and mean recoveries from actual drinking water samples of 98, 100, and 101% were obtained for glyphosate, aminomethylphosphonic acid, and glufosinate, respectively. A multiresidue method was developed by Kamel et al. (2010) for analyzing thirteen neonicotinoid pesticides and metabolites and nine macrocyclic lactone pesticides and veterinary drugs using solid phase extraction (SPE) and ultra–performance liquid chromatography /MS/MS. The recoveries of the analytes from the spiked matrices were mostly between 70 and 120%, and the limits of detection ranged between 0.001 and 2 ng/g.

A matrix solid–phase dispersion sample cleanup procedure was developed for analyzing pesticide residues in lanolin (Perez et al., 2010). In this method, lanolin is dispersed on C–18 bonded silica, and the pesticides are eluted with acetonitrile saturated with *n*–hexane, yielding recoveries from 83 to 118%. Gilbert–Lopez, Garcia–Reyes, Fernandez–Alba, et al. (2010) evaluated two sample treatment methodologies for the multiresidue fast liquid chromatography–electrospray time–of–flight mass spectrometry analyses of pesticides in olive oil using methodologies based on liquid–liquid partitioning and dispersive solid–phase extraction clean–up or liquid partitioning followed by matrix solid–phase dispersion. Recoveries at 10 and 100 µg/kg levels were 70 to 130% for most of the analytes using either treatment method, and the limits of detection were lower than 10 µg/kg for most of the analytes.

Dulaurent et al. (2010) developed a general unknown screening procedure based on solid–phase extraction (SPE) and liquid chromatography coupled with a single linear ion trap mass spectrometer to analyze for pesticides and their metabolites in whole blood. The limits of detection for 320 compounds were studied by extracting 1 mL spiked blood at concentrations between 10 µg/L and 10 mg/L of the analytes. Dujakovic et al. (2010) developed a method for detecting the 14 most common agricultural pesticides in Serbian surface and ground waters. The protocol involved a solid–phase extraction followed by LC–MS/MS. When used on actual Serbian water samples, the following pesticides were detected: carbendazim, dimethoate, carbofuran, and propazine with carbendazim being the most frequent. Wang, Zou, et al. (2010) developed a method to analyze for enestroburin residues in wheat grain, wheat straw, and soil by solid–phase extraction (SPE) and HPLC–UV. They reported a recovery greater than 88.0% and a sensitivity of 0.005 mg/kg. The Japanese positive list system for residual agricultural chemicals in food led Noda et al. (2010) to develop a protocol that simultaneously detects a majority of the 116 chemicals on the list for salt products. The authors intentionally added all 116 chemicals to salt samples that were then treated with their method, and 94 of the 116 chemicals were successfully extracted with solid phase extraction with recoveries higher than 70% according to GC–MS.

A method was developed to analyze for eight pyrethroids in water samples by liquid–liquid microextraction based on solidification of floating organic droplets followed by gas chromatography with electron capture detection (Chang et al., 2010). Under optimal conditions, enrichment factors ranged from 824 to 1,432, and the limit of detection (LOD) ranged from 2.0 to 50 ng/L, and the recoveries ranged from 79.0% to 113.6%. Jia, Mao, et al. (2010) reported on a multiresidue method for analyzing 74 pesticides and metabolites in traditional Chinese herbal medicines using accelerated solvent extraction (ASE) coupled with HPLC/MS/MS. Analysis of extracts spiked at three concentration levels ranging from 0.005 to 0.125 mg/kg the analytes yielded recoveries from 70 to 110%, and the limit of detection (LOD) for most of the tested pesticides and metabolites was below 0.01 mg/kg.

Several methods were evaluated for breaking emulsions in problematic environmental surface water samples containing 11 typical pyrethroid and phenylpyrazole pesticides extracted by liquid–liquid extraction (LLE) using methylene chloride and using GC–ECD analysis (Wu, Lu, Wilson, et al., 2010). The average recoveries of spiked pyrethroid and phenylpyrazole pesticides were 88 to 123% for samples with moderate emulsions and 93 to 117% for water samples with severe emulsions. Lu et al. (2010) developed a protocol for the simultaneous quantification of the six major constituents of the pyrethrin (pyrethrin I and II, jasmolin I and II, and cinerin I and II) in tea. The method used acetonitrile extraction, with multilayer solid phase extraction cartridge clean–up, followed by sample analysis by ultra–high performance (UHP) LC–MS/MS. Lee et al. (2010) developed a method for the simultaneous determination of phoxim and its photo–transformation product, O,O–diethyl alpha–cyanobenzylideneamino–thiophosphonate (DCTP), in chicken and quail eggs by blending eggs with anhydrous magnesium sulfate for sample pretreatment, extracting with acetonitrile, and purifying the extracts with SPE silica gel tubes deactivated with trimethylamine for analyses via a reversed phase–liquid chromatography–tandem mass spectrometry (RP–LC–MS/MS) in positive–ion electrospray ionization (ESI) mode. At the levels of fortification tested (0.02, 0.05, and 0.125 mg/kg), the recoveries were from 81.3% to 93.6% for phoxim and 83.3% to 90.1% for DCTP, and the matrix effect was <2%.

