

Simultaneous Determination of 346 Multiresidue Pesticides in Grapes by PSA-MSPD and GC-MS-SIM

YU-JING LIAN,[†] GUO-FANG PANG,^{*‡} HUAI-RUI SHU,[†] CHUN-LIN FAN,[‡]
YONG-MING LIU,[§] JIE FENG,[§] YAN-PING WU,[§] AND QIAO-YING CHANG[‡]

[†]Shandong Agricultural University, 61 Daizong Street, Taian, Shandong 271018, China,

[‡]Chinese Academy of Inspection and Quarantine, No. 3 Gaobeidian North Road, Chaoyang District, Beijing 100123, China, and [§]Qinhuangdao Entry-Exit Inspection and Quarantine Bureau, No. 1 Liupanshan Road, Qinhuangdao, Hebei 066004, China

The article demonstrates a method of simultaneous determination for 352 pesticide residues in grapes using primary–secondary amine (PSA) matrix solid phase dispersion (MSPD) cleanup and gas chromatography–mass spectrometry–selected ion monitoring (GC-MS-SIM). Grape samples (15 g) were mixed with 6 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride, and then extracted with acetonitrile (15 mL) and cleaned up with 0.3 g of dispersive PSA. The analytes were determined by GC-MS-SIM. Four injections for one sample were acquired to cover a total of 352 pesticides. The limit of detection (LOD) for the method was 0.0017–0.2667 mg kg⁻¹, depending on the nature of compounds. The linear correlation coefficient (*r*) was equal to or greater than 0.95; at low, medium, and high fortification levels, recoveries ranged from 45% to 136% for 352 pesticides, among which the recoveries between 60%–120% accounted for 97%. The pesticides for which the relative standard deviations (RSD) were equal to or below 20% accounted for 95%. A positive of nine varieties of grape samples was detected out, one of which was obtained Changli city, Hebei province, China. Pesticides were identified by the retention time, molecule ions, fragment ions, and the abundance ratio of the selected ions. The analytical method was rugged, quick, cheap and effective, and suitable for the determination of a wide scope of 346 pesticides in grapes.

KEYWORDS: Grape; pesticide residues; PSA-MSPD; GC-MS-SIM

INTRODUCTION

It is well known that pesticides make important contributions to boost modern agricultural production but that they bring potential hazards to both the environment and food safety. Many international organizations and countries are greatly concerned about pesticide residues. To protect public health and ensure food safety, pesticide maximum residue limit (MRL) detection is established and has become an international threshold of entrance in international trade. As the consumption of grapes increase, the problem of pesticide residues in grape cause great concern. Up to now, CAC prescribes the MRL in grapes for 70 pesticides (1), USA is 102 (2), EU is 436 (3), Japan is 345 (4), and China is 5 (5). Presently, with the progress of scientific technique and the public's concern on food safety, more pesticides of MRL in grapes have been prescribed, and pesticide residual level is getting stricter. Naturally, there is much research on analytical techniques of residue pesticides, especially simultaneous determination of multiresidues. It has already become a hot topic. Many studies, which can simultaneously determine over a hundred pesticide residues, have been constantly published about agricultural produce such as fruits and vegetable (6), grains and

cereals (7), animal tissues (8), aquatic products (9, 10), edible fungi (11, 12), Chinese medicinal herbs (13), teas (14), etc. The author has retrieved 12 methods about the simultaneous determination of pesticide multiresidues in grapes since 2000. The varieties of pesticide that have been determined are 11 (15), 12 (16), 13 (17), 15 (18), 17 (19), 27 (20), 38 (21), 50 (22, 23), 67 (24), 74 (25), and 171 (26). Some methods are exclusively devoted to detect residue pesticides in grapes, and others can be applied to different fruits and vegetables, including grapes. Gonzalez et al. proposed a multiresidue method of determination of 11 new fungicides in grapes. Samples were extracted with ethyl acetate/hexane (1:1, v/v) and cleaned up with graphitized carbon blacks (GCB)/PSA solid phase extraction (SPE) cartridges using acetonitrile/toluene (3:1, v/v) as eluent, and the target compound was determined by gas chromatography–ion trap mass spectrometry (GC-ITMS). LOD < 0.01 mg kg⁻¹, RSD < 16% (15). Oliva et al. and Navarro et al. described a multiresidue method for determining 12 insecticides and 17 fungicides in grapes. The matrix once extracted with an acetone/dichloromethane (1:1, v/v) mixture was filtered and concentrated. Nitrogen phosphorus detector (NPD) and electron capture detector (ECD) were used to identify and quantify the target, the findings being confirmed using a mass spectrometry detector (MSD); no cleanup was necessary for either NPD or ECD (16, 17). Fernández et al.

*Corresponding author. Tel: (86 335) 5997609. Fax: (86 335) 5997608. E-mail: qhdcqgfpan@yahoo.com.cn.

researched a multiresidue method for the determination of 13 carbamate residues in grapes by matrix solid phase dispersion–liquid chromatography mass spectrometry (MSPD-LC-MS). The efficiency of different solid phases (C18, C8 cyano, amine and phenyl) for the MSPD was compared. Mean recoveries using C8 varied from 64–106% with a RSD of 5–15% in the concentration range of 0.01–10 mg kg⁻¹. The LOD ranged from 0.001 to 0.01 mg kg⁻¹ (18). Alberro et al. developed a rapid multiresidue method for the determination of 15 herbicides in grapes. Samples were extracted and cleaned up by MSPD with Florisil, determined by GC-MS-SIM. The LOD ranged from 0.1 to 1.6 µg kg⁻¹, and recoveries ranged from 80 to 115%; RSD ≤ 10% (19). Cunha et al. used acetonitrile as extracting solvent and Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) for cleanup to analyze 27 representative pesticides in grapes. Recoveries were acceptable at three different spiking levels (from 0.04 to 2.5 µg g⁻¹), and RSD was from 3 to 21%. Limit of quantitation (LOQ) was from 0.02 to 5 µg g⁻¹ (20). Taylor et al. extracted 38 pesticides from grapes with crude extracts and no sample clean up before analysis by LC-MS-MS. Mean recoveries ranged from 63% to 96% with RSD < 20% (21). Savant et al. determined 50 pesticides in grape by GC-ITMS-multiple reaction monitoring (MRM). The samples were extracted by ethyl acetate and cleaned up by dispersive solid phase extraction with PSA (25 mg). Sample preparation was done by using a programmable temperature vaporizer (PTV) with a large volume injection (20 µL) through a multibaffled linear vessel. Recoveries ranged within 70–120% at 10, 20, and 50 ng g⁻¹ with RSD < 20% (*n* = 6) (22). Alberro et al. developed a multiresidue method for the simultaneous determination of 50 pesticides in commercial juices. The extraction procedure was carried out in C18 columns, eluted by hexane-ethyl acetate (1:1, v/v), and determined by GC-MS-SIM. Average recoveries were higher than 91% with a RSD lower than 9% in the concentration range of 0.02–0.1 µg mL⁻¹; LOD ranged from 0.1 to 4.6 µg L⁻¹ (23). Cesnik et al. described pesticide monitoring of 47 samples of wine grapes. Two internal analytical methods were used for the determination of pesticides by GC-MS. Multiple residues were found in 41 samples (87.2%). The highest number of pesticides detected was seven. Folpet (97.9%), cyprodinil (51.1%), dithiocarbamates (44.7%), chlorothalonil (23.4%), chlorpyrifos (19.1%), and Pyrimethanil (14.9%) were the most frequently found pesticides in grapes (24). Didier et al. described a multiresidue method for the determination of 74 pesticides in grapes and extracted them with ethyl acetate, with no additional cleanup steps, and determined them by LC-ESI-MS-MS. The LOQ was 0.01 mg kg⁻¹. Recoveries, RSD, and accuracy values of the method fulfilled the criteria of validation commonly admitted (25). Hiemstra et al. developed a multiresidue method for the simultaneous target analysis of 171 pesticides and metabolites in grapes by LC-MS-MS. Pesticide residues were extracted/partitioned from the samples with acetone/dichloromethane/light petroleum. Recoveries ranged from 70% to 110%, with RSD < 15%. The LOQ was 0.01 mg kg⁻¹ (26).

According to the 2006 statistical data of Organisation Internationale de la Vigne et du Vin (OIV), the area of the globe producing grapes was 7.812 million hectares, and production was 667.7 millions of quintals. The world production of wine in 2006 was, without juices and musts, 282.8 millions of hectolitres. China ranked the seventh after France, Italy, Spain, USA, Australia, and Argentina (27). Pesticide residue monitoring and control no doubt is an undertaking of great magnitude. Changli city, Hebei province, P.R. China is mainly a dry red wine producing area in China, with the same latitude as that of Bordeaux France, and the soil and coastal moist climate is similar to that of Bordeaux France. Vineyard area measurements are several

hundred thousand acres, and the task of monitoring the pesticide residues was heavy. Meanwhile, grapes are a complicated matrix containing a great deal of sugar, pigment, and organic acid, etc. It is necessary to select an analytical technique with higher sensitivity and selectivity for enlarging the varieties of pesticides detected and enhancing analysis speed. Compared with the three-sample preparation and cleanup methods such as those of Lehotay et al. (28), Pang et al. (29), and Wong et al. (30), this work established a method of the 346 of pesticides multiresidue method by acetonitrile extraction and PSA MSPD cleanup and GC-MS-SIM detection. The method, which is efficient, fast, and of low cost, is applicable to routine screening, validation, and quantification of hundreds of pesticides in grapes.

MATERIALS AND METHODS

Materials and Reagents. Pesticide standards, all ≥95% Purity, were obtained from LGC Promochem (Wesel, Germany). Acetonitrile, hexane and toluene were of LC grade for residue analysis obtained from J. T. Baker (Phillipsburg, NJ). Magnesium sulfate (anhydrous) was purchased from Sigma Inc. (USA), heated to 550 °C for five hours, and kept in a desiccator. PSA (40 µm) was obtained from Varian Inc. (USA). Envi-18 cartridges (12 mL/2.0 g) and Envi-Carb cartridges (6 mL/0.5 g) were from Supelco Inc. (USA). Sep-Pak NH₂ cartridges (3 mL/0.5 g) and Oasis HLB cartridges (6 mL/500 mg) were purchased from Waters (USA). Deionized water purified by Milli-Q. Grapes were purchased from Vineyard in Changli, Hebei province, China.

Solutions. *Stock Standard Solutions.* Accurately weighed individual pesticide standards were dissolved in toluene, toluene–acetone, dichloromethane, and methanol, etc., and the concentration of pesticides were at the range of 500–1000 ng µL⁻¹, which were based on the solubility of each compound.

Mixture Standard Solution. Three hundred fifty-two pesticides were divided into four groups, A, B, C, D, and each group was diluted in toluene; the concentrations were between 2.5 and 240 ng µL⁻¹.

Internal Solution. Accurately weighed 3.5 mg of heptachlor-epoxide was placed in a 100 mL volumetric flask, dissolved, and diluted to volume with toluene.

Instrumentation. *GC-MS.* Model 6890 N gas chromatograph performed with an Agilent 5973N MSD equipped with EI. Capillary column DB-1701 (30 m × 0.25 mm × 0.25 µm). GC-MS conditions: column temperature was maintained at 40 °C for 1 min and then programmed to 130 at 30 °C min⁻¹, to 250 at 5 °C min⁻¹, then attached to 300 at 10 °C min⁻¹, and held for 5 min. Carrier gas at a rate of 1.2 mL min⁻¹ was helium, with a purity ≥99.999%. The injection port temperature was 290 °C; 1 µL was injected in splitless linear with the purge on after 1.5 min. The ionization energy was 70 eV, the ion source temperature 230 °C, and the GC-MS interface temperature 280 °C. The monitoring mode was set to selected ion monitoring.

Homogenizer. The homogenizer was T-25B (Janke & Kunkel, Staufen, Germany); centrifuge, Z320 (B. Hermlle AG, Gosheim, Germany); rotary evaporator-Buchi, EL131 (Flawil, Switzerland); nitrogen evaporator, EVAP 112 (Organomation Associates, Inc., New Berlin, MA).

Extraction and Cleanup. *Method A.* A 15.0 g portion of homogenized grapes was accurately weighed to a 50 mL glassware centrifuge tube with stopper and mixed with 15 mL of acetonitrile. The capped tube was shaken for 1 min before 6.0 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride were added into it, then the cap was screwed, the tube was vigorously shaken for about 10 s by hand, transferred to the oscillator for shaking for 5 min, and eventually put into the centrifuge for 5 min at 3000 rpm. The extracts (upper layer) were transferred to another 50 mL glassware centrifuge tube with stopper containing 0.3 g of PSA and 1.8 g of anhydrous magnesium sulfates. The tube was capped, shaken for 5 min, and centrifuged for 3 min at 3000 rpm; 7.5 mL of the supernatant was pipetted to a pear-shaped flask, concentrated to ca. 0.5 mL by rotary evaporation at 40 °C, exchanged with hexane (2 × 5 mL) twice, and diluted to ca. 1 mL. Forty microliters of internal standard solution was added. The solution, after thoroughly mixing and passing the 0.20 µm filter membrane, was ready for GC-MS analysis.

Method B. Grape samples (20 g) were extracted with 40 mL of acetonitrile, salted out with sodium chloride. The coextractive was concentrated, cleaned up with Envi-18, Envi-carb, and sep-pak NH₂ cartridges in series, and eluted with 25 mL of acetonitrile-toluene (3 + 1). The eluate was concentrated to ca. 1 mL, added to internal standard solution after solvent exchange, and then was ready for GC-MS analysis.

Method C. Grape samples (20 g) were extracted with 40 mL of acetonitrile, salted out with sodium chloride, the coextractive concentrated to ca. 1 mL, added to 20 mL of water, then the aqueous solution transferred to the Oasis HLB cartridge. While all the liquid passed through the cartridge, the cartridge was dried under vacuum. Then it was attached to NH₂ cartridges, the tandem cartridge was eluted with 5 mL each of 80:20, 50:50, and 20:80 ethyl acetate/hexane. The eluate was concentrated, dissolved, and then was ready for GC-MS analysis.

Qualitative and Quantitative Analysis. The qualitative pesticide was based on the retention time of the peaks and the abundance ratios of the selected ions. If the abundance ratios of the selected ions were within the expected limits (32), the sample was confirmed to contain the pesticide. If the results were not definitive, the sample was reinjected with one or two additional confirmatory ions of the suspicious pesticides. Quantification was performed by using an internal standard. To compensate for the matrix effect, quantitation was based on a mixed standard solution prepared in blank matrix extract. The LOD was set with concentration levels giving a signal-to-noise ratio ≥ 5 . LOQ was calculated to $2 \times \text{LOD}$.

RESULTS AND DISCUSSION

Designing Test Procedures Based on Interval Grouping. All pesticides were conducted to proceed full scan, then base peaks, fragment ions, and retention times were selected. To enlarge the variety scope of the pesticides determined, on the basis of the concept of combining information acquisition and analytical technology, and designed monitoring program the groups were divided according to the time frame so that determination of multiresidues was as simple as single residue determination. On the basis of the retention times, the 352 compounds were divided into four groups: A, B, C, and D. One quantifying ion and two or three qualifying ions were selected for each compound. The analysis parameters optimized for the compounds are listed in **Table 1**. SIM program for the four groups A, B, C, D are listed in **Table 2**.

Comparison and Selection of Extraction and Cleanup Condition of Samples. Diverse extraction and cleanup procedures purposed to acquire the better recovery of analyte and less matrix interference (31). Three methods were conducted in this work, including Lehotay (labeled method A) (28), Pang (labeled method B) (29), and Wong (labeled method C) (30). In comparative experiments, fortification recovery experiments were conducted at low level, and then samples were extracted and cleaned up, respectively, with the three methods described above and analyzed by the monitoring mode described above. The data of recoveries and RSDs are listed in **Table 3**. The results of the statistic data are shown in **Table 4**.

From **Table 4**, comparing the three different methods, the number of pesticides that the average recoveries ranged from 60% to 120% were 340, 332, and 269, respectively, accounting for 96.6%, 92.9%, and 76.4% of all results. Among the results mentioned above, the recoveries were between 80% and 120%, and 288 analytes were determined by method A, 320 by method B, and 163 by method C.

Pesticides determined by method A whose recoveries were less than 40% contained resemthrin, cycloxydim, fenamiphos sulfone, and carboxim. Recoveries for all pesticides disposed by method B were higher than 60%. The elute solvents used in method C were nonpolar so as to influence the eluate of some polar pesticides including some organophosphorus, carbamate, and triazine herbicide, etc.. Recoveries were below 40%, for

instance, allidochlor, etridiazol, chlormephos, prothion, cycloate, diphenylamine, ethalfuralin, phorate, thiometon, quintozone, atrazine-desethyl, clomazone, diazinon, fonofos, etrimfos, simazine, propetamphos, and sebumeton. The number of pesticide recoveries which were over 120%, determined by method A is 3, by method B is 25, and by method C is 14. From the above, three methods for the determination of multiresidue pesticides were applied widely. From the comparative experiments, methods A and B were superior to method C.

From the experiments, the number of RSDs lower than 10% was 287, 292, 262 pesticides by method A, B, and C, respectively, among which 341, 340, and 334 analyte RSDs were lower than 20%, accounting for 96.9%, 96.6%, and 94.9%, respectively. It showed that these methods have relatively good repeatability.

For the effect of removing pigment, the coextractive cleaned up by method B was colorless no matter what the matrix color was. However, for the dark color matrix, the samples were light yellow, which was cleaned up by methods A and C. Therefore, on the effect of removing pigment, method B was better than A and C, but if considering the time of sample preparation and consumption of solvents, method A was rapid and was of low cost. The advantage was especially suitable for the determination of multiple samples. Thus, the following validation experiment for the selected method A was conducted. However, after about 300 of injections for samples disposed by method A, the inner tube must be cleaned or changed. Otherwise, recoveries for some pesticides will fall, e.g., pyrethroids.

Linear Range and LOD for the Proposed Method. The linear correlation coefficients for 352 pesticides were greater than 0.95, among which 318 pesticide linear correlation coefficients greater than 0.99 account for 90.3%, 34 pesticides below 0.99, accounting for 9.7%. It can be seen that the method has relatively higher sensitivity and a good linear range. LOD for 352 pesticides was between 0.0017 to 0.2661 mg kg⁻¹, among which the LOD was below 0.01 mg kg⁻¹ accounting for 40%; between 0.01 to 0.05 mg kg⁻¹ accounting for 56%; over 0.05 mg kg⁻¹, accounting for 4%. The LOD of the method, the linear range, and the linear correlation coefficient for 352 pesticides are shown in **Table 1**.

Evaluation of the Method Efficiency. The method efficiency was evaluated by recovery tests at low, medium, and high fortification levels using 8 different grapes (including wine grapes, wild wine grapes, and table grapes, etc.), while matrix standards were substituted for solution standards to quantify targeting pesticides correct for the matrix effect. See **Table 5** for test results. It could be seen from **Table 5** that the 352 compounds with their recoveries between 60% and 120% at low, medium, and high fortification levels were, respectively, 341, 341, and 346, accounting for 97%, 97%, and 98.2% of all results. The compounds with recoveries less than 40% at three fortification levels were resemthrin (2 isomers), cycloxydim, fenamiphos sulfoxide, and carboxin; the compounds with recoveries higher than 120% were bromfenfos, chlorfenson, aziprotryne, hexaflumuron, and chlorflazuron. The compounds with RSDs less than 20% at three fortification levels were 333, 322, and 327, respectively, which accounted for 94.6%, 91.5%, and 92.9%. The compounds with RSDs higher than 30% were only 7, 6, and 7 varieties, accounting for less than 2%. In general, some pesticides that were not detected by the method because their recoveries were less than 40%, and RSD was over 20%. Besides, the RSD of fenamiphos sulfone was relatively high, and its recoveries were unstable, which were not applicable to the detection of this method. In conclusion, the method is applicable to a quantified determination of 346 compounds.

The above test results further proved that the method has broad applicability for the determination of pesticide

Table 1. Parameter for the Simultaneous Determination of 352 Pesticide Residues in Grapes by GC-MS

no.	pesticide	retention time (min)	quantification ion ($m z^{-1}$)	qualification ion 1 ($m z^{-1}$)	qualification ion 2 ($m z^{-1}$)	qualification ion 3 ($m z^{-1}$)	linear range (ng)	correlation coefficient	LOD ($mg kg^{-1}$)
ISTD	Heptachlor-epoxide	22.10	353(100)	355(79)	351(52)				
A Group									
1	Allidochlor	8.83	138(100)	158(10)	173(15)		0.1250–5.000	0.9986	0.0167
2	Etridiazol	10.36	211(100)	183(73)	140(19)		0.1875–7.500	0.9945	0.0250
3	Chlormephos	10.53	121(100)	234(70)	154(70)		0.1250–5.000	0.9928	0.0167
4	Propham	11.38	179(100)	137(66)	120(51)		0.0625–2.500	0.9975	0.0083
5	Cycloate	13.52	154(100)	186(5)	215(12)		0.0625–2.500	0.9986	0.0083
6	Diphenylamine	14.52	169(100)	168(58)	167(29)		0.0625–2.500	0.9975	0.0083
7	Ethalfuralin	15.12	276(100)	316(81)	292(42)		0.2500–10.00	0.9926	0.0333
8	Phorate	15.48	260(100)	121(160)	231(56)	153(3)	0.0625–2.500	0.9969	0.0083
9	Thiometon	16.21	88(100)	125(55)	246(9)		0.0625–2.500	0.9967	0.0083
10	Quintozene	16.70	295(100)	237(159)	249(114)		0.1250–5.000	0.9958	0.0167
11	Atrazine-desethyl	16.74	172(100)	187(32)	145(17)		0.0625–2.500	0.9956	0.0083
12	Clomazone	17.00	204(100)	138(4)	205(13)		0.0625–2.500	0.9976	0.0083
13	Diazinon	17.17	304(100)	179(192)	137(172)		0.0625–2.500	0.9980	0.0083
14	Fonofos	17.30	246(100)	137(141)	174(15)	202(6)	0.0625–2.500	0.9975	0.0083
15	Etrimfos	17.92	292(100)	181(40)	277(31)		0.0625–2.500	0.9976	0.0083
16	Simazine	17.88	201(100)	186(62)	173(42)		0.0625–2.500	0.9965	0.0083
17	Propetamphos	18.06	138(100)	194(49)	236(30)		0.0625–2.500	0.9968	0.0083
18	Secbumeton	18.38	196(100)	210(38)	225(39)		0.0625–2.500	0.9952	0.0083
19	Dichlofenthion	18.82	279(100)	223(78)	251(38)		0.0625–2.500	0.9986	0.0083
20	Pronamide	18.82	173(100)	175(62)	255(22)		0.0625–2.500	0.9905	0.0083
21	Dimethoate	19.00	125(100)	143(21)	229(19)		0.5000–10.00	0.9962	0.0667
22	Aldrin	19.54	263(100)	265(65)	293(40)	329(8)	0.1250–5.000	0.9990	0.0167
23	Dinitramine	19.48	305(100)	307(38)	261(29)		0.2500–10.00	0.9956	0.0333
24	Ronnel	19.77	285(100)	287(67)	125(32)		0.1250–5.000	0.9978	0.0167
25	Prometryne	20.17	241(100)	184(78)	226(60)		0.0625–2.500	0.9961	0.0083
26	Cyprazine	20.21	212(100)	227(58)	170(29)		0.0625–2.500	0.9978	0.0083
27	Chlorothalonil	20.22	266(100)	264(72)	268(49)		2.5000–5.000	1.0000	0.3333
28	Vinclozolin	20.38	285(100)	212(109)	198(96)		0.0625–2.500	0.9973	0.0083
29	beta-HCH	20.45	219(100)	217(78)	181(94)	254(12)	0.0625–2.500	0.9992	0.0083
30	Metalaxyl	20.70	206(100)	249(53)	234(38)		0.1875–7.500	0.9982	0.0250
31	Chlorpyrifos (-ethyl)	20.97	314(100)	258(57)	286(42)		0.0625–2.500	0.9985	0.0083
32	Methyl-Parathion	20.89	263(100)	233(66)	246(8)	200(6)	0.2500–10.00	0.9892	0.0333
33	Anthraquinone	21.41	208(100)	180(84)	152(69)		0.0625–2.500	0.9862	0.0083
34	Delta-HCH	21.26	219(100)	217(80)	181(99)	254(10)	0.1250–5.000	0.9986	0.0167
35	Fenthion	21.71	278(100)	169(16)	153(9)		0.0625–2.500	0.9973	0.0083
36	Malathion	21.62	173(100)	158(36)	143(15)		0.2500–10.00	0.9974	0.0333
37	Fenitrothion	21.71	277(100)	260(52)	247(60)		0.1250–5.000	0.9957	0.0167
38	Paraoxon-ethyl	22.09	275(100)	220(60)	247(58)		0.2500–10.00	0.9892	0.0333
39	Triadimefon	22.33	208(100)	210(50)	181(74)		0.1250–5.000	0.9976	0.0167
40	Parathion	22.43	291(100)	186(23)	235(35)	263(11)	0.2500–10.00	0.9935	0.0333
41	Pendimethalin	22.63	252(100)	220(22)	162(12)		0.2500–10.00	0.9960	0.0333
42	Chlorbenside	22.94	268(100)	270(41)	143(11)		0.1250–5.000	0.9946	0.0167
43	Bromophos-ethyl	23.09	359(100)	303(77)	357(74)		0.0625–2.500	0.9973	0.0083
44	Quinalphos	23.12	146(100)	298(28)	157(66)		0.0625–2.500	0.9959	0.0083
45	trans-Chlordane	23.27	373(100)	375(96)	377(51)		0.0625–2.500	0.9989	0.0083
46	Phenthoate	23.33	274(100)	246(24)	320(5)		0.1250–5.000	0.9970	0.0167
47	Metazachlor	23.37	209(100)	133(120)	211(32)		0.1875–7.500	0.9984	0.0250
48	Prothiophos	24.06	309(100)	267(88)	162(55)		0.0625–2.500	0.9957	0.0083
49	Chlorfurenol	24.14	215(100)	152(40)	274(11)		0.1875–7.500	0.9960	0.0250
50	Dieldrin	24.37	263(100)	277(82)	380(30)	345(35)	0.1250–5.000	0.9994	0.0167
51	Procymidone	24.47	283(100)	285(70)	255(15)		0.0625–2.500	0.9979	0.0083
52	Methidathion	24.52	145(100)	157(2)	302(4)		0.1250–5.000	0.9948	0.0167
53	cyanazine	24.59	225(100)	240(56)	198(61)		0.1875–7.500	0.9952	0.0250
54	Napropamide	24.90	271(100)	128(111)	171(34)		0.1875–7.500	0.9963	0.0250
55	Oxadiazone	25.18	175(100)	258(62)	302(37)		0.0625–2.500	0.9980	0.0083
56	Fenamiphos	25.40	303(100)	154(56)	288(31)	217(22)	0.1875–7.500	0.9864	0.0250
57	Tetrasul	25.79	252(100)	324(64)	254(68)		0.0625–2.500	0.9958	0.0083
58	Aramite	25.73	185(100)	319(37)	334(32)		0.0625–2.500	0.9927	0.0083
59	Bupirimate	26.12	273(100)	316(41)	208(83)		0.0625–2.500	0.9954	0.0083
60	Carboxin	26.29	235(100)	143(168)	87(52)		0.1875–7.500	0.9930	0.0250
61	Flutolanil	26.41	173(100)	145(25)	323(14)		0.0625–2.500	0.9951	0.0083
62	p,p'-DDD	26.64	235(100)	237(64)	199(12)	165(46)	0.0625–2.500	0.9930	0.0083
63	Ethion	26.81	231(100)	384(13)	199(9)		0.1250–5.000	0.9944	0.0167
64	Sulprofos	26.93	322(100)	156(62)	280(11)		0.1250–5.000	0.9959	0.0167

