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# Chromatographic Determination of Pesticides in Foods and Food Products

**Tibor Cserhati and Maria Szogyi***

*Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, Budapest, Hungary*

**Abstract**

The newest results in the chromatographic analysis of pesrticides present in foods and food products are collected and the results are critically evaluated. Examples for the employment of preconcentration and prepurification technologies, gas chromatography using ECD, NPD, MS and MS/MS detection methods, liquid chromatographic methodologies such as thin-layer chromatography, high performance liquid chromatographic methods as well as electrically driven systems are presented. The advantages and disasdvantages of the various chromatographic technologies are shortly discussed and the efficacies of the methodologies are compared. Pesticides included in the review are insecticides, herbicides, acaricides, organophosphorous and organochlorine compounds The application of the chromatographic methods for the determination of pesticides in a wide variety foods and food products is discussed in detail.

**Keywords:** Pesticides; Gas chromatography; Liquid chromatography; Electrically driven systems

**Abbrevations:** ACN: Acetonitrile; ASE: Accelerated Solvent Extraction; BCF: Bioconcentration Factor; BUVs: Benzotriazole UV stabilizers; DAD: Diode Array Detector; DDTs: Dichlorodiphenyltrichloroethanes; DPX: Disposable Pipette Extraction; DSPE: Disperase Solid Phase Extraction; ECD: Electron Capture Detector; ESI: Electrospray Ionizaton; FID: Flame Ionization Detection; FL: Fluorescence Detection; GC: Gas Chromatography; GCB: Graphitized Carbon Black; GPC: Gel Permeation Chromatography; HCB: Hexachlorobenzene; HCHs: Hexachlorocyclohexanes; HILIC: Hydrophilic Interaction Liquid Chromatography; HPLC: High Performance Liquid Chromatography; HS-SPME: Headspace Solid-Phase Microextraction; i.d : Internal Diameter; LLE: Liquid-Liquid Extraction; LOD: Limit of Detection; LOQ: Limit of Quantitation; LTP: Low Temperature Purification; MRL: Maximum Residue Level; MRM: Multiple Reaction Monitoring; NCI: Negative Chemical Ionization; NP: Nonylphenol isomers; NPD: Nitrogen-Phodphorous Detector; OCPs: Organochlorine Pesticides; ODS: Octadecyl Silica; OPP: Organophosphorous Pesticides; PA: Polyacrylate; PBDC: Propylenebisdithiocarbamate; PCI: Positive Chemical Ionization; PDMS: Poly(dimethylsiloxane); PHI: Pre-Harvest Interval; PSA: Primary Secondary Amine; PTU: Propylenethiourea, 4-methylimidazoline-2-thione; QuEChERS: Quick, Easy, Cheap, Effective, Rugged and Safe; RP-HPLC: Reversed-Phase High Performance Liquid Chromatography; RSD: Relative Standard Deviation; SPE: Solid-Phase Extration; SPME: Solid-Phase Microextraction; SRM: Selected Reaction Monitoring; SWCNTs: Single-Walled Carbon Nanotubes; TCHMs: Traditional Chinese Herbal Medicines; TLC: Thin Layer Chromatography; UHPL: Ultra High Performance Liquid Chromatography; UA-DLLME: Ultrasonic-Assisted Dispersive Liquid-Liquid Microextraction; UPLC-MS-MS: Ultra-Performance Liquid Chromatography Combined with Tandem Spectrometry

## Introduction

Chromatography has been developed as a powerful separation technique suitable for the quantitative analysis of compounds with very similar chemical structure. Various chromatographic techniques such as Gas Chromatography (GC), liquid chromatographic procedures (Thin Layer Chromatography, TLC, High Performance Liquid Chromatography, HPLC, Ultra Performane Liqid Chromatography, UPLC) and electrically driven systems found application in biology, medicine, chemical technology and in the analysis of natural products contributing to the isolation and identification of new molecules. These methodologies were successfully employed in analytical quality control and environmental sciences. Moreover, chromatography has been applied for the residue analysis of xenobiotics in air, ground and surface water, sludge, soil matrices, foods and food products and in human and veterinary health care.

The objectives of the recent review are the compilation and concise evaluation of the newest results obtained in the chromatographic analysis of pesticides in foods and food products, the brief enumeration of the techniques employed and the critical evaluation of the results.

## Gas Chromatography

Gas chromatographic (GC) methods are suitable for the separation and quantitative determination of compounds which are volatile or semi-volatile and thermally stable at the temperature of the measurement. Unfortunately, the overwhelming majority of compounds are not volatile, consequently, the application of GC methods is limited.

### Fruits, juices and vegetables

Gas chromatography coupled with mass spectrometry was employed for the determination of the residues of kresoxim-methyl and boscalid in fruits, vegetables and soil.

Both fungicides were successfully applied to control a considerable number of diseases caused by *Venturia inaequalis* [1], powdery

***Corresponding author:** Maria Szogyi, Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, Budapest, P.O.B. 17, H-1525 Hungary, E-mail: szogyim@t-online.hu

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mildew [2], *Sclerotinia blight* [3] and *Botritis cinerea* [4]. It was also established that the mixture of these two insecticides show good capacity to control straw-berry grey mould disease [5]. Because of their con- siderable commercial importance these insecticides have been many times investigated by various chromatographic methods such as GC combined with electron capture detector (ECD) [6], liquid chromatography tandem mass spectrometry [7], HPLC-DAD [8], GC-ECD and GC-MS [9], on column liquid-liquid extraction following liquid chromatography-tandem mass spectrometry [10], etc. Samples of 10 g (melon, cucumber, tomato, apple, pear, pepper, eggplant and soil) were extracted with 50 mL of acetone or methanol, filtered, mixed with 50 mL of water, 50 g NaCl and 40 ml of *n*-hexane or dichloromethane or their mixture of 1:1, v/v ratio. The organic phase was dried with anhydrous $Na_2SO_4$. The extraction step was repeated three times, the combined extracts were evaporated to dryness and redissolved in 1 mL of *n*-hexane. Separations were performed on a capillary column (30 m x 0.25 mm i.d.; film thickness, 0.25 µm). The injector temperature was 250°C. Temperature gradient initiated at 50°C (1 min), ramped to 150°C at 30°C/min), then to 250°C at 10°C/min, final hold, 10 min. MS conditions were: electron ionization (EI) mode at 70 eV. The temperature of the ion source was 250°C, the temperature of the transfer line was 25°C. Helium was the carrier gas. Analytes were identified at full scan (m/z 50-400) Quantitative measurements were carried out at selected ion monitoring (SIM). The LODs were 0.006 and 0.015 mg/kg for kresoxim-methyl and boscalid and the LOQs were 0.02 and 0.05 mg/kg, respectively. The RSD of the intra- and inter-day precision were lower than 13.8 and 14.5%, recoveries varied between 77.1 and 98.7% for kresoxim-methyl and 72.8-105.1% for boscalid. It was established that the good validation parameters of the method advocate its application for the analysis of kresoxim and boscalid in mixed formulations [11]. Five pesticide residues (gamma-HCH, clorothalonil, fenithrothion, chlorpyrifos and pocymidone) were determined in tomatoes. Analytes were extracted with ACN and a mixture of dichloromethane:petroleum ether (1:1, v/v). The suspension was centrifuged and the supernatant was filtered through $Na_2SO_4$. Analytes were separated and quntified by GC-ECD. The recoveries varied between 70% and 110%. LOD and LOQ were 0.04-0.06 ng/kg and 0.014-0.02 mg/kg [12].