A multi–residue method for the determination of organochlorine pesticides in fish feed samples was developed using liquid–liquid extraction on diatomaceous earth cartridges with *n*–hexane/acetonitrile (80/20, v/v) followed by solid phase extraction (SPE) with silica gel–SCX cartridge, before analyzing the residues by gas chromatography–triple quadrupole tandem spectrometry (GC–MS/MS) (Nardelli et al., 2010). Instrumental limits of detection ranged from 0.01 to 0.11 µg/L, and the limits of quantification were in the range of 0.02 to 0.35 µg/L, with recoveries between 92 and 116% of spiked extracted fat samples at 100 µg/kg. He et al. (2010) reported using the ionic liquid 1,3–dibutylimidazolium hexafluorophosphate for extracting and preconcentrating the organophosphorus pesticides fenitrothion, parathion, fenthion, and phoxim from water and fruit samples by dispersive liquid–liquid microextraction and analyzing the pesticides using high–performance liquid chromatography. An extraction recovery of over 75% and enrichment factor of over 300–fold were obtained, and linearity was observed in the range of 5–1000 µg/L, and limits of detection were from 0.01 to 0.05 µg/L for the four analytes.

Guo et al. (2010) developed a procedure for extracting benzimidazole fungicides and related transformation products in various raw agricultural commodities for direct analysis by liquid chromatography–tandem mass spectrometry (LC–MS/MS) by using a conservative homogenizing extraction and multifunction adsorption cleanup (CHEMAC) method involving extraction with acetate–buffered acetonitrile at low–temperature and sequential rapid solid–phase dispersive cleanup with a ternary sorbent mixture. By using CHEMAC, extraction recoveries were improved for several previously problematic fungicides. Rashid et al. (2010) developed a procedure to analyze nineteen organochlorine pesticides which involved extraction of hydrated soil samples using acetonitrile and cleanup by liquid–liquid partition into *n*–hexane for analyses using gas chromatography–tandem mass spectrometry (GC–MS/MS). The method was validated by analysis of soil samples spiked at levels between 1 and 200 µg/kg, with recoveries generally between 70 and 100% and limits of detection of 0.3 µg/kg and limits of quantification of 1.0 µg/kg.

A rapid method for the simultaneous analyses of pesticide residues in edible seaweed was developed by Garcia–Rodriguez, Carro–Diaz, Lorenzo–Ferreira, et al. (2010) for three pyrethroids, a carbamate, and two organophosphorus pesticides. The procedure used pressurized liquid extraction (PLE) with integrated clean–up followed by gas chromatography coupled to tandem mass spectrometry and yielded detection limits of 0.3 pg/g for chlorpyrifos–ethyl, 3.0 pg/g for carbaryl, and 23.1 pg/g for deltamethrin and recoveries from 82 to 108%. Liu, Dong, et al. (2010) developed a GC–MS procedure for simultaneously analyzing kresoxim–methyl and boscalid in fruit, vegetable, and soil matrices using a one–step liquid–liquid extraction process with acetone and dichloromethane. The limits of detection were 0.006 mg/kg for kresoxim–methyl and 0.015 mg/kg for boscalid, and the limits of quantification were 0.02 and 0.05 mg/kg for kresoxim–methyl and boscalid, respectively.

Nguyen et al. (2010) reported on the analyses of 95 pesticides in soybean oil using liquid–liquid extraction and centrifugation, freezing, and dispersive solid phase extraction followed by gas chromatography with mass spectrometric detection. The recoveries using this method ranged from 80 to 114%. Guan et al. (2010) analyzed organochlorine and organophosphate pesticides and fungicides in fruits and vegetables using disposable pipette extraction (DPX) followed by gas chromatography–mass spectrometry–selective ion monitoring (GC/MS–SIM). The limits of detection were found to be lower than 0.1 µg/mL for all targeted pesticides in carrot and orange sample matrices, with recoveries between 72 and 116% for nonpolar and slightly polar pesticides but poor recoveries for polar pesticides.

A multiresidue method to extract and determine 42 pesticides in various tea products was developed and validated by Kanrar et al. (2010) for analyses using liquid chromatography–tandem mass spectrometry (LC–MS/MS). The recoveries of all the pesticides were between 70% and 120%. Brutti et al. (2010) developed a pressurized liquid extraction and LC–MS/MS method for determining nine benzoylureas in fruit, vegetable, cereals, and animal products. Limits of quantitation were between 0.002 and 0.01 mg/kg, and the recoveries ranged from 58 to 97%.

Wang, Li, et al. (2010) reported a method for simultaneously detecting 21 organochlorine (OC) and 6 pyrethroid pesticides in hot pot condiments. The pesticides were extracted with acetonitrile, and then purified with gel permeation chromatography and Florisil solid phase extraction before analysis by GC. Zhuang et al. (2010) presented a method for detecting 33 different pesticides, a variety of organophosphorus (OP), numerous OCs, and pyrethroid, from honeysuckle by extraction with a 1:1 volume mixture of ethyl–acetate/cyclohexane, followed by gel–permeation chromatography, and then GC–MS analysis.

Hu et al. (2010) reported a method for the simultaneous analysis of two herbicides (metribuzin and quizalofop–*p*–ethyl) in potato and soil samples. The herbicides were extracted with an acetone/methanol/water mixture, followed with solid phase extraction with a Florisil cartridge to remove co–extractives before analysis by GC. Mariani et al. (2010) used GC–MS with nitrogen–phosphorous detection (NPD) to identify 26 different OP pesticides in cereals. They extracted the pesticides from samples using petroleum ether, acetone, or dichloromethane. The extracts were cleaned up with one of the following three methods: a multicartridge system, a low–temperature lipid precipitation, or with acid and neutral alumina. Fenoll et al. (2010) were able to detect 54 different pesticides in peppers, tomatoes, oranges, and lemons obtained from orchards and greenhouses in the Murcia, Spain region. They used an acetonitrile extraction, followed by liquid–liquid partition aided by a sodium chloride “salting out” process. The samples were then analyzed by LC–MS/MS.