Table 1. Continued

no.	pesticide	retention time (min)	quantification ion (m z ⁻¹)	qualification ion 1 (m z ⁻¹)	qualification ion 2 (m z ⁻¹)	qualification ion 3 (m z ⁻¹)	linear range (ng)	correlation coefficient	LOD (mg kg ⁻¹)
65	Etaconazole-1	26.97	245(100)	173(85)	247(65)		0.1875–7.500	0.9948	0.0250
66	Etaconazole-2	26.97	245(100)	173(85)	247(65)		0.1875–7.500	0.9976	0.0250
67	Myclobutanil	27.39	179(100)	288(14)	150(45)		0.0625–2.500	0.9958	0.0083
68	Diclofop-methyl	28.14	253(100)	281(50)	342(82)		0.0625–2.500	0.9958	0.0083
69	Propiconazole	28.24	259(100)	173(97)	261(65)		0.1875–7.500	0.9942	0.0250
70	Fensulfothin	28.13	292(100)	308(22)	293(73)		0.1250–5.000	0.9900	0.0167
71	Bifenthrin	28.66	181(100)	166(25)	165(23)		0.0625–2.500	0.9936	0.0083
72	Mirex	28.94	272(100)	237(49)	274(80)		0.0625–2.500	0.9974	0.0083
73	Benodanil	28.73	231(100)	323(38)	203(22)		0.1875–7.500	0.9899	0.0250
74	Nuarimol	28.95	314(100)	235(155)	203(108)		0.1250–5.000	0.9962	0.0167
75	Methoxychlor	29.38	227(100)	228(16)	212(4)		0.0625–2.500	0.9921	0.0083
76	Oxadixyl	29.24	163(100)	233(18)	278(11)		0.0625–2.500	0.9897	0.0083
77	Tetramethrin	29.70	164(100)	135(3)	232(1)		0.1250–5.000	0.9928	0.0167
78	Tebuconazole	29.65	250(100)	163(55)	252(36)		0.1875–7.500	0.9941	0.0250
79	Norflurazon	30.15	303(100)	145(101)	102(47)		0.0625–2.500	0.9879	0.0083
80	Pyridaphenthion	30.29	340(100)	199(48)	188(51)		0.0625–2.500	0.9841	0.0083
81	Phosmet	30.53	160(100)	161(11)	317(4)		0.1250–5.000	0.9861	0.0167
82	Tetradifon	30.76	227(100)	356(70)	159(196)		0.0625–2.500	0.9966	0.0083
83	cis-Permethrin	31.49	183(100)	184(15)	255(2)		0.0625–2.500	0.9935	0.0083
84	Trans-Permethrin	31.75	183(100)	184(15)	255(2)		0.0625–2.500	0.9911	0.0083
85	Pyrazophos	31.73	221(100)	232(35)	373(19)		0.1250–5.000	0.9904	0.0167
86	Cypermethrin	33.19 33.38 33.46 33.56	181(100)	152(23)	180(16)		0.1875–7.500	0.9919	0.0250
87	Fenvalerate	34.45 34.79	167(100)	225(53)	419(37)	181(41)	0.2500–10.00	0.9928	0.0333
B Group									
88	EPTC	8.54	128(100)	189(30)	132(32)		0.1875–7.500	0.9993	0.0250
89	Butylate	9.50	156(100)	146(115)	217(27)		0.1875–7.500	0.9994	0.0250
90	Dichlobenil	9.79	171(100)	173(68)	136(15)		0.0250–0.500	0.9998	0.0033
91	Pebulate	10.19	128(100)	161(21)	203(20)		0.1875–7.500	0.9994	0.0250
92	Nitrapyrin	10.86	194(100)	196(97)	198(23)		0.1875–7.500	0.9957	0.0250
93	Mevinphos	11.30	127(100)	192(39)	164(29)		0.1250–5.000	0.9957	0.0167
94	Chloroneb	11.83	191(100)	193(67)	206(66)		0.0625–2.500	0.9990	0.0083
95	Tecnazene	13.52	261(100)	203(135)	215(113)		0.2500–5.000	0.9985	0.0333
96	Heptenophos	13.81	124(100)	215(17)	250(14)		0.1875–7.500	0.9979	0.0250
97	Hexachlorobenzene	14.57	284(100)	286(81)	282(51)		0.0625–2.500	0.9996	0.0083
98	Ethoprophos	14.45	158(100)	200(40)	242(23)	168(15)	0.1875–7.500	0.9982	0.0250
99	cis-Diallate	14.76	234(100)	236(37)	128(38)		0.1250–5.000	0.9998	0.0167
100	Propachlor	14.80	120(100)	176(45)	211(11)		0.1875–7.500	0.9998	0.0250
101	trans-Diallate	15.32	234(100)	236(37)	128(38)		0.1250–5.000	0.9994	0.0167
102	Trifluralin	15.41	306(100)	264(72)	335(7)		0.1250–5.000	0.9930	0.0167
103	Chlorpropham	15.57	213(100)	171(59)	153(24)		0.1250–5.000	0.9971	0.0167
104	Sulfotep	15.65	322(100)	202(43)	238(27)	266(24)	0.0625–2.500	0.9991	0.0083
105	Sulfallate	15.78	188(100)	116(7)	148(4)		0.1250–5.000	0.9958	0.0167
106	Alpha-HCH	16.10	219(100)	183(98)	221(47)	254(6)	0.0625–2.500	0.9994	0.0083
107	Terbufos	16.90	231(100)	153(25)	288(10)	186(13)	0.1250–5.000	0.9977	0.0167
108	Terbumeton	17.22	210(100)	169(66)	225(32)		0.1875–7.500	0.9983	0.0250
109	Profluralin	17.54	318(100)	304(47)	347(13)		0.2500–10.00	0.9955	0.0333
110	Dioxathion	17.56	270(100)	197(43)	169(19)		0.2500–10.00	0.9993	0.0333
111	Propazine	17.75	214(100)	229(67)	172(51)		0.0625–2.500	0.9988	0.0083
112	Chlorbufam	17.93	223(100)	153(53)	164(64)		0.2500–10.00	0.9921	0.0333
113	Dicloran	17.96	206(100)	176(128)	160(52)		0.2500–10.00	0.9963	0.0333
114	Monolinuron	18.26	61(100)	126(45)	214(51)		0.2500–10.00	0.9888	0.0333
115	Flufenoxuron	18.92	305(100)	126(67)	307(32)		0.1875–7.500	0.9929	0.0250
116	Cyanophos	18.86	243(100)	180(8)	148(3)		0.1250–5.000	0.9980	0.0167
117	Chlorpyrifos-methyl	19.39	286(100)	288(70)	197(5)		0.0625–2.500	0.9972	0.0083
118	Desmetryn	19.66	213(100)	198(60)	171(30)		0.0625–2.500	0.9964	0.0083
119	Dimethachlor	20.11	134(100)	197(47)	210(16)		0.1875–7.500	0.9989	0.0250
120	Alachlor	20.11	188(100)	237(35)	269(15)		0.1875–7.500	0.9990	0.0250
121	Pirimiphos-methyl	20.34	290(100)	276(86)	305(74)		0.0625–2.500	0.9977	0.0083
122	Terbutryn	20.66	226(100)	241(64)	185(73)		0.1250–5.000	0.9980	0.0167
123	Thiobencarb	20.66	100(100)	257(25)	259(9)		0.1250–5.000	0.9987	0.0167
124	Dicofol	21.36	139(100)	141(72)	250(23)	251(4)	0.1250–5.000	0.9980	0.0167
125	Metolachlor	21.42	238(100)	162(159)	240(33)		0.0625–2.500	0.9977	0.0083
126	Oxy-chlordane	21.60	387(100)	237(50)	185(68)		0.2500–2.500	0.9997	0.0333
127	Pirimiphos-ethyl	21.66	333(100)	318(93)	304(69)		0.1250–5.000	0.9980	0.0167
128	Methoprene	21.84	73(100)	191(29)	153(29)		0.1250–5.000	0.9980	0.0167

Table 1. Continued

no.	pesticide	retention time (min)	quantification ion (m z ⁻¹)	qualification ion 1 (m z ⁻¹)	qualification ion 2 (m z ⁻¹)	qualification ion 3 (m z ⁻¹)	linear range (ng)	correlation coefficient	LOD (mg kg ⁻¹)
129	Bromofos	21.76	331(100)	329(75)	213(7)		0.2500–10.00	0.9975	0.0333
130	Dichlofluanid	21.75	224(100)	226(74)	167(120)		0.3750–15.00	0.9977	0.0500
131	Ethofumesate	21.99	207(100)	161(54)	286(27)		0.1250–5.000	0.9987	0.0167
132	Isopropalin	22.36	280(100)	238(40)	222(4)		0.3750–15.00	0.9977	0.0500
133	Chlorthiamid	23.06	205(100)	170(203)	171(86)		2.0000–10.00	0.9986	0.2667
134	endosulfan I	23.06	241(100)	265(66)	339(46)		0.1250–5.000	0.9992	0.0167
135	Propanil	23.05	161(100)	217(21)	163(62)		0.3750–15.00	0.9991	0.0500
136	Isofenphos	23.11	213(100)	255(44)	185(45)		0.1250–5.000	0.9930	0.0167
137	Crufomate	23.04	256(100)	182(154)	276(58)		0.1250–5.000	0.9982	0.0167
138	Chlorfenvinphos	23.29	323(100)	267(139)	269(92)		0.3750–15.00	0.9914	0.0500
139	Cis-Chlordane	23.56	373(100)	375(96)	377(51)		0.1250–5.000	0.9994	0.0167
140	Tolyfluanide	23.55	238(100)	240(71)	137(210)		0.1875–7.500	0.9981	0.0250
141	p,p'-DDE	23.92	318(100)	316(80)	246(139)	248(70)	0.0625–2.500	0.9994	0.0083
142	Butachlor	23.94	176(100)	160(75)	188(46)		0.1250–5.000	0.9970	0.0167
143	Chlozolinate	24.01	259(100)	188(83)	331(91)		0.1250–5.000	0.9994	0.0167
144	Crotoxyphos	24.08	193(100)	194(16)	166(51)		0.3750–15.00	0.9904	0.0500
145	Iodofenphos	24.36	377(100)	379(37)	250(6)		0.1250–5.000	0.9940	0.0167
146	Tetrachlorvinphos	24.44	329(100)	331(96)	333(31)		0.1875–7.500	0.9975	0.0250
147	Profenofos	24.74	339(100)	374(39)	297(37)		0.3750–15.00	0.9969	0.0500
148	Fluorochloridone	25.11	311(100)	313(64)	187(85)		0.5000–5.000	0.9994	0.0667
149	Buprofezin	24.95	105(100)	172(54)	305(24)		0.1250–5.000	0.9987	0.0167
150	o,p'-DDD	25.23	235(100)	237(65)	165(39)	199(15)	0.1250–5.000	0.9987	0.0167
151	Endrin	25.10	263(100)	317(30)	345(26)		0.1250–2.500	0.9997	0.0167
152	Hexaconazole	25.04	214(100)	231(62)	256(26)		0.7500–30.00	0.9982	0.1000
153	Chlorfenson	25.15	302(100)	175(282)	177(103)		0.7500–15.00	0.9989	0.1000
154	o,p'-DDT	25.54	235(100)	237(63)	165(37)	199(14)	0.1250–5.000	0.9980	0.0167
155	Paclobutrazol	25.40	236(100)	238(37)	167(39)		0.1250–5.000	0.9978	0.0167
156	Methoprottryne	25.74	256(100)	213(24)	271(17)		0.1875–7.500	0.9941	0.0250
157	Chloropropylate	25.97	251(100)	253(64)	141(18)		0.0625–2.500	0.9970	0.0083
158	Flamprop-methyl	26.04	105(100)	77(26)	276(11)		0.0625–2.500	0.9983	0.0083
159	Nitrofen	26.22	283(100)	253(90)	202(48)	139(15)	0.3750–15.00	0.9905	0.0500
160	Oxyfluorfen	26.33	252(100)	361(35)	300(35)		0.2500–10.00	0.9945	0.0333
161	Chlorthiophos	26.61	325(100)	360(52)	297(54)		0.1875–7.500	0.9978	0.0250
162	endosulfan II	26.76	241(100)	265(66)	339(46)		1.5000–15.00	0.9994	0.2000
163	Flamprop-Isopropyl	26.87	105(100)	276(19)	363(3)		0.0625–2.500	0.9974	0.0083
164	p,p'-DDT	27.27	235(100)	237(65)	246(7)	165(34)	0.1250–5.000	0.9971	0.0167
165	Carbofenthiol	27.29	157(100)	342(49)	199(28)		0.1250–5.000	0.9955	0.0167
166	Benalyxyl	27.62	148(100)	206(32)	325(8)		0.0625–2.500	0.9972	0.0083
167	Edifenphos	28.02	173(100)	310(76)	201(37)		0.1250–5.000	0.9912	0.0167
168	Triazophos	28.38	161(100)	172(47)	257(38)		0.1875–7.500	0.9944	0.0250
169	Cyanofenphos	28.59	157(100)	169(56)	303(20)		0.0625–2.500	0.9967	0.0083
170	Chlorbenside sulfone	29.03	127(100)	99(14)	89(33)		0.1250–5.000	0.9966	0.0167
171	Endosulfan-Sulfate	29.17	387(100)	272(165)	389(64)		0.1875–7.500	0.9985	0.0250
172	Bromopropylate	29.41	341(100)	183(34)	339(49)		0.1250–5.000	0.9963	0.0167
173	Benzoylprop-ethyl	29.53	292(100)	365(36)	260(37)		0.1875–7.500	0.9983	0.0250
174	Fenprothrin	29.71	265(100)	181(237)	349(25)		0.1250–5.000	0.9966	0.0167
175	Leptophos	30.22	377(100)	375(73)	379(28)		0.2500–10.00	0.9913	0.0333
176	EPN	30.19	157(100)	169(53)	323(14)		0.1875–7.500	0.9970	0.0250
177	Hexazinone	30.30	171(100)	252(3)	128(12)		0.2500–10.00	0.9826	0.0333
178	Bifenox	30.76	341(100)	189(82)	310(75)		0.2500–10.00	0.9826	0.0333
179	Phosalone	31.38	182(100)	367(30)	154(20)		0.1250–5.000	0.9955	0.0167
180	Azinphos-methyl	31.50	160(100)	132(71)	77(58)		0.7500–15.00	0.9971	0.1000
181	Fenarimol	31.73	139(100)	219(70)	330(42)		0.1250–5.000	0.9993	0.0167
182	Azinphos-ethyl	32.13	160(100)	132(103)	77(51)		0.1250–5.000	0.9930	0.0167
183	Prochloraz	33.23	180(100)	308(59)	266(18)		0.0375–15.00	0.9919	0.0050
184	Cyfluthrin	32.94 33.12	206(100)	199(63)	226(72)		0.7500–30.00	0.9966	0.1000
185	Coumaphos	33.39	362(100)	226(56)	364(39)	334(15)	0.3750–15.00	0.9919	0.0500
186	Fluvalinate	34.94 35.02	250(100)	252(38)	181(18)		0.7500–30.00	0.9954	0.1000
C Group									
187	Dichlorvos	7.90	109(100)	185(34)	220(7)		0.3750–15.00	0.9990	0.0500
188	Biphenyl	9.03	154(100)	153(40)	152(27)		0.0625–2.500	0.9998	0.0083
189	Vernolate	9.92	128(100)	146(17)	203(9)		0.0625–2.500	0.9997	0.0083
190	3,5-Dichloroaniline	11.32	161(100)	163(62)	126(10)		0.0625–2.500	0.9905	0.0083
191	Molinate	11.99	126(100)	187(24)	158(2)		0.0625–2.500	0.9996	0.0083
192	Methacrifos	11.99	125(100)	208(74)	240(44)		0.0625–2.500	0.9994	0.0083
193	2-Phenylphenol	12.56	170(100)	169(72)	141(31)		0.0625–1.250	0.9946	0.0083

Table 1. Continued

no.	pesticide	retention time (min)	quantification ion (m z ⁻¹)	qualification ion 1 (m z ⁻¹)	qualification ion 2 (m z ⁻¹)	qualification ion 3 (m z ⁻¹)	linear range (ng)	correlation coefficient	LOD (mg kg ⁻¹)
194	Cis-1,2,3,6-Tetrahydrophthalimide	13.55	151(100)	123(16)	122(16)		0.1875–7.500	0.9985	0.0250
195	Fenobucarb	14.78	121(100)	150(32)	107(8)		0.1250–5.000	0.9982	0.0167
196	Benfluralin	15.50	292(100)	264(20)	276(13)		0.0625–2.500	0.9888	0.0083
197	Hexaflumuron	16.41	176(100)	279(28)	277(43)				0.0500
198	Prometon	16.81	210(100)	225(91)	168(67)		0.1875–7.5000	0.9988	0.0250
199	Triallate	17.23	268(100)	270(73)	143(19)		0.1250–5.000	0.9993	0.0167
200	Pyrimethanil	17.36	198(100)	199(45)	200(5)		0.0625–2.500	0.9980	0.0083
201	Gamma-HCH	17.64	183(100)	219(93)	254(13)	221(40)	0.1250–5.000	0.9998	0.0167
202	Disulfoton	17.79	88(100)	274(15)	186(18)		0.0625–2.500	0.9983	0.0083
203	Atrazine	17.83	200(100)	215(62)	173(29)		0.0625–2.500	0.9991	0.0083
204	Heptachlor	18.54	272(100)	237(40)	337(27)		0.1875–7.500	0.9990	0.0250
205	Iprobenfos	18.63	204(100)	246(18)	288(17)		0.1875–7.500	0.9895	0.0250
206	Isazofos	18.76	161(100)	257(53)	285(39)	313(14)	0.1250–5.000	0.9994	0.0167
207	Plifenate	19.01	217(100)	175(96)	242(91)		0.2500–5.000	0.9992	0.0333
208	Fenpropimorph	19.36	128(100)	303(5)	129(9)		0.0625–2.5000	0.9989	0.0083
209	Transfluthrin	19.26	163(100)	165(23)	335(7)		0.0625–2.500	0.9991	0.0083
210	Fluchloralin	19.26	306(100)	326(87)	264(54)		0.2500–10.00	0.9952	0.0333
211	Tolclofos-methyl	19.82	265(100)	267(36)	250(10)		0.0625–2.500	0.9991	0.0083
212	Ametryn	20.27	227(100)	212(53)	185(17)		0.1875–7.500	0.9989	0.0250
213	Simetryn	20.33	213(100)	170(26)	198(16)		0.1250–5.000	0.9990	0.0167
214	Metobromuron	20.32	61(100)	258(11)	170(16)		0.3750–15.00	0.9986	0.0500
215	Metribuzin	20.49	198(100)	199(21)	144(12)		0.1875–7.500	0.9986	0.0250
216	Dipropetryn	21.01	255(100)	240(42)	222(20)		0.0625–2.500	0.9976	0.0083
217	Formothion	21.12	170(100)	224(97)	257(63)		0.5000–5.000	0.9993	0.0667
218	Diethofencarb	21.66	267(100)	225(98)	151(31)		0.3750–15.00	0.9977	0.0500
219	Bioallethrin-1	22.59	123(100)	136(24)	107(29)		0.2500–10.00	0.9941	0.0333
220	Bioallethrin-2	22.59	123(100)	136(24)	107(29)		0.2500–10.00	0.9983	0.0333
221	o,p'-DDE	22.73	246(100)	318(34)	176(26)	248(65)	0.0625–2.500	0.9998	0.0083
222	Fenson	22.73	141(100)	268(53)	77(104)		0.0625–2.500	0.9993	0.0083
223	Diphenamid	23.07	167(100)	239(30)	165(43)		1.250–2.500	1.0000	0.1667
224	Chlorthion	23.11	297(100)	267(162)	299(45)		0.0625–2.500	0.9989	0.0083
225	Prallethrin	23.36	123(100)	105(17)	134(9)		0.5000–5.000	0.9857	0.0667
226	Penconazole	23.42	248(100)	250(33)	161(50)		0.1875–7.500	0.9964	0.0250
227	Mecarbam	23.73	131(100)	296(22)	329(40)		0.1875–7.500	0.9992	0.0250
228	Tetraconazole	23.72	336(100)	338(33)	171(10)		0.2500–10.00	0.9986	0.0333
229	Flumetralin	24.39	143(100)	157(25)	404(10)		0.1250–5.000	0.9873	0.0167
230	Triadimenol	24.52	112(100)	168(81)	130(15)		0.1875–7.500	0.9972	0.0250
231	Pretilachlor	24.92	162(100)	238(26)	262(8)		0.1250–5.000	0.9980	0.0167
232	Kresoxim:methyl	25.19	116(100)	206(25)	131(66)		0.0625–2.500	0.9976	0.0083
233	Fluazifop-butyl	25.52	282(100)	383(44)	254(49)		0.0625–2.500	0.9961	0.0083
234	Chlorfluazuron	25.50	321(100)	323(71)	356(8)		0.3750–7.500	0.9980	0.0500
235	Chlorobenzilate	26.12	251(100)	253(65)	152(5)		0.0625–2.500	0.9977	0.0083
236	Flusilazole	26.49	233(100)	206(33)	315(9)		0.1875–7.500	0.9983	0.0250
237	Fluorodifen	27.00	190(100)	328(35)	162(34)				0.0167
238	Diniconazole	27.30	268(100)	270(65)	232(13)		0.1875–7.500	0.9936	0.0250
239	Piperonyl butoxide	27.70	176(100)	177(33)	149(14)		0.0625–2.500	0.9919	0.0083
240	Propargite	28.13	135(100)	350(7)	173(16)		0.1250–5.000	0.9995	0.0167
241	Mepronil	28.20	119(100)	269(26)	120(9)		0.0625–2.500	0.9941	0.0083
242	Diffufenican	28.70	266(100)	394(25)	267(14)		0.0625–2.500	0.9950	0.0083
243	Fenzaaquin	29.12	145(100)	160(46)	117(10)		0.0625–2.500	0.9950	0.0083
244	Phenothrin	29.08 29.21	123(100)	183(74)	350(6)		0.0625–2.500	0.9950	0.0083
245	Fludioxonil	29.21	248(100)	127(24)	154(21)		0.0625–2.500	0.9963	0.0083
246	Fenoxycarb	29.81	255(100)	186(82)	116(93)		0.3750–15.00	0.9902	0.0500
247	Sethoxydim	29.95	178(100)	281(51)	219(36)		0.5625–22.50	0.9868	0.0750
248	Anilofos	30.92	226(100)	184(52)	334(10)		0.2500–5.000	0.9954	0.0333
249	Acrinathrin	31.38	181(100)	289(31)	247(12)		0.1250–5.000	0.9939	0.0167
250	Lambda-Cyhalothrin	31.40	181(100)	197(100)	141(20)		0.0625–2.500	0.9982	0.0083
251	Mefenacet	31.48	192(100)	120(35)	136(29)		0.1875–7.500	0.9928	0.0250
252	Permethrin	31.75	183(100)	184(14)	255(1)		0.1250–5.000	0.9967	0.0167
253	Pyridaben	32.05	147(100)	117(11)	364(7)		0.0625–2.500	0.9958	0.0083
254	Fluoroglycofen-ethyl	32.29	447(100)	428(20)	449(35)		0.7500–30.00	0.9826	0.1000
255	Bitertanol	32.45	170(100)	112(8)	141(6)		0.1875–7.500	0.9931	0.0250
256	Etofenprox	32.92	163(100)	376(4)	183(6)		0.0625–2.500	0.9961	0.0083
257	Cycloxydim	33.30	178(100)	279(7)	251(4)		0.7500–30.00	0.9907	0.1000
258	Esfenvalerate	34.98	419(100)	225(158)	181(189)		0.2500–10.00	0.9983	0.0333
259	Alpha-Cypermethrin	33.59	163(100)	181(84)	165(63)		0.1250–5.000	0.9974	0.0167