HS-SPME followed by GC-ECD was employed for the determination of organophosphorous (diazinon, malathion, chloropyrofos, quinalphos, profenofos) and organochlorine (chorothanoil, alpha-endosulfane, beta-endosulfan) pesticide residues in vegetable (cucumber) and fruit (strawberry) samples. GC-ECD measurements indicated that the washing with acetic acid, sodium carbonate, sodium chloride and tap water decreased the amount of pesticides, acetic acid being the most efficace. It was established that the efficacy of washing is correlated with the water solubility and, and vapour pressure of the washing solution [13].

A fast, low-pressure GC-time-of-flight MS (LC-GC/TOFMS) was developed and applied for the identification and quantitative determination of of 150 pesticides in tomato, strawberry, potato, orangeand lettuce samples. Dispersive solid-phase extraction (d-SPE) and disposable pipette extraction (DPX) were employed for clean-up. The stationary phase for clean-up consisted of 150 mg $MgSO_4$, 50 mg primary secondary amine (PSA) 50 mg ODS, and 7.5 mg graphitized carbon black (GCB) per mL extract was applied. It was established that the method is suitable for the analysis of a wide rage of pesticide residues [14]. Insecticides (ethoprophos, diazinon, chlorpyrophos-methyl, fenitrothion, malathion, chlorpyrophos, and fenamiphos) were extracted from banana leaves by applying a modified QuEChERS technique. Insecticides were separated and quantitated by GC followed with nitrogen phophorous detection (NPD). Recovery values of the method ranged from 89% to 104% with a RSD values of lower than 9.1%. LOD varied between 0.002 – 0.064 mg/kg.

The confirmation of the identity of target compounds was performed by GC tandem mass spectrometry [15]. A hyperspectral imaging system was applied for the determination of dichlorvos residues on the surface of navel orange. It was established that the method meet the requirements of online fast nondestructive detection [16].

GC-MS technology was employed for the determination of organophosphate and pyrethroid insecticide residues in fruits, vegetables and fruit juices. The investigation revealed that 14% or 5% of the food samples consumed by children contain pesticide residues [17]. Seven strobilurin and six oxazole fungicides were determined in fruits and juices. Target compounds were preconcentrated by two procedures: ultrasound-assisted emulsification liquid-liquid microextraction and single-drop liquid-liquid microextraction. Analytes were separated by GC-MS used in the SIM mode. Enrichment factors were between 140-1140 and 80-1600 for the first and second extraction technique, respectively. It was established that LODs were below the MRLs t by the European Comission [18]. Organochlorine pesticides residues have also been determined in commercial fruits and baby food samples. Target compounds were extracted and prepurified by QuEChERS (quick, easy, cheap, effective rugged and safe) technique followed by GC-MS analysis. The recovery of the method varied from 70% to 120%, RSD was lower than 17% in the majority of cases. LOQ varied between 0.001-0.013 mg/kg [19]. Ultrasonic-assisted dispersive liquid-liquid microextraction (UA-DLLME) followed with GC-FID (flame ionization detection) was applied for the analysis of cypermethrin ans permethrin residues in pear juice. The enrichment factors for cypermethrin ans permethrin residues were 344 and 351, respectively. LODs ranged from 3.1 to 2.2 µg/kg, recoveries were 92.1 – 107.1%. The intra-day and inter-day RSDs were less than 4.0%. The method was successfully employed for the measurement of these pesticide residues in pear juices [20]. Azole fungicide residues were determined in grape and wine using offline disperse solid phase extraction (DSPE) coupled with GC-positive chemical ionization mass spectrometry (GC-MS/PCI). Target comounds were extracted from grape and wine with ACN and ethyl acetate, respectively. Recoveries of the method varied between 71.2% and 102.2%, RSDs were less than 10.6%, LODs were lower than 10 µg/kg. It was stated that the method can be applied for the determination of azole fungicide residues in grape and wine [21]. A GC-MS procedure was applied for the detection and quantitation of 23 pesticides in 160 different vegetables marketed in Saudi Arabia. The pesticide residues were over the MRL in 53 samples. The most frequently occurring pesticides were carbaryl, biphenyl and carbofuran. Cabbage, carrot, green peper, cucumber and egg-plant were the most polluted vegetables. The monitoring of the environmental pollutants in vegetable has been advocated [22]. GC technology has been employed for the analysis of organophosphate pesticide residues in broccoliheads. The measurements indicated that malathion, diazinon, chlorphenvinphos, fenthion and ethion are the most freqeunt pollutants [23].