Li et al. (2010) studied using desorption corona beam ionization (DCBI) in combination with poly(dimethylsiloxane) (PDMS) substrate sampling for microextraction to analyze for pesticides in water samples by mass spectrometry. The device was shown to be able to identify the five pesticides acephate, isoprocarb, dimethoate, dichlorvos, and dicofol in water with detection limits of 1 μg/L. Vinas et al. (2010) developed two procedures based on ultrasound–assisted emulsification and single–drop liquid–liquid microextraction to analyze for seven strobilurin and six oxazole fungicides in fruits and juice samples, with both extraction techniques coupled to gas chromatography with mass spectrometry in the selected ion monitoring mode, GC–MS(SIM). Enrichment factors were found to be between 140–1140 for one technique and 80–1600 for the other.

Chen, Zou, et al. (2010) prepared a grapheme–based solid–phase microextraction (SPME) fiber and studied its use for extracting six pyrethroid pesticides for gas chromatographic determination. The limit of detection (LOD) was found to range from 3.69 to 69.4 ng/L, and the recoveries were 83–110% with spikings of 1 μg/L of the pesticides. A vortex–assisted liquid–liquid microextraction (VALLME) method coupled with gas chromatography micro electron–capture detector (GC–microECD) was developed to analyze for pesticide residues in water samples (Jia, Zhu, et al., 2010). Under optimal conditions, enrichment factors in the range of 835–1115 and limits of detection below 0.010 µg/L were achieved for the analysis of target pesticides in water. Wu, Lu, Chen, et al. (2010) used single–walled carbon nanotubes (SWCNTs) as the stationary phase of solid–phase microextraction (SPME) fibers to extract 13 pesticides in tea samples for gas chromatography–mass spectrometry analyses. Under optimized conditions, the developed method showed linearity from 0.125 to 25 ng/mL, and the limits of detections were 0.027 to 0.23 ng/mL.

A solid–phase microextraction (SPME)/gas chromatography (GC)–mass spectrometry (MS) multiresidue analytical method for 16 pesticides with disparate physicochemical properties was developed by Passeport et al. (2010). The limits of quantification for this method were in the 0.05 to 0.5 µg/L range, and the recoveries varied from 75% to 140% for pesticides such as diphenyl ether, triazine, ureas, acetamides, benzofuran, thiocarbamate, pyridine carboxamides, chloronitrile, piperedine, and azoles,. Faraji and Helalizadeh (2010) developed a dispersive liquid–liquid microextraction (DLLME) method coupled with gas chromatography–electron capture detection (GC–ECD) to analyze for 14 organochlorine pesticides and pesticide degradation products (hexachlorocyclohexanes (α–HCH, β–HCH and δ–HCH), Lindane (γ–HCH), Aldrin, Dieldrin, Endrin, Heptachlor, Heptachlor epoxide, α –Chlordane, β–Chlordane and *p,p′*–DDT, *p,p′*–DDD, *p,p′*–DDE) in river water samples. In this method 13.5 µL of carbon disulfide as the extraction solvent 0.50 mL acetone as the disperser solvent, and the detection limit of the method ranged from 0.05 to 0.001 µg/L.

Du, Yan, et al. (2010) developed a new method for the simultaneous determination of cypermethrin and permethrin residues in pear juice by ultrasound–assisted dispersive liquid–liquid microextraction (UA–DLLME) and gas chromatography–flame ionization detection (GC–FID) by injecting 3.5 mL of methanol as the dispersant and 30 µL of C_2Cl_4 as the extractant into 5.0 mL of pear juice sample and then emulsifying by ultrasound for 2.0 min to form a cloudy solution. The enrichment factors for cypermethrin and permethrin were 344 and 351–fold, respectively, and good linearity was observed from 0.009–1.52 µg/g, and the limits of detection were 3.1 and 2.2 µg/kg for cypermethrin and permethrin, respectively. A stir bar microextraction (SBSE) procedure was developed to analyze seven strobilurin fungicides in fruit samples using liquid chromatography (LC) and diode array detection (DAD) (Campillo et al., 2010). The detection limits were from 0.3 to 2 ng/g for trifloxystrobin and metominostrobin, respectively, and recoveries of 80–105% from spiked samples were achieved.

A method was developed for the simultaneous analysis of 14 pesticide residues, clofentezine, carbo–furan, diazinon, methyl parathion, malathion, fenthion, thiabendazole, imazalil, bifenthrin, permethrin, prochloraz, pyraclostrobin. Difenoconazole, and azoxystrobin, in mango fruit using solid–phase micro extraction (SPME) coupled to gas chromatography–mass spectrometry (GC–MS) (Menezes et al., 2010). The best results were obtained using polyacrylate fiber and direct immersion mode at 50

°C for 30 min, along with stirring at 250 rpm and desorption for 5 min at 280 °C, with average recoveries from 71.6 to 117.5% and detection and quantification limits from 1.0 to 3.3 µg/kg and 3.33 to 33.33 µg/kg, respectively. Liquid–phase microextraction (LPME) based on polypropylene hollow fibers was evaluated for extracting the fungicides thiabendazole (TBZ), carbendazim (CBZ), and imazalil (IMZ) from orange juices using 500 µL of 840 mM NaOH added to 3 mL of orange juice for pH adjustment (Barahona et al., 2010). Analytes were extracted through a supported liquid membrane of 2–octanone into 20 µL of an aqueous solution of 10 mM HCl inside the lumen of the hollow fiber. Limits of detection less than 0.1 µg/L were achieved.

Padilla–Sanchez et al. (2010) developed and validated a method for analyzing 13 phenolic compounds, including chlorophenols, alkylphenol,, nitrophenols, and cresols in agricultural soils using a *quick, easy, cheap, effective, rugged, and safe* (QuEChERS)–based procedure. The developed procedure yielded average extraction recoveries in the range 69–103% (10 µg/kg), 65–98% (50 µg/kg), 76–112% (100 µg/kg) and 76–112% (300 µg/kg). Lewis and Khan (2010) published an article on the determination of pesticides in grapes using QuEChERS, a dispersive solid–phase extraction (SPE) technique for extracting pesticides. The publication demonstrated that QuEChERS was an effective sample preparation method for the GC–MS analysis of pesticides in grapes.