Table 1. Continued

no.	pesticide	retention time (min)	quantification ion (m z ⁻¹)	qualification ion 1 (m z ⁻¹)	qualification ion 2 (m z ⁻¹)	qualification ion 3 (m z ⁻¹)	linear range (ng)	correlation coefficient	LOD (mg kg ⁻¹)
260	Difenconazole	35.64	323(100)	325(66)	265(83)		0.3750–15.00	0.9860	0.0500
261	Flumiclorac-pentyl	36.79	423(100)	308(51)	318(29)		0.1250–5.000	0.9903	0.0167
D Group									
262	Dimefox	5.66	110(100)	154(75)	153(17)		0.1875–7.500	0.9992	0.0250
263	Disulfoton-sulfoxide	8.45	212(100)	153(61)	184(20)		0.1250–5.000	0.9993	0.0167
264	Pentachlorobenzene	11.05	250(100)	252(64)	215(24)		0.0625–2.500	0.9998	0.0083
265	Crimidine	13.21	142(100)	156(90)	171(84)		0.0625–2.500	0.9987	0.0083
266	Chlorfenprop-methyl	13.65	165(100)	196(87)	197(49)		0.0625–2.500	0.9989	0.0083
267	Thionazin	14.13	143(100)	192(39)	220(14)		0.0625–2.500	0.9969	0.0083
268	2,3,5,6-Tetrachloroaniline	14.20	231(100)	229(76)	158(25)		0.0625–2.500	0.9994	0.0083
269	Tri- <i>N</i> -Butyl Phosphate	14.48	155(100)	211(61)	167(8)		0.1250–5.000	0.9972	0.0167
270	2,3,4,5-Tetrachloroanisole	14.64	246(100)	203(70)	231(51)		0.0625–2.500	0.9997	0.0083
271	Pentachloroanisole	15.15	280(100)	265(100)	237(85)		0.0625–2.500	0.9996	0.0083
272	Tebutam	15.40	190(100)	106(38)	142(24)		0.1250–5.000	0.9990	0.0167
273	Dioxabenzofos	16.23	216(100)	201(26)	171(5)		0.6250–25.00	0.9980	0.0833
274	Methabenzthiazuron	16.37	164(100)	136(81)	108(27)		0.6250–25.00	0.9930	0.0833
275	Simetone	16.76	197(100)	196(40)	182(38)		0.0125–5.0	0.9984	0.0167
276	Atraton	16.80	196(100)	211(68)	197(105)		0.0625–2.500	0.9988	0.0083
277	Desisopropyl-atrazine	16.75	173(100)	158(84)	145(73)		0.5000–20.00	0.9975	0.0667
278	Terbufos Sulfone	16.90	231(100)	288(11)	186(15)		0.0625–2.500	0.9980	0.0083
279	Tefluthrin	17.40	177(100)	197(26)	161(5)		0.0625–2.500	0.9980	0.0083
280	Bromocylen	17.44	359(100)	357(99)	394(14)		0.0625–2.500	0.9990	0.0083
281	Trietazine	17.63	200(100)	229(51)	214(45)		0.0625–2.500	0.9987	0.0083
282	Etrifos oxon	17.92	292(100)	277(35)	263(12)		0.0625–2.500	0.9990	0.0083
283	Cycluron	18.08	89(100)	198(36)	114(9)		0.1875–7.500	0.9982	0.0250
284	2,6-Dichlorobenzamide	18.06	173(100)	189(36)	175(62)		0.1250–5.000	0.9983	0.0167
285	DE-PCB 28	18.18	256(100)	186(53)	258(97)		0.0625–1.250	0.9997	0.0083
286	DE-PCB 31	18.18	256(100)	186(53)	258(97)		0.0625–1.250	0.9997	0.0083
287	Desethyl-sebuthylazine	18.41	172(100)	174(32)	186(11)		0.1250–5.000	0.9985	0.0167
288	2,3,4,5-Tetrachloroaniline	18.58	231(100)	229(76)	233(48)		0.1250–5.000	0.9989	0.0167
289	Pentachloroaniline	18.89	265(100)	263(63)	230(8)		0.0625–2.500	0.9990	0.0083
290	Aziprotryne	19.16	199(100)	184(83)	157(31)		0.5000–20.00	0.9979	0.0667
291	Sebutylazine	19.40	200(100)	214(14)	229(13)		0.0625–2.500	0.9989	0.0083
292	Isocarbamid	19.39	142(100)	185(2)	143(6)		0.3125–12.50	0.9965	0.0417
293	DE-PCB 52	19.49	292(100)	220(88)	255(32)		0.0625–2.500	0.9997	0.0083
294	Prosulfocarb	19.60	251(100)	252(14)	162(10)		0.0625–2.500	0.9988	0.0083
295	Dimethenamid	19.68	154(100)	230(43)	203(21)		0.0625–2.500	0.9990	0.0083
296	Fenchlorphos oxon	19.77	285(100)	287(70)	270(7)		0.1250–5.000	0.9991	0.0167
297	Paraoxon-methyl	20.03	230(100)	247(93)	200(40)		0.5000–5.000	0.9852	0.0667
298	Monalide	20.24	197(100)	199(31)	239(45)		0.1250–5.000	0.9988	0.0167
299	Isobenzan	20.55	311(100)	375(31)	412(7)		0.0625–2.500	0.9995	0.0083
300	Octachlorostyrene	20.54	380(100)	343(94)	308(120)		0.0625–2.500	0.9997	0.0083
301	Isodrin	20.98	193(100)	263(46)	195(83)		0.0625–2.500	0.9982	0.0083
302	Isomethiozin	21.17	225(100)	198(86)	184(13)		0.1250–5.000	0.9970	0.0167
303	Trichloronat	21.18	297(100)	269(86)	196(16)		0.0625–2.500	0.9989	0.0083
304	Dacthal	21.32	301(100)	332(31)	221(16)		0.0625–2.500	0.9995	0.0083
305	4,4-Dichlorobenzophenone	21.35	250(100)	252(62)	215(26)		0.0625–2.500	0.9959	0.0083
306	Nitrothal-isopropyl	21.92	236(100)	254(54)	212(74)		0.1250–5.000	0.9911	0.0167
307	Rabenzazole	21.77	212(100)	170(26)	195(19)		0.0625–2.500	0.9931	0.0083
308	Cyprodinil	21.97	224(100)	225(62)	210(9)		0.0625–2.500	0.9977	0.0083
309	Dicapthon	22.61	262(100)	263(10)	216(10)		0.3125–12.50	0.9946	0.0417
310	DE-PCB 101	22.64	326(100)	254(66)	291(18)		0.0625–2.500	0.9994	0.0083
311	MCPA-Butoxyethyl ester	22.77	300(100)	200(71)	182(41)		0.0625–2.500	0.9966	0.0083
312	Phorate sulfone	23.42	199(100)	171(30)	215(11)		0.0625–2.500	0.9946	0.0083
313	Chlorfenethol	23.42	251(100)	253(66)	266(12)		0.0625–2.500	0.9975	0.0083
314	Trans-Nonachlor	23.69	409(100)	407(89)	411(63)		0.0625–2.500	0.9990	0.0083
315	DEF	24.23	202(100)	226(51)	258(55)		0.1250–5.000	0.9971	0.0167
316	Fluorochloridone	24.62	311(100)	187(74)	313(66)		0.1250–5.000	0.9979	0.0167
317	Bromfenvinfos	24.77	267(100)	323(56)	295(18)		0.0625–2.500	0.9937	0.0083
318	Perthane	24.91	223(100)	224(20)	178(9)		0.0625–2.500	0.9974	0.0083
319	DE-PCB 118	25.12	326(100)	254(38)	184(16)		0.0625–2.500	0.9985	0.0083
320	4,4-Dibromobenzophenone	25.39	340(100)	259(30)	185(179)		0.1250–2.500	0.9945	0.0167
321	Flutriafol	25.48	219(100)	164(96)	201(7)		0.1250–5.000	0.9985	0.0167
322	Mephosfolan	25.51	196(100)	227(49)	168(60)		0.1250–5.000	0.9890	0.0167
323	Athidathion	25.68	145(100)	330(1)	129(12)		0.1250–5.000	0.9995	0.0167
324	DE-PCB 153	25.69	360(100)	290(62)	218(24)		0.0625–2.500	0.9992	0.0083

Table 1. Continued

no.	pesticide	retention time (min)	quantification ion ($m z^{-1}$)	qualification ion 1 ($m z^{-1}$)	qualification ion 2 ($m z^{-1}$)	qualification ion 3 ($m z^{-1}$)	linear range (ng)	correlation coefficient	LOD ($mg\ kg^{-1}$)
325	Diclobutrazole	26.16	270(100)	272(68)	159(42)		0.2500–10.00	0.9957	0.0333
326	Disulfoton sulfone	26.48	213(100)	229(4)	185(11)		0.1250–5.000	0.9966	0.0167
327	Hexythiazox	26.72	227(100)	156(158)	184(93)		0.500–20.00	0.9981	0.0667
328	DE-PCB 138	26.88	360(100)	290(68)	218(26)		0.0625–2.500	0.9991	0.0083
329	Resmethrin-1	27.57	171(100)	143(83)	338(7)		0.1250–5.000	0.9823	0.0167
330	Cyproconazole	27.42	222(100)	224(35)	223(11)		0.0625–2.500	0.9983	0.0083
331	Resmethrin-2	27.57	171(100)	143(80)	338(7)		0.1250–5.000	0.9953	0.0167
332	Phthalic acid, benzyl butyl	27.72	206(100)	312(4)	230(1)		0.0625–2.500	0.9973	0.0083
333	Clodinafop-propargyl	27.92	349(100)	238(96)	266(83)		0.1250–5.000	0.9820	0.0167
334	Fenthion sulfoxide	28.30	278(100)	279(290)	294(145)		0.2500–5.000	0.9863	0.0333
335	Fluotrimazole	28.53	311(100)	379(60)	233(36)		0.0625–2.500	0.9981	0.0083
336	Fluoroxypyr-1-methylheptyl ester	28.66	366(100)	254(67)	237(60)		0.1250–2.500	0.9974	0.0167
337	Fenthion sulfone	28.80	310(100)	136(25)	231(10)		0.2500–10.00	0.9839	0.0333
338	Triphenyl phosphate	28.78	326(100)	233(16)	215(20)		0.0625–2.500	0.9990	0.0083
339	Metamitron	28.79	202(100)	174(52)	186(12)		2.500–25.00	0.9994	0.3333
340	DE-PCB 180	29.11	394(100)	324(70)	359(20)		0.0625–2.500	0.9989	0.0083
341	Tebufenpyrad	29.19	318(100)	333(78)	276(44)		0.0625–2.500	0.9965	0.0083
342	Cloquintoletole-methyl	29.46	192(100)	194(32)	220(4)		0.0625–2.500	0.9915	0.0083
343	Lenacil	29.84	153(100)	136(6)	234(2)		0.6250–25.00	0.9967	0.0833
344	Bromuconazole-1	30.05	173(100)	175(65)	214(15)		0.1250–5.000	0.9916	0.0167
345	Desbrom-leptophos	30.22	377(100)	171(97)	375(72)		0.0625–2.500	0.9990	0.0083
346	Bromuconazole-2	30.86	173(100)	175(67)	214(14)		0.1250–2.500	0.9994	0.0167
347	Nitralin	31.22	316(100)	274(58)	300(15)		0.6250–25.00	0.9907	0.0833
348	Fenamiphos sulfoxide	31.23	304(100)	319(29)	196(22)		0.2500–10.00	0.9561	0.0333
349	Fenamiphos sulfone	31.60	320(100)	292(57)	335(7)		0.2500–10.00	0.9954	0.0333
350	Fenpiclonil	32.53	236(100)	238(66)	174(36)		0.2500–10.00	0.9981	0.0333
351	Fluquinconazole	32.72	340(100)	342(37)	341(20)		0.0625–2.500	0.9991	0.0083
352	Fenbuconazole	34.28	129(100)	198(51)	125(31)		0.1250–5.000	0.9896	0.0167

Table 2. Monitoring Program of Selected Ions for A, B, C, and D Groups of Pesticides

no.	time (min)	monitored ions (amu)	dwelt time (ms)
Group A			
1	8.30	138, 158, 173	200
2	9.60	124, 140, 166, 172, 183, 211	90
3	10.50	121, 154, 234	200
4	10.75	120, 137, 179	200
5	11.70	154, 186, 215	200
6	14.40	167, 168, 169	200
7	14.90	121, 142, 143, 153, 183, 195, 196, 198, 230, 231, 260, 276, 292, 316	30
8	16.20	88, 125, 246	200
9	16.70	137, 138, 145, 172, 174, 179, 187, 202, 204, 205, 237, 246, 249, 295, 304	30
10	17.80	138, 173, 175, 181, 186, 194, 196, 201, 210, 225, 236, 255, 277, 292	30
11	18.80	150, 165, 173, 175, 222, 223, 251, 255, 279	50
12	19.20	125, 143, 229, 261, 263, 265, 293, 305, 307, 329	50
13	19.80	125, 261, 263, 265, 285, 287, 293, 305, 307, 329	50
14	20.10	170, 181, 184, 198, 200, 206, 212, 217, 219, 226, 227, 233, 234, 241, 246, 249, 254, 258, 263, 264, 266, 268, 285, 286, 314	10
15	21.40	143, 152, 153, 158, 169, 173, 180, 181, 208, 217, 219, 220, 247, 254, 256, 260, 275, 277, 278, 351, 353, 355	10
16	22.30	61, 143, 160, 162, 181, 186, 208, 210, 220, 235, 248, 252, 263, 268, 270, 291, 351, 353, 355	20
17	23.00	133, 143, 146, 157, 209, 211, 246, 268, 270, 274, 298, 303, 320, 357, 359, 373, 375, 377	20
18	23.70	72, 104, 133, 145, 152, 157, 160, 162, 209, 211, 215, 253, 255, 260, 263, 267, 274, 277, 283, 285, 297, 302, 309, 345, 380	10
19	24.80	128, 145, 154, 157, 171, 175, 198, 217, 225, 240, 255, 258, 271, 283, 285, 288, 302, 303	20
20	25.50	154, 185, 217, 252, 253, 254, 288, 303, 319, 324, 334	50
21	26.00	87, 139, 143, 145, 165, 173, 199, 208, 231, 235, 237, 251, 253, 273, 316, 323, 384	20
22	26.80	145, 150, 156, 165, 173, 179, 199, 231, 235, 237, 245, 247, 280, 288, 322, 323, 384	20
23	27.90	165, 166, 173, 181, 253, 259, 261, 281, 292, 293, 308, 342	40
24	28.60	118, 160, 165, 166, 181, 203, 212, 227, 228, 231, 235, 237, 272, 274, 314, 323	30
25	29.30	135, 163, 164, 212, 227, 228, 232, 233, 250, 252, 278	40
26	30.00	102, 145, 159, 160, 161, 188, 199, 227, 303, 317, 340, 356	40
27	31.00	175, 183, 184, 220, 221, 223, 232, 250, 255, 267, 373	40
28	33.00	127, 180, 181	200
29	34.40	167, 181, 225, 419	150
30	35.70	172, 174, 181	200

Table 2. Continued

no.	time (min)	monitored ions (amu)	dwell time (ms)
Group B			
1	7.80	128, 132, 189	200
2	8.80	146, 156, 217	200
3	9.70	128, 136, 161, 171, 173, 203	90
4	10.70	127, 164, 192, 194, 196, 198	90
5	11.70	191, 193, 206	200
6	13.40	124, 203, 215, 250, 261	100
7	14.40	158, 168, 200, 242, 282, 284, 286	80
8	14.70	116, 120, 128, 148, 153, 171, 176, 188, 202, 211, 213, 234, 236, 238, 264, 266, 282, 284, 286, 306, 322, 335	10
9	16.00	116, 148, 183, 188, 219, 221, 254	80
10	16.80	153, 186, 231, 288	150
11	17.10	153, 160, 164, 169, 172, 173, 176, 197, 206, 210, 214, 223, 225, 229, 270, 318, 330, 347	20
12	18.20	61, 126, 160, 173, 176, 206, 214, 229	60
13	18.70	126, 127, 134, 148, 164, 171, 172, 180, 192, 197, 198, 210, 213, 223, 243, 286, 288, 305, 307	20
14	19.90	134, 171, 188, 197, 198, 210, 213, 237, 269, 276, 290, 305	40
15	20.60	100, 185, 211, 226, 241, 253, 257, 259, 378	50
16	21.20	73, 139, 141, 153, 161, 162, 167, 185, 191, 207, 213, 224, 226, 237, 238, 240, 250, 251, 286, 304, 318, 329, 331, 333, 351, 353, 355, 387	10
17	22.00	161, 167, 207, 222, 224, 226, 238, 264, 280, 286, 351, 353, 355	40
18	22.70	161, 163, 170, 171, 182, 185, 205, 213, 217, 241, 255, 256, 265, 267, 269, 276, 323, 339	20
19	23.40	137, 160, 176, 188, 238, 240, 246, 248, 259, 267, 269, 316, 318, 323, 331, 373, 375, 377	20
20	23.90	61, 160, 166, 176, 188, 193, 194, 246, 248, 250, 259, 292, 294, 297, 316, 318, 329, 331, 333, 339, 374, 377, 379	20
21	24.90	61, 105, 165, 167, 172, 175, 177, 187, 199, 214, 231, 235, 236, 237, 238, 256, 263, 292, 294, 297, 302, 305, 311, 313, 317, 339, 345, 374	10
22	25.60	77, 105, 139, 141, 165, 169, 171, 199, 202, 213, 223, 235, 237, 251, 252, 253, 256, 271, 276, 283, 297, 300, 325, 360, 361	10
23	26.70	105, 157, 165, 195, 199, 235, 237, 246, 276, 297, 325, 339, 342, 360, 363	30
24	27.60	148, 157, 161, 169, 172, 173, 201, 206, 257, 303, 310, 325	40
25	28.90	89, 99, 126, 127, 157, 161, 169, 172, 181, 183, 257, 260, 265, 272, 292, 303, 339, 341, 349, 365, 387, 389	10
26	29.80	79, 181, 183, 265, 311, 349	90
27	30.00	128, 157, 169, 171, 189, 252, 310, 323, 341, 375, 377, 379	40
28	31.20	132, 139, 154, 160, 161, 182, 189, 251, 310, 330, 341, 367	40
29	32.90	180, 199, 206, 226, 266, 308, 334, 362, 364	50
30	34.00	181, 250, 252	200
Group C			
1	7.30	109, 185, 220	200
2	8.70	152, 153, 154	200
3	9.30	58, 128, 129, 146, 188, 203	90
4	11.20	126, 161, 163	200
5	11.75	125, 126, 141, 158, 169, 170, 187, 208, 240	50
6	13.50	122, 123, 124, 151, 215, 250	90
7	14.70	107, 121, 150, 264, 276, 292	90
8	16.00	174, 202, 217	200
9	16.50	126, 141, 143, 156, 168, 176, 198, 199, 200, 210, 225, 268, 270, 277, 279	30
10	17.60	88, 173, 183, 186, 200, 215, 219, 254, 274	50
11	18.40	104, 130, 159, 161, 204, 237, 246, 257, 272, 285, 288, 313, 337	40
12	18.90	128, 129, 161, 163, 165, 175, 204, 217, 242, 246, 257, 264, 285, 288, 303, 306, 313, 326, 335	20
13	19.80	73, 89, 146, 162, 185, 212, 223, 227, 250, 265, 267	50
14	20.30	61, 144, 146, 162, 170, 185, 198, 199, 212, 213, 223, 227, 258	40
15	20.70	61, 103, 118, 144, 170, 181, 198, 199, 210, 217, 219, 222, 240, 254, 255	30
16	21.35	108, 117, 151, 160, 161, 170, 219, 221, 224, 225, 257, 267, 351, 353, 355	30
17	22.20	107, 108, 119, 123, 136, 145, 176, 219, 221, 246, 248, 263, 318, 351, 353, 355	20
18	22.70	77, 141, 165, 167, 174, 176, 206, 234, 239, 246, 248, 267, 268, 297, 299, 318	20
19	23.20	105, 123, 134, 161, 248, 250, 267, 297, 299	50
20	23.50	131, 143, 157, 161, 171, 220, 248, 250, 262, 296, 304, 329, 336, 338, 404	30
21	24.30	112, 130, 162, 168, 238, 262	90
22	25.10	112, 116, 130, 131, 162, 168, 206, 233, 234, 235, 238, 262	40
23	25.30	254, 282, 321, 323, 356, 383	90
24	26.00	131, 152, 206, 233, 234, 236, 251, 253, 315	50
25	26.90	149, 162, 176, 177, 190, 232, 268, 270, 328	50
26	27.90	105, 119, 120, 135, 140, 173, 266, 267, 269, 350, 394	50
27	28.80	105, 117, 123, 140, 145, 160, 183, 266, 267, 350, 394	50
28	29.00	117, 123, 127, 145, 154, 160, 183, 248, 350	50
29	29.60	116, 178, 186, 191, 219, 255	90
30	30.30	132, 162, 178, 184, 219, 226, 281, 293, 334	50
31	31.10	120, 136, 141, 147, 181, 183, 184, 192, 197, 247, 255, 289, 309, 364	30
32	32.00	112, 141, 147, 170, 183, 184, 255, 309, 364, 428, 447, 449	40
33	32.60	112, 141, 163, 170, 183, 376, 428, 447, 449	50

Table 2. Continued

no.	time (min)	monitored ions (amu)	dwelt time (ms)
34	33.10	163, 165, 178, 181, 251, 279	90
35	33.80	157, 199, 451	200
36	34.70	181, 225, 250, 252, 419	100
37	35.40	259, 265, 287, 323, 325, 354	90
38	36.40	308, 318, 423	200
Group D			
1	5.50	110, 153, 154	200
2	8.00	153, 184, 212	200
3	11.00	139, 155, 211, 215, 250, 252	90
4	13.00	142, 156, 165, 171, 196, 197, 200, 201, 202	50
5	14.00	143, 155, 158, 167, 192, 203, 211, 220, 229, 231, 246	40
6	15.00	106, 142, 190, 237, 265, 280	90
7	16.00	108, 136, 145, 158, 164, 171, 173, 182, 186, 196, 197, 201, 211, 216, 213, 288	20
8	17.20	161, 174, 177, 197, 200, 202, 214, 229, 246, 357, 359, 394	40
9	17.90	89, 114, 128, 172, 173, 174, 175, 186, 189, 198, 223, 229, 230, 231, 233, 253, 256, 258, 263, 265, 268, 277, 282, 292, 297	10
10	19.20	142, 143, 154, 157, 162, 184, 185, 199, 200, 201, 202, 203, 214, 220, 229, 230, 247, 251, 252, 255, 263, 264, 270, 278, 285, 287, 292	10
11	20.00	153, 180, 197, 199, 200, 201, 202, 230, 239, 247, 251, 252, 266, 305, 308, 311, 343, 375, 380, 412	15
12	21.00	115, 184, 193, 195, 196, 198, 215, 221, 225, 250, 252, 263, 269, 276, 285, 297, 301, 332	20
13	21.60	128, 170, 194, 195, 210, 212, 224, 225, 236, 254, 279, 294	40
14	22.10	129, 155, 182, 184, 200, 201, 210, 212, 216, 224, 225, 229, 230, 254, 262, 263, 291, 300, 314, 326, 351, 353, 355	10
15	23.00	136, 171, 199, 215, 230, 251, 253, 266, 289, 407, 409, 411	40
16	23.90	130, 148, 178, 187, 202, 211, 223, 224, 226, 240, 258, 267, 295, 299, 311, 313, 323	20
17	25.00	129, 130, 145, 148, 164, 168, 184, 185, 196, 201, 218, 219, 227, 254, 259, 290, 299, 326, 330, 340, 360	15
18	26.00	156, 159, 184, 185, 213, 218, 227, 229, 270, 272, 290, 360	40
19	27.10	143, 160, 171, 206, 222, 223, 224, 230, 238, 251, 266, 294, 312, 338, 349	30
20	28.00	136, 174, 186, 202, 215, 231, 233, 237, 254, 278, 279, 294, 310, 311, 326, 366, 379	20
21	29.00	136, 153, 192, 194, 220, 234, 276, 318, 324, 333, 359, 394	40
22	30.00	160, 161, 171, 173, 175, 214, 317, 375, 377	50
23	30.80	173, 175, 196, 213, 230, 274, 292, 300, 304, 316, 319, 320, 335, 373	30
24	32.40	147, 236, 238, 340, 341, 342	90
25	34.00	125, 129, 198	200

Table 3. Comparison of Recoveries and RSD of 352 Pesticides in Grapes Conducted by Three Different Methods