A novel technique was developed for the simultaneous determination of 346 multiresidue pesticides in grapes. The method applied PSA matrix solid phase dispersion followed with GC-MS-SIM. Samples of

15 g were mixed with 6 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride, then extracted with 15 mL of ACN and cleaned up with 0.3 g dispersive PSA. Four injections from one sample was enough to separate all the 352 pesticides. LOD varied between 0.0017-0.2667 mg/kg, recoveries ranged from 45% to 136%. RSD value of 95% of pesticides was equal or lower than 20%. The identification of pesticides was performed by the retention time, molecule ions, fragment ions, and the abundance ratio of the selected ions. It was stated that the technique suitable for the determination of 346 pesticide residues ing rapes [24]. Pesticide residues in grapes were detrmined by MSPD combined with GC-MS. Target compounds included in the measurements were vinclozolin, dichlofluanid, penconazol, captan, quinoxyfen, fluquinconazol, boscalid and pyraclostrobin. GC-MS analyses were performed in the SIM mode. The optimal MSDP method consisted of 0.5 g of grapes, 1.0 g of silica as clean-up sorbent, 1.5 g of ODS as bonded phase and 10 mL of dichloromethane/ethyl acetate (1:1, v/v) as eluting solvent. Recoveries were over 80% except for captan. LOQs ranges from 3.4 to 8.7 μg/kg being lower than MRLS [25]. Organochlorine pesticide residues have also been determined in honey from various geographic regions. The concentration of hexachlorocyclohexanes (HCHs), dichlorodiphenyltrichloroethanes (DDTs), chlordane and hexachlorobenzene (HCB) in honey was measured after accelerated solvent extraction with GC-ion trap mass spectrometry. The measurements indicted that the amount of organochlorine pesticide residues ranged 0.21-8.70. 0.10-4.35, 0.02-3.75 and 0-1.16 ng/g for HCHs, DDTs, chlordanes and HCB, respectively. It was established that honey samples from developed countries show considerable differences in the concentration of organochlorine pesticide residues [26]. Chlorpyrifos, lamda-cyhalothrin, cypermethrin and deltamethrin residues were measured in honeys. The method employed liquid-liquid extraction (LLE) and low temperature purification (LTP). LTP cleanup used ACN-ethyl acetate mixture (6.5 mL:1.5 mL) as extracting agent. Final cleanup was performed with 2 g of florisil. Analytes were separated and quantitated by GC-ECD. LOD and LOQ were below 0.016 and 0.032 μg/g, respectively. The presence of target compounds was confirmed by GC-MS [27]. GC-ECD procedure was applied for the determination of 24 organochlorine pesticide residues in 109 honey samples. It was established that aldrin, cis-chlordane, trans-chlordane, oxy-chlordane, 2,4'-DDE and 4,4'-DDE was present in each samples. The concentration of pesticide residues was over the MRL values in 55 samples. A strict control of organochlorine pesticide residues in honey was proposed [28]. The acaricide residues in commercial beeswax were determined by GC using ECD, NPD and MS detection. The target compounds measured were chlorfenvinphos, fluvalinate, amitraz, bromopropylate, acrinathrin, flumethrin, coumaphos, chlorpyrophos, chlordimeform, endosulfan and malathion. Recoveries varied between 86-108%. LOQ ranged from 0.10 to 0.30 mg/kg for ECD and NPD detection and from12 to 85 μg/kg for MS detection [29]. Herbicides metribuzin and quizalofop-p-ethyl have been etermined in potato and soil by GC-ECD. Herbicides were extracted with acetone and methanol-water, the liquid phase was cleaned by SPE using florisil cartridges (500 mg, 3 mL). Target compounds were eluted with petroleum ether-acetic ether (9:1, v/v, 5 mL) and petroleum ether-acetic ether (8:2, v/v, 2 mL). LOQ was 0.01 mg/kg, recoveries varied between 72.9 – 109.5%. RSD ranged 0.7 – 9.2%. Identity of analytes was confirmed by GC-MS [30].

## Miscellaneous foods and food products

Beside of fruits, juices and vegetables various GC technologies have been frequently applied for the pesticide residue analysis of a wide variety of other foods and food products. Thus, the occurrence of pollutants in tea has been vigorously investigated. Single-walled carbon nanotube (SWCNTs) was employed as stationary phase for SPME preconcentration of pesticide residues in tea samples. Analytes were separated and quantified by GC-MS. LODs were 0.027-0.23 ng/mL. RSDs of measurements with single fiber, fiber-to-fiber, and day-to-day were 2.3-13.0, 8.2 – 14.6, and 4.1 – 12.5%, respectively. Recoveries were between 75.1 and 118.4%. The efficacy of SWCNTs was higher than the commerial poly (dimethylsiloxane) (PDMS) and plyacrylate (PA) fibers. The results indicated the presence of chlorfenapyr and lambda-cyhalothrin in some tea samples. It was stated that the method is simple, efficient and can be applied for the determination of pesticide residues in complicated accompanying matrices [31]. QeEChERS method followed by ion-trap GC/MS/MS was applied for the analysis of 22 pesticides in tea samples. Teas were homogenized with water and the analyted were extracted with ACN containing 1% of acetic acid. Initial cleanup was performed with dispersive dSPE, followed with solvent exchange and dSPE again. The recoveries ranged from 78 to 115% except for diazinon (130%) and malathion (122%). Average RSD was 8.7%, the LOD values except for terbufos were below the MRL limit set by EU and Japan [32]. Pyrethroid residues have also been determined in tea samples. Measurements were performed with GC and ion trap mass spectrometry [33]. Gas chromatography with micro-ECD was employed for the measurement of 21 organochlorine and 6 pyrethroid pesticides inhotpot condiment. Analytes were extracted with ACN, cleaned up with GPC, florisil SPE and PSA. LODS were 0.082-2.3 g/kg for organochlorine pesticides and 1.5-13.0 g/kg for pyrethroid pesticides. Recoveries ranged from 70.1% to 116.0% RSD being 0.2%-6.1%. It was stated that the method is rapid, sensitive and reliable, and can be applied for the simultaneous detection of multi-residues of pesticides in hotpot condiment [34]. Pesticides have also been investigated in milk and dairy poducts. The influence of lactic acid fermentation and heat treatment of bovine milk on the decomposition of seven organophosphorous pesticides (denthion, dimethoate, malathion, methyl-parathion, monocrotophos, phorate and trichlorphon) was studied in detail. Measurements were carried out by GC-MS. The results indicated that fermentation process and heat treatment accelerate the decomposition of pesticides [35]. GC-NPD was applied for the separation and quantitation of seven organophophorous pesticides (OPPs) in raw milk and infant formulas. Recoveries ranged from 62.2% to 97.25% [36]. GC-ECD technique was applied for the determination of organochlorine pesticides (DDT and derivatives, HCH, lindane, heptechlor and endosulfan) in raw and processed milk. LOD was 0.11 mg/kg. The measurements indicated that the concentration of organochlorine pesticides in the samples was below the MRL. It was further concluded from the data that the continuous monitoring and control of pesticides in milk is of paramount importance for public health [37]. GC was also applied for the determination of organochlorine pesticides OCPs in kaymak and butter marketed in Afyonkarahisar province of Turkey. It was found that the majority of samples was contaminated with organochlorine pesticide residues (672.46 ng/g .in kaymak and 308.95 ng/g in butter). It was further established that the concentration of OCPs such as beta-HCH (90.01 ng/g), aldrin (528.04 ng/g, and endrin (7.31 ng/g) in kaymak was higher than the MRL value. The amount of beta-HCH (214.18 ng/g), heptachlor (10.38 ng/g), aldrin (12.34 ng/g), dieldrin (12.69 ng/g), and endosulfan sulfate (8.08 ng/g) in butter was over the MRL. It was assumed that the consumption of contaminated products can be potential risk for public health [38]. Pyrethroid residues were also investigated in butter. Target compounds were concentrated with matrix solid phase dispersion methof followed with purification at