Ferrer, Aguera, et al. (2010) compared the performance of three multiresidue methods, the QuEChERS method, the mini–Luke method, and the ethyl acetate method for analyzing amitraz and its metabolites N–2,4–dimethylphenyl–N–methylformamidine and 2,4–dimethylformanilide, in fruits using HPLC–electrospray ionization–MS/MS. The study showed that the pesticide residue is underestimated if only parent compounds are targeted. Lehotay, Son, et al. (2010) compared different versions of a QuEChERS sample preparation approach for analyzing pesticide residues in fruits and vegetables by concurrent use of gas and liquid chromatography (GC and LC) coupled to mass spectrometry (MS) for detection. Analyses using each method were tested from 50 to 1000 ng/g in apple–blueberry sauce, peas, and limes spiked with 32 representative pesticides, and an average recovery of 98% was obtained.

Pinto et al. (2010) developed a simplified version of the QuEChERS method to extract chlorinated pollutant compounds from soil samples using ethyl acetate liquid extraction of the soil sample followed by the addition of anhydrous $MgSO_4$ and analyses by gas chromatography/electron capture detection (ECD) without any other sample pretreatment. Recoveries between 62 and 93% were achieved using the method. Asensio–Ramos et al. (2010) developed a modified version of the QuEChERS method to analyze for a group of ten organophosphorus pesticides (ethoprofos, dimethoate, diazinon, malaoxon, chlorpyrifos–methyl, fenitrothion, malathion, chlorpyrifos, fenamiphos, and phosmet) and one thiadiazine pesticide (buprofezin) in three different types of soils (forestal, ornamental and agricultural). Recoveries between 45 and 96% were obtained for all the pesticides and soils, except for malathion and malaoxon in forestal and ornamental soils, which did not yield quantitative extraction.

Research on the extraction and analyses of the pesticides thiosultap sodium, thiocyclam, and nereistoxin in pepper was conducted by Ferrer, Mezcua, et al. (2010), who used acetonitrile extraction in an acidic medium with ultrasonic extraction followed by a cleanup step with anhydrous magnesium sulfate. The analyses were performed on a Linear Ion Trap Quadrupole LC–MS/MS. Recovery studies carried out on peppers spiked at 20 and 200 µg/kg yielded recoveries from 58–87%, while the instrument detection limits were in the low µg/kg range. An ultrasound–assisted emulsification microextraction (USAEME) method was developed by Su and Jen (2010) to extract organophosphorus pesticides (OPPs) from water samples for analyses by gas chromatography (GC) using a microelectron capture detector (µECD). Detections were linear in the concentration of 0.01 to 1 µg/L with detection limits between 1ng/L and 2ng/L for the OPPs, and the enrichment factors ranged from 330 to 699.

Several reviews on extraction methods for pesticides and herbicides were published in 2010. The determination of organophosphorus and organonitrogen pesticides in water samples was analyzed by Tankiewicz et al. (2010). The techniques most commonly used for extracting and determining these pesticides were examined, and the analytical problems arising at each stage of the analytical procedures were discussed. Sharma et al. (2010) published a review of methods for analyzing organophosphate pesticide residues. The review focused on various extraction and quantification procedures used throughout the world to analyze for organophosphorus residues in vegetables and fruits. Herrera–Herrera et al. (2010) published a review article on applications of dispersive liquid–liquid microextraction (DLLME) for extracting organic analytes such as pesticides, pharmaceuticals, polychlorinated biphenyls, and polybrominated diphenyl ethers. The review focused on developments that represented improvements in the technique and on the most challenging applications of DLLME. Capriotti et al. (2010) reviewed the use of matrix solid–phase dispersion (MSPD). A general overview and an update of the last developments in MSPD are provided in the review.

Chromatographic or Mass Spectrometric Techniques

The separation of mixtures containing pesticides or herbicides for analyses has been commonly accomplished using a variety of chromatographic methods. In general, these techniques fall into two basic categories: gas chromatography (GC) and liquid chromatography (LC). Frequently, the GC or LC method is coupled with mass spectrometry (MS) techniques to assist in the identification and quantification of a variety of environmental pesticides and herbicides. In this section, the recent advances in both chromatographic and mass spectrometric techniques are reviewed.

Niell et al. (2010) developed two sample preparation methods for the multiresidue analyses of herbicides in loam soil using HPLC with a diode array detector. Residues of metsulfuron methyl, bensulfuron ethyl, bentazone, cyhalofop butyl, pyrazosulfuron ethyl, propanil, and clomazone were analyzed simultaneously using this method, which represents the first time that the simultaneous detection of these analytes has been achieved. Nakata et al. (2010) used GC high resolution (HR) MS to quantify benzotriazole UV stabilizers (BUVs) found in porpoise blubber. The bioconcentrations of certain BUVs were an order of magnitude higher in porpoises than in small fish obtained from the same waters. There results suggested that BUVs were bioaccumulating within the aquatic food chain.

Wong et al. (2010) developed a method using isotope dilution GC–MS to detect three matrix–bound pyrethroids (cypermethrin, permethrin, and bifenthrin) in apple juice. The method identified the analytes by comparing two dominant ion fragments to those in a series of calibration standards. Quantification of the analytes was obtained by comparing the natural and isotope analogues between the samples and the standards. Since a labeled form of bifenthrin was not accessible, it was compared to the labeled *cis*–permerthrin as its standard. Vujic et al. (2010) developed a GC protocol for detecting an OC pesticide, lindane (γ–HCH), and its impurity α–hexachlorocyclohexane (HCH) in bulk and pharmaceutical products such as shampoo, gels, and emulsions.

