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Use of gas–liquid chromatography with electron-capture and thermionic-sensitive detection for the quantitation and identification of pesticide residues

F. Sicbaldi[a,*], A. Sarra[b], D. Mutti[b], P.F. Bo[b]

[a]*Istituto di Chimica Agraria ed Ambientale, Sez. Vegetale, Università Cattolica del Sacro Cuore, Via Emilia Parmense 84, 29100 Piacenza, Italy*

[b]*CADIR-LAB, Via Alessandria 13, 15044 Quargnento (AL), Italy*

Abstract

A gas chromatographic methodology that exploits combination of different columns and different detectors for testing residues of over 150 pesticides is proposed. Two gas–liquid chromatographs were set up each with two capillary columns of different polarities, DB-5 and DB-1701. Each column was connected to a precolumn with deactivated silica which was in turn connected to the injector. Each column was coupled to an electron-capture detector in one instrument and a thermionic-sensitive detector in the other. Standard solutions containing each pesticide, with ethion used as a reference peak to calculate the relative retention time (RRT), were injected into each gas chromatograph. RRT values for each column-detector combination are reported. Two columns of different polarities allowed identification and separation of most of the pesticides studied. Qualitative confirmations achieved by comparing the RRT values obtained by columns of different polarities on the same detectors as well as on different detectors are discussed. The instrumental configuration and the analytical conditions used may be a useful tool for the determination and confirmation of the great number of pesticides residues that may be extracted by multiresidue methods.

Keywords: Detection, GC; Pesticides

1. Introduction

Multiresidue analysis for pesticides has usually been applied in monitoring foodstuffs destined for human consumption. Fruits and vegetables usually receive direct applications of pesticides in the field or in post-harvest treatments and may retain a proportion as residues in or on the edible portion delivered to the consumer.

In Italy the number of registered pesticides is 385 [1] and imported foodstuffs may contain pesticide residues normally banned in Europe. Both these factors and the continuing development of new pesticides provide a constant challenge to the analytical methodology.

Compound classifications based on chemical structure have been useful in defining analytical strategies but methods based on such classifications cannot adequately determine many classes or even all pesticides within a class.

Multiresidue (MR) methods have, therefore, been required to determine residues of a wide range of pesticides in samples. MR methodology has increas-

*Corresponding author.

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ingly become dependent on instrumental analyses techniques. Gas chromatography is still the basis for determination of pesticide residues in most standard multiresidue methods.

It is more than a decade since high-resolution gas chromatography (HRGC) using long, narrow-bore capillary columns (25–30 m×0.20–0.32 mm I.D.) began to be used for routine residue screening in fruits and vegetables [2,3]. Initial developments concentrated on class analysis. Recently full MR procedures based on HRGC determination use columns connected to electron-capture (ECD), thermionic flame (NPD) or flame photometric (FPD) detection systems.

The aim of this work was to carry out a systematic study on the GC analyses of over 150 pesticides. In Italy government agencies as well as food retailer chains usually require routine residue analyses for such a wide variety of pesticides.

Pesticides retention times were studied in two different columns, DB-5 and DB-1701 both coupled to ECDs in one instrument and TSDs in the other. The best column-detector combinations were studied in order to identify and confirm the pesticides under investigation. After setting the GC instrumental conditions a multiresidue method was developed and presented elsewhere [4].

2. Experimental

Two Varian gas chromatographs were set up each with two columns of different polarities (DB-1701 fused-silica column, containing 14% cyanopropyl-phenyl-methylpolysiloxane, 30 m×0.25 mm I.D., 0.25 μm (J&W Scientific) and DB-5 fused-silica column, containing 5% phenyl-methyl-polysiloxane, 30 m×0.25 mm I.D., 0.25 μm) connected by a Y connection to a precolumn in silica de-activated (2.5 m) which was in turn connected to the injector. Each column was connected to ECD in one instrument and TSD in the other.