low temperature. Analytes were separated and quantified by GC. LOD of cypermethrin and deltamethrin were 0.082 and 0.11 μg/g, LOQs were0.28 and 0.32 μg/g. The recovery was about 90% with a RSD of less than 10% [39]. Simple and environmental friend GC-MS methodology was developed for the analysis of pesticide residues in cattle feed and soil samples. Investigation included 36 pesticides belonging to different classes. Measurements indicated that organochlorine and organophosphorous pesticides were commonly detected, while the occurence of pyrethroid and chloroacetanilide pesticides was markedly lower. It was further established that residue levels were generally below the MRL [40].

The pesticide content of various animal products has also been frequently investigated by using GC technolgies. Thus, OCPs (alfa-, beta-, gamma-HCH, heptachlor, heptachlor epoxide, aldrin, dieldrin, and eldrin) were determined in water and fishes by GC-MS. It was established that the amount of OCPs was higher in fish gonads than in the muscle tissue. Gonads of roach and bream contained mainly gamma-HCH while muscle tissue concentrated beta-HCH. The amount of OCPs in gonads varied between 0.385 - 0.554 ng/g wet weight (alpha-HCH) . 0.745 – 0.832 ng/g (gamma-HCH), 0.479 – 0.576 ng/g (dieldrin), and 0.381 – 0.684 ng/g (eldrin). Water samples taken from the Odera River contained pesticides residues in the following order: endrin, gamma-HCH, alpha-HCH, dieldrin, beta-HCH, heptachlor = aldrin, heptachlor epoxide. It was assumed that the accumulation of OCPs in fish gonads may results in decreased reproduction of fish potentially leading to their extinction [41]. OCPs were also measured in fish feed. The first step of the analysis included the liquid-liquid extraction of fat. It was carried out on diatomaceous earth cartridge using n-hexane.ACN (80:20, v/v). Further purification was achieved by SPE employing silica gel cartridge. Analytes were identified and quantificated by GC-triple quadrupole tandem spectrometry. LODs of the method varied between 0.01 – 0.11 mg/L, LOQs ranged 0.02 – 0.35 mg/L. Repeatability were 3 – 15%, and the recoveries were 92 – 116% [42]. The accumulation of 18 current use and 8 OCPs in crab embrios was monitored by GC-MS. It was found that the level of contamination of crab embrios of site specific and can be used as an indicator of ecosystem health [43]. The accumulation of benzotriazole UV stabilizers (BUVs) in the blubbers of finless porpoises (*Neophocaena phocaenoides*) was studied by GC-high resolution mass spectrometry (GC-HRMS). The mean concentrations and standard deviations of two benzotriazole UV stabilizers (BUVs) were 38 ± 28 ng/g and 19 ± 19 ng/g, respectively. The bioconcentration factor (BCF) was 33 300 similar to that of persistent HCH. The strict monitoring of BUVBs was proposed to understand more in detail their potential risk to wildlife and human [44]. Microwave-assisted extraction followed with SPE and coupled to large-volume injection GC-MS/MS was employed for the determination of 17 pesticides in wild and aquaculture edible seaweeds. The optimal microwave conditions were 125°C and 12 min, extraction solvent being 24 mL of n-hexane/ethyl acetate (80:20, v/v). SPE purification was performed on graphitized carbon and Florisil supports. Recoveries were near to 100%, RSD being lower than 13%. LODs varied between 0.3 – 23.1 pg/g, LOQs ranged from 2.3 to 76.9 pg/g below MRL. The method using microwave-assisted extraction was proposed for the routine analysis of pesticides in aquaculture and wild seaweed samples [45]. Pesticide residues were determined in four processed food (frozen Chinese dumpling, eel kabayaki, corned beef and retort curry) employing ion trap GC/MS technique. Target compounds were extracted with ethyl acetate-cyclohexane (1:1, v/v) in the presence of anhydrous sodium sulfate. The solvent was evaporated and the residue was redissolved in n-hexane. Lipids were removed from the solution by ACN-n-hexane partitioning. Pesticide residues in the ACN fraction was preconcentrated on ODS and graphite carbon/PSA silica mini-cartridge columns. LOQs were below 0.01 μg/g, recoveries ranged from 70% to 120%, and RSD was 15%. It was assumed that the technique can be applied for the multi-residue analysis of pesticides in processed foods prepared employing livestock and seafoods as main raw materials [46]. A GC method was applied for the detemination of carbofuran using broiler chickens as experimental model. The amount of carbofuran was measured in blood, muscle, liver and stomach contents. It was stated that carbofuran in the edible tissue of poisoned birds may result in the secondary poisoning of predators and may be harmful for humans [47].

## Simultaneous Application of GC and HPLC Technologies

The molecutar base of separation is markedly different in GC and HPLC. As the separation in GC is mainly governed by the volatility of the analytes; the acidity or alkalinity, the adsorption capacity, the lipophilicity and polarity etc. of the target compounds play a considerable role in their HPLC retention behavior. Because of their diverse separation parameters the simultaneous application of GC and HPLC methodologies may facilitate and substantiate the analysis of target compounds present in complicated accompanying matrices. The application of GC and HPLC for the analysis of pesticides in bee and bee products has been previously reviewed [48]. The extraction by QueEChERS technology and the use of subsequent chromatographic methodology for the analysis of pesticide residues present in food matrices has also been rviewed [49]. The analytical methods employed for the determination of organophosphorous pesticide (OPP) residues in frutis and vegetables have also been compiled. The various extraction techniques and the chromatographic separation procedures were discussed and the results were compared [50]. The concentration of pesticide residues present in crushed grapes, cake, most, lees and wine was monitored by multiresidual GC-MS (71 pesticides) and multiresidual LC-MS-MS technology (45 pesticides). It was established that the amount of boscalid, cyprodynil, dimethomorph, fengexamid, metalaxyl and procymidone was the highest during the vinification process, their concentration was 0.01-0.02, 0.04, 0.01-0.08, 0.12-0.13, 0.09-0.11 and 0.07-0.13 mg/L, respectively [51]. A new technique has been developed for the determination of pestcide residues on processed tea leaves. The procedure includes extraction and dispersive d-SPE extraction followed with GC/NCI/tandem MS and UHPLC-MS/MS. LOD was below 1 μg/kg for GC and 10 μg/kg for UHPLC. The majority of recovery was over 70%. GC-NCI dected endosulfan sulfate and kelthane, while UHPLC detected imidacloprid and acetamiprid in the samples. It was established that the price of tea and the pesticide residue level did not correlated [52].