Nevado et al. (2010) also presented a GC–MS method for detecting a variety of pesticides in fish oil: various HCHs, dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichloroethane (DDE) and numerous polychlorinated biphenyls (PCBs). The method involved a single step silica column clean–up and fractionation, followed by GC–MS analysis. The effectiveness of the method was demonstrated by detecting DDT and PCBs in Spanish fish oil pills sold as omega–3 fatty acid dietary supplements. Portoles et al. (2010) presented GC–MS analysis results involving a new atmospheric pressure ionization source. Protonated molecular ion (MH^+) formation was facilitated in this new ion source by the addition of water as a modifier, and the source was used in the screening of around 100 pesticides from various foods. Toledano et al. (2010) showed that large volume injections (5mL) of aqueous samples containing OP, OC, and triazine pesticides could be analyzed using capillary GC–MS when using a Through Oven Transfer Adsorption Desorption (TOTAD) interface. The TOTAD interface eliminated the majority of the water, so that the pesticides could be detected by MS.

Ennaceur and Driss (2010) reported the GC analysis of blood serum obtained from delivering mothers in Tunisia. They measured for PCBs, dichlorodiphenyltrichloroethane and its metabolites, hexachlorobenzene, and hexachlorocyclohexane isomers. The results showed that the mothers tested from Tunisia exhibited lower than normal concentrations of these species when compared with mothers from other nations. Budnik et al. (2010) reported on a new thermal desorption GC–MS analysis of atmospheric samples obtained from freight containers arriving at Hamburg, Germany and Rotterdam, Netherlands over a 2.5 year period. The containers had been fumigated with methyl and ethyl halide gases, and the authors contend that their protocol will serve as a valuable strategy to measure atmospheric exposure of these highly toxic chemicals.

Dasgupta et al. (2010) reported a 2–D GC–time of flight (TOF) MS method for the simultaneous analysis of 160 pesticides, 12 PCBs, 12 polyaromatic hydrocarbons (PAHs), and bisphenol A from grape and wine samples. Greater than 85% of all of analytes matched the mass–spectral data in the National Institute of Standards and Technology data base. Norli et al. (2010) detailed the results of their evaluation of Agilent Technologies' GC–MS deconvolution reporting software (DRS) as a screening tool for detecting numerous pesticides extracted via the Mini–Luke method. A blind study of 52 samples revealed the presence of 158 different pesticides. The screening rate of GC–MS/DRS was about 5–10 times faster than a comparable GC–NPD/electron capture detector (ECD) method. Yoshida and Tobino (2010) used both GC–MS and LC–MS/MS methods to detect a total of 68 aqueous pesticides that are highly toxic toward fish. The analysis times for these procedures were approximately one hour, and the authors expect that these methods could assist in understanding the causes of different fish mortality incidents.

Advances in urine analysis to detect organophosphate (OP) pesticides have been made the past year. For example, Odetokun et al. (2010) developed a safe, quick, and sensitive protocol for analyzing urine samples for six common dialkylphosphate (DAP) metabolites of OP insecticides. The authors used weak anion exchange cartridges to perform a solid phase extraction, and then analyzed the extracts using HPLC. Wu, Liu, et al. (2010) also developed a method for analyzing urine containing OP pesticide metabolites with GC–Flame Photometric Detection (FPD) after derivatization with pentafluorobenzyl bromide (PFBBr). The samples were obtained from workers from Eastern China; 30 who experienced occupational exposure to OP pesticides and a control of 60 workers that were not exposed at their work. OP pesticides were detected in both groups, although at higher levels for those that were occupationally exposed. Ueyama et al. (2010) employed a method of liquid–liquid extraction, derivatization with PFBBr, and clean–up for GC–MS analysis; however, they also implemented an improved pre–derivatization dehydration step for DAPs that resulted in improved analyses when compared with previous methods.

Park, et al. (2010) presented a LC–MS/MS protocol for detecting 44 pesticide residues in watermelon samples. The author used a Zorbax XDB–C–18 LC column to separate the pesticides and positive ion electrospray ionization (ESI) MS to identify the pesticides. The method was applied to watermelons obtained from 8 local markets in South Korea, but no pesticide residues were observed. Caporossi et al. (2010) used LC with diode array detection (DAD) to detect oxamyl residues on greenhouse employees. Samples were obtained using a wipe test from greenhouse and office workers. The results obtained from LC–DAD were repeated with LC–MS/MS to show that the UV detector overestimated the amount of oxamyl residue.

Dong et al. (2010) reported a method to quantify the herbicide 4–chloro–2–methlphoxyacetic acid (MCPA) in wheat products and soil. MCPA was first extracted with acetonitrile and then a portion was taken for cleanup with primary secondary amine before analysis by UHP LC–MS/MS. UHP LC was also investigated by Grimalt et al. (2010) with three different MS detectors: triple quadrupole (QqQ), TOF, and quadrupole time of flight (QTOF). The researchers analyzed 11 pesticide residues from fruit and vegetable samples. In all three cases, the limits of quantification set for baby–food analysis was met with the TOF and QTOF being the most sensitive. The investigators also demonstrated that UHP LC–TOF MS to be an effective analytical tool for detecting over 400 pesticides in 50 commercial fruits and vegetables.