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
A Group					
1	Allidochlor	0.0333	77.0(6.5)	69.9 (15.4)	97.1(1.6)
2	Etridiazol	0.0500	69.2 (6.3)	65.4 (24.7)	73.0 (6.1)
3	Chlormephos	0.0333	95.6 (5.9)	103.0 (16.1)	91.8 (6.6)
4	Propham	0.0167	91.2 (5.5)	93.3 (6.9)	103.8 (0.1)
5	Cycloate	0.0167	87.0 (3.1)	98.8 (3.8)	93.6 (7.9)
6	Diphenylamin	0.0167	79.8 (8.0)	101.3 (3.2)	87.4 (9.0)
7	Ethalfuralin	0.0667	86.7 (8.8)	100.5 (3.7)	75.9 (9.0)
8	Phorate	0.0667	82.7 (10.5)	97.5 (4.2)	84.5 (11.7)
9	Thiometon	0.0167	78.0 (8.3)	97.7 (1.7)	80.8 (7.9)
10	Quintozene	0.0167	84.2 (4.6)	104.7 (3.3)	84.2 (16.1)
11	Atrazine-desethyl	0.0333	84.2 (2.1)	93.0 (7.2)	107.3 (3.6)
12	Clomazone	0.0167	84.8 (6.0)	100.9 (2.3)	105.4 (3.6)
13	Diazinon	0.0167	91.3 (7.7)	101.0(2.3)	95.3 (9.3)
14	Fonofos	0.0167	89.1 (9.3)	102.3(2.7)	87.4 (10.1)
15	Etrimfos	0.0167	92.0 (9.4)	105.5 (5.1)	96.0 (9.3)
16	Simazine	0.0167	83.2 (8.4)	107.9(5.2)	113.9 (5.8)
17	Propetamphos	0.0167	89.5 (9.0)	102.6(2.4)	102.7 (5.8)
18	Secbumeton	0.0167	83.8 (6.0)	96.1(2.0)	105.4 (3.3)
19	Dichlofenthion	0.0167	82.2 (8.4)	93.8(2.3)	92.1 (5.0)
20	Pronamide	0.0167	91.5 (6.3)	103.4(1.6)	104.9 (3.4)
21	Dimethoate	0.0667	72.1 (9.8)	94.7 (1.6)	106.0 (3.8)
22	Aldrin	0.0333	91.1 (6.8)	106.9 (1.9)	76.1 (8.8)
23	Dinitramine	0.0667	90.0 (7.9)	98.4 (3.4)	73.6 (13.5)
24	Ronnel	0.0333	94.1 (7.8)	104.4 (2.0)	78.5 (11.7)
25	Prometrye	0.0167	90.0 (8.9)	100.2 (3.0)	97.5 (4.3)
26	Cyprazine	0.0167	86.8 (11.1)	86.5 (36.9)	107.8 (5.4)
27	Chlorothalonil	0.0333	40.7 (50.2)	91.8 (11.1)	86.6 (4.4)

Table 3. Continued

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
28	Vinclozolin	0.0167	89.0 (7.4)	113.2 (3.1)	72.0 (8.6)
29	beta-HCH	0.0167	87.3 (4.3)	112.9 (1.8)	81.6 (9.8)
30	Metalaxyl	0.0500	83.6 (11.7)	101.9 (3.0)	107.1 (4.6)
31	Chlorpyrifos(-ethyl)	0.0167	96.5 (5.7)	102.6 (5.3)	77.3 (13.0)
32	Methyl-parathion	0.0667	89.5 (7.9)	99.8 (4.8)	107.4 (4.1)
33	Anthraquinone	0.0167	92.3 (17.0)	90.0 (5.8)	97.5 (5.1)
34	delta-HCH	0.0333	90.4 (6.8)	109.1 (1.3)	84.0 (10.1)
35	Fenthion	0.0167	94.8 (6.9)	101.7 (3.1)	81.8 (13.3)
36	Malathion	0.0667	94.1 (8.0)	101.9 (3.3)	103.4 (7.7)
37	Fenitrothion	0.0333	89.6 (9.9)	99.7 (4.8)	99.9 (5.9)
38	Paraoxon-ethyl	0.0667	93.8 (7.4)	102.3 (5.3)	94.5 (5.3)
39	Triadimefon	0.0333	87.7 (14.2)	99.8 (2.3)	106.7 (3.5)
40	Parathion	0.0667	92.5 (6.8)	100.8 (4.5)	91.6 (11.2)
41	Pendimethalin	0.0667	94.4 (6.5)	105.4 (3.3)	77.3 (11.4)
42	Chlorbenside	0.0333	97.2 (9.7)	102.3 (2.2)	76.3 (12.2)
43	Bromophos-ethyl	0.0167	93.2 (4.1)	103.9 (1.3)	75.0 (7.6)
44	Quinalphos	0.0167	93.8 (7.4)	102.8 (4.0)	92.1 (9.5)
45	trans-Chlodane	0.0167	96.7 (6.0)	107.4 (0.9)	76.8 (10.5)
46	Phenthoate	0.0333	95.4 (5.7)	112.2 (9.7)	69.7 (8.5)
47	Metazachlor	0.0500	87.8 (13.2)	100.5 (2.3)	108.6 (3.1)
48	Prothiophos	0.0167	92.7 (7.9)	97.0 (4.1)	72.2 (10.8)
49	Chlorfuremol	0.0500	88.1 (11.3)	100.1 (3.4)	104.2 (4.2)
50	Dieldrin	0.0333	96.5 (5.2)	112.3 (5.1)	80.2 (10.1)
51	Procymidone	0.0167	92.5 (8.2)	108.4 (3.5)	100.5 (7.7)
52	Methidathion	0.0333	95.8 (9.6)	96.3 (4.4)	115.1 (5.3)
53	Cyanazine	0.0500	82.7 (11.5)	93.7 (3.1)	113.2 (4.3)
54	Napropamide	0.0500	86.3 (14.1)	99.5 (3.5)	107.5 (3.9)
55	Oxadiazone	0.0167	97.3 (6.0)	103.6 (2.1)	83.1 (12.3)
56	Fenamiphos	0.0500	84.6 (5.7)	81.8 (10.8)	98.2 (7.1)
57	Tetrasul	0.0167	99.1 (11.3)	102.4 (2.7)	75.4 (8.4)
58	Aramite	0.0167	93.6 (6.5)	110.8 (11.7)	81.9 (0.5)
59	Bupirimate	0.0167	89.8 (12.8)	96.5 (1.8)	92.3 (4.8)
60	Carboxin	0.0500	61.6 (13.1)	82.7 (1.7)	66.6 (15.0)
61	Flutolanil	0.0167	88.7 (11.0)	97.1 (3.4)	102.7 (4.9)
62	p,p'-DDD	0.0167	95.7 (6.3)	103.9 (2.9)	78.2 (6.2)
63	Ethion	0.0333	96.2 (7.4)	98.2 (4.5)	77.9 (10.6)
64	Sulprofos	0.0333	97.1 (5.8)	99.1 (2.9)	74.8 (11.7)
65	Etaconazole-1	0.0500	79.4 (7.0)	98.7 (4.1)	101.8 (3.2)
66	Etaconazole-2	0.0500	79.4 (7.3)	99.5 (3.3)	101.1 (0.8)
67	Myclobutanil	0.0167	83.1 (12.7)	96.0 (0.7)	104.7 (3.2)
68	Dichlorofop-methyl	0.0167	97.9 (11.6)	95.4 (7.1)	96.1 (3.9)
69	Propiconazole	0.0500	85.2 (8.8)	101.7 (3.3)	106.4 (1.4)
70	Fensulfotion	0.0333	81.4 (10.4)	98.0 (2.8)	112.3 (5.3)
71	Bifenthrin	0.0167	99.1 (6.5)	100.7 (2.8)	75.3 (7.9)
72	Mirex	0.0167	84.5 (5.1)	109.3 (1.6)	72.1 (10.2)
73	Benodanil	0.0500	80.5 (17.3)	93.1 (4.6)	109.9 (1.8)
74	Nuarimol	0.0333	75.6 (20.3)	91.6 (3.6)	107.1 (3.5)
75	Methoxychlor	0.0167	70.2 (9.3)	88.7 (16.4)	46.5 (20.5)
76	Oxadixyl	0.0167	67.8 (11.7)	94.1 (1.8)	104.5 (3.8)
77	Tetramethrin	0.0333	94.2 (7.7)	98.5 (4.3)	79.4 (11.0)
78	Tebuconazole	0.0500	77.4 (21.8)	91.8 (2.1)	100.1 (0.8)
79	Norflurazon	0.0167	77.2 (26.4)	91.9 (10.3)	102.5 (6.6)
80	Pyridaphenthion	0.0167	76.1 (24.5)	95.9 (4.4)	97.6 (4.9)
81	Phosmet	0.0333	90.5 (10.1)	90.2 (7.5)	35.4 (11.8)
82	Tetradifon	0.0167	93.8 (10.0)	103.2 (3.6)	83.5 (8.4)
83	cis-Permethrin	0.0167	99.3 (7.3)	97.5 (3.9)	104.9 (30.2)
84	trans-Permethrin	0.0167	98.6 (6.5)	98.3 (3.7)	77.5 (5.7)
85	Pyrazophos	0.0333	92.5 (10.7)	93.1 (6.4)	95.5 (7.2)
86	Cypermethrin	0.0500	97.7 (6.8)	98.8 (3.0)	77.9 (5.5)
87	Fenvalerate	0.0667	95.4 (5.8)	101.4 (1.5)	80.7 (5.0)
B Group					
88	EPTC	0.0500	76.9 (3.6)	72.4 (31.1)	98.7 (8.2)
89	Butylate	0.0500	79.2 (6.4)	82.5 (24.3)	83.8 (7.1)
90	Dichlobenil	0.0033	80.7 (1.1)	71.9 (32.5)	102.4 (8.3)
91	Pebulate	0.0500	84.7 (6.0)	73.4 (23.9)	93.4 (6.7)
92	Nitrapyrin	0.0500	86.9 (5.0)	92.6 (10.2)	78.1 (11.7)
93	Mevinphos	0.0333	72.7 (7.3)	98.6 (11.7)	60.4 (33.7)
94	Chloroneb	0.0167	84.0 (7.7)	98.0 (12.7)	97.0 (5.8)

Table 3. Continued

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
95	Tecnazene	0.0333	78.2 (7.3)	100.6 (10.5)	76.6 (5.2)
96	Heptanophos	0.0500	77.2 (5.9)	105.7 (7.1)	80.2 (2.3)
97	Hexachlorobenzene	0.0167	80.8 (6.3)	100.8 (8.2)	78.7 (7.4)
98	Ethoprophos	0.0500	79.0 (6.6)	110.5 (6.8)	102.4 (8.3)
99	<i>cis</i> -Diallate	0.0333	86.7 (5.1)	107.0 (8.5)	80.0 (5.7)
100	Propachlor	0.0500	78.7 (5.2)	106.9 (6.8)	107.7 (7.7)
101	<i>trans</i> -Diallate	0.0333	82.4 (6.8)	104.7 (8.3)	82.6 (5.2)
102	Trifluralin	0.0333	84.7 (4.5)	109.6 (5.4)	69.0 (6.3)
103	Chlorpropham	0.0333	88.3 (6.3)	103.5 (5.7)	97.4 (2.0)
104	Sulfotep	0.0167	82.2 (6.6)	109.1 (6.9)	81.9 (5.7)
105	Sulfallate	0.0333	79.3 (6.8)	107.5 (7.6)	85.0 (5.3)
106	alpha-HCH	0.0167	85.3 (6.6)	92.4 (7.7)	79.7 (5.2)
107	Terbufos	0.0333	85.4 (4.8)	109.5 (6.7)	72.1 (4.7)
108	Terbumeton	0.0500	82.0 (3.1)	107.3 (3.8)	99.5 (5.8)
109	Profluralin	0.0667	90.2 (3.6)	114.2 (6.7)	66.4 (5.9)
110	Dioxathion	0.0667	92.1 (1.7)	108.0 (8.4)	72.6 (6.3)
111	Propazine	0.0167	91.3 (4.1)	110.9 (4.8)	106.3 (7.4)
112	Chlorbufam	0.0333	86.9 (6.7)	118.5 (5.9)	97.9 (5.4)
113	Dicloran	0.0333	86.2 (5.3)	112.7 (6.7)	96.4 (2.5)
114	Monolinuron	0.0667	83.2 (11.2)	109.7 (4.8)	109.3 (8.3)
115	Flufenoxuron	0.0500	90.8 (5.4)	117.7 (5.8)	66.8 (3.3)
116	Cyanohos	0.0333	86.6 (4.4)	109.4 (6.1)	101.6 (8.1)
117	Chlorprifos-methyl	0.0167	90.8 (4.9)	110.9 (5.1)	71.2 (4.3)
118	Desmetryn	0.0167	81.1 (3.5)	107.6 (6.2)	102.5 (6.3)
119	Dimethachloro	0.0500	86.5 (5.5)	112.4 (4.7)	105.5 (7.6)
120	Alachlor	0.0500	89.9 (4.2)	112.5 (5.4)	102.5 (7.2)
121	Pirimiphos-methyl	0.0167	93.8 (5.9)	111.6 (4.3)	84.7 (6.9)
122	Terbutryn	0.0333	85.9 (4.7)	108.7 (4.4)	91.5 (6.6)
123	Thiobencarb	0.0333	91.2 (3.1)	110.4 (6.2)	84.7 (4.0)
124	Dicofol	0.0333	135.8 (17.8)	85.4 (55.5)	57.2 (2.5)
125	Metolachlor	0.0167	88.8 (3.3)	114.2 (6.3)	107.2 (4.8)
126	Oxy-chlordane	0.0167	89.3 (1.7)	112.4 (6.6)	63.5 (18.6)
127	Pirimiphos-ethyl	0.0333	91.8 (5.6)	110.9 (3.7)	71.4 (7.6)
128	Methoprene	0.0667	94.3 (3.3)	112.2 (7.1)	65.6 (3.0)
129	Bromofos	0.0333	91.0 (4.1)	109.2 (3.7)	68.8 (1.9)
130	Dichlofluanid	0.1000	70.0 (7.1)	108.5 (8.1)	19.5 (7.8)
131	Ethofumesate	0.0333	92.8 (2.4)	110.8 (6.1)	103.1 (7.7)
132	Isopropalin	0.0333	92.4 (4.4)	113.6 (7.1)	37.9 (70.4)
133	Chlorthiamid	0.5334	87.4 (7.7)	104.0 (6.9)	80.6 (1.4)
134	endosulfan-I	0.1000	92.0 (2.0)	110.9 (3.7)	73.9 (3.3)
135	Propanil	0.0333	82.3 (13.1)	109.7 (5.5)	100.1 (2.6)
136	Isofenphos	0.0333	92.2 (3.0)	113.4 (5.7)	77.5 (5.6)
137	Crufomate	0.1000	81.0 (5.7)	119.0 (10.4)	96.2 (1.6)
138	Chlorfenvinphos	0.0500	90.4 (5.1)	114.9 (6.4)	89.4 (4.1)
139	<i>cis</i> -Chlordane	0.0333	92.1 (3.5)	109.9 (4.9)	70.1 (10.6)
140	Tolyfluanide	0.0500	73.7 (11.2)	111.0 (5.5)	18.2 (5.5)
141	<i>p,p'</i> -DDE	0.0167	92.6 (4.3)	110.9 (5.1)	71.4 (9.2)
142	Butachlor	0.0333	92.1 (1.9)	111.9 (5.8)	73.3 (4.6)
143	Chlozolate	0.0333	88.4 (2.4)	106.6 (10.5)	65.1 (12.2)
144	Crotoxyphos	0.1000	85.1 (12.3)	120.0 (4.9)	68.4 (11.2)
145	Iodofenphos	0.0333	92.0 (3.4)	114.2 (3.9)	68.0 (2.2)
146	Tetrachlorvinphos	0.0500	83.0 (6.4)	114.8 (5.2)	74.7 (4.8)
147	Profenofos	0.1000	91.5 (2.8)	117.0 (6.6)	42.4 (14.3)
148	Fluorochloridone	0.0333	87.7 (9.9)	106.7 (7.1)	52.5 (6.1)
149	Buprofezin	0.0333	91.5 (4.2)	108.6 (5.0)	100.5 (7.6)
150	<i>o,p'</i> -DDD	0.0167	90.5 (3.6)	97.1 (14.8)	96.7 (11.7)
151	Endrin	0.2000	88.3 (3.0)	112.3 (5.0)	63.1 (3.6)
152	Hexaconazole	0.1000	86.3 (6.0)	111.0 (6.7)	83.2 (2.5)
153	Chlorfenson	0.0333	94.2 (3.8)	109.8 (3.1)	80.6 (5.8)
154	<i>o,p'</i> -DDT	0.0333	80.3 (5.8)	127.1 (6.3)	40.5 (5.3)
155	Paclobutrazol	0.0500	80.1 (10.9)	112.9 (5.2)	89.2 (4.1)
156	Methoprotryne	0.0500	83.3 (6.0)	107.8 (4.4)	96.3 (3.7)
157	Chlorpropylate	0.0167	92.9 (3.0)	109.1 (6.2)	68.2 (4.9)
158	Flamprop-methyl	0.0167	87.7 (5.0)	108.7 (5.6)	104.4 (5.4)
159	Nitrofen	0.1000	90.4 (5.7)	115.4 (6.7)	63.1 (6.6)
160	Oxyfluorfen	0.0667	93.5 (4.7)	114.7 (7.2)	58.8 (3.7)
161	Chlorthiophos	0.0500	92.3 (2.3)	110.3 (5.3)	68.9 (4.3)
162	endosulfan-II	0.1000	126.4 (40.8)	114.3 (3.5)	112.8 (12.5)
163	Flamprop-Isopropyl	0.0167	91.4 (4.4)	111.7 (6.6)	88.6 (5.1)

Table 3. Continued

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
164	<i>p,p'</i> -DDT	0.0333	83.0 (4.8)	136.0 (11.3)	31.3 (4.9)
165	Carbofenthoion	0.0333	92.3 (4.2)	114.2 (5.2)	65.8 (2.4)
166	Benalaxyl	0.0167	89.4 (4.3)	111.3 (6.1)	96.9 (4.9)
167	Edifenphos	0.0333	84.4 (6.0)	116.7 (6.7)	58.0 (15.7)
168	Triazophos	0.0500	90.9 (5.5)	113.5 (5.4)	91.9 (3.5)
169	Cyanofenphos	0.0167	97.0 (2.5)	112.5 (5.5)	75.2 (5.3)
170	Chlorbenside sulfone	0.0333	90.2 (5.4)	101.1 (5.8)	97.9 (3.8)
171	Endosulfan-sulfate	0.0500	91.8 (2.8)	110.6 (4.8)	72.0 (6.3)
172	Bromopropylate	0.0333	92.1 (4.6)	112.2 (6.7)	66.3 (4.2)
173	Benzoylprop-ethyl	0.0500	91.8 (4.2)	109.0 (5.5)	88.6 (5.5)
174	Fenpropathrin	0.0333	98.4 (3.8)	111.3 (7.5)	66.9 (5.6)
175	Leptophos	0.0333	95.0 (4.2)	109.9 (6.3)	68.1 (9.5)
176	EPN	0.0667	96.7 (5.0)	104.0 (8.9)	65.6 (5.5)
177	Hexazinone	0.0500	83.0 (5.2)	104.1 (11.1)	104.9 (7.7)
178	Bifenox	0.0333	94.0 (5.9)	113.6 (5.6)	67.9 (3.6)
179	Phosalone	0.0333	92.4 (4.3)	111.3 (5.8)	97.5 (7.2)
180	Azinphos-methyl	0.1000	86.8 (11.7)	114.2 (4.8)	99.2 (4.9)
181	Fenarimol	0.0333	82.8 (5.8)	107.8 (4.3)	96.4 (4.6)
182	Azinphos-ethyl	0.0333	90.1 (7.8)	116.4 (6.0)	93.0 (4.8)
183	Prochloraz	0.1000	68.5 (13.5)	105.6 (7.6)	66.2 (3.9)
184	Cyfluthrin	0.2000	92.1 (1.9)	108.6 (5.8)	58.9 (10.3)
185	Coumaphos	0.1000	92.0 (5.0)	112.8 (4.7)	76.9 (3.4)
186	Fluvalinate	0.2000	93.2 (3.1)	110.7 (3.8)	64.3 (9.8)
C Group					
187	Dichlorvos	0.1000	82.4 (7.2)	85.3 (13.9)	6.2 (27.0)
188	Biphenyl	0.0167	85.9 (5.3)	81.6 (14.9)	45.1 (10.3)
189	Vernolate	0.0167	76.9 (6.1)	89.4 (12.2)	65.1 (8.0)
190	3,5-Dichloroaniline	0.0167	86.4 (3.1)	97.8 (17.3)	76.7 (13.4)
191	Molinate	0.0167	83.8 (4.6)	97.9 (9.2)	77.3 (4.5)
192	Methacrifos	0.0167	84.3 (4.6)	94.4 (10.9)	80.8 (8.5)
193	2-Phenylphenol	0.0167	89.3 (9.3)	100.2 (9.3)	74.5 (3.6)
194	<i>cis</i> -1,2,3,6-Tetrahydrophthalimide	0.0500	71.5 (8.5)	94.1 (8.5)	61.8 (6.1)
195	Fenobucarb	0.0333	89.9 (9.1)	100.5 (6.8)	80.2 (2.9)
196	Benfluralin	0.0167	84.7 (3.4)	96.8 (8.1)	53.1 (7.6)
197	Hexaflumuron	0.1000	98.0 (7.8)	99.9 (5.3)	49.3 (9.0)
198	Prometon	0.0500	81.1 (8.3)	101.0 (5.7)	77.2 (1.4)
199	Triallate	0.0333	82.6 (5.7)	101.1 (6.8)	51.5 (7.3)
200	Pyrimethanil	0.0167	82.7 (4.6)	96.3 (6.9)	73.8 (4.9)
201	gamma-HCH	0.0333	83.3 (5.6)	101.7 (7.5)	58.0 (9.3)
202	Disulfoton	0.0167	62.3 (13.0)	100.6 (7.2)	51.7 (10.3)
203	Atrazine	0.0167	85.4 (6.7)	103.2 (6.2)	78.6 (2.5)
204	Heptachlor	0.0500	86.5 (7.0)	103.6 (7.3)	48.2 (4.7)
205	Iprobenfos	0.0500	79.9 (11.3)	103.3 (11.2)	76.1 (2.6)
206	Isazofos	0.0333	86.1 (7.6)	102.6 (7.0)	77.4 (2.6)
207	Plifenate	0.0333	78.3 (3.5)	106.0 (4.7)	45.4 (7.9)
208	Fenpropimorph	0.0167	82.8 (12.5)	102.1 (6.5)	55.5 (4.4)
209	Transfluthrin	0.0167	94.9 (1.2)	103.2 (7.0)	53.8 (10.0)
210	Fluchloralin	0.0667	85.9 (4.9)	104.1 (7.1)	48.2 (7.2)
211	Tolclofos-methyl	0.0167	87.4 (4.5)	101.2 (6.8)	52.8 (11.1)
212	Ametryn	0.0500	83.5 (6.7)	102.1 (6.6)	73.5 (1.2)
213	Simetryn	0.0333	82.8 (2.0)	98.3 (7.1)	65.1 (12.2)
214	Metobromuron	0.1000	90.0 (16.1)	91.7 (25.3)	70.0 (8.0)
215	Metribuzin	0.0500	84.5 (3.2)	101.6 (7.9)	78.5 (1.5)
216	Dipropetryn	0.0167	86.7 (5.0)	101.1 (6.5)	56.5 (4.1)
217	Formothion	0.0333	37.3 (39.7)	75.1 (10.0)	33.1 (3.4)
218	Diethofencarb	0.1000	85.5 (8.8)	105.0 (7.6)	88.7 (3.5)
219	Bioallethrin-1	0.0667	111.5 (8.8)	104.3 (10.0)	56.7 (10.1)
220	Bioallethrin-2	0.0667	113.0 (11.1)	103.3 (13.7)	42.2 (7.2)
221	<i>o,p'</i> -DDE	0.0167	94.8 (3.2)	102.3 (7.1)	53.1 (9.9)
222	Fenson	0.0167	92.6 (4.4)	104.4 (7.4)	60.7 (9.4)
223	Diphenamid	0.0167	80.8 (13.9)	105.3 (6.5)	79.4 (1.5)
224	Chlorthion	0.0333	83.8 (7.2)	104.7 (10.5)	57.4 (7.5)
225	Prallethrin	0.0500	85.9 (6.7)	103.3 (8.4)	51.4 (7.6)
226	Penconazole	0.0500	81.2 (9.5)	104.9 (7.6)	61.7 (6.5)
227	Mecarbam	0.0667	84.7 (7.0)	107.8 (4.0)	47.5 (8.3)
228	Tetraconazole	0.0500	79.8 (12.5)	103.6 (9.0)	63.5 (5.1)
229	Flumetralin	0.0333	91.0 (3.3)	104.4 (8.1)	50.0 (9.3)
230	Triadimenol	0.0500	77.5 (5.5)	103.1 (10.0)	73.2 (1.6)