## Liquid Chromatographic Technologies

Oppositely to GC, liquid chromatographic (LC) methods can be used for the separation and quantitative determination of non-volatile target compounds in a very large range of molecular mass and surface acidity or alkalinity.Moreover, LC technologies can be easily applied for the analysis of both water-soluble and water-insoluble analytes. The analysis of 280 pesticides in food samples was performed using QuEChERS extracts followed by liquid chromatography tandem mass spectrometry. Separation were carried out on an ODS stationary phase, analytes were identified with ion ratio calculation and mass spectral library searching [53].

## Reversed-phase high perfomance liquid chromatography

The overwhelming majority of LC analyses are performed in reversed-phase high performance liquid chromatographic (RPHPLC) separation mode. The advantages of this technique are the high variety of both stationary and mobile phases resulting in widely different separation characteristics, the reduced consumption of organic solvent, the increased safety of laboratory staff, and the lower environmental loading.

**Fruits, juices and vegetables:** A QuEChERS extraction method combined with LC-MS/MS measurements was applied for the determination of the residues of 150 pesticides in grapes. Pesticides were extracted with ACN. Phase separation was achieved by shaking the sample with a salt mixture containing magnesium sulfate, sodium chloride, disodium hydrogen citrate sesquihydrate, and trisodium citrate dihydrate. The mixture was centrifuged and an aliquot of the clear supernatant was dried with magnesium sulfate. After a new centrifugation step and aliquot of the supernatant was evaporated, redissolved in methanol-water buffer solution and used for LC analysis. Quantitation and identification were performed by using atmospheric pressure electrospray positive ionization in multiple reaction monitoring (MRM) mode. Recoveries ranged from 70% to 110%, the RSD varied between 1% and 25% [54].

Some organophosphorous pesticides (fenitrothion, parathion, fenthion and foxim were determined in pear and water. A new ionic liquid, 1,3-dibutylimidazolium hexafluorophosphate was employed for the dispersive liquid-liquid microextraction of target compounds. Separation was carried out by HPLC. Recovery was over 75%, enrichment factor over 300-fold. LODs were 0.01-0.05 mg/L. RSD ranged from 1.3-2.7, 1.4-1.9, and 1.1-1.7, respectively [55]. A LC-MS/MS technique was applied for the analysis of 69 pesticides in fruits and vegetables (zucchini, melon, cucumber, watermelon, tomato, garlic eggplant, lettuce and pepper). Target compounds were extracted by QuEChERS. MS measurements were carried out in MRM mode. Two selected reaction monitoring (SRM) were employed for the quantitation and identification of target compounds in one run. Recoveries range from70% to 120% RSD being lower than 20%. It was stated that the method is rapid, simple and sensitive and can be employed in routine analytical laboratories [56]. A QuEChERS –HPLC-UV method was applied for the investigation of the dissipation of carbaryl on greenhouse cucumbers during the pre-harvest interval (PHI). It was established that the dissipation depends on the initial dose and follows first order kinetics. A waiting period of more than 14 days was proposed for the safe consumption of cucumber [57]. It was further established that washing, peeling and refrigeration storge markedly decreased the amount of carbaryl in cucumber samples [58]. The concentration of propylenethiourea (PTU), 4-methylimidazoline-2-thione, the main degradation product of propylenebisdithiocarbamate (PBDC) was measured in tomatoes. Analytes were extracted with dispersive solid-phase clean up followed by high performance hydrophilic interaction liquid chromatography atmospheric pressure electrospray ionization mass spectrometry (HILIC-ESI-MS-MS) [59]. LC-MS/MS technology was employed for the simultaneous determination of 54 pesticides. Samples of pepper, tomato, orange and lemon were extracted with ACN and liquid-liquid partitioned by salting out procedure applying NaCl. GC measurements were performed with the triple quadrupole in SRM mode. Recoveries ranged from 65.5% to 114.5%. RSD being 2.3 -8.3%. LOD varied between 0.03-14.9 μg/kg [60]. An enzyme-linked immunosorbent assay was developed and applied for the determination of fenhexamide residues in grape must, kiwifruit and strawberry. The results were compared with those obtained by HPLC. It was established that excellent correlation can be found between the data determined by HPLC and enzyme-linked immunosorbent assay [61]. An LC-MS/MS method was developed for the first screen of 300 pesticides in fruits and vegetables with a commercially enhanced product ion method. The probably positive extracts were further investigated using LC-MS/MS optimized for 55 pesticides. It was found that no false-negative and no-false postive were encountered during the analysis [62]. A chromatographic analitical method was developed for the determination of N-methyl carbamate pesticide residues in foods. The method was simple and rapid and makes possible the simultaneous determination of pesticide residues in foods.

It was stated that the method ensure safe products for consumers [63]. Benzimidazole fungicides and their degradation products were separated and identified in various raw agricultural commodities. Pretreatment of samples included the direct extraction and multifunctional adsorption cleanup (CHEMAC) involving salting out-partitioning /extraction with acetate-buffered ACN at low-temperature followed with sequential rapid solid-phasedispersive cleanup with a ternary sorbent mixture. Recoveries were between 70% and 92% , Intr-day and inter-day reproducibility ranged from 15% and 20% [64].

A comprehensive and sensitive multi-residue LC-MS/MS method was developed and applied for the separation and quantitation of 73 pesticides and related products in edible oil, meat, egg, cheese, chocolate, coffe, rice, tree nuts, citric fruits, vegetables etc. Cleanup was performed by QuEChERS methodology LC-MS/MS was operated in MRM mode. Recoveries varied between 70-120% RSD being lower than 20%. It was established that the method can be successfully applied for the analysis of organophosphorous pesticide and carbamate residues in various foods and food products [65]. Fungicides (metalexyl-M, azoxystrobin myclobutanil, fusilazole, penconazole, tebuconazole, propiconazole, diniconazole, and difenoconazole) were determined in wine. Target compounds were purified employing mixed-mode anion exchange and reversed-phase SPE cartridges. LC-MS/MS detection of analytes was performed applying atmospheric pressure electrospray ionization.