Pico et al. (2010) reported the UHP LC–QTOF–MS analysis of 40 apple and pear samples intended for human consumption. The authors detected imazalil/ethoxyquin and thiabendazole/diphenylamine. They were also able to identify several metabolites of both ethoxyquin and diphenylamine. The metabolite concentrations were several times more concentrated than their parent compounds. Benvenuto et al. (2010) developed a qualitative and quantitative analysis of 11 compounds from the European Water Policy list of pollutants commonly found in surface and wastewater. Their method involved using UHP LC–MS/MS for analysis with isotope–labeled internal standards. It detected the following compounds plus their transformation products: atrazine, simazine, terbuthylazine, terbumeton, and terbutryn.

Qian et al. (2010) developed a method for measuring chlorantraniliprole and flubendiamide residues obtained from a variety of vegetables and grains. The authors extracted the residues with acetonitrile, and then homogenized the extracts with magnesium sulfate and sodium chloride. After centrifugation, the top layer was analyzed with HPLC–MS/MS.

Fan et al. (2010) reported a procedure for the determination of cyromazine and dicyclanil residues from greasy wool using HPLC–MS/MS. The samples were extracted with 1% trichloroacetic acid under ultrasonication. The extracts were cleaned up with a MCX SPE column, and then analyzed by HPLC and confirmed by HPLC–MS/MS. Kruve et al. (2010a) studied the matrix effects of HPLC ESI–MS analysis of pesticide residues in vegetables and fruits. From their work, the authors constructed a matrix effect graph for estimating the resulting uncertainty in the analysis of thiabendazole, aldicarb, imazalil, and thiocarb. The graph was used to identify vegetables and fruits with extreme matrix effects. Kruve et al. (2010b) also examined various optimization protocols for LC ESI with quadrupole ion trap MS analysis of the following three pesticides: thiabendazole, aldicarb, and imazalil. The authors concluded that the factors that most enhanced sensitivity was capillary voltage, direct current of the first octopole, trap drive, and the second lens, while maxima intensity was best obtained by using software–based optimizations.

Advances in chiral analytical methods for the resolution of pesticides have been reported by several groups. John et al. (2010) developed an enantioselective assay for analyzing a racemic mixture of S– and R–hyoscyamine, a class of toxic tropane alkaloids. The protocol used LC–MS/MS and resulted in precise, accurate, and selective determination of total and individual hyoscyamine levels in vitro and in vivo. The protocol was shown to be immune to poisoning from OP compounds and thiono–pesticides; however it was affected by the presence of oxon–pesticides. Cheng et al. (2010) also reported new method for the resolution and determination of enantiomers of indoxacarb residues obtained from cucumber, tomato, apple, pear, and soil samples. Their approach used HPLC with a Chiralpak (R) AS–H column and an n–hexane–ethanol mixture as the mobile phase.

Garrison et al. (2010) reported the use of chiral GC columns for the analysis of both chiral and non–chiral OC pesticides hypothesized to be causing reproductive disorders in American alligators residing in Lake Apopka, Florida. The authors sampled the livers of 12 alligators inhabiting Lake Apopka and 10 alligators from a control lake and analyzed the samples using GC–MS. The most abundant pesticide detected from Lake Apopka alligators were *p, p'*–DDE, *trans*– and *cis*– nonachlor, and *p,p'*–DDD, plus five chiral OC pesticides. The samples obtained from alligators in the control lake exhibited only five chiral OC pesticides and those were at significantly lower concentrations than those in Lake Apopka. Desorption atmospheric pressure photoionization (DAPPI) MS was shown by Luosujarvi et al. (2010) to detect a series of analytes commonly found in environmental and food samples: five polyaromatic hydrocarbons (PAHs), a N–PAH, a brominated flame retardant, and nine pesticides. The samples were analyzed using sprays of acetone and toluene in the positive ion mode and anisole in the negative ion mode. Molecular ions as well as fragmentation and substitution products were detected.

A nanoliter level sampling technique was developed by Wang, Yang, et al. (2010) to analyze untreated commercial juices. The authors used desorption atmospheric pressure chemical ionization (DAPCI) MS and a homemade nanoliter sampler to detect trace levels of dimethoate, an OP pesticide, in the juices. Barker et al. (2010) used single particle aerosol mass spectrometry (SPAMS) to detect compounds from the following four classes of pesticides: OPs, pyrethroids, chloronicotinyls, and carbamates. Analytical standards as well as commercial products containing each compound were analyzed and the characteristic MS peaks from the standards were used to confirm the presence of the pesticides in the commercial products. The authors suggested that their method would be useful in detecting pesticides in various environmental and agricultural situations.

Numerous examples of MS analysis of food samples prepared by QuEChERS sample preparation method were reported. For example, Koesukwiwat et al. (2010) presented a test for 150 different pesticides from a variety of vegetables and fruit samples prepared with QuEChERS and analyzed with low pressure GC–TOF MS. Chung and Chan (2010) used a QuEChERS method and LC–MS/MS to detect 98 different OP pesticides and carbamates in a variety of food samples. Also, Steiniger et al. (2010) used QuEChERS methods and GC–MS/MS to analyze for pesticides in green tea, and Lacina et al. (2010) used ultra–high performance (UHP) LC–TOF MS to detect 212 pesticides from four different plant matrices prepared by a QuEChERS method. UHP LC MS was used by Chen, Dong, et al. (2010) to detect mancozeb residues, a germicide found in apple samples. The mancozeb residue was first methylated with dimethyl sulfate and then extracted with QuEChERS before analysis by UHP LC MS where the precursor and two daughter ions were detected.

In addition, Lehotay, Mastovska, et al. (2010) applied three different calibration methods for high pressure LC–MS/MS in the determination of 14 different pesticides in samples prepared by the QuEChERS method. Meanwhile, Gilbert–Lopez, Garcia–Reyes, Lozana, et al. (2010) compared the QuEChERS method and a matrix solid phase dispersion (MSPD) method for analyzing pesticides in olives with LC–MS/MS. Wiley et al. (2010) used low–temperature plasma (LTP) MS to measure various agrochemicals in fruit peels and fruit and vegetable extracts prepared with a QuEChERS protocol showing that LTP–MS can be successfully applied to detect agrochemicals in market–purchased produce.