Table 3. Continued

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
231	Pretilachlor	0.0333	89.9 (5.8)	104.0 (7.1)	66.3 (5.9)
232	Kresoxim:methyl	0.0167	90.4 (6.9)	103.8 (6.1)	67.2 (7.2)
233	Fluazifop-butyl	0.0167	87.9 (5.8)	103.0 (7.4)	49.7 (7.8)
234	Chlorfluazuron	0.0500	85.7 (1.7)	122.4 (17.1)	45.9 (19.2)
235	Chlorobenzilate	0.0167	87.5 (3.8)	102.6 (6.7)	52.6 (7.3)
236	Flusilazole	0.0500	79.2 (12.1)	103.6 (7.4)	54.9 (9.5)
237	Fluorodifen	0.0167	87.8 (7.3)	102.5 (8.4)	54.8 (9.1)
238	Diniconazole	0.0500	78.2 (9.0)	102.5 (10.6)	53.2 (6.5)
239	Piperonyl butoxide	0.0167	92.0 (4.8)	104.0 (10.3)	54.5 (6.6)
240	Propargite	0.0333	102.6 (6.1)	98.3(17.7)	51.5 (10.7)
241	Mepronil	0.0167	86.9 (7.9)	101.7 (8.1)	70.3 (1.6)
242	Diflufenican	0.0167	82.2 (11.4)	102.9 (8.5)	50.5 (7.0)
243	Fenazaquin	0.0167	88.0 (3.5)	100.6 (8.7)	46.9 (9.8)
244	Phenothrin	0.0167	97.7 (10.4)	98.9 (7.4)	52.5 (12.1)
245	Fludioxonil	0.0167	67.4 (25.4)	102.4 (8.7)	62.9 (6.4)
246	Fenoxycarb	0.1000	99.1 (7.2)	134.3 (15.8)	66.7 (8.9)
247	Sethoxydim	0.1500	82.8 (3.8)	80.8 (11.3)	9.8 (8.8)
248	Anilofos	0.0333	85.6 (10.0)	104.4 (8.0)	48.9 (5.7)
249	Acrinathrin	0.0333	81.9 (4.8)	111.0 (6.2)	15.8 (19.9)
250	Lambda-cyhalothrin	0.0167	86.8 (4.4)	99.7 (7.6)	38.7 (13.3)
251	Mefenacet	0.0500	85.4 (0.4)	105.1 (10.1)	75.0 (3.0)
252	Permethrin	0.0333	92.5 (2.6)	103.0 (8.0)	51.7 (8.9)
253	Pyridaben	0.0167	88.2 (1.9)	102.5 (8.4)	50.6 (11.1)
254	Fluoroglycofen-ethyl	0.2000	84.1 (11.6)	102.9 (14.2)	38.5 (9.4)
255	Bitertanol	0.0500	78.4 (11.3)	102.4 (11.0)	56.0 (6.8)
256	Etofenprox	0.0167	101.4 (3.4)	104.4 (6.8)	53.1 (11.5)
257	Cycloxydim	0.2000	23.6 (11.3)	64.0 (17.8)	4.1 (12.7)
258	Esfenvalerate	0.0667	78.5 (26.6)	101.0 (7.7)	46.1 (33.0)
259	alpha-Cypermethrin	0.0333	86.7 (4.5)	98.5 (7.0)	40.7 (11.4)
260	Difenconazole	0.1000	82.1 (12.1)	122.9 (24.5)	47.4 (9.0)
261	Flumiclorac-pentyl	0.0333	80.7 (8.8)	101.1 (11.9)	26.5 (15.1)
D Group					
262	Dimefox	0.0500	74.2 (6.9)	108.1 (49.1)	10.0 (5.5)
263	Disulfoton-sulfoxide	0.0333	87.2 (5.3)	108.4 (7.3)	114.4 (2.4)
264	Pentachlorobenzene	0.0167	80.4 (5.9)	101.1 (7.0)	74.9 (14.6)
265	Crimidine	0.0167	76.7 (13.4)	99.1 (8.7)	112.0 (1.6)
266	Chlorfenprop-methyl	0.0167	72.7 (13.2)	111.3 (4.6)	100.3 (5.8)
267	Thionazin	0.0167	70.4 (14.0)	110.9 (3.7)	124.0 (4.3)
268	2,3,5,6-Tetrachloroaniline	0.0167	72.0 (9.8)	110.5 (4.1)	83.8 (11.5)
269	tri- <i>n</i> -Butyl phosphate	0.0333	77.6 (12.8)	113.7 (4.9)	10.3 (44.8)
270	2,3,4,5-Tetrachloroanisole	0.0167	73.1 (10.2)	112.6 (4.3)	81.4 (12.1)
271	Pentachloroanisole	0.0167	76.3 (8.8)	109.9 (8.4)	81.7 (15.0)
272	Tebutam	0.0333	81.6 (6.5)	112.6 (4.8)	115.1 (3.4)
273	Dioxabenzofos	0.1667	77.6 (10.1)	116.3 (4.8)	112.3 (2.1)
274	Methabenzthiazuron	0.1667	94.6 (3.5)	114.4 (7.9)	128.3 (5.5)
275	Simeton	0.0333	75.5 (6.1)	97.7 (10.4)	92.1 (17.9)
276	Atratone	0.0167	86.9 (10.0)	100.5 (9.6)	109.5 (6.4)
277	Desisopropyl-atrazine	0.1333	64.3 (1.9)	109.0 (11.6)	76.8 (24.1)
278	Terbufos sulfone	0.0167	82.9 (8.0)	114.9 (4.7)	83.5 (10.3)
279	Tefluthrin	0.0167	102.2 (6.8)	113.7 (4.8)	74.3 (10.3)
280	Bromocycen	0.0167	88.2 (11.9)	111.6 (4.8)	86.2 (14.1)
281	Trietazine	0.0167	96.9 (5.8)	114.2 (5.4)	112.2 (2.9)
282	Etrimfos oxon	0.0167	94.6 (5.9)	114.6 (4.7)	101.7 (5.4)
283	Cycluron	0.0500	91.3 (2.8)	118.8 (10.2)	133.6 (9.1)
284	2,6-Dichlorobenzamide	0.0333	58.5 (2.0)	113.5 (11.2)	75.6 (50.2)
285	DE-PCB 28	0.0167	96.7 (6.9)	113.1 (4.6)	81.1 (14.0)
286	DE-PCB 31	0.0167	97.0 (6.8)	113.5 (4.9)	82.6 (14.5)
287	Desethyl-sebutylazine	0.0333	86.2 (3.5)	111.3 (8.0)	117.7 (2.0)
288	2,3,4,5-Tetrachloroaniline	0.0333	96.8 (7.2)	112.6 (5.9)	83.9 (9.4)
289	Pentachloroaniline	0.0167	95.0 (7.2)	116.5 (6.0)	82.9 (13.0)
290	Aziprotryne	0.1333	92.2 (10.0)	118.4 (3.2)	107.9 (1.3)
291	Sebutylazine	0.0167	99.9 (4.7)	114.7 (5.3)	114.2 (2.2)
292	Isocarbamid	0.0833	58.1 (17.3)	109.3 (11.5)	90.4 (21.1)
293	DE-PCB 52	0.0167	99.9 (8.8)	115.7 (5.8)	76.8 (12.9)
294	Prosulfocarb	0.0167	99.4 (5.5)	116.0 (3.9)	89.8 (9.9)
295	Dimethenamid	0.0167	96.4 (4.3)	113.6 (5.2)	116.4 (2.7)
296	Fenchlorphos oxon	0.0333	102.5 (7.1)	117.2 (4.1)	85.2 (11.1)
297	Paraoxon-methyl	0.0333	81.5 (5.3)	126.3 (10.7)	44.7 (20.1)

Table 3. Continued

no.	pesticide	spiked level (mg kg ⁻¹)	method A	method B	method C
			ave. (RSD%)	ave. (RSD%)	ave. (RSD%)
298	Monalide	0.0333	99.5 (5.6)	116.3 (4.7)	108.7 (5.2)
299	Isobenzan	0.0167	98.6 (7.5)	114.8 (3.1)	78.1 (11.5)
300	Octachlorostyrene	0.0167	92.2 (6.9)	111.0 (4.7)	66.0 (6.1)
301	Isodrin	0.0167	108.0 (6.7)	112.3 (6.0)	76.8 (13.0)
302	Isomethiozin	0.0333	91.1 (9.0)	107.1 (6.8)	51.8 (6.4)
303	Trichloronat	0.0167	103.8 (7.9)	114.7 (4.6)	81.7 (12.9)
304	Dacthal	0.0167	103.9 (6.3)	115.6 (5.4)	86.4 (11.2)
305	4,4-Dichlorobenzophenone	0.0167	103.3 (6.1)	119.3 (4.7)	87.4 (10.4)
306	Nitrothal-isopropyl	0.0333	105.7 (7.9)	125.7 (6.3)	90.0 (5.4)
307	Rabenzazole	0.0167	99.0 (5.7)	97.7 (16.6)	99.3 (4.9)
308	Cyprodinil	0.0167	103.4 (7.1)	104.8 (10.1)	99.7 (4.7)
309	Dicaphon	0.0833	102.3 (8.6)	126.6 (5.3)	95.9 (6.5)
310	DE-PCB 101	0.0167	103.2 (8.1)	113.3 (5.5)	70.8 (8.9)
311	MCPA-Butoxyethyl ester	0.0167	105.2 (7.2)	115.8 (5.1)	91.5 (7.0)
312	Phorate sulfone	0.0167	101.1 (5.4)	120.9 (6.5)	120.1 (4.2)
313	Chlorfenethol	0.0167	103.6 (6.7)	119.5 (5.1)	91.1 (6.1)
314	trans-Nonachlor	0.0167	104.0 (7.2)	114.4 (5.2)	71.4 (9.4)
315	DEF	0.0333	109.1 (7.0)	121.8 (5.2)	82.1 (6.9)
316	Fluorochloridone	0.0333	102.2 (5.2)	121.3(5.8)	103.1 (2.5)
317	Bromfenvinfos	0.0167	101.5 (5.8)	123.9 (5.6)	106.5 (6.4)
318	Perthane	0.0167	108.4 (7.2)	116.1 (4.5)	73.4 (8.6)
319	DE-PCB 118	0.0167	105.6 (8.0)	115.0 (6.0)	68.6 (6.5)
320	4,4-Dibromobenzophenone	0.0167	102.6 (7.2)	123.5 (3.4)	85.3 (10.6)
321	Flutriafol	0.0333	72.8 (2.7)	109.9 (8.6)	121.5 (9.3)
322	Mephosfolan	0.0333	69.6 (15.7)	123.1 (13.4)	133.2 (24.8)
323	Athidathion	0.0333	112.8 (11.4)	120.5 (15.2)	64.5 (6.3)
324	DE-PCB 153	0.0167	103.9 (7.0)	111.9 (4.8)	63.6 (3.1)
325	Diclobutrazole	0.0667	94.8 (3.3)	116.7 (9.5)	116.2 (8.5)
326	Disulfoton sulfone	0.0333	107.6 (4.1)	118.1 (7.2)	120.1 (7.1)
327	Hexythiazox	0.1333	104.5 (6.8)	123.8 (4.8)	92.7 (10.1)
328	DE-PCB 138	0.0167	104.2 (8.6)	114.5 (6.1)	64.2 (2.8)
329	Resmethrin-1	0.0333	28.8 (1.3)	103.9 (6.6)	50.2 (55.7)
330	Cyproconazole	0.0167	93.3 (2.7)	209.3 (32.0)	60.5 (8.0)
331	Resmethrin-2	0.0333	9.6 (34.4)	106.6 (7.8)	31.4 (49.2)
332	Phthalic acid,Benzyl butyl ester	0.0167	104.6 (5.2)	116.8 (5.5)	91.0 (7.0)
333	Clodinafop-propargyl	0.0333	101.7 (7.5)	140.8(7.8)	102.6 (11.9)
334	Fenthion sulfoxide	0.0667	81.0 (5.6)	112.2(7.8)	121.5 (4.0)
335	Fluotrimazole	0.0167	104.1 (6.1)	116.8(7.8)	96.2 (13.6)
336	Fluoroxypyr-1-methylheptyl ester	0.0167	105.7 (5.9)	123.3(5.0)	79.3 (9.8)
337	Fenthion sulfone	0.0667	95.7 (3.3)	119.5(7.3)	124.1 (5.6)
338	Triphenyl phosphate	0.0167	104.0 (6.8)	117.3(5.8)	93.2 (8.3)
339	Metamitron	0.1667	141.5 (19.5)	142.4(7.7)	240.6 (21.0)
340	DE-PCB 180	0.0167	94.1 (6.9)	109.4(3.4)	60.8 (6.4)
341	Tebufenpyrad	0.0167	106.1 (4.7)	117.5(5.1)	92.0 (11.4)
342	Cloquintocet-mexyl	0.0167	103.8 (5.2)	112.7(12.4)	96.0 (12.0)
343	Lenacil	0.1667	75.5 (5.7)	113.9(9.9)	122.0 (5.4)
344	Bromuconazole-1	0.0333	101.4 (4.0)	115.9(5.6)	121.1 (14.3)
345	Desbrom- leptophos	0.0167	106.4 (7.8)	121.6(4.6)	76.2 (9.8)
346	Bromuconazole-2	0.0333	92.9 (4.7)	115.8(8.3)	115.6 (6.5)
347	Nitralin	0.1667	105.6 (6.9)	128.3(9.1)	102.7 (6.3)
348	Fenamiphos sulfoxide	0.0667	30.0 (47.4)	130.1(30.0)	53.6 (74.1)
349	Fenamiphos sulfone	0.0667	78.2 (3.1)	117.7(14.0)	121.5 (12.3)
350	Fenpiclonil	0.0667	52.9 (16.7)	126.4(16.2)	114.7 (4.0)
351	Fluquinconazole	0.0167	92.1 (2.9)	115.6(7.3)	105.3 (2.2)
352	Fenbuconazole	0.0333	83.2 (6.8)	110.7(8.7)	107.4 (3.3)

Table 4. Statistical Distributions of Recoveries and RSD of Contrast Experiments Using Three Methods ($n = 9$)

experimental method	number of pesticides (percentage of pesticides)									
	average recoveries (%)						RSDs (%)			
	≤40	40–60	60–80	80–100	100–120	≥120	≤10	10–20	20–30	≥30
method A	5(1.4)	4(1.1)	52(14.8)	249(70.7)	39(11.1)	3(0.9)	287(81.5)	54(15.3)	6(1.7)	5(1.4)
method B	0	0	7(2.0)	70(19.9)	250(71.0)	25(7.1)	292(83.0)	48(13.6)	6(1.7)	6(1.7)
method C	18(5.1)	51(14.5)	106(30.1)	95(27.0)	68(19.3)	14(4.0)	262(74.4)	72(20.5)	7(2.0)	11(3.1)

Table 5. Recovery and Precision Data for the Residues of 352 Pesticides in Eight Varieties of Grapes by Method A

no.	pesticide	recovery (%)				recovery (%)				recovery (%)						
		low level (mg kg ⁻¹)	Dragon eye	Cabernet sauvignon ave.	RSD (%)	medium level (mg kg ⁻¹)	Vamu- rensis	Sirah Merlot	Gamay ave.	RSD (%)	high level (mg kg ⁻¹)	Muscat hambury	Vamu- rensis	Caberner france	Gamay ave.	RSD (%)
A Group																
1	Allidochlor	0.0333	76.6	73.0	14.6	0.0667	103.1	81.3	73.0	72.5	82.5	64.5	73.3	66.9	66.7	7.1
2	Etridiazol	0.0500	67.3	72.3	16.9	0.1000	94.7	74.6	60.4	62.8	73.1	76.9	66.9	76.6	73.9	6.4
3	Chlormephos	0.0333	73.3	83.9	77.5	0.0667	102.4	81.2	74.6	71.2	82.4	80.3	78.4	65.7	72.6	10.7
4	Propham	0.0167	86.1	90.7	115.3	0.0333	112.5	87.9	71.9	75.1	86.9	70.5	86.9	78.6	80.0	9.0
5	Cycloate	0.0167	81.3	89.4	100.8	0.0333	107.5	85.1	88.5	67.4	87.1	70.0	91.0	81.1	82.1	10.9
6	Diphenylamin	0.0167	97.5	110.3	113.7	0.0333	102.6	90.1	87.6	62.4	85.7	72.2	88.4	77.9	81.1	9.2
7	Ethalfuralin	0.0667	91.2	84.5	96.6	0.1333	113.1	86.3	86.3	72.8	89.6	79.8	97.5	89.8	89.2	8.1
8	Phorate	0.0667	83.5	88.3	100.6	0.1333	103.4	81.0	81.3	75.0	85.2	67.9	83.9	79.7	77.9	8.9
9	Thiometon	0.0167	80.3	82.7	93.8	0.0333	93.6	78.2	82.9	89.2	81.0	80.6	81.2	84.9	83.2	3.3
10	Quintozene	0.0167	107.6	110.9	97.9	0.0333	105.7	89.9	76.7	78.4	87.7	82.9	94.2	84.9	87.2	5.6
11	Atrazine-desethyl	0.0333	82.6	85.2	86.2	0.0667	88.7	100.0	86.3	84.6	89.9	72.3	77.4	95.5	78.2	15.7
12	Clomazone	0.0167	89.0	88.2	96.8	0.0333	104.6	89.0	84.9	77.7	89.1	80.6	89.3	88.3	87.2	5.1
13	Diazinon	0.0167	93.7	94.7	101.9	0.0333	106.1	93.3	92.0	85.8	94.3	92.4	97.1	94.9	92.5	7.4
14	Fomofos	0.0167	87.6	88.4	100.8	0.0333	105.1	89.7	82.0	80.1	89.2	93.7	91.8	85.5	90.2	3.9
15	Etrifos	0.0167	96.6	92.6	105.9	0.0333	105.4	93.7	82.3	87.0	92.1	94.9	98.3	94.5	95.8	1.8
16	Simazine	0.0167	105.8	88.3	103.7	0.0333	106.3	96.3	88.0	88.0	94.6	86.6	111.4	119.4	103.9	14.0
17	Propetamphos	0.0167	100.3	97.4	114.2	0.0333	116.5	100.8	89.7	91.5	99.7	80.4	82.8	100.1	86.9	4.3
18	Sebumeton	0.0167	88.6	85.8	101.4	0.0333	108.6	96.2	91.4	88.0	96.1	80.4	82.8	100.1	86.9	10.3
19	Dichlofenthion	0.0167	91.6	91.7	96.9	0.0333	90.6	92.0	84.0	87.7	88.6	80.7	93.5	89.2	93.5	13.3
20	Pronamide	0.0167	93.6	93.1	103.2	0.0333	90.0	93.8	87.7	86.1	89.4	81.7	93.5	95.1	91.4	7.1
21	Dimefthoate	0.0667	84.0	61.9	83.2	0.1333	79.2	98.5	89.6	81.5	87.2	70.5	72.5	104.2	81.4	19.2
22	Aldrin	0.0333	93.5	88.5	99.7	0.0667	96.6	87.9	78.0	82.5	86.3	87.2	92.5	87.3	89.8	3.2
23	Dinitramine	0.0667	96.5	93.2	107.4	0.1333	108.8	95.2	85.4	84.8	93.6	85.2	92.8	100.7	94.8	7.2
24	Ronnel	0.0333	94.3	93.2	102.2	0.0667	96.9	94.7	86.5	88.3	91.6	83.2	94.3	92.9	91.5	6.1
25	Prometrye	0.0167	82.6	75.8	91.2	0.0333	107.7	89.1	87.4	83.9	92.0	80.7	92.0	99.6	91.9	8.8
26	Cyprazine	0.0167	94.7	87.3	99.8	0.0333	90.6	91.9	90.6	90.1	90.8	79.3	92.5	98.9	90.9	9.1
27	Chlorothalonil	0.0333	0.0	0.0	0.0	0.0667	79.5	126.6	79.2	100.9	96.5	46.6	71.0	89.6	68.0	26.1
28	Vindozolin	0.0167	94.9	89.7	101.1	0.0333	107.0	93.4	89.2	92.1	95.4	80.1	93.2	92.7	89.7	7.1
29	beta-HCH	0.0167	88.4	81.1	90.7	0.0333	98.4	86.1	81.5	79.1	86.3	80.2	86.8	85.5	86.2	5.7
30	Metaxyl	0.0500	86.6	75.9	91.2	0.1000	98.4	92.3	89.8	86.0	91.6	70.5	83.0	94.2	83.7	11.9
31	Chlorpyrifos(-ethyl)	0.0167	94.2	96.4	106.9	0.0333	102.2	96.3	91.6	89.7	94.9	81.0	97.2	95.3	92.5	8.3
32	Methyl-parathion	0.0667	96.5	91.7	111.3	0.1333	101.6	103.7	87.3	88.4	95.3	80.0	97.2	108.5	103.1	4.3
33	Anthraquinone	0.0167	97.3	92.6	96.4	0.0333	99.0	106.9	88.7	87.3	95.5	73.5	88.3	92.1	86.0	9.8
34	delta-HCH	0.0333	100.6	89.8	107.5	0.0667	95.7	92.5	86.3	87.1	90.4	86.8	91.0	93.7	91.0	3.3
35	Fenithion	0.0167	87.8	87.6	96.2	0.0333	98.2	88.5	82.4	80.9	87.5	86.9	94.3	93.1	91.8	3.6
36	Malathion	0.0667	95.5	87.8	104.4	0.1333	87.9	100.7	88.6	89.2	91.6	63.8	99.8	100.5	90.9	19.9
37	Fenitrothion	0.0333	94.0	88.4	107.7	0.0667	101.4	102.4	90.2	91.2	96.3	67.6	100.5	104.7	98.9	18.4
38	Paraoxon-ethyl	0.0667	97.9	88.2	98.5	0.1333	90.0	89.7	88.9	97.8	92.2	97.6	98.9	99.0	98.2	0.9
39	Triadimefon	0.0333	95.7	83.9	102.1	0.0667	100.4	98.8	92.8	90.2	95.6	80.5	90.5	97.3	90.0	7.7
40	Parathion	0.0667	94.9	90.0	108.4	0.1333	104.4	100.7	90.9	90.5	96.6	82.5	101.8	104.8	97.1	10.3
41	Pendimethalin	0.0667	96.3	93.7	107.3	0.1333	105.3	100.8	91.4	92.3	97.4	86.5	100.3	103.1	96.4	7.5
42	Chlorbenside	0.0333	101.0	95.0	107.8	0.0667	98.8	100.2	87.4	88.4	93.7	86.9	91.6	94.7	91.7	3.7
43	Bromophos-ethyl	0.0167	90.9	94.4	103.3	0.0333	101.4	101.2	94.4	95.9	98.3	88.8	96.6	97.2	95.2	4.6