Optimal extraction condtions were: 10 mL of wine was diluted with 1 L of ultrapure water and passed through the mixed-mode SPE cartridge at a flow-rate of 5 mL/min. Cartridge was washed with with 5 mL of aqueius $NH_4OH$ (5% w/v). Target compounds were removed with 1 mL of methanol. RSDs were below 11%, LOQs ranged 0.01-0.79 ng/mL being below the MRLs for fungicides in grapes and wine [66]. Permethrin (3-phenoxybenzyl (1RS)-cis-trans-3-(2,2-dichlorovinyl)2,2-dimethyl-cyclopropanecarboxylate residues have also been investigated in wines using an isocratic HPLC technology. RP-HPLC measurements were performed on an ODS column (25 cm x 4 mm i.d.; particle size, μm). Isocratic mobile phase consisted of ACN-water (70:30, v/v). The flow rate was 2 mL/min, tartget compounds were detected at 215 nm and column was thermostated at 25°C. Recoveried ranged from 93.95% to 86.58%. RSD varied between 0.89-3.69%. The method was proposed for the routine analysis of permethrin in wines because of the simple sample preparation technique, acceptable analysis time, and low cost.

An octadecylsilica (ODS) column was employed for the determination of the residues of three acidic herbicides (2-4-dichlorophenoxyacetic acid, 2,4-D, Dicamba, 4-chlorophenoxyacetic acid, 4-CPA) in food crops. Because of their water solubility they can pollute soils,

groundwater and air [67] causing serious environmental pollution [68] and organ damage in humans [69]. A considerable number of extraction procedures were developed and applied for the preconcentration and prepurification of acidic herbicides. Thus, the application of stir-bar sorptive extraction-liquid desorption [70], solid-phase microextraction (SPME) [71] and solid-phase extraction (SPE)-solid phase derivatization have been reported [72]. Liquid-liquid extraction (LLE) has been recently employed for the analysis of various target compounds such as Chinese cabbage *(Brassica rapa* spp. pekinensis), apple (*Malus pumila* Mill), pepper (*Capsicum annuum* L.), brown rice (*Oryza sativa* L.), and soybean (*Glycine max* L.) Target analytes were extracted from the samples of 2,4-D, Dicamba and 4-CPA by homogenization 25 g of samples with 100 mL of acetone. The suspension was filtered and evaporated to 20 – 25 mL than it was mixed with 15 g of sodiumchloride dissolved in 100 mL of deionized water. The aqueous phase was washed twice with 50 mL of n-hexane. The aqueous phase was acidified with 10 mL of 10% sulfuric acid. The acidic solution was extracted twice with 50 mL of dichloromethane. Anhydrous sodium sulfate was added to the organic phase. The solvent was evaporated to dryness, the residue was redissolved in 50% acetone and used for GC measurements. Because of their elevated fat content the extraction of brown rice and soybean was slightly modified. The powdered samples (25 g each) were moistened with 25 mL of deionized water for 30 min, then homogenized with 100 mL of 5 M HCl:ACN (10:90, v/v). The suspension was filtered, the liquid phase was evaporated to 20-25 mL and mixed with 10 g of NaCl The liquid phase was alkalinized and parted rwice with 50 mL of n-hexane. The aqueous fraction was acidified with sulfuric acid, then extracted twice with 50 mL of dichloromethane and dryied with anhydrous sodium sulfate. The extract was evaporated to dryness and dissolved in 10 mL of water:ACN (1:1, v/v). Cleanup step used a glass column (600 mm x 12 mm) filled with a slurry of 10 g of activated silica in dichloromethane. The slurry contained 2 g of anhydrous sodium sulfate. The extract was loaded on the column and washed with 50 mL of acetone-dichloromethane (1:1, v/v) followed by 50 mL (40 mL for brown rice and soybean samples) methanol-dichloromethaned methanol-dichloromethane (20:80, v/v). Target compounds were removed from the column by 80 mL of (100 mL for brown rice and soybean) methanol-dichloromethane (30:70, v/v). HPLC-UV measurements were performed on an ODS column (250 x 4.6 mm i.d., partivle size, 5 μm). Isocratic mobile phase for the separation of 2,4-D, Dicamba and 4-CPA consisted of aqueous 0.02% phosphoric acid (pH 2.5)-ACN (60:40, v/v). Chinese cabbage, apple and pepper were analysed by the following mobile phase: aqueous solution of 0.1% formic acid (pH 3.0)-ACN (60-40, v/v). The flow rate was 0.8 mL/min, analytes were detected at 230 nm. LC-MS/MS measurements were carried on an $NH_2$ column at 35°C. The components of the mobile phase were ACN-methanol (3:7, v/v; solution A) and aqueous solution of 0.1% formic acid (solution B). The flow rate was 0.5 mL/min. It was established that average recoveries for Chinese cabbage ranged from 94.30 to 102.63%, for apple it was 94.67-108.47%; for pepper 97.52-102.27%. The recoveries were similar in the Scase of analytes with elevated fat content: brown-rice 76.19-101.90%; soybean 74.60-107.39%. It was concluded from the results that the method is suitable for the determination of 2,4-D, dicamba and 4-CPA in Chinese cabbage, apple, pepper, brown rice and soybean. The method was proposed for the routine analyses of these pesticide residues [73].

Insecticides are extensively applied to protect agricultural poducts against harmful insects, to improve quality and to enhance yield [74,75]. Acetampidrid, a neonicotinoid insecticide has also been frequently used in the up-to-date agricultural practice. Various chromatographic technologies have been frequently employed for the determination of the amount of acetamiprid residues in foods and food products such as in tea [76,77]), and bovine milk [78]. Both GC [5] and HPLC [79] techniques were employed for the separation and quantitative determination of the target compound. The behaviour of acetamiprid residues in zuccini has been investigated in detail. Samples of chopped zucchini (20 g) were mixed with 100 mL of water-methanol (50:50, v/v), the suspension was homogenized and filtered. The filtrate was partially evaporated and extracted with n-hexane and redissolved in dichloromethane. RP-HPLC-DAD measurements were performed on an ODS column (250 mm x 4.6 mm i.d.; particle size, 5 μm). The flow rate was 1.0 mL/min. The mobile phase for zucchini and zucchini leaves were water-ACN (7:3, v/v) and water-ACN (75:25, v/v), respectively. Analyte was detected at 248 nm. LC-ESI-MS/MS analyses were carried out on an octylsilica column (150 x 4.6 mm i.d.; particle size, μm). Components of the gradient elution were methanol, 10 mM ammonium acetate solution and water. The flow rate was 0.5 mL/min the column temperature was 40°C. MS/MS conditions were: nebulizing gas and drying gas were nitrogen; ion spray voltage, 4000 V; source temperature, 350°C. LOD and LOQ values were 0.01 and 0.03 μg/g and 0.02 and 0.06 μg/g for zucchini and zucchini leaves, respectively. Recoveries varied between 85.7 – 92.2% for zucchini and 90.5 – 101.9% for zucchini leaves. RSD was lower than 12% [80].