Three separate reports were released this past year on the use of capillary electrophoresis (CE) for pesticide analysis in different food products. Sun, Chen, et al. (2010) reported a new method to detect acetamiprid, imidacloprid and thiamethoxam in vegetables. Their assay used CE in an electrokinetic injection mode with sweeping during the analysis. The effects of buffer concentration, pH, sodium dodecyl sulfate concentration, separating voltage and sample size were reported. Gu and Shamsi (2010) used capillary electrochromatography (CEC) atmospheric pressure photoionization (APPI) MS to separate and identify N–methylcarbamate pesticides in apple juice, while Kumar et al. (2010) reviewed recent advances in CE–UV and CE–MS methods for analysis of pesticide residue in various food products.

Electrochemical Techniques

A capillary electrochromatography with indirect amperometric detection technique was developed for analyzing certain non–electroactive organophosphorus pesticides (Wu, Wu, et al., 2010). The detection limits achieved using this technique ranged from 0.008 to 0.2 mg/kg. Sarigul et al. (2010) investigated the adsorptive stripping voltammetric behavior of halosulfuron methyl. At the optimized conditions, the peak current was linear to halosulfuron methyl concentrations from 4.1 to 50.0 µg/mL, and the limit of detection and limit of quantification were 1.23 and 4.10 µg/mL, respectively. Mazzotta et al. (2010) developed an electrochemical sensor based on a molecularly imprinted polymer (MIP) of a cobalt porphyrin complex for detecting the organochlorine pesticide 4–(2,4–dichlorophenoxy)butyric acid. The amperometric response showed linearity between 200 µM and 2 mM and sensitivity to 5.89 µA/mM.

Sanchez–Bayo et al. (2010) analyzed the herbicides amitrole, glyphosate, and its metabolite, aminomethyl–phosphonic acid (AMPA), in water samples using high–performance liquid chromatography and an electrochemical detector. Limits of detection of 0.3 µg/mL for glyphosate, 0.05 µg/mL for AMPA, and 0.03 µg/mL for amitrole were achieved. A method for the electrochemical analysis of methylparathion (MP) by a gemini surfactant–intercalated clay–modified electrode was reported by Tcheumi et al. (2010). After optimization, a linear calibration curve for MP was obtained from 4×10^{-7} to 8.5×10^{-6} M with a detection limit of 7×10^{-8} M. A new enzyme electrode for analyzing organophosphorus pesticides based on a polypyrrole (PPy) composite and the enzyme acetylcholinesterase (AChE) was developed by Sulak (2010). Under optimal conditions, the enzyme electrode could measure 0.2 µM paraoxon with a sensitivity of 1.77 nA/µM.

A sampler system to analyze for paraquat by square–wave voltammetry with a hanging mercury drop electrode using a sequential injection method for automation was developed (dos Santos et al., 2010). In this method, 212 analyses per hour can be made, and the limits of detection and quantification were 2.0 and 7.0 µg/L, respectively. Xie, Li, et al. (2010) developed a surface molecular self–assembly strategy for molecular imprinting of chlorpyrifos (CPF) in electropolymerized polyaminothiophenol (PATP) membranes at the surface of a gold nanoparticle–modified glassy carbon electrode. The electrochemical detection of CPF was achieved with electrochemical selectivity for CPF over other pesticides.

Spectrophotometric Techniques

A new method for analyzing carbaryl pesticide in waters was developed based on using a long pathlength (100 cm) flow cell to increase the sensitivity in detecting indophenol which is formed from the reaction between carbaryl and *p*–aminophenol (Melchert et al., 2010). Linearity was observed from 5 to 200 µg/L, and the detection limit was 1.7 µg/L.

Chemiluminescence and Fluorescence Methods

A molecularly imprinted polymer (MIP) was synthesized which selectively absorbs triazophos (TAP) from solution and enhances the chemiluminescence (CL) of luminol–H_2O_2 for use in the on–line enrichment and detection of TAP (Xie, Zhou, et al., 2010). The observed change in CL intensity was linear from 1.0 µM to 4.0 nM, and the detection limit was 2.5 nM. Chouhan et al. (2010) developed a CL–based technique for the detection of methyl parathion (MP). The technique showed linearity from 0.001 to 500 ng/mL mL with a limit of quantification of 0.005 ng/mL and a limit of detection of 0.001 ng/mL. The increase in fluorescence intensity due to supramolecular–complex formation between carbendazim and cucurbit[7]uril was studied and used to develop a selective method for benzoimidazole–type pesticide determination in fruit samples (del Pozo et al., 2010). Under optimal conditions, the developed method allowed for the detection of carbendazim at 5.0×10^{-9} M, and the limit of detection and limit of quantification for real samples were 0.10 and 0.52 mg/kg, respectively.

A method with high sensitivity and good selectivity was developed to analyze for picloram using immunorecognition and fluorescence (Zhang, Zeng, et al., 2010). The detection limit of the method was 1.0×10^{-5} ng/mL for picloram, and picloram concentrations in actual wastewater samples were accurately measured by the method and verified by HPLC. Zou et al. (2010) developed a portable fluorescent biosensor for monitoring trichloropyridinol (TCP), which is a metabolite and biomarker of exposure to the organophosphorus pesticide chlorpyrifos. The biosensor can detect 1.0 ng/mL TCP standard analyte in 15 min and detected TCP spiked in rat plasma with an average recovery of 102.0%.