Table 5. Continued

no.	pesticide	recovery (%)					recovery (%)					recovery (%)										
		low level (mg kg ⁻¹)	Dragon eye	Cabernet sauvignon ave.	RSD (%)	medium level (mg kg ⁻¹)	Vamu- rensis	Sirah	Merlot	Gamay ave.	RSD (%)	high level (mg kg ⁻¹)	Muscat hambury	Vamu- rensis	Caberner france	Gamay ave.	RSD (%)					
44	Quinaphos	0.0167	98.0	91.2	108.5	91.6	97.3	8.3	0.0333	104.7	100.0	92.1	92.4	97.3	6.3	0.0667	98.8	92.2	96.4	101.4	97.2	4.0
45	trans-Chlodane	0.0167	92.7	90.0	98.8	93.1	93.6	4.0	0.0333	97.6	92.9	87.8	89.2	91.9	4.8	0.0667	94.6	84.0	92.0	92.5	90.8	5.1
46	Phenthoate	0.0333	98.8	91.6	107.1	95.1	98.2	6.8	0.0667	101.6	101.3	93.1	91.8	96.9	5.4	0.1333	98.9	92.0	99.5	99.2	97.4	3.7
47	Melazachlor	0.0500	92.7	83.4	97.9	76.9	87.7	10.7	0.1000	94.3	97.5	89.7	86.6	92.0	5.2	0.2000	94.2	92.5	91.9	99.3	94.5	3.6
48	Prothiophos	0.0167	93.4	96.6	109.9	104.2	102.0	6.1	0.0333	107.4	98.2	91.1	91.6	97.1	7.8	0.0667	97.9	89.9	98.2	98.0	96.0	4.2
49	Chlorfuretol	0.0500	97.5	83.8	101.3	86.4	91.2	8.6	0.1000	102.4	99.1	91.4	90.0	95.8	6.2	0.2000	96.3	80.4	94.1	99.2	92.5	9.0
50	Dieldrin	0.0333	92.3	75.4	83.6	87.5	84.7	8.4	0.0667	105.5	94.2	90.3	89.5	94.9	7.8	0.1333	93.7	81.4	93.1	94.8	90.8	6.9
51	Procyimidone	0.0167	93.5	93.9	98.2	87.1	93.2	4.9	0.0333	98.3	99.4	90.5	93.1	95.3	4.4	0.0667	94.0	84.2	92.9	96.1	91.8	5.7
52	Methidathion	0.0333	105.7	76.7	118.1	79.5	95.0	21.3	0.0667	98.2	76.4	93.2	91.7	89.9	10.4	0.1333	100.5	98.2	99.7	110.0	102.1	5.2
53	Cyanazine	0.0500	85.4	78.8	97.0	85.6	86.7	8.7	0.1000	79.4	84.7	89.9	88.1	85.5	5.4	0.2000	83.8	81.8	91.1	105.5	90.6	11.9
54	Napropamide	0.0500	95.7	84.7	101.4	84.2	91.5	9.3	0.1000	99.2	101.6	92.7	90.7	96.0	5.4	0.2000	95.6	92.6	92.5	99.2	95.0	3.3
55	Oxadiazone	0.0167	94.2	93.3	99.6	92.0	94.8	3.5	0.0333	102.4	100.8	93.6	92.1	97.2	5.3	0.0667	98.0	76.9	95.6	98.4	92.2	11.1
56	Fenamiphos	0.0500	85.0	86.3	97.0	80.8	87.3	7.9	0.1000	122.8	109.9	86.4	80.6	99.9	19.8	0.2000	90.0	90.5	95.4	108.6	96.1	9.0
57	Teprusal	0.0167	92.1	88.1	99.7	91.4	92.9	5.3	0.0333	95.8	101.1	100.9	100.2	99.5	2.5	0.0667	94.0	89.1	92.1	97.7	93.2	3.9
58	Aramite	0.0167	98.8	92.9	98.8	89.1	94.9	5.1	0.0333	106.8	114.2	94.3	94.6	102.5	9.5	0.0667	103.6	81.1	104.1	106.0	98.7	12.0
59	Bupirimate	0.0167	95.2	81.4	101.6	78.5	88.2	12.4	0.0333	105.1	100.9	91.6	90.5	97.0	7.4	0.0667	95.0	91.4	91.3	100.5	94.6	4.6
60	Carboxin	0.0500	63.6	60.7	71.7	67.5	65.9	7.3	0.1000	20.0	19.5	12.9	14.8	16.8	20.8	0.2000	33.9	7.0	23.3	47.4	27.9	61.1
61	Flutolanil	0.0167	94.2	80.9	102.1	77.7	88.8	12.9	0.0333	110.0	106.8	93.9	90.5	100.3	9.5	0.0667	95.0	85.4	94.3	98.7	93.4	6.1
62	p,p'-DDD	0.0167	94.0	91.4	101.6	97.8	96.2	4.6	0.0333	98.1	99.6	90.4	91.0	94.8	5.0	0.0667	97.5	95.2	95.3	97.5	96.4	1.4
63	Ethion	0.0333	94.9	81.7	96.1	93.6	91.6	7.3	0.0667	104.1	106.0	96.3	92.3	99.7	6.5	0.1333	96.8	89.7	94.4	101.3	95.5	5.1
64	Sulprofos	0.0333	94.0	87.9	96.3	97.5	94.7	5.3	0.0667	98.0	93.8	83.6	80.5	89.0	9.3	0.1333	93.1	86.3	95.3	97.6	93.1	5.3
65	Etaconazole-1	0.0500	103.4	92.0	111.8	88.7	99.0	10.8	0.1000	97.6	106.1	91.5	88.5	95.9	8.1	0.2000	109.9	76.6	100.3	122.8	102.4	19.1
66	Etaconazole-2	0.0500	103.1	92.2	111.4	78.7	96.3	14.7	0.1000	95.8	107.3	91.1	88.1	95.6	8.9	0.2000	107.8	77.7	100.1	121.0	101.7	17.9
67	Myclobutanil	0.0167	91.9	86.6	122.5	70.5	92.9	23.4	0.0333	82.7	96.2	83.1	79.4	85.3	8.7	0.0667	78.3	70.8	76.8	93.6	79.9	12.2
68	Dichlorofop-methyl	0.0167	97.3	77.0	94.4	97.9	91.6	10.8	0.0333	93.6	103.6	92.8	93.6	95.9	5.4	0.0667	96.2	97.1	97.3	97.1	96.9	0.5
69	Propiconazole	0.0500	86.6	78.5	77.7	60.8	75.9	14.3	0.1000	95.3	102.1	92.9	88.7	94.7	5.9	0.2000	85.6	83.5	85.6	97.5	88.0	7.2
70	Fenulfthion	0.0333	64.3	73.1	62.4	60.4	65.0	8.6	0.0667	76.6	77.9	87.6	85.6	81.9	6.7	0.1333	74.8	70.5	69.9	82.0	74.3	7.5
71	Bifenthrin	0.0167	94.7	87.8	94.9	97.2	93.7	4.3	0.0333	103.9	102.2	95.4	95.5	99.2	4.5	0.0667	97.1	90.1	98.6	101.3	96.8	4.9
72	Mirex	0.0167	92.5	87.8	93.4	86.3	90.0	3.9	0.0333	102.1	80.7	76.7	79.7	84.8	13.7	0.0667	94.9	81.4	81.7	85.4	85.8	7.4
73	Benodanil	0.0500	96.5	75.4	93.1	78.9	86.0	12.1	0.1000	94.5	102.1	91.6	89.0	94.3	6.0	0.2000	93.6	92.5	93.1	100.4	94.9	3.9
74	Nuarimol	0.0333	90.8	79.0	93.8	77.4	85.2	19.7	0.0667	92.4	97.3	86.5	82.5	89.7	7.2	0.1333	87.8	90.8	88.2	96.6	90.8	4.5
75	Methoxychlor	0.0167	85.2	61.7	71.8	62.4	70.3	15.6	0.0333	89.9	77.7	78.6	83.7	82.5	6.8	0.0667	97.5	62.3	77.8	60.3	74.5	23.1
76	Oxadixyl	0.0167	79.7	53.0	75.1	60.3	67.0	18.6	0.0333	65.4	92.6	84.1	76.6	79.7	14.5	0.0667	66.2	60.2	73.7	95.0	73.8	20.6
77	Tetraamethrin	0.0333	91.9	89.3	103.6	88.8	93.4	7.4	0.0667	100.1	103.0	92.4	91.8	96.8	5.8	0.1333	97.8	81.7	101.5	105.5	96.6	10.8
78	Tebuconazole	0.0500	90.1	73.8	90.3	70.5	81.2	12.9	0.1000	98.3	99.2	90.4	86.6	93.6	6.6	0.2000	78.5	70.6	81.4	99.8	82.6	15.0
79	Norfurazone	0.0167	84.1	62.6	76.7	54.2	69.4	19.5	0.0333	82.9	99.4	89.3	83.1	88.7	8.7	0.0667	79.8	74.6	80.7	99.5	83.7	13.0
80	Pyridaphenthion	0.0167	92.1	74.2	92.0	72.9	82.8	12.9	0.0333	98.0	120.5	97.2	93.6	102.3	12.0	0.0667	104.2	82.7	106.2	115.8	102.2	13.7
81	Phosmet	0.0333	93.2	83.4	100.0	75.5	88.0	12.2	0.0667	81.9	130.3	103.2	101.5	104.2	19.1	0.1333	112.9	82.6	117.8	133.6	111.7	19.1
82	Tetraclon	0.0167	110.8	95.1	117.0	122.0	111.2	10.5	0.0333	90.1	97.6	96.1	94.6	94.6	3.4	0.0667	94.2	76.4	95.9	97.5	91.0	10.8
83	cis-Permethrin	0.0167	86.5	83.2	90.6	96.0	89.1	6.2	0.0333	102.8	105.2	97.7	96.9	100.6	4.0	0.0667	102.5	90.1	101.7	104.8	99.8	6.6
84	trans-Permethrin	0.0167	91.2	88.4	99.2	101.9	95.2	6.7	0.0333	98.9	105.1	98.2	98.5	100.2	3.3	0.0667	101.3	89.6	99.0	103.3	99.3	6.1
85	Pyrazophos	0.0333	74.4	88.5	101.4	77.7	85.5	14.3	0.0667	94.2	110.3	99.7	97.8	100.5	6.9	0.1333	105.5	99.0	105.8	111.4	105.4	4.8
86	Cypermethrin	0.0500	88.4	95.2	105.9	105.4	98.7	8.6	0.1000	99.2	105.6	95.5	95.8	99.0	4.8	0.2000	103.7	84.7	102.3	103.2	98.5	9.3
87	Fenvalerate	0.0667	90.6	95.7	98.1	136.0	105.1	19.8	0.1333	95.0	97.7	94.1	97.2	96.0	1.8	0.2667	104.4	79.9	95.6	104.3	96.1	12.0

Table 5. Continued

no.	pesticide	recovery (%)						recovery (%)						recovery (%)																												
		Dragon eye			Cabernet sauvignon ave.			Muscat hambury			Cabernet france			Vamu- rensis			Muscat hambury			high level (mg kg ⁻¹)			RSD (%)			Gamay ave.			RSD (%)													
		low level (mg kg ⁻¹)																																								
B Group																																										
88	EPTC	0.0500	65.4	79.7	84.3	78.4	11.4	0.1000	77.9	61.2	75.6	68.1	70.7	10.7	0.2000	69.4	74.3	75.4	66.7	71.5	5.7																					
89	Butylate	0.0500	94.6	113.9	120.2	108.5	10.2	0.1000	83.1	79.7	84.8	90.6	84.5	5.4	0.2000	82.2	83.1	93.6	81.5	85.1	6.7																					
90	Dichlobenil	0.0033	72.2	114.9	87.7	96.2	20.7	0.0067	76.7	88.5	80.6	76.7	80.6	6.9	0.0133	69.1	84.4	73.8	69.9	74.3	9.5																					
91	Pebulate	0.0500	77.1	87.8	88.4	84.4	6.2	0.1000	82.3	74.6	75.0	85.3	79.3	6.7	0.2000	75.6	79.0	80.7	79.0	76.3	3.0																					
92	Nitrapyrin	0.0500	75.9	79.0	85.4	78.8	5.0	0.1000	71.6	70.4	77.9	72.2	74.3	5.1	0.2000	64.3	73.6	73.6	71.7	63.6	7.4																					
93	Mevinphos	0.0333	70.6	89.8	103.0	75.6	17.3	0.0667	78.0	91.2	82.9	62.8	78.7	15.2	0.1333	76.5	92.3	56.3	90.7	79.0	21.2																					
94	Chloroneb	0.0167	76.7	98.5	90.6	91.1	11.4	0.0333	88.7	80.7	64.6	67.2	75.3	15.1	0.0667	77.1	71.4	71.4	85.1	80.8	7.4																					
95	Teconazene	0.0333	89.4	94.4	91.5	93.3	3.9	0.0667	91.3	83.6	67.0	63.3	76.3	17.5	0.1333	78.1	79.7	79.7	86.8	82.5	4.6																					
96	Heptanophos	0.0500	81.3	97.4	82.0	90.2	11.1	0.1000	71.7	94.9	64.9	73.9	76.3	16.9	0.2000	83.8	71.6	71.6	95.0	95.3	86.4	13.0																				
97	Hexachlorobenzene	0.0167	115.7	97.5	99.3	101.0	10.3	0.0333	90.8	88.2	69.0	65.0	78.3	16.8	0.0667	76.4	77.8	77.8	83.2	84.2	4.8																					
98	Ethionophos	0.0500	89.2	95.0	104.8	78.3	12.1	0.1000	82.4	98.4	72.2	79.9	83.2	13.2	0.2000	87.7	70.5	70.5	94.0	95.8	87.5	13.3																				
99	cis-Diallate	0.0333	93.5	94.3	101.3	86.9	6.3	0.0667	91.8	92.7	86.0	72.0	85.6	11.2	0.1333	87.5	87.8	87.8	91.9	91.2	2.6																					
100	Propachlor	0.0500	85.6	95.3	97.9	90.3	8.2	0.1000	73.7	92.4	79.6	75.8	80.4	10.4	0.2000	86.4	73.6	73.6	82.5	81.8	4.6																					
101	trans-Diallate	0.0333	92.9	98.8	101.8	92.2	9.4	0.0667	91.6	96.3	80.1	75.6	85.9	11.3	0.1333	89.6	83.2	83.2	95.0	92.7	5.7																					
102	Trifluralin	0.0333	94.1	99.3	104.4	93.0	5.4	0.0667	92.9	103.8	79.3	81.2	89.3	12.7	0.1333	94.1	100.2	100.2	102.8	100.3	3.7																					
103	Chlorpropham	0.0333	88.7	101.7	107.9	91.9	9.0	0.0667	83.1	101.4	85.8	89.0	89.8	9.0	0.1333	96.1	81.9	81.9	100.2	98.8	4.2																					
104	Sulfotep	0.0167	90.3	98.7	98.8	90.7	5.0	0.0333	83.7	94.0	70.1	75.7	80.9	12.8	0.0667	92.2	76.4	76.4	96.2	93.7	10.0																					
105	Sulfallate	0.0333	92.2	100.8	102.1	93.0	5.3	0.0667	86.5	87.5	79.1	75.0	82.0	7.3	0.1333	83.3	85.7	85.7	92.3	91.1	4.9																					
106	alpha-FCH	0.0167	86.1	64.1	101.4	90.6	18.3	0.0333	87.0	107.4	70.8	76.2	85.3	18.9	0.0667	91.6	80.9	80.9	94.7	93.1	6.9																					
107	Tebuifos	0.0333	94.9	107.4	102.5	93.1	6.7	0.0667	90.3	82.6	78.1	72.9	81.0	9.1	0.1333	80.2	88.7	88.7	95.5	93.6	7.6																					
108	Terbuteton	0.0500	91.2	92.6	101.1	74.7	89.9	12.3	0.1000	59.5	101.7	96.4	95.3	97.8	3.5	0.2000	87.0	47.1	47.1	94.6	99.0	6.5																				
109	Profluralin	0.0667	95.0	102.2	113.1	92.1	100.6	9.3	0.1333	91.4	96.9	84.3	84.3	89.2	6.8	0.2667	95.4	91.7	91.7	102.8	100.2	5.1																				
110	Dioxathion	0.0667	105.6	101.2	113.3	86.9	101.8	10.9	0.1333	83.2	96.3	97.8	96.9	93.5	7.4	0.2667	98.4	88.4	88.4	97.5	93.9	4.8																				
111	Proprazine	0.0167	95.0	97.7	101.7	86.9	95.3	6.5	0.0333	82.9	103.2	95.0	96.3	94.3	9.0	0.0667	94.7	82.8	82.8	99.2	98.4	8.1																				
112	Chlorbutam	0.0333	89.8	106.0	111.4	103.3	102.6	9.0	0.0667	76.7	113.1	92.4	94.0	94.1	15.8	0.1333	99.7	86.4	86.4	109.2	109.9	10.3																				
113	Dicloran	0.0333	70.9	95.3	93.3	85.9	86.4	12.8	0.0667	76.6	97.6	81.0	82.5	84.4	10.8	0.1333	75.9	72.1	72.1	103.1	97.7	17.7																				
114	Monolinuron	0.0667	91.5	95.6	97.2	78.6	90.7	9.3	0.1333	64.2	98.1	82.5	84.8	82.4	16.9	0.2667	92.9	60.8	60.8	101.4	99.0	4.5																				
115	Flufenoxuron	0.0500	100.2	101.4	106.1	106.1	100.4	4.9	0.1000	75.0	136.5	84.1	96.3	98.0	27.7	0.2000	111.5	121.2	121.2	129.0	105.1	9.0																				
116	Cyanohos	0.0333	92.7	97.7	105.0	82.8	94.5	9.8	0.0667	76.0	95.6	82.8	85.2	84.9	9.6	0.1333	93.6	73.2	73.2	98.3	97.9	13.1																				
117	Chlorpirifos-methyl	0.0167	93.1	96.8	101.9	88.5	95.1	6.0	0.0333	87.9	98.8	85.5	86.8	89.7	6.8	0.0667	96.4	84.8	84.8	100.3	100.4	7.7																				
118	Desmetryn	0.0167	81.2	86.5	96.7	65.9	82.6	15.5	0.0333	60.9	97.7	91.2	91.9	85.4	19.5	0.0667	87.4	51.1	51.1	91.6	96.1	4.7																				
119	Dimethachloro	0.0500	88.5	91.2	97.5	67.2	86.1	15.3	0.1000	70.0	99.7	89.6	91.3	87.6	14.3	0.2000	95.3	73.4	73.4	99.2	98.7	13.4																				
120	Alachlor	0.0500	87.8	93.6	99.9	78.3	89.9	10.2	0.1000	82.8	99.1	91.5	92.8	91.5	7.3	0.2000	95.4	78.7	78.7	97.9	96.8	9.8																				
121	Pirimiphos-methyl	0.0167	109.0	99.2	105.2	86.2	99.9	10.0	0.0333	86.0	101.2	92.5	92.0	92.9	6.7	0.0667	95.7	87.5	87.5	100.1	98.5	5.9																				
122	Terbutryn	0.0333	94.7	98.7	103.8	75.1	93.1	13.5	0.0667	77.5	102.9	94.5	93.0	92.0	11.5	0.1333	93.2	75.3	75.3	95.3	97.8	11.3																				
123	Thiencarb	0.0333	96.6	101.8	106.1	85.2	97.4	9.3	0.0667	90.0	101.3	90.9	91.7	93.5	5.6																											

Table 5. Continued

no.	pesticide	recovery (%)				recovery (%)				recovery (%)												
		low level (mg kg ⁻¹)	Dragon eye	Cabernet sauvignon ave.	RSD (%)	medium level (mg kg ⁻¹)	Vamu-rensis	Sirah Merlot	Gamay ave.	RSD (%)	high level (mg kg ⁻¹)	Muscat hambury	Vamu-rensis	Cabernet france	Gamay ave.	RSD (%)						
132	Isopropalin	0.0333	96.7	100.7	109.6	94.0	100.2	6.8	0.0667	92.0	107.0	95.9	96.1	97.8	6.6	0.1333	98.6	97.9	106.6	105.3	102.1	4.4
133	Chlorthiamid	0.5334	88.7	97.7	114.5	89.2	97.5	12.4	1.0668	90.7	119.3	95.4	81.8	96.8	16.5	2.1336	139.4	96.0	77.0	110.1	105.6	24.9
134	endosulfan-I	0.1000	95.9	101.0	104.8	99.7	100.4	3.6	0.2000	93.8	96.1	89.3	92.3	92.9	3.0	0.4000	97.4	86.6	98.4	98.7	95.3	6.1
135	Propanil	0.0333	81.5	94.9	104.4	86.6	91.8	10.9	0.0667	67.2	113.9	90.0	90.1	90.3	21.1	0.1333	90.4	66.4	99.7	98.3	88.7	17.4
136	Isfenphos	0.0333	97.8	105.3	106.7	89.6	99.8	7.9	0.0667	85.0	103.1	94.2	94.8	94.3	7.7	0.1333	97.5	81.3	102.2	101.9	95.7	10.3
137	Cruformate	0.1000	82.5	89.5	107.7	67.6	88.8	19.2	0.2000	62.2	111.5	93.2	95.1	90.5	22.7	0.4000	90.7	60.8	105.0	107.5	91.0	23.6
138	Chlorfenvinphos	0.0500	92.2	98.5	105.9	75.8	93.1	13.8	0.1000	75.9	104.5	93.6	93.8	91.9	12.9	0.2000	96.2	62.7	103.2	102.6	100.7	3.9
139	dis-Chlordane	0.0333	95.8	99.6	103.7	93.2	98.1	4.7	0.0667	94.3	98.4	91.5	91.4	93.9	3.5	0.1333	97.0	90.0	97.6	97.7	95.6	3.9
140	Tolylfluamide	0.0500	78.3	95.7	93.9	82.5	87.6	9.7	0.1000	73.6	94.6	78.9	87.0	83.5	11.1	0.2000	91.8	69.9	92.6	89.3	85.9	12.5
141	p,p'-DDE	0.0167	98.3	101.1	104.0	92.0	98.9	5.2	0.0333	97.7	99.9	93.4	93.9	96.2	3.2	0.0667	97.2	93.4	100.2	100.8	97.9	3.5
142	Butachlor	0.0333	93.2	96.4	108.5	86.8	96.2	9.5	0.0667	88.7	103.8	94.3	94.2	95.2	6.6	0.1333	97.6	83.8	99.4	99.9	95.2	8.0
143	Chlortalat	0.0333	98.7	102.6	110.3	87.5	99.8	9.5	0.0667	78.5	102.4	96.6	95.0	93.1	11.0	0.1333	92.8	74.3	98.1	98.5	90.9	12.5
144	Crotoxyphos	0.1000	86.3	97.8	118.6	67.4	92.5	23.2	0.2000	56.5	119.2	85.3	89.2	87.5	29.3	0.4000	97.3	56.7	112.3	115.4	95.4	28.3
145	Iodofenphos	0.0333	93.8	97.8	108.8	93.2	98.4	7.4	0.0667	90.4	108.4	92.4	89.5	95.2	9.3	0.1333	101.1	94.6	104.2	104.1	101.0	4.5
146	Tetrachlorvinphos	0.0500	88.4	96.8	110.1	74.7	92.5	16.0	0.1000	71.9	108.2	88.6	90.6	89.8	16.5	0.2000	98.5	63.9	108.4	108.3	105.1	5.4
147	Profenofos	0.1000	89.4	102.7	109.4	88.4	97.5	10.5	0.2000	86.2	108.7	92.6	90.7	94.5	10.4	0.4000	97.1	79.5	102.1	104.0	95.7	11.7
148	Fluorochloridone	0.0333	106.9	97.1	117.5	95.2	104.1	9.8	0.0667	85.2	85.3	83.5	77.8	82.9	4.3	0.1333	100.4	86.8	100.3	117.9	101.4	12.6
149	Buprofazin	0.0333	94.4	100.7	107.1	78.6	95.2	12.8	0.0667	81.6	108.7	98.5	93.9	95.7	11.7	0.1333	96.7	79.0	99.6	99.7	93.8	10.6
150	o,p'-DDD	0.0167	98.3	108.1	108.1	92.8	101.8	7.5	0.0333	96.6	108.2	104.4	98.6	102.0	5.2	0.0667	103.3	110.4	118.6	108.5	110.2	5.8
151	Endrin	0.2000	96.7	98.1	102.3	86.7	95.9	6.9	0.4000	83.0	96.0	86.1	88.8	88.5	6.3	0.8000	95.6	84.3	97.2	97.6	93.7	6.7
152	Hexaconazole	0.1000	95.9	100.7	113.5	69.7	95.0	19.3	0.2000	45.8	100.4	95.9	93.3	96.5	3.7	0.4000	89.1	50.9	94.7	98.1	94.0	4.8
153	Chlorfenson	0.0333	116.9	134.3	140.2	102.7	123.5	13.8	0.0667	87.7	98.5	92.5	88.3	91.7	5.4	0.1333	99.3	85.2	103.1	101.6	97.3	8.5
154	o,p'-DDT	0.0333	95.0	94.9	97.7	79.9	91.9	8.8	0.0667	79.2	82.5	71.2	77.6	77.6	6.1	0.1333	95.2	81.1	89.5	85.1	87.8	6.9
155	Paclobutrazol	0.0500	81.9	83.8	95.5	54.3	78.9	22.1	0.1000	31.4	107.8	95.6	93.5	99.0	7.8	0.2000	83.8	83.1	95.2	97.8	90.0	8.5
156	Methoprotine	0.0500	90.6	88.9	96.2	92.0	91.9	3.4	0.1000	60.4	106.2	97.0	94.2	89.5	22.4	0.2000	88.0	52.7	94.1	98.5	93.5	5.7
157	Chlorpropylate	0.0167	95.5	100.2	106.9	89.7	98.1	7.4	0.0333	89.4	109.4	94.1	94.3	96.8	9.0	0.0667	97.4	89.4	101.9	101.0	97.4	5.8
158	Flamprop-methyl	0.0167	91.3	98.6	105.2	73.9	92.3	14.7	0.0333	68.4	107.8	96.1	94.4	91.7	18.1	0.0667	96.1	64.5	100.0	99.7	90.1	19.0
159	Nitrofen	0.1000	89.7	100.5	108.3	92.8	97.8	8.5	0.2000	90.4	115.2	91.4	94.2	97.8	12.0	0.4000	95.4	91.6	106.0	106.2	99.8	7.5
160	Oxyfluorfen	0.0667	93.6	98.6	113.8	95.6	100.4	9.1	0.1333	86.0	112.6	97.1	98.3	98.5	11.1	0.2667	97.2	81.6	103.2	104.0	96.5	10.7
161	Chlorthiophos	0.0500	97.5	100.5	105.4	93.3	99.2	5.1	0.1000	91.5	108.2	95.8	96.2	94.9	8.0	0.2000	96.0	88.3	100.0	100.1	96.1	5.8
162	endosulfan-II	0.1000	89.1	96.3	85.2	83.8	88.6	6.3	0.2000	84.8	103.2	95.2	96.2	94.9	8.0	0.4000	94.8	81.0	97.1	101.8	93.7	9.6
163	Flamprop-Isopropyl	0.0167	93.1	98.6	108.5	79.4	94.9	12.8	0.0333	76.5	108.1	94.8	94.9	93.6	13.9	0.0667	97.8	76.3	102.3	101.0	94.3	12.9
164	p,p'-DDT	0.0333	99.2	96.7	103.9	78.6	94.6	11.7	0.0667	73.4	74.6	66.2	73.0	71.8	5.3	0.1333	94.4	77.5	86.1	80.9	84.7	8.7
165	Carbendofen	0.0333	101.3	100.7	110.1	95.0	101.8	6.1	0.0667	92.6	108.9	94.2	93.9	97.4	7.9	0.1333	96.4	92.3	101.4	100.9	97.7	4.4
166	Benalaxyl	0.0167	91.7	100.6	106.9	75.8	93.8	14.4	0.0333	73.2	108.7	97.7	98.3	94.5	15.9	0.0667	95.2	61.1	99.5	90.8	95.2	4.5
167	Ectfenphos	0.0333	89.3	95.3	107.7	76.4	92.2	14.1	0.0667	78.4	116.7	91.5	91.1	94.4	17.0	0.1333	97.6	77.8	111.2	112.2	99.7	16.1
168	Triazophos	0.0500	92.8	93.2	103.4	75.8	91.3	12.5	0.1000	70.0	111.6	96.3	96.4	93.6	18.5	0.2000	96.1	61.1	103.6	103.9	101.2	4.3
169	Cyanofenphos	0.0167	96.2	104.9	109.4	94.1	101.2	7.2	0.0333	83.3	106.1	95.6	94.2	94.8	9.9	0.0667	97.1	86.0	100.8	99.8	95.9	7.1
170	Chlorbenside sulfone	0.0333	91.7	103.1	107.0	80.3	95.5	12.6	0.0667	76.1	106.3	94.6	93.7	92.7	13.5	0.1333	96.2	74.8	100.7	100.1	92.9	13.2
171	Endosulfan-sulfate	0.0500	95.0	102.3	108.6	80.4	96.6	12.5	0.1000	76.2	100.9	95.8	95.6	92.1	11.8	0.2000	96.5	67.8	98.1	100.5	90.7	17.0
172	Bromopropylate	0.0333	98.8	101.7	108.6	94.0	100.8	6.1	0.0667	90.5	106.5	95.5	94.8	96.8	7.1	0.1333	98.4	89.7	101.9	103.9	98.5	6.4
173	Benzoilprop-ethyl	0.0500	90.4	99.1	105.6	78.4	93.4	12.6	0.1000	75.2	104.9	96.3	96.9	93.3	13.6	0.2000	96.0	74.5	99.0	100.1	92.4	13.0
174	Fenprothathrin	0.0333	100.4	107.3	107.7	96.5	103.0	5.3	0.0667	95.0	116.2	101.9	101.6	103.7	8.6	0.1333	96.6	86.0	100.2	100.0	95.7	7.0
175	Leptophos	0.0333	96.8	102.3	98.0	94.2	97.8	3.5	0.0667	93.6	106.8	96.2	94.6	97.8	6.2	0.1333	94.7	92.0	100.9	100.1	96.9	4.4
176	EPN	0.0667	94.2	102.8	112.2	100.8	102.5	7.2	0.1333	86.3	113.7	96.0	102.4	99.6	11.5	0.2667	96.3	87.8	110.2	105.1	99.9	9.9
177	Hexazinone	0.0500	69.5	78.8	83.8	61.6	73.4	13.4	0.1000	61.0	97.4	93.1	89.7	85.3	19.4	0.2000	68.3	61.7	85.3	95.9	77.8	20.1