Carbamate pesticides are frequently applied in plant protection. although, they are considered to be toxic for the environment and for human beings. A considerable number of chromatographic method was developed for the measurement of carbamate rersidues in various accompanying matrices. Because of the low amount of carbamate pesticides in the samples, preconcentration and prepurification techniques are often employed before the chromatographic separation step. Single-drop microextraction followed by GC-MS was used for the measurement of carbamate pesticides in water [81], hot water extraction coupled with LC-MS for the analysis of bovine milk [82], and SPME for the determination of pesticides in fruit juices [83]. SPE followed by LC-MS was employed for the determination of pesticide residues in water [84]. Gel-permeation chromatography (GPC) has also been employed as prepurification and preconcentration step in the analysis of animal tissues [85] and oils. Gel permeation chromatography (GPC) followed with ultra-performance liquid chromatography and tandem mass spectrometric detection (UPLC-MS-MS) has been applied for the determination of the residues of 18 carbamate pesticides in chestnut and pine nut. Target compounds were extracted from chestnut and pine nut by homogenizing 2.000 g sample with 20 mL of ACN. Liquid fraction was filtered trough a sodium sulfate bed, rehomogenized with 20 mL ACN. The combined extracts were evaporated to dryness. Mobile phase for GPC procedure was cyclohexane-ethyl acetate (1:1, v/v); the flow rate was 4.7 mL/min; injection volume 5 mL; collecting time started at 8.2 min and finished at 14.2 min. Fractions collected between 8.2 and 14.2 min were collected and evaporated to dryness. UPLC-MS-MS measurements were performed on a column of 50 mm x 2.1 mm the particle size being 1.8 μm. The flow rate was 0.3 mL/min. Analytes were separated by gradient elution started with 10% ACN and finished 100% 10 mM ammonium acetate. MS detection was carried out with an electrospray interface in the positive ionization mode. The capillary voltage was 3 kV, Source temperature and desolvation temperature were 110°C and 350°C, respectively. Nitrogen was employed as nebulizing, desolvation, and cone gas. Recoveries were

between 70.21 and 89.56%, and the RSD values ranged from 2.26 to 4.07% [86].

The endocrine-disrupting activity of nonylphenol isomers (NP), linea nonylphenol and short chain ethoxylated derivatives has been many time demonstrated. It has been established that NPs can occur in food-contact materials [87]. A considerable number of chromatographic techniques was developed for the separation and quantitative determination of endocrin-disrupting compounds in various matrices such as milk [88], eggs [89], and environmental samples [90]. Linear nonylphenol, nonylphenol isomers, short chain ethoxylated derivarives ($NPEO_1$ and $NPEO_2$) were determined in commercial powdered milk infant formula employing HPLC with fluorescence detection (FL). Analytes were determined by reconstituted milk-based method (method A) and powdered milk-based method (method B). Samples for method A were prepared by mixing 4.3 g milk powder with 30 mL of HPLC grade water. Saponification was performed by adding 6 mL of 0.4 M sodium hydroxy solution to 3 g of reconsituted milk.

NaOH was dissolved in ethanol-water (9:1, v/v). Saponification was carried out at 60°C for 30 min. The solution wasa acidified by formic acid (pH = 4). SPE was carried out in ODS cartridges conditioned with two column volume of ACN, one column volume of methanol, 2 x 1 mL of methanol and 1 x 1 mL mL of ethanol-water 9:1 v/v. Target compounds were eluted with 2 x 1 mL of methanol followed with 2 x 1 mL of ACN. Samples were concentrated to 0.5 mL, redissolved and used for HPLC-FL measurements. Method B empolyed SPE columns. Columns were immersed in ultrasonic bath the extraction time being 15 min at ambient temperature. Separations were performed on an octylsilica column (150 x 4.6 mm i.d., particle size, 3 μm). Target compounds were eluted in isocratic separation mode using mobile phase ACN-water (65:35, v/v) at the flow rate of 1 mL/min. LC-MS-MS conditions were: nitrogen was used as drying gas (9 L/min) and nebulizer gas (40 psi); capillary voltage, 400 V; flow rate 1 mL/min. Isocratic mobile phase consisted of 0.04% aqueous acetic acid solution-ACN (35:65, v/v). Desolvation temperature was 200°C. The recoveries were 96.8%, 94.0%, 92.7% and 89.2% for $NPEO_x$, $NPEO_{1+2}$, NP, and 4-*n*-NP, respectively. RSDs (%) were 8.4, 7.0, 6.7 and 8.5, respectively. LOD values (μg/g) were 0.89, 0.48, 0.51 and 0.047, respectively. It was established that the selectivity and sensitivity of the non-reconstructed milk-base method was higher than those of the other analytical procedure. It was further stated that the method can be applied by ruotine laboratories for the quality control of powdered milk infant formulas [91].

**Miscellaneous foods and food products:** A considerable number of other foods and food products was analysed by RP-HPLC technologies. Although the stationary phases used for the measurements were markedly similar the differences in preconcentration and prepurification methods, in the type of mobile phases and in detecting system is responsible for the considerable differences among the RP-HPLC methodologies.