Sensors

Meng et al. (2010) investigated the ultrasensitive detection Aldrin and DDT using a sub–10 nm SnO_2/ multi–walled carbon nanotube–based gas sensor. The sensing material showed improved sensitivity compared to conventional SnO_2 sensors and forms stable attachments between the carbon nanotubes and the SnO_2 nanoparticles. An acetylcholinesterase biosensor with dual–layer membranes of chitosan and Prussian Blue on glassy carbon electrode was synthesized and tested (Sun and Wang, 2010). The detection limits of the sensor were found to be 2.5 ng/L for dichlorvos, 15 ng/L for omethoate, 5 ng/L for trichlorfon, and 10 ng/L for phoxim. An acetylcholinesterase biosensor based on immobilized enzyme membrane–electrode for detecting organophosphate pesticides was also developed by Sun, Wang, and Zhao (2010). The biosensor registered response currents that were proportional to the concentration of dichlorvos from 5 to 50 µg/L, and the detection limit was 3 µg/L.

Firdoz et al. (2010) synthesized an amperometric biosensor based on single–walled carbon nanotubes and acetylcholine esterase for the detection of carbaryl. The biosensor achieved a detection limit of pg/L for the analysis of carbaryl pesticides in water. Arduini et al. (2010) reviewed research from the previous nine years on biosensors for insecticides and nerve agents that are based on cholinesterase inhibition. In the review article, the authors emphasized the optimization of irreversible inhibition by OP and carbamic insecticides to improve biosensors.

Du, Wang, et al. (2010) reported on an amperometric biosensor for OP pesticides based on immobilization of acetylcholinesterase (AChE) on multiwall carbon nanotubes (MWCNTs)–beta–cyclodextrin (beta–CD) composite modified glassy carbon electrode. The inhibition of AChE by dimethoate was proportional to its concentration from 0.01 to10.00 µM, and a detection limit of 2 nM was determined.

Flampouri et al. (2010) developed and validated a cellular biosensor for detecting pesticide residues in tomatoes. It was shown that the sensor could detect concentrations of pesticides down to 3 nM. An amperometric biosensor for the detection of paraoxon was designed by using zinc oxide as a matrix for immobilizing acetylcholinesterase (Sinha et al., 2010). The biosensor detected paraoxon in the range of 0.035 to 1.38 mg/L and could also detect other acetylchlolinesterase inhibiting organophosphorus pesticides.

Biochemical Assays and Immunoassays

Two enzyme–linked immunosorbent assays were developed to analyze for the fungicide picoxystrobin in beers (Esteve–Turrillas et al., 2010). Limits of detection below 0.2 µg/L were determined, and recoveries between 79 to 122% were achieved. Huang et al. (2010) developed both dip–and–dry and flow–through methods using a piezoelectric immunosensor to detect triazophos. The immunosensors could detect triazophos within 40 to 60 min, and the limits of detection were 0.04 mg/L for the dip–and–dry method and 1 mg/L for the flow–through method.

A qualitative and semi–quantitative immunochromatography using a colloidal gold–antibody probe was developed for detecting chlorpyrifos–methyl in water in under 10 min (Hua et al., 2010). The limit of detection for chlorpyrifos–methyl was 0.6 μg/mL, and the recoveries obtained were 102.5 to 107.6%. A high–throughput suspension array technology for the simultaneous detection of multiple pesticides was developed by Liu, Su, et al. (2010). The developed method could simultaneously determine seven analytes within 1–2 h.

Sun, Dong, et al. (2010) developed an enzyme linked immunosorbent assay for the simultaneous analysis of carbaryl and metolcarb in agricultural products. The limits of detection of carbaryl and metolcarb were 0.15 μg/L and 1.2 μg/L, respectively, and recoveries of spiked samples were greater than 70%. An immunostrip test for thiabenzadole detection was developed by Blazkova et al. (2010) that could be used either qualitatively by visual detection or quantitatively by scanning densitometry. When optimized, the test could be completed in 10 min, and the visual detection limit was 0.25 ng/mL.

Ramon–Azcon et al. (2010) applied particle manipulation with negative dielectrophoresis to develop an immunosensing system for detecting atrazine and bromopropylate with limits of detection of 4 and 1.5 μg/L, respectively. The assay was accelerated by particle manipulation with negative dielectrophoresis and could be completed within 5 min. Senyuva and Gilbert (2010) wrote a critical review of the use of immunoaffinity columns for sample clean–up in the food analyses. The use of immunoaffinity columns for sample clean–up in the areas such as analyzing pesticide residues were discussed, and extraction methods and achievable recoveries were compared.

Miscellaneous Techniques

Pohanka et al. (2010) developed a colorimetric dipstick to assay nerve agents and OP pesticides based on the hydrolysis of acetylcholine into acetic acid and choline by acetylcholinesterase and using pH paper to detect the acidification from the buildup of acetic acid. The presence of an OP pesticide or a nerve agent resulted in irreversible inhibition of acetylcholinesterase and no change in the pH, and limits of detection were 5 x 10^{-8} M for paraoxon–ethyl and 5 x 10^{-9} M for sarin and VX.

Several review articles on different aspects of analytical methods for pesticides and herbicides were published in 2010. For example, the analysis of bee and bee products for pesticides was reviewed by Barganska and Namiesnik (2010). The review discussed the latest developments of methods to determine the levels of many different pesticides. Koskela (2010) published a review on the use of nuclear magnetic resonance (NMR) spectroscopy for analyzing OP pesticides and chemical warfare agents. Using NMR spectroscopy to analyze food and environmental samples and characterize OP pesticide degradation in the environment is also discussed. Souza et al. (2010) reviewed different analytical methods for studying pesticide degradation in water and environmental samples. The critical role of gas and liquid chromatography in combination with mass spectrometry is emphasized.

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