The instruments were equipped as follows:

(1) GC Varian 3400 series Cx equipped with two ^{63}Ni electron-capture detectors; interfaced respectively to the two columns described above; capillary split/splitless injector model 1077. Operating conditions: injector 250 °C; detectors 300 °C; temperature program, column 120 °C, hold 2 min, increase to 190 °C at 3 °C min^{-1}, no hold time, increase to 220 °C at 2 °C min^{-1}, no hold time, increase to 280 °C at 5 °C min^{-1}, hold time 30 min; helium carrier gas (1.5 ml min^{-1}), N_2 make-up (25 ml min^{-1}), split 30 ml min^{-1}; injection volume: 2 μl; manual injection, type "sandwich", split mode operation before injection, splitless mode for 0.75 min after injection and then split mode.

(2) GC Varian 3400 equipped with two thermionic-sensitive detectors interfaced respectively to the two columns described above; same injector; operating conditions: injector 250 °C; detectors 300 °C; temperature program, 120 °C, hold 2 min, increase to 190 °C at 5 °C min^{-1}, hold time 2 min, increase to 220 °C at 3 °C min^{-1}, hold time 5 min, increase to 260 °C at 5 °C min^{-1} hold 30 min; helium carrier gas (1.5 ml min^{-1}), H_2 working gas (2.4 ml min^{-1}), N_2 make-up (25 ml min^{-1}); air 190 ml min^{-1}; split 30 ml min^{-1}. Operating TSD bead current: 2.9 A. Injection volume, injection mode and injector mode operation as reported above. Chromatographic signals were registered with an IBM 433 DX/S computer using STAR 4.0 chromatography software.

Pesticides standards were certified with purities ranging from 92 to 99.9% (Ehrenstorfer, Germany; Krasiejko, Poland). Stock standard solutions were prepared dissolving each standard in pure acetone (DAB-BP-Merck). Standard solutions were at 0.4 mg ml^{-1} and were then diluted so as to obtain a working range of 1–5 μg ml^{-1}.

Ethion was used as reference peak to calculate the relative retention times (RRT) values and added to solutions containing each pesticide to obtain a concentration of 1 μg ml^{-1}. At least two injections of each compound were performed to increase confidence in assignation of chromatographic peaks.

3. Results

The advantage of the HRGC is the high resolution and the reproducibility of retention times which were less than 0.05% RSD for most compounds.

Although peaks were separated from each other

when RRT values differed of ±0.003 min, the analytes were identified only when the RRT values differed at least ±0.004 min among two close eluting peaks. This procedure was not applicable to the configuration previously used where two wide-bore columns of different polarities (RTX-5 and DB-17) were coupled to the detectors (data not shown) because the resolution was poorer than that obtained with the columns tested in the present work.

Some of the pesticides studied could not be directly determined by the GC configurations used (Table 1), 16% with the columns coupled with ECD and 28% with those coupled with TSD.

Some were not detectable, others were hydrolysed at room temperature (e.g. phoxim) or thermally labile (e.g. dinocap). Some polar pesticides that eluted at the beginning of the chromatogram were often not separated from the co-extracted compound and that rendered their determination difficult. Some compounds were detectable either by ECD or TSD (e.g., benzoximate, dithianon, rimsulfuron) but their detection limits (5, 2 and 4 $\mu g\,ml^{-1}$, respectively) were higher than the levels normally found in crops as well as their maximum residue limits and thus their determination was not practicable.

Figs. 1 and 2 demonstrate the performance of this system for one standard mix run on the columns considered.

Table 2 reports the relative retention times of each pesticide for each column(s) detector(s) combination and the best detector used for quantitation. This study was necessary to supply preliminary results on the chromatography of the selected pesticides in order to develop a multiresidue extraction/clean-up method. The method reported elsewhere [4] is based on matrix solid-phase dispersion with diatomaceous earth of a fruit sample; no further clean-up was required for subsequent GC analysis.