Table 5. Continued

no.	pesticide	recovery (%)					recovery (%)					recovery (%)										
		Dragon eye	Cabernet sauvignon ave.	Muscata hambury	Cabernet sauvignon ave.	RSD (%)	medium level (mg kg ⁻¹)	Vamu-rensis	Sirah Merlot	Gamay ave.	RSD (%)	high level (mg kg ⁻¹)	Muscata hambury	Vamu-rensis	Cabernet france	Gamay ave.	RSD (%)					
178	Bifenox	0.0333	94.4	98.9	114.9	112.4	105.2	9.6	0.0667	84.1	112.2	93.4	94.4	96.0	12.2	0.1333	92.1	83.7	112.1	108.9	99.2	13.6
179	Phosalone	0.0333	96.9	101.9	105.3	84.3	97.1	9.5	0.0667	82.5	106.9	94.6	93.3	94.3	10.6	0.1333	98.3	76.4	105.3	105.3	96.3	14.2
180	Azinphos-methyl	0.1000	98.5	108.8	113.3	75.0	98.9	17.3	0.2000	61.3	124.3	92.1	95.8	93.4	27.6	0.4000	94.4	61.1	102.2	108.2	91.5	23.0
181	Fenarimol	0.0333	90.3	99.8	102.1	69.5	90.4	16.4	0.0667	55.4	99.8	92.4	91.1	94.4	5.0	0.1333	86.3	56.9	94.7	95.6	83.4	21.8
182	Azinphos-ethyl	0.0333	85.1	92.5	98.0	69.3	86.2	14.4	0.0667	73.9	108.5	92.8	91.0	91.6	15.4	0.1333	95.3	67.3	104.4	100.5	91.9	18.3
183	Prochloraz	0.1000	78.1	78.2	92.4	82.0	82.7	8.2	0.2000	87.3	78.3	93.3	86.6	86.4	9.1	0.4000	57.0	78.4	76.5	107.7	79.9	26.2
184	Cyfluthrin	0.2000	100.5	105.8	111.3	96.0	103.4	6.4	0.4000	90.2	111.9	95.7	96.6	98.6	9.4	0.8000	92.7	85.5	102.5	102.7	95.8	8.7
185	Coumaphos	0.1000	94.8	101.2	105.7	76.8	94.6	13.4	0.2000	72.5	106.2	95.7	95.3	92.4	15.4	0.4000	95.2	63.3	100.4	103.4	99.7	4.2
186	Fluvalinate	0.2000	99.4	110.8	113.9	98.6	105.7	7.4	0.4000	87.1	108.1	93.0	95.1	95.8	9.3	0.8000	94.0	84.0	98.7	97.1	93.4	7.1
C Group																						
187	Dichlorvos	0.1000	67.5	68.8	84.9	64.9	71.5	12.7	0.2000	89.5	66.8	66.7	60.5	70.9	18.0	0.4000	78.1	70.4	73.8	69.9	73.1	5.2
188	Biphenyl	0.0167	88.4	85.2	89.8	99.3	90.7	6.7	0.0333	91.0	88.4	80.2	83.4	85.7	5.7	0.0667	90.9	95.6	97.5	97.2	95.3	3.2
189	Vernolate	0.0167	75.8	78.4	92.8	95.2	85.6	11.5	0.0333	98.0	70.1	69.2	63.2	75.1	20.7	0.0667	76.7	70.2	83.3	62.1	73.1	12.4
190	3,5-Dichloroaniline	0.0167	91.9	88.6	88.5	80.3	85.8	5.6	0.0333	87.0	83.0	85.0	88.8	86.0	2.9	0.0667	75.6	77.4	78.5	75.7	76.8	1.8
191	Molinate	0.0167	75.5	83.8	93.4	92.0	86.2	9.6	0.0333	101.0	74.0	73.4	75.6	81.0	16.5	0.0667	81.7	80.5	88.5	77.4	82.0	5.7
192	Methacrifos	0.0167	79.7	88.8	94.8	96.0	89.8	8.3	0.0333	107.8	77.9	79.7	79.0	86.1	16.8	0.0667	90.6	82.6	93.6	81.7	87.1	6.8
193	2-Phenylphenol	0.0167	85.5	83.8	93.5	92.5	88.8	5.5	0.0333	100.6	87.5	78.1	75.8	85.5	13.2	0.0667	88.0	81.5	95.7	74.7	84.9	10.6
194	cis-1,2,3,6-Tetrahydrophthalimide	0.0500	83.4	75.3	61.3	79.2	74.8	12.8	0.1000	65.3	75.7	86.0	79.8	76.7	11.4	0.2000	69.2	60.6	68.8	73.5	68.1	8.0
195	Fenobucarb	0.0333	93.8	90.8	97.2	85.2	91.8	5.5	0.0667	96.2	92.3	90.1	88.0	91.6	3.9	0.1333	89.5	80.2	95.1	86.7	87.9	7.1
196	Benfluralin	0.0167	90.3	96.4	100.7	104.3	97.9	6.2	0.0333	102.5	95.0	91.1	87.4	94.0	6.8	0.0667	92.2	102.2	100.8	83.9	94.8	9.0
197	Hexaflumuron	0.1000	114.7	97.3	100.8	108.7	105.4	7.4	0.2000	85.5	176.7	115.5	131.2	127.2	29.9	0.4000	64.4	114.8	120.0	111.3	115.4	3.8
198	Prometon	0.0500	94.8	79.2	86.3	80.3	85.2	8.4	0.1000	74.8	105.8	107.4	106.8	98.7	16.2	0.2000	87.7	72.0	76.8	96.5	83.3	13.2
199	Triallate	0.0333	94.8	97.8	99.0	103.8	98.8	3.8	0.0667	106.5	95.1	89.8	86.8	94.5	9.2	0.1333	92.5	85.7	97.7	86.1	90.5	6.3
200	Pyrimethanil	0.0167	90.8	80.2	92.4	69.9	83.3	12.5	0.0333	95.0	104.0	101.0	103.0	100.8	4.0	0.0667	89.7	70.2	89.2	93.8	85.7	12.3
201	gamma-HCH	0.0333	98.0	94.4	97.9	97.4	97.0	1.8	0.0667	99.4	74.6	75.6	75.8	81.4	14.8	0.1333	91.2	73.3	86.5	78.0	82.7	10.3
202	Disulfoton	0.0167	73.3	72.8	68.2	78.8	73.3	5.9	0.0333	76.5	73.6	74.5	68.2	73.2	4.8	0.0667	89.9	72.0	86.5	70.1	79.6	12.6
203	Atrazine	0.0167	95.2	88.8	95.9	84.7	91.1	5.9	0.0333	90.4	105.6	104.2	102.4	100.6	6.9	0.0667	90.4	76.0	92.0	96.1	88.6	9.9
204	Heptachlor	0.0500	93.8	96.3	98.2	97.7	96.5	2.0	0.1000	107.4	71.7	73.1	69.6	71.5	2.5	0.2000	89.2	82.9	87.5	73.4	83.2	8.5
205	Iprobenfos	0.0500	95.0	94.0	102.5	81.5	93.2	9.3	0.1000	103.0	121.3	117.1	118.6	115.0	7.1	0.2000	88.4	83.1	92.2	95.6	89.8	5.9
206	Isazofos	0.0333	84.9	83.8	99.1	102.5	81.5	8.7	0.0667	101.4	93.8	91.0	89.6	93.9	5.6	0.1333	95.9	84.0	95.7	88.3	91.0	6.4
207	Pilfenate	0.0333	95.0	126.6	91.1	93.7	101.6	16.5	0.0667	95.4	62.1	69.0	78.4	76.2	18.9	0.1333	83.7	71.3	80.1	67.9	75.7	9.7
208	Fenpropiimorph	0.0167	86.7	71.2	83.1	70.4	77.8	10.6	0.0333	70.0	96.3	92.1	97.2	88.9	14.4	0.0667	87.9	65.2	61.8	89.9	76.2	19.4
209	Transfluthrin	0.0167	96.4	94.5	101.5	102.3	98.7	3.9	0.0333	104.1	100.9	100.4	96.2	100.4	3.2	0.0667	95.0	83.8	97.8	94.3	92.7	6.6
210	Fluchloralin	0.0667	80.7	91.2	96.2	99.1	91.8	8.8	0.1333	102.2	97.0	98.9	94.5	98.2	3.3	0.2667	95.4	81.2	99.1	93.3	92.3	8.4
211	Tolclofos-methyl	0.0167	98.6	95.4	107.7	100.5	100.5	5.2	0.0333	101.4	94.9	95.3	92.4	96.0	4.0	0.0667	93.3	77.4	96.6	89.6	89.2	9.4
212	Ametryn	0.0500	96.0	82.1	91.3	60.6	82.5	19.0	0.1000	86.5	102.1	102.0	100.9	97.9	7.8	0.2000	89.7	72.3	84.3	94.6	85.2	11.3
213	Simetryn	0.0333	95.0	60.7	85.2	62.8	75.9	22.2	0.0667	78.3	100.3	104.0	101.9	96.1	12.4	0.1333	86.4	60.5	76.1	92.2	78.8	17.6
214	Metobromuron	0.1000	93.8	83.9	92.0	72.8	85.6	11.2	0.2000	93.4	93.1	105.1	107.4	99.7	7.6	0.4000	82.1	75.7	83.0	89.3	82.5	6.7
215	Metribuzin	0.0500	95.7	79.0	86.3	64.5	81.4	16.2	0.1000	80.2	87.1	99.3	95.8	90.6	9.5	0.2000	86.2	68.4	79.0	90.3	81.0	11.9
216	Dipropetryn	0.0167	94.9	89.5	71.7	88.3	71.7	13.0	0.0333	98.0	103.3	105.0	100.2	101.6	3.1	0.0667	92.2	69.2	92.2	95.0	87.2	13.8
217	Fomothion	0.0333	54.0	70.8	71.7	42.5	59.8	23.6	0.0667	60.5	29.0	71.2	53.3	53.5	33.5	0.1333	49.9	60.7	42.7	48.0	50.2	15.1
218	Diethofencarb	0.1000	97.6	87.7	99.4	78.8	90.9	10.5	0.2000	90.9	106.1	108.1	106.4	102.9	7.8	0.4000	90.9	86.7	94.0	98.9	92.6	5.6
219	Bioallethrin-1	0.0667	66.4	66.1	82.3	75.8	72.7	10.8	0.1333	103.1	95.0	113.9	124.0	109.0	11.6	0.2667	83.4	71.1	86.5	90.5	82.9	10.1
220	Bioallethrin-2	0.0667	84.3	90.4	111.0	97.5	95.8	12.0	0.1333	92.4	150.0	96.7	121.8	115.2	23.0	0.2667	86.6	70.1	82.3	82.1	80.3	8.8

Table 5. Continued

no.	pesticide	recovery (%)						recovery (%)						recovery (%)								
		low level (mg kg ⁻¹)	Dragon eye	Caberner hambury	Muscato hambury	Cabernet sauvignon	ave.	RSD (%)	medium level (mg kg ⁻¹)	Vamu- rensis	Sirah	Merlot	Gamay	ave.	RSD (%)	high level (mg kg ⁻¹)	Muscato hambury	Vamu- rensis	Caberner france	Gamay	ave.	RSD (%)
221	o,p'-DDE	0.0167	101.0	99.7	102.8	106.6	102.5	2.9	0.0333	107.2	99.8	100.7	98.3	101.5	3.8	0.0667	94.1	90.6	99.0	95.2	94.7	3.6
222	Fenson	0.0167	116.4	113.6	117.5	101.8	112.3	6.4	0.0333	99.5	90.4	100.1	98.9	97.2	4.7	0.0667	92.3	72.7	96.7	92.3	88.5	12.2
223	Diphenamid	0.0167	94.9	80.2	91.9	65.2	83.1	16.2	0.0333	85.2	101.3	100.6	99.7	96.7	8.0	0.0667	90.5	73.4	83.5	95.8	85.8	11.3
224	Chlorothion	0.0333	92.3	88.7	101.5	95.7	94.5	5.8	0.0667	99.2	117.1	112.3	111.8	110.1	7.0	0.1333	89.0	67.3	95.1	94.9	86.6	15.2
225	Prallethrin	0.0500	78.6	69.8	87.1	74.4	77.5	9.5	0.1000	99.6	106.5	95.4	102.0	100.9	4.6	0.2000	93.1	68.1	96.1	99.2	89.1	16.0
226	Penconazole	0.0500	91.8	70.9	77.0	70.3	77.5	12.9	0.1000	76.7	102.5	104.0	103.5	96.7	13.8	0.2000	86.2	82.4	78.5	95.3	85.6	8.4
227	Mecarbam	0.0667	89.9	85.3	109.3	81.6	91.5	13.5	0.1333	90.9	104.9	100.8	102.2	99.7	6.1	0.2667	95.1	61.2	97.1	98.5	96.9	1.8
228	Tetraconazole	0.0500	87.2	62.1	74.3	80.6	76.0	14.1	0.1000	86.9	101.8	101.6	99.7	97.5	7.3	0.2000	84.7	78.5	70.0	93.3	81.6	12.1
229	Flumetralin	0.0333	92.1	93.2	102.0	102.2	97.4	5.6	0.0667	104.0	122.8	108.8	107.2	110.7	7.5	0.1333	93.1	80.9	97.4	96.6	92.0	8.3
230	Triadimenol	0.0500	88.9	65.3	73.2	80.2	76.9	13.1	0.1000	74.5	111.5	104.4	103.3	98.4	16.6	0.2000	78.2	76.4	70.8	95.4	80.2	13.2
231	Pretilachlor	0.0333	98.2	93.3	103.2	93.7	97.1	4.8	0.0667	101.9	110.4	103.5	102.6	104.6	3.8	0.1333	95.4	71.4	98.9	98.6	91.1	14.5
232	Kresoxim:methyl	0.0167	113.6	98.7	100.7	101.9	103.7	6.5	0.0333	92.5	109.1	102.3	103.4	101.8	6.8	0.0667	95.0	95.7	98.4	97.1	96.6	1.6
233	Fluzifop-butyl	0.0167	101.9	98.0	101.5	101.5	100.7	1.8	0.0333	100.1	110.7	104.7	103.9	104.8	4.2	0.0667	95.3	82.7	100.2	97.8	94.0	8.3
234	Chlorfluazuron	0.0500	100.1	111.1	89.6	81.1	95.5	13.6	0.1000	78.8	171.4	171.8	124.1	136.5	32.6	0.2000	74.9	76.7	81.5	102.2	83.8	15.0
235	Chlorobenzilate	0.0167	98.4	94.5	101.9	102.1	99.2	3.6	0.0333	102.7	113.6	106.8	105.1	107.1	4.4	0.0667	93.5	81.4	99.5	98.8	93.3	9.0
236	Flusilazole	0.0500	89.6	64.6	70.9	90.3	78.8	16.6	0.1000	74.2	109.4	106.5	102.3	98.1	3.6	0.2000	84.6	80.2	70.5	96.6	83.0	13.0
237	Fluoridifen	0.0167	100.3	95.6	92.9	100.5	97.3	3.8	0.0333	92.6	145.7	112.9	152.1	116.6	18.8	0.0667	88.1	65.2	89.0	93.2	83.9	15.1
238	Dimiconazole	0.0500	93.8	73.5	83.5	60.0	77.7	18.5	0.1000	50.2	120.4	110.6	108.3	113.1	5.7	0.2000	85.1	59.8	83.0	96.7	81.1	19.0
239	Piperonyl butoxide	0.0167	89.9	92.4	94.7	99.6	94.2	4.4	0.0333	105.2	126.2	110.3	110.7	113.1	8.0	0.0667	93.9	94.1	97.9	95.0	90.0	2.0
240	Propargite	0.0333	112.7	114.2	102.9	135.5	116.3	11.8	0.0667	83.8	78.9	77.1	77.2	79.3	4.0	0.1333	92.3	96.9	107.4	89.0	96.4	8.3
241	Mepronil	0.0167	96.4	86.0	94.2	74.6	87.8	11.2	0.0333	90.7	106.8	98.2	94.1	97.5	7.1	0.0667	80.2	80.4	82.8	94.7	84.5	8.1
242	Diflufenican	0.0167	104.4	98.4	104.5	99.9	101.8	3.1	0.0333	98.9	110.9	104.2	104.8	104.7	4.7	0.0667	94.0	72.3	100.4	99.0	91.4	14.3
243	Fenazaquin	0.0167	98.5	95.6	107.6	90.5	98.0	7.3	0.0333	95.7	111.7	106.0	104.4	104.4	6.4	0.0667	93.8	77.4	97.2	100.5	92.2	11.1
244	Phenothrin	0.0167	94.6	83.2	94.4	98.2	92.6	7.0	0.0333	102.3	117.9	97.7	103.3	105.3	8.3	0.0667	97.4	93.2	104.0	101.3	99.0	4.8
245	Fludioxonil	0.0167	123.2	84.8	76.4	60.4	86.2	31.0	0.0333	71.4	84.2	66.1	67.6	72.3	11.3	0.0667	52.2	65.8	57.9	67.4	60.8	11.7
246	Fenoxycarb	0.1000	95.4	122.6	99.4	60.9	94.6	26.9	0.2000	69.1	69.8	92.1	58.2	72.3	19.7	0.4000	107.4	95.9	90.2	101.0	98.6	7.4
247	Sethoxydim	0.1500	86.0	80.0	77.6	88.9	83.1	6.3	0.3000	82.4	81.8	81.6	83.8	82.4	1.2	0.6000	68.1	64.5	85.3	81.2	74.8	13.4
248	Anilofos	0.0333	100.3	93.4	101.2	79.2	93.5	10.9	0.0667	87.8	107.9	109.8	109.7	103.8	10.3	0.1333	92.2	83.5	100.0	101.8	94.6	8.9
249	Acinathrin	0.0333	94.4	96.3	99.9	99.8	97.6	2.8	0.0667	99.1	102.7	100.0	100.8	100.7	1.5	0.1333	97.2	82.9	107.0	97.5	96.2	10.3
250	Lambda-cyhalothrin	0.0167	101.6	103.8	114.3	109.4	107.3	5.3	0.0333	96.6	100.6	105.5	103.7	101.6	3.8	0.0667	92.4	82.7	99.0	94.1	92.0	7.5
251	Mefenacet	0.0500	104.7	82.5	95.7	80.4	90.8	12.7	0.1000	83.5	107.9	117.9	111.6	105.2	14.3	0.2000	84.1	72.5	84.7	103.6	86.2	14.9
252	Permethrin	0.0333	101.3	98.5	103.9	107.2	102.8	3.6	0.0667	106.2	109.3	106.7	107.9	107.5	1.3	0.1333	95.7	88.5	101.5	100.8	96.6	6.2
253	Pyridaben	0.0167	97.6	80.9	104.5	76.7	89.9	14.8	0.0333	106.9	106.5	103.2	103.0	104.9	2.0	0.0667	94.3	81.5	96.5	93.4	91.4	7.4
254	Fluroglycofen-ethyl	0.2000	106.7	111.5	113.8	111.4	110.9	2.7	0.4000	92.1	121.5	111.3	113.7	109.6	11.4	0.8000	93.2	64.1	95.0	96.7	87.2	17.8
255	Bifenthrin	0.0500	93.8	71.7	74.0	90.3	82.5	13.6	0.1000	29.4	110.3	111.4	108.3	110.0	1.4	0.2000	76.5	75.6	70.4	97.0	79.9	14.7
256	Etofenprox	0.0167	100.8	96.8	102.2	106.6	101.6	4.0	0.0333	106.6	106.9	106.9	104.6	106.3	1.0	0.0667	96.3	91.2	102.6	99.7	97.5	5.0
257	Cycloxydim	0.2000	26.0	10.2	28.2	5.2	17.4	65.6	0.4000	76.3	39.9	3.5	11.6	32.8	100.3	0.8000	54.6	23.6	43.9	42.8	41.3	31.3
258	Estenvertate	0.0667	98.5	100.2	117.0	98.3	103.5	8.7	0.1333	98.1	89.1	87.8	86.0	90.2	6.0	0.2667	98.5	86.2	119.0	91.9	99.4	13.8
259	alpha-Cypermethrin	0.0333	95.0	98.4	104.7	96.3	98.6	4.3	0.0667	86.5	87.9	96.3	94.7	91.4	5.3	0.1333	91.7	76.8	97.5	93.3	89.8	10.0
260	Difenconazole	0.1000	87.5	63.7	77.4	60.5	72.3	17.3	0.2000	67.3	100.8	115.7	128.4	103.0	25.6	0.4000	72.4	63.5	61.6	91.8	72.3	19.1
261	Flumidlorac-pentyl	0.0333	101.6	96.2	107.5	91.7	99.2	6.9	0.0667	88.7	103.2	105.2	105.6	100.6	8.0	0.1333	91.3	93.7	97.8	99.0	95.4	3.7
D Group																						
262	Dimefox	0.0500	81.8	76.5	72.5	60.5	72.8	12.4	0.1000	65.8	95.0	72.2	76.3	79.8	15.3	0.2000	66.3	69.9	89.5	77.1	75.7	13.5
263	Disulfoton-sulfoxide	0.0333	90.0	84.3	97.0	64.3	83.9	16.8	0.0667	70.9	106.6	104.1	101.3	88.2	35.9	0.1333	84.4	82.3	96.9	100.1	90.9	9.7