HPLC-DAD was employed for the simultaneous determination of eight pesticides frequently applied in rice cultivation (ponoxsulam tricycazole, propanil, azoxystrobin, molinate, profoxydim, cyhalofop-butyl, deltamethrin and 3,4-dichloroaniline, the main metabolite of propanil). Extraction and cleanup of samples was achieved by solid-phase dispersion (MSDP) on neutral alumina (5 g) ACN being the elution solvent. Components of gradient elution system consisted of ACN-water starting with 30% ACN ramped to 100% ACN in 14 min at a flow-rate of 0.8 mL/min.LOD and LOQ ranged from 0.002 to 0.200 mg/kg and 0.006-0.60 mg/kg. Recoveries were 74-127% with an RSD below 12%. It was established that the method is suitable for the analysis of the majority of rice pesticides in rice grains at levels below MRLs [92]. The separation of 1o4 pesticides was achieved by LC-MS-MS. Target compounds were purified by two different methods. Method 1 used liquid-liquid extraction ACN being the extracting agent. Following step was dispersive solid-phase extraction using GCB, PSA and ODS sorbents (QuEChERS method modified for for fatty vegetables). Method 2 employed MSDP and preconcentration on Florisil stationary phase. Analytes were separated on an ODS column (50 mm x 4.6 mm i.d.; particle size, 1.8 μm). LODs were lower than 10 μg/g for 89% of analytes using both preconcentratzion methods. Recoveries ranged form 70% to 120% for QuEChERS technique and 50-70% for MSPD [93]. Simazine and terbutylazine residues were determined in olive oil. The technolgy applies liquid-liqued partitioning extraction and low temperature precipitation followed by MSPD. using aminopropyl as dispersant. Cleanup was performed with Florisil and graphitized carbon supports. (60:40, v/v). Separations were carried out by HPLC followed with UV detection. The recoveries were over 91%, LODs and LOQs 0.0127 and 0.0540 μg/g for simazine and 0.027 and 0.14 μg/g for terbutylazine, respectively [94]. Ultra-performance liquid chromatography followed with tandem mass spectrometry (UPLC-MS/MS) was employed for the analysis of pyrethrins in tea. Pyrethrins included in the investigation were pyrethrin I and II, jasmolin I and II and cinerin I and II. Analytes were extracted with ACN and the extract was passed through a multilayer solid phase extraction cartridge. MS used an electrospray ionization source in posotive mode (ESI+). Recoveries ranged from 76.15% to 101.86% the RSDs being 2.71 – 12.93%. LODs were lower than 0.009 mg/kg and LOQs did not exceed 0.03 mg/kg. It was stated that the data may help to draw up MRLs for pyrethrins in teas [95]. A LC-MS/MS technology was applied for the separation and quantitation of phoxim and its degradation product, O,O-diethyl-alpha-cyanobenzylideneamino-thiophosphonate (DCTP) in chicken and quail eggs. Eggs (1 g) were mixed with 1 g of anhydrous magnesium sulfate and extracted with ACN. The extract was passed through a SPE silica cartridge deactivated with trimethylamine. After RP-HPLC separation target compounds was detected with tandem mass spectrometry in positive-ion electrospray ionization (ESI) mode RSDs of intra- and inter-day variations varied between 2.1% - 6.7% and 2.8% and 6.4%for phoxim and DCTP, respectively. Recoveries ranged from 81.3% to 93.6% for phoxim and from 83.3% to 90.1% for DCTP [96]. Thiosultap sodium, thiocyclam, and nereistoxin were determined in pepper. The optimal extraction procedure combined ACN extraction in acidic medium with ultrasonic extraction followed with a cleanup step with anhydrous $MgSO_4$. Separation and quantitative determination of target compounds was carried out on a linear ion trap quadrupole LC-MS/MS in negative mode for thiosultap sodium and in positive mode for thiocyclam and nereistoxin. Recoveries ranged from 58% to 87%, RSDs were lower than 20%. The method has been successfully applied for the investigation of the decomposition of thiosultap sodium [97]. Pesticide present in onion were analysed by LC-ESI-MS/MS. The optimal conditions of MSPD were: 0.5 g of sample mixed with 1.0 g reused ODS; interaction time, 1 h; dispersion time, 5 min, elution solvent, ACN. Recoveries varied between 78.3 – 120.4%, RSD was lower than 20%. LOD and LOQ ranged from 0.003 to 0.03 mg/kg, and from 0.01 to 0.1 mg/kg [98]. Pesticide residues in flesh of *Cirrhinius mrigala* were determined by HPLC. The method revealed the presence of endosulfan a, p,p'-DDT, methamidophos, carbofuran, diazinon, parathion methyl, dimethoate malathion, chlorpyriphos,

cypermetryn, carbosulfan and isoproturon in the flesh of farmed fish. it was established that the amount of pesticide residues was higher in farmed than in wild species [99]. The concentration of acephate, methamidophos, and omethoate was determined in animal and fishery products, their processed foods, and honey. Samples were extracted with ethyl acetate in the presence of anhydrous $Na_2SO_4$ (diatomaceous earth in the case of honey). The extract was further purified on a PSA mini-column and the analytes were detected by LC-MS in the ESI-SIM mode. Recoveries of the method ranged from 71.4% to 98.4%. RSD of repeatability was ≤ 12.5% [100]. A multiresidue method was developed and applied for the analysis of 74 pesticides and metabolites in traditional Chinese herbal medicines (TCHMs). The amount of pesticides were assessed in Cortex Cinnamoni, Flos Carthami, Folium Gingko, Herba Pogostemonis, Radix Ginseng and Semen Gingko. Target compounds were preconcentrated with accelerated solvent extraction (ASE), further purified by gel permeation chromatography and graphitized carbon black/primary, secondary amine SPE. Pesticide residues and metabolites were separated by LC-MS using gradient elution. Recoveries varied between 70-110% RSD being below 15%. In the majority of cases the LOD was lower than 0.01 mg/kg [101]. Systemic insecticide residues (fipronil, imidacloprid and thiametoxam) and their metabolites (fipronil sulfon, fipronyl sulfide, fipronil desulfinyl, and fipronyl carboxamide) were determined in honey and pollen. Prior to the extraction the samples were centrifuged and 1 g of the lower phase was mixed with 3 mL of methanol-water (10:90, v/v). The extract was passed through an florisil cartridge, eluted with methanol and analysed by LC.MS/MS in selective reaction monitoring mode [102].

## Electrically Driven Separation Systems

Electrically driven separation sytems (capillary electrophoresis, micellar electrokinetic capillary chromatography, capillary isoelectric focusing, capillary gel electrophoresis, capillary isotachophoresis) show marked advantageous characteristics such as high separation efficiency, short analysis time, high resolution power, low consumption of samples and reagents, etc. Interestingly, the number of studies dealing with the application of electrically driven separation technologies in the analysis of pesticides in foods and food products is surpridingly low. The sample preparation methods employed for the determination of pesticides in foods have been recently reviewed [103].

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