Table 1
Pesticides not directly determinable by HRGC

Anilazine	Cyhexatin	Hexithiazox	Sethoxidim
Azocyclotin	Dithianon	Imidacloprid	Teflubenzuron
Benomyl	Dinocap	Methomyl	Tiophanate-methyl
Bentazone	Dodine	Phoxim	Triforine
Benzoximate	Diflubenzuron	Propargite	
Carbendazim	Etoxiquin	Rimsulfuron	

Some of the pesticides in Table 2 showed more than one chromatographic peak. Some are isomers (e.g. chlorfenvinphos), others split during the GC run (e.g. fenthion, simazine) resulting in different fragments that can be detected.

When possible peak areas of all components or isomers should be summed in the quantitation process (e.g. pyrethroid residues) [5]. If that operation is not possible because of overlapping of chromatographic peaks belonging to pesticide residues, presence of contaminants that co-elute with the peak of interest, poor sensitivity or resolution, etc., the main chromatographic peak should be used in the quantitation process (e. g. the second peak of chlorfenvinphos reported in Figs. 1 and 2) after ensuring that the area ratio repeatability of the peaks considered is consistent inter-analysis.

Of the pesticides that can be determined directly by the GC configuration used (Table 2) 50% were detected on four detectors; among the pesticides detected by ECD three compounds (captafol, chlorothalonil and dimethomorf) were detected on the DB-5 column only, while among those detected by TSD six compounds (alphamethrine, chlorothalonil, ethiofencarb, fenpropatrin, fenvalerate hexaflumuron) were detected on DB-5 only.

The identification of those pesticides detected on columns of analogous polarity was possible by comparing their RRT values obtained on columns coupled to different detectors.

Very few compounds were detected on one column–detector combination only, respectively on DB-5–ECD (captafol and dimethomorf) and on DB-5–TSD (ethiofencarb). For such compounds other procedures to confirm their presence are recommended such as the use of HPLC.

In the rarest case that the detected pesticides studied are simultaneously present in a sample, only 17% of the chemicals are not cross confirmed because their peaks overlap. This is the worst scenario but it is usually very rare that 150 pesticides are simultaneously present in environmental samples.

4. Discussion

The configuration proposed allowed detection and

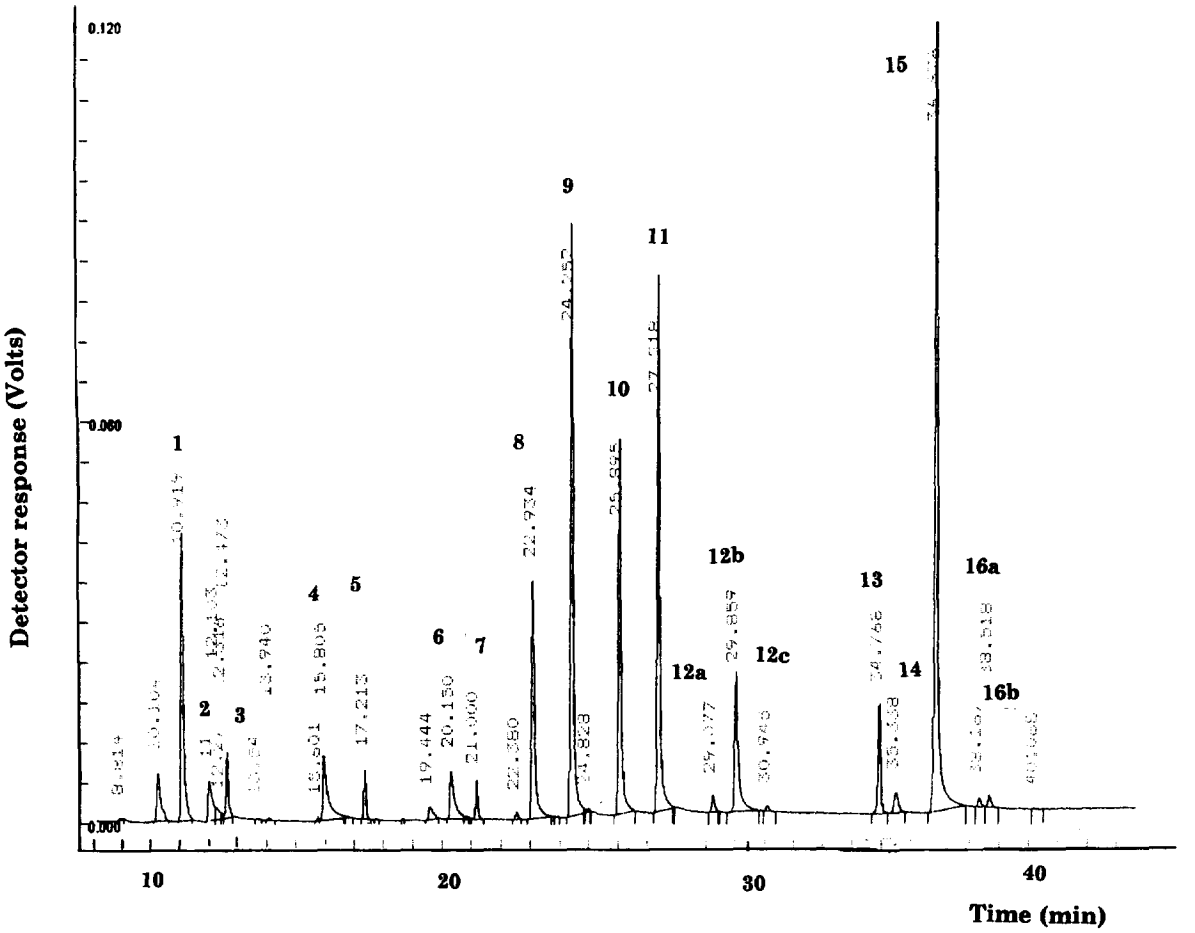

Fig. 1. Chromatogram of a standard mixture on DB-5–TSD. 1=Propamocarb; 2=acephate; 3=propham; 4=omethoate; 5=chlorpropham; 6=cyromazine; 7=propyzamide; 8=formothion; 9=parathion-methyl; 10=fenithrothion; 11=chlorpyrophos-ethyl; 12a=chlorfenvinphos; 12b=chlorfenvinphos; 12c=chlorfenvinphos; 13=bupirimate; 14=cyproconazole; 15=ethion; 16a=propiconazole; 16b=propiconazole.

identification of most of the pesticides studied. Configuration of instruments equipped with single column and effluent splitting to two detectors [6] gave the same combination obtained with two columns and two detectors. The configuration proposed in the present work is preferable because the different polarities of two columns associated with the temperature programming increase the separation power and reduce the risk of false positive due to co-eluting substances. This drawback often occurs with the less specific ECD because extract cleaned-up by solid matrix partition yields a minor purification of the extracts in comparison with gel permeation or liquid–liquid chromatography. The extracts often contain electron-capturing materials other than pesticides and this can lead to incorrect identification even if two capillary columns of different polarity are employed.

However the detailed interpretation and correlation to distinguish matrix and pesticide peaks in ECD are helped by the easier identification in TSD so residues can be positively identified. This procedure is being automated by an approach already reported [7]. The development of software that identifies the analytes,

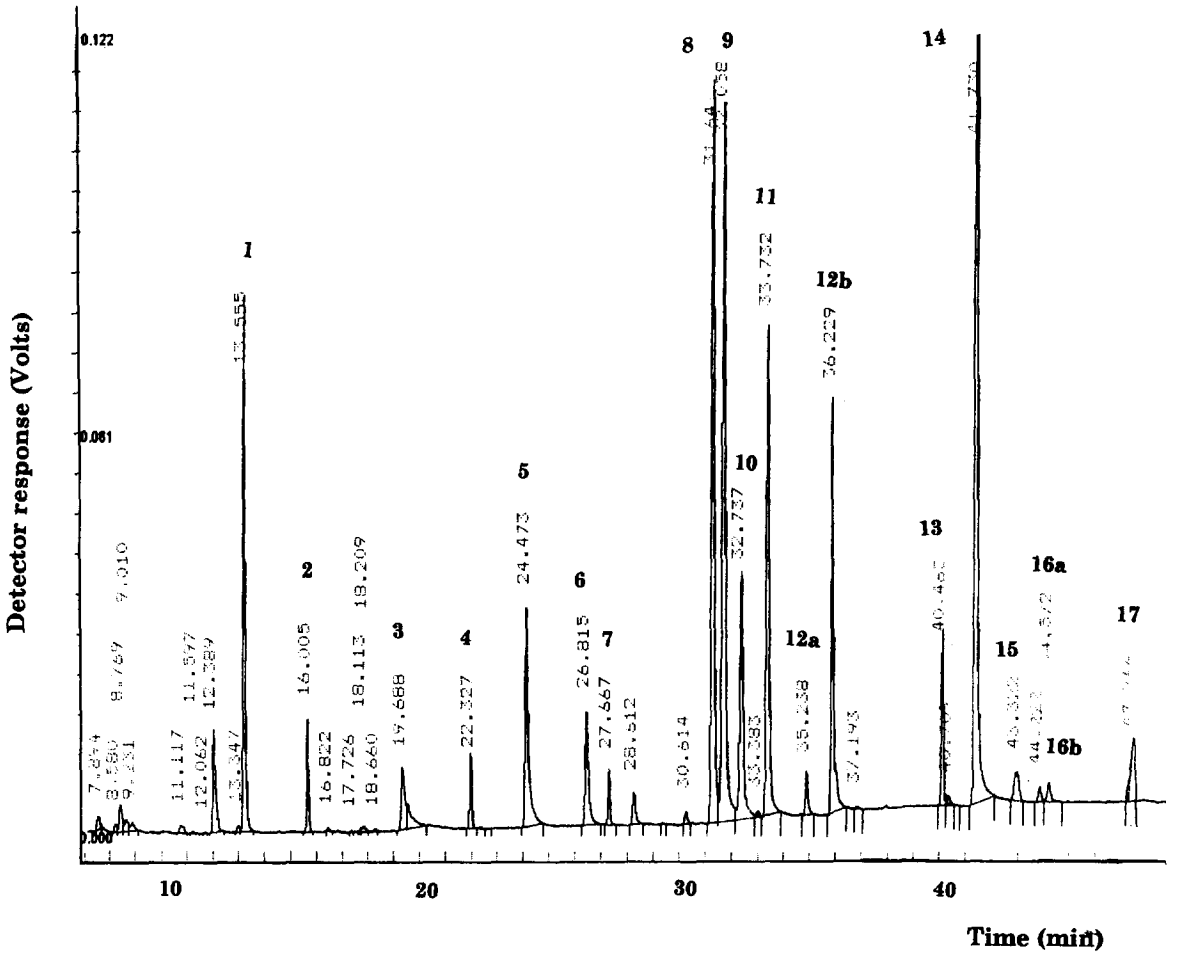

Fig. 2. Chromatogram of the same standard mixture as Fig. 1 on DB-1701–TSD. 1=Propamocarb; 2=propham; 3=acephate; 4=chlorpropham; 5=omethoate; 6=cyromazine; 7=propyzamide; 8=chlorpyriphos-ethyl; 9=parathion-methyl; 10=formothion; 11=fenithrothion; 12a=chlorfenvinphos; 12b=chlorfenvinphos; 13=bupirimate; 14=ethion; 15=cyproconazole; 16a=propiconazole; 16b=propiconazole; 17=oxadixyl.

exploiting their different RRT values on four columns helps reduce the time required for the interpretation of the chromatogram.

On the one hand it is better to use TSD instead of ECD because the former is more specific, only slightly influenced by the co-extracts often present in environmental samples and also less sensitive to molecular fragments usually detected by ECDs (e.g. pirimiphos-ethyl, simazine). On the other hand the use of ECD is superimposed by the chemical structure of those analytes that are electron capturing such as the organochlorine pesticides.

5. Conclusions

Two columns of different polarities allowed identification and separation of most of the pesticides studied. Qualitative confirmations were easily achieved by comparing the RRT values obtained by

Table 2
Relative retention times of each column–detector combination and best detector used in the quantitation process of the pesticides studied

Compound	RRT ECD		RRT TSD		Detection
	DB-5	DB1701	DB-5	DB1701	
Abamectin	*1.065*	1.070	ND	ND	ECD
Acephate	0.305	0.485	0.321	0.470	TSD
Aclonifen	0.994	1.014	0.995	1.022	TSD
Acrinathrin	1.179	*1.154*	*1.193*	*1.305*	ECD
			1.250	1.330	
Alachlor	*0.692*	*0.745*	*0.677*	0.722	ECD
Aldrin	0.747	*0.713*	ND	ND	ECD
Alphametrine	1.300	1.332	*1.495*	ND	ECD
Amitraz	ND	ND	*1.216*	*1.208*	TSD
Atrazine	0.558	0.645	0.544	0.624	TSD
Azinphos-ethyl	1.187	*1.220*	1.258	*1.440*	TSD
Azinphos-methyl	1.147	1.183	1.188	*1.353*	TSD
Benalaxyl	ND	ND	1.033	1.038	TSD
Benfuracarb	ND	ND	*1.263*	1.328	TSD
Bifentrin	1.108	1.047	ND	ND	ECD
Bitertanol	*1.212*	*1.237*	*1.306*	*1.491*	TSD
	1.219				
Bromophos-ethyl	*0.870*	0.871	*0.854*	0.859	TSD
Bromophos-methyl	0.782	*0.817*	*0.773*	0.805	TSD
Brompropylate	1.106	1.079	ND	ND	ECD
Bupirimate	0.951	*0.978*	*0.948*	*0.969*	TSD
Buprofezin	0.945	*0.938*	0.943	0.923	TSD
Captafol	*1.069*	ND	ND	ND	ECD
Captan	0.830	0.927	ND	ND	ECD
Carbofenothion	*1.025*	1.012	*1.026*	1.018	TSD
	1.029	1.016	1.031	1.024	
		1.030	*1.045*	*1.046*	
Carbofuran	ND	ND	0.532	0.623	TSD
Carbosulfan	ND	ND	1.120	*1.087*	TSD
Chlorfenson	*0.905*	*0.952*	ND	ND	ECD
Chloridazon	1.040	*1.177*	ND	ND	ECD
Chlorfenvinphos	*0.392*	*0.435*	*0.792*	0.843	TSD
	0.814	*0.452*	0.813	*0.867*	
	0.835	0.858	0.843	0.871	
Chlormephos	*0.316*	*0.353*	*0.333*	*0.357*	TSD
Chlorothalonil	0.617	ND	*0.604*	ND	ECD
Chlortoluron	*0.182*	*0.191*	ND	ND	ECD
Chlorpropham	*0.479*	*0.523*	*0.468*	0.534	ECD
Chlorpyriphos-ethyl	0.761	*0.779*	0.741	*0.758*	TSD
Chlorpyriphos-methyl	0.676	0.710	0.661	0.688	TSD
Chlozolinate	0.827	0.910	0.805	*0.893*	ECD
Clofentezine	0.155	0.203	ND	ND	ECD
Cyflutrin	*1.260*	*1.289*	ND	ND	ECD
	1.267	1.307			
	1.274	1.313			
Cymoxanil	*0.203*	*0.392*	*0.227*	0.386	ECD
Cypermethrin	1.286	1.306	ND	ND	ECD
	