Table 5. Continued

no.	pesticide	recovery (%)											RSD (%)									
		Dragon eye					Cabernet sauvignon ave.					RSD (%)										
		low level (mg kg ⁻¹)	Cabernet hambury	Muscata hambury	Cabernet hambury	RSD (%)	medium level (mg kg ⁻¹)	Vamu-rensis	Sirah	Merlot	Gamay	ave.		RSD (%)	high level (mg kg ⁻¹)	Muscata hambury	Vamu-rensis	Cabernet france	Gamay	ave.	RSD (%)	
264	Pentachlorobenzene	0.0167	96.8	87.0	94.7	88.9	10.1	0.0333	93.7	95.5	80.7	81.3	87.8	9.0	0.0667	68.2	68.5	79.9	69.5	71.5	7.8	
265	Crimidine	0.0167	72.9	94.2	80.7	82.5	10.7	0.0333	66.1	101.6	71.5	62.5	75.4	23.7	0.0667	80.8	60.3	88.9	61.1	72.8	19.7	
266	Chlorfenprop-methyl	0.0167	105.0	101.1	92.0	96.8	7.7	0.0333	95.6	106.3	70.5	72.9	86.3	20.2	0.0667	91.2	80.0	104.7	79.1	88.8	13.5	
267	Thionazin	0.0167	93.5	93.2	75.0	87.5	9.9	0.0333	87.6	109.3	80.9	82.4	90.0	14.6	0.0667	91.4	69.3	105.5	69.9	84.1	21.0	
268	2,3,5,6-Tetrachloroaniline	0.0167	105.0	100.2	98.2	98.0	7.0	0.0333	100.9	103.1	71.4	67.6	85.7	22.0	0.0667	91.5	87.7	103.0	66.6	87.2	17.4	
269	tri-n-Butyl phosphate	0.0333	103.9	107.9	92.1	99.5	7.7	0.0667	98.4	109.3	97.4	97.8	97.9	0.5	0.1333	94.6	86.6	104.5	94.9	95.1	7.7	
270	2,3,4,5-Tetrachloroanisole	0.0167	108.3	101.2	98.9	99.8	7.2	0.0333	102.8	103.0	82.0	78.7	91.6	14.3	0.0667	91.6	89.5	102.8	86.7	92.7	7.6	
271	Pentachloroisole	0.0167	107.8	97.1	112.1	102.0	9.5	0.0333	103.9	103.9	90.0	85.5	95.8	10.1	0.0667	91.2	91.9	103.5	85.1	92.9	8.3	
272	Tebutam	0.0333	106.5	103.1	82.0	99.3	6.6	0.0667	95.1	108.8	94.5	90.4	97.2	8.3	0.1333	94.6	83.4	105.7	92.1	93.9	9.8	
273	Dioxabenzofos	0.1667	106.5	102.2	81.3	95.7	11.7	0.3333	78.0	110.4	82.2	75.3	86.5	18.8	0.6667	92.1	75.5	104.8	75.6	87.0	16.3	
274	Methabenzthiazuron	0.1667	97.7	98.5	82.1	94.0	8.4	0.3333	74.9	112.4	109.3	105.2	100.4	17.2	0.6667	90.7	71.2	102.3	100.2	91.1	15.6	
275	Simeiton	0.0333	91.6	74.6	85.4	82.4	9.2	0.0667	77.3	103.7	101.5	102.6	96.3	13.2	0.1333	77.6	61.3	83.5	97.8	80.1	18.9	
276	Atratore	0.0167	78.3	84.4	80.3	78.1	17.6	0.0333	68.6	115.2	105.3	109.0	109.8	4.6	0.0667	83.4	80.5	90.5	101.4	89.0	10.5	
277	Desisopropyl-atrazine	0.1333	107.9	81.6	72.5	85.4	18.1	0.2667	66.0	110.7	86.1	90.6	88.4	20.8	0.5333	53.3	54.2	77.2	88.4	68.3	25.5	
278	Tebuifos sulfone	0.0167	80.6	84.5	72.7	82.2	9.4	0.0333	101.6	82.6	70.8	71.4	81.6	17.7	0.0667	95.5	86.8	105.1	80.9	92.1	11.4	
279	Tefluthrin	0.0167	120.3	111.3	105.6	110.0	7.0	0.0333	108.8	111.5	100.1	99.1	104.9	6.0	0.0667	99.1	96.5	109.2	98.4	100.8	5.7	
280	Bromocyclo	0.0167	109.9	115.8	98.6	111.5	8.9	0.0333	106.4	94.3	78.9	81.2	90.2	14.1	0.0667	94.8	91.9	100.6	78.7	91.5	10.1	
281	Trietazine	0.0167	107.9	103.5	90.0	100.2	7.6	0.0333	97.7	108.3	102.3	103.5	102.9	4.2	0.0667	98.8	89.5	105.6	100.5	98.6	6.8	
282	Eirimfos oxon	0.0167	112.5	105.4	94.9	102.9	7.5	0.0333	101.5	107.7	96.6	93.5	99.9	6.2	0.0667	99.2	88.9	108.6	94.0	97.7	8.6	
283	Cycluron	0.0500	92.7	96.6	90.5	93.9	3.0	0.1000	71.9	99.8	102.4	99.2	93.3	15.4	0.2000	87.0	83.8	97.7	98.9	91.9	8.3	
284	2,6-Dichlorobenzamide	0.0333	107.0	59.7	69.4	76.9	27.0	0.0667	63.0	82.2	63.5	64.8	68.4	13.5	0.1333	68.9	57.6	53.6	72.6	63.2	14.2	
285	DE-PCB 28	0.0167	110.7	102.6	101.5	102.8	5.7	0.0333	105.4	107.3	93.0	91.4	99.3	8.3	0.0667	98.4	96.5	107.6	93.5	99.0	6.2	
286	DE-PCB 31	0.0167	109.3	105.3	101.9	103.5	4.8	0.0333	106.0	106.6	91.4	91.9	99.0	8.6	0.0667	98.1	97.3	104.5	93.0	98.2	4.8	
287	Desethyl-sebutylazine	0.0333	88.1	91.6	84.3	90.7	6.8	0.0667	45.7	108.1	99.0	102.3	103.1	4.5	0.1333	79.3	69.3	97.6	100.3	86.6	17.2	
288	2,3,4,5-Tetrachloroaniline	0.0333	94.8	98.8	95.3	96.4	1.9	0.0667	93.2	101.1	95.5	93.0	95.7	3.9	0.1333	95.5	86.7	103.0	89.2	93.6	7.8	
289	Pentachloroaniline	0.0167	103.8	106.6	94.7	100.7	5.4	0.0333	98.6	104.8	96.6	96.7	99.2	3.9	0.0667	97.8	92.6	104.9	95.7	97.8	5.4	
290	Aziprotine	0.1333	110.4	105.4	96.0	102.8	6.1	0.2667	102.9	132.5	124.3	127.7	121.8	10.7	0.5333	97.5	91.7	115.2	127.3	107.9	15.1	
291	Sebutylazine	0.0167	108.6	105.9	83.4	98.8	11.3	0.0333	84.8	106.5	107.2	107.0	101.4	10.9	0.0667	95.2	85.8	105.4	104.6	97.8	9.4	
292	Isocarbamid	0.0833	56.9	66.4	60.7	69.0	22.9	0.1667	67.0	108.9	100.7	97.4	93.5	19.6	0.3333	53.3	61.8	80.3	96.4	73.0	26.4	
293	DE-PCB 52	0.0167	111.2	107.1	99.9	104.5	5.3	0.0333	107.8	107.9	99.0	98.4	103.3	5.1	0.0667	99.0	98.1	108.5	98.5	101.0	5.0	
294	Prosulfocarb	0.0167	103.5	96.9	87.0	94.2	7.9	0.0333	105.5	107.8	100.7	101.1	103.8	3.3	0.0667	100.7	91.9	109.1	99.7	100.4	7.0	
295	Dimethenamid	0.0167	94.3	96.3	86.1	94.4	6.6	0.0333	86.4	104.7	95.3	96.0	95.6	7.8	0.0667	97.3	83.3	106.7	100.0	96.8	10.2	
296	Fenchlorphos oxon	0.0333	97.6	108.9	104.2	97.3	102.0	5.5	0.0667	103.2	107.4	97.7	97.2	101.4	4.8	0.1333	99.4	94.8	108.3	97.6	100.0	5.8
297	Paraoxon-methyl	0.0333	79.9	85.7	26.3	85.9	7.1	0.0667	88.2	130.3	104.2	97.8	105.1	17.2	0.1333	75.9	76.5	96.7	96.8	86.5	13.7	
298	Monsalde	0.0333	108.2	106.2	87.2	99.9	9.5	0.0667	96.2	105.5	102.7	100.5	101.2	3.9	0.1333	99.3	85.3	108.2	103.7	99.1	10.0	
299	Isobenzan	0.0167	117.5	112.1	108.7	111.4	4.1	0.0333	106.1	107.3	99.9	99.1	103.1	4.1	0.0667	97.5	95.8	105.9	96.7	99.0	4.7	
300	Octachlorostyrene	0.0167	106.4	100.9	98.3	100.6	4.2	0.0333	106.5	107.5	98.4	98.7	102.8	4.8	0.0667	97.1	96.2	105.7	98.1	99.3	4.4	
301	Isodin	0.0167	87.8	93.3	94.3	89.4	6.3	0.0333	115.4	108.0	95.4	100.8	104.9	8.3	0.0667	100.4	97.6	107.5	96.6	100.5	4.9	
302	Isomethiozin	0.0333	95.9	97.3	73.9	88.9	12.0	0.0667	92.1	103.4	96.1	98.2	97.5	4.8	0.1333	96.1	80.3	104.7	99.9	95.2	11.1	
303	Trichloronat	0.0167	110.4	106.9	98.4	103.4	6.0	0.0333	107.1	108.3	102.5	102.1	105.0	3.0	0.0667	99.6	97.0	107.8	100.0	101.1	4.6	
304	Dachal	0.0167	111.6	105.7	93.6	102.3	7.7	0.0333	99.7	108.7	102.6	102.9	103.5	3.6	0.0667	100.1	90.2	110.3	102.6	100.8	8.2	
305	4,4-Dichlorobenzophenone	0.0167	102.7	100.1	87.7	95.7	7.2	0.0333	103.1	110.1	100.4	101.9	103.9	4.2	0.0667	98.5	93.0	108.1	100.0	99.9	6.3	
306	Nitrothial-isopropyl	0.0333	99.6	95.7	75.2	90.5	11.8	0.0667	104.1	114.7	105.9	110.6	108.8	4.4	0.1333	94.7	85.8	106.9	101.1	97.1	9.3	
307	Rabenzazole	0.0167	83.3	81.2	88.1	87.2	7.6	0.0333	70.2	110.9	113.0	100.7	98.7	20.0	0.0667	78.3	77.8	70.0	84.4	77.6	7.6	
308	Cyprodinil	0.0167	91.4	84.6	42.3	88.7	4.0	0.0333	93.7	108.9	109.1	112.3	106.0	7.9	0.0667	96.3	85.7	100.1	105.3	96.8	8.6	
309	Dicaphon	0.0833	92.0	99.6	82.0	92.9	8.5	0.1667	79.7	136.4	104.1	106.3	106.6	21.8	0.3333	93.8	82.2	108.2	101.3	96.4	11.6	

Table 5. Continued

no.	pesticide	recovery (%)				recovery (%)				recovery (%)											
		Dragon eye	Cabernet sauvignon	Muscata hambury	Cabernet sauvignon	RSD (%)	medium level (mg kg ⁻¹)	Vamu-rensis	Sirah Merlot	Gamay	RSD (%)	high level (mg kg ⁻¹)	Muscata hambury	Vamu-rensis	Cabernet france	Gamay	RSD (%)				
310	DE-PCB 101	100.0	110.6	105.9	102.8	104.8	4.3	0.0333	106.9	107.4	104.9	105.4	106.1	1.1	0.0667	100.3	100.1	109.0	106.0	103.9	4.2
311	MCPA-Butoxyethyl ester	98.9	113.2	115.9	95.8	106.0	9.5	0.0333	98.9	118.8	104.8	107.4	107.5	7.8	0.0667	98.3	91.3	107.8	101.6	99.7	6.9
312	Phorate sulfone	98.0	104.4	102.9	67.8	93.2	18.4	0.0333	90.5	110.3	100.5	101.4	100.7	8.1	0.0667	96.7	63.9	107.1	103.5	92.8	21.3
313	Chlorfenehol	95.1	106.3	105.3	93.0	99.9	6.9	0.0333	92.1	108.6	107.1	106.6	103.6	8.3	0.0667	98.9	90.0	105.7	105.7	100.8	8.3
314	trans-Nonachlor	96.1	104.6	104.0	96.7	100.3	4.6	0.0333	104.5	105.9	103.5	103.4	104.3	1.1	0.0667	100.5	95.3	106.5	103.2	101.4	4.7
315	DEF	92.5	93.2	95.3	72.2	88.3	12.2	0.0667	107.3	114.8	99.8	94.0	104.0	8.7	0.1333	98.6	92.1	104.6	103.0	99.6	5.6
316	Fluorochloridone	96.2	102.7	106.0	69.0	93.5	18.0	0.0667	91.9	93.7	90.4	98.5	93.6	3.8	0.1333	98.3	67.1	108.6	100.9	93.7	19.5
317	Bromfeninfos	97.8	138.5	170.3	79.8	121.6	33.5	0.0333	81.8	115.3	98.3	109.5	101.2	14.6	0.0667	93.0	74.0	105.0	100.9	93.2	14.8
318	Perthane	98.4	108.7	106.0	97.6	102.7	5.4	0.0333	102.1	111.4	104.9	108.0	106.6	3.8	0.0667	101.2	96.6	107.8	106.0	102.9	4.9
319	DE-PCB 118	103.4	109.4	105.8	102.7	105.3	2.9	0.0333	104.5	109.8	106.7	108.4	107.4	2.2	0.0667	99.2	98.5	109.5	108.1	103.8	5.6
320	4,4-Dibromobenzophenone	99.1	110.3	91.1	87.2	96.9	10.6	0.0333	103.7	116.5	105.4	107.5	108.3	5.3	0.0667	97.5	94.0	106.6	104.7	100.7	5.9
321	Flutriafol	92.3	53.9	77.5	65.3	72.2	22.8	0.0667	85.0	108.9	100.8	99.3	98.5	10.1	0.1333	80.0	77.3	90.5	100.5	87.1	12.2
322	Mephostolan	97.7	91.0	78.0	78.2	86.2	11.3	0.0667	70.3	119.0	106.7	99.9	99.0	20.0	0.1333	65.8	76.2	89.5	101.1	83.1	18.5
323	Athidathion	106.4	107.5	97.8	99.4	102.8	4.7	0.0667	112.7	103.2	105.1	99.5	105.1	5.3	0.1333	79.5	94.5	104.1	90.8	92.2	11.0
324	DE-PCB 153	100.7	108.8	102.9	100.1	103.1	3.8	0.0333	105.7	111.3	108.0	107.9	108.2	2.1	0.0667	101.2	99.9	108.7	108.8	104.7	4.6
325	Diclobutrazole	93.7	77.9	92.7	90.5	88.7	8.3	0.1333	88.8	114.5	107.5	105.9	104.2	10.5	0.2667	91.7	92.6	100.3	104.5	97.3	6.4
326	Disulfoton sulfone	95.6	98.7	102.0	70.4	91.7	15.7	0.0667	71.5	130.1	100.0	103.1	101.2	23.7	0.1333	95.1	90.9	109.0	102.3	99.3	8.0
327	Hexythiazox	97.7	102.0	104.7	87.5	98.0	7.7	0.2667	71.2	120.9	112.5	114.5	116.0	3.8	0.5333	98.1	90.8	108.0	110.2	101.8	8.9
328	DE-PCB 138	96.0	106.8	107.7	96.3	101.7	6.3	0.0333	105.0	109.9	105.6	108.6	107.3	2.2	0.0667	98.9	99.5	107.7	108.5	103.6	5.0
329	Resmethrin-1	2.9	3.9	1.9	0.0	2.2	77.2	0.0667	44.8	3.6	1.8	2.1	2.5	38.9	0.1333	58.8	27.2	142.6	70.6	74.8	65.2
330	Cyproconazole	97.7	64.1	93.8	44.5	75.0	33.7	0.0333	69.9	125.8	109.3	112.3	104.3	23.0	0.0667	77.3	48.5	81.2	87.2	73.6	23.4
331	Resmethrin-2	3.4	3.6	2.6	2.4	3.0	20.4	0.0667	44.8	3.6	1.9	2.2	2.6	35.4	0.1333	61.2	26.7	126.7	105.8	80.1	56.0
332	Phthalic acid, Benzyl butyl ester	100.8	104.0	103.9	99.9	102.1	2.1	0.0333	93.1	120.1	114.0	109.9	109.3	10.6	0.0667	103.3	90.5	110.9	109.5	103.6	9.0
333	Clodimatop-propargyl	97.3	100.9	105.6	96.6	100.1	4.1	0.0667	88.7	132.3	109.2	113.2	110.8	16.1	0.1333	98.0	93.6	107.4	105.2	101.0	6.3
334	Fenithion sulfoxide	91.4	77.3	92.9	90.3	88.0	8.2	0.1333	87.0	89.1	97.9	91.7	91.4	5.2	0.2667	77.5	86.5	84.6	137.8	96.5	28.8
335	Fluotrimazole	98.2	98.4	106.5	85.8	97.2	8.8	0.0333	88.9	88.2	108.8	97.7	95.9	10.0	0.0667	105.9	120.0	123.2	104.5	113.1	8.5
336	Fluoroxypyr-1-methylheptyl ester	98.4	110.8	104.0	95.9	102.3	6.5	0.0333	93.8	115.5	107.7	109.2	106.6	8.6	0.0667	100.7	91.4	108.2	106.9	101.8	7.5
337	Fenithion sulfone	98.9	93.1	102.1	60.7	98.0	4.6	0.1333	35.6	114.7	105.7	102.6	107.7	5.8	0.2667	96.9	51.5	107.5	108.2	104.2	6.0
338	Triphenyl phosphate	96.4	107.8	110.6	75.1	97.5	16.5	0.0333	81.1	111.8	107.2	107.9	102.0	13.8	0.0667	100.2	75.9	109.9	106.5	98.1	15.6
339	Meltamiron	75.4	83.4	84.1	72.9	79.0	7.2	0.3333	73.0	97.6	98.2	116.8	96.4	18.6	0.6667	92.0	77.7	86.7	102.7	89.8	11.6
340	DE-PCB 180	100.7	108.0	104.5	99.5	103.1	3.7	0.0333	108.3	112.4	110.1	110.7	110.4	1.5	0.0667	98.4	97.3	107.9	109.6	103.3	6.1
341	Tebuconazole	100.6	108.5	103.8	82.5	98.8	11.5	0.0333	101.5	116.5	109.7	108.9	109.1	5.6	0.0667	99.5	90.5	107.4	108.7	101.5	8.3
342	Cloquintocet-mexyl	99.5	97.0	104.8	90.5	97.9	6.0	0.0333	87.6	135.7	116.1	121.9	115.3	17.6	0.0667	91.7	73.0	107.0	118.9	97.7	20.3
343	Lenacil	95.0	72.5	75.3	70.8	78.4	14.3	0.3333	72.3	98.1	87.7	102.4	90.1	14.9	0.6667	77.2	78.2	95.0	104.0	88.6	14.8
344	Bromuconazole-1	95.5	70.9	89.6	79.0	83.8	13.1	0.0667	73.2	115.4	105.0	107.0	100.1	5.5	0.1333	90.6	89.0	102.2	108.5	97.6	9.6
345	Desbrom- leptophos	98.6	109.3	106.0	99.2	103.3	5.0	0.0333	104.3	116.3	108.2	110.9	109.9	4.6	0.0667	100.8	99.4	111.2	108.4	105.0	5.5
346	Bromuconazole-2	98.6	62.6	82.0	62.6	80.4	15.3	0.0667	88.2	107.4	103.9	99.6	99.8	3.9	0.1333	87.1	88.4	95.5	107.3	84.6	9.7
347	Nitralin	98.3	95.2	91.2	61.9	86.6	19.4	0.3333	95.3	110.3	103.5	109.8	104.7	3.6	0.6667	93.8	94.6	111.3	110.6	102.6	9.4
348	Fenamiphos sulfoxide	63.7	27.9	40.2	5.5	34.3	70.8	0.1333	26.6	110.6	98.5	101.3	103.4	6.1	0.2667	10.7	8.8	29.3	98.0	16.3	69.8
349	Fenamiphos sulfone	86.7	50.6	72.2	16.7	56.6	53.8	0.1333	91.1	105.3	94.3	93.8	96.1	6.7	0.2667	46.5	20.5	70.3	96.0	58.3	55.4
350	Fenpiconil	119.9	44.0	51.2	60.5	51.9	15.9	0.1333	37.1	66.1	37.4	40.2	45.2	21.0	0.2667	38.3	32.3	70.2	54.7	48.9	35.0
351	Fluquinconazole	97.2	91.8	99.1	94.3	95.6	3.3	0.0333	92.5	109.3	108.5	105.7	104.0	7.5	0.0667	95.4	99.1	107.7	108.0	102.6	6.2
352	Fenbuconazole	89.9	89.9	80.6	70.2	82.6	11.3	0.0667	70.0	105.3	103.2	101.9	95.1	17.7	0.1333	77.1	90.1	88.7	101.1	89.3	11.0

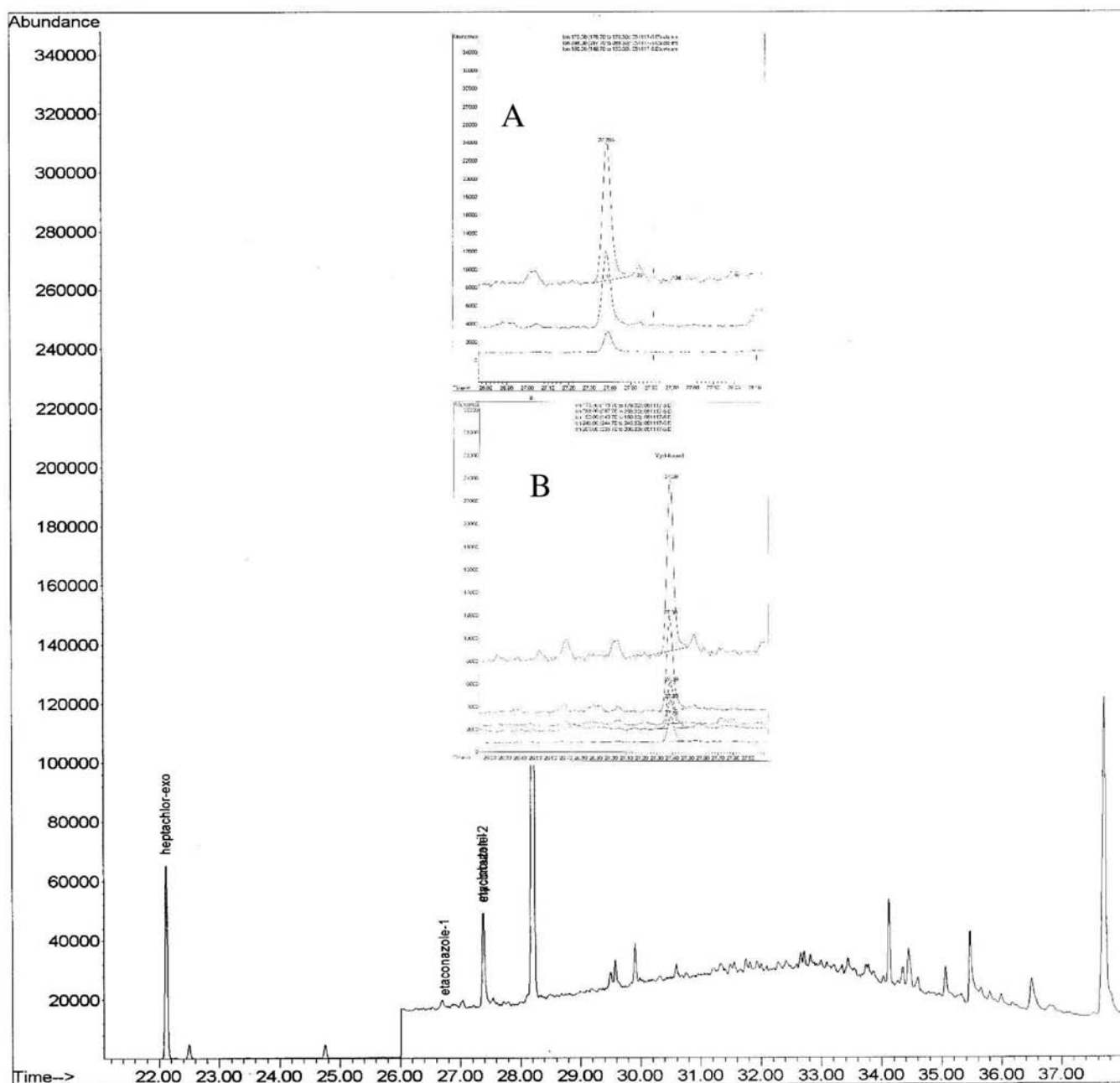


Figure 1. Total ion chromatogram of a blank grape sample cleaned up by dispersive SPE. (Inset) Extracted ion chromatogram for myclobutanil at 27.38 min; extracted ion for (A) myclobutanil, m/z 179.0, 288.0, and 150.0; (B) m/z 179.0, 288.0, 150.0, 245.0, and 206.0.

multiresidues in grapes. The stable and reliable repeatability of this method makes it an effective routine analytical method in international trade and in residue monitoring by countries all over the world.

Determination in Real Samples. According to the detecting program described above, 352 pesticides and related chemicals from nine varieties of grapes, such as Dragoneye, Caberner, Muscathambury, Cabemetsauvignon, V. amurensis, Sirah, Merlot, Gamay, and Murasaki, were determined. A peak of an unknown compound was found at 27.38 min in Dragoneye. It was preliminarily identified as myclobutanil, which is shown in **Figure 1A**, by comparing the retention time, three fragment ions 179.0, 288.0, and 150.0, and the abundance ratio of selected ions of the unknown peak with that of the standard compounds peak. To further verify the unknown compound, additional two fragment ions, 245.0 and 206.0, were monitored, as shown in **Figure 1B**.

The result showed that fragment ions of the target analyte were the same as those of the standard compound, and the relative abundance ratio of selected ions was in the range of the maximum tolerant deviation. The unknown compound was ascertained as myclobutanil, the concentration of which was 0.029 mg kg^{-1} , and then upon communication with the grape planter, we knew that the pesticide was used to prevent and cure *Phoma citricarpa* and that it can be detected due to the mistaken utility.

Conclusions. Qualitative and quantitative analyses and determination for grape samples were performed, and the results showed that the method has a higher sensitivity and good selectivity and that 352 pesticides and related chemical residues were determined at a time suitable for monitoring and screening for multiresidue pesticides of grape samples.

ABBREVIATIONS USED

PSA, primary secondary amine; MSPD, matrix solid phase dispersion; LOD, limit of detection; RSD, relative standard deviation; MRL, maximum residue limit; GCB, graphitized carbon blacks; SPE, solid phase extraction; GC-ITMS, gas chromatography-ion trap mass spectroscopy; NPD, nitrogen phosphorus detector; ECD, electron capture detector; MSD, mass spectroscopy detector; MSPD-LC-MS, matrix solid phase dispersion-liquid chromatography-mass spectrometer; QuEChERS, Quick, Easy, Cheap, Effective, Rugged and Safe; LOQ, limit of quantitation; MRM, multiple reaction monitoring; PTV, programmable temperature vaporizer; OIV, Organisation Internationale de la Vigne et du Vin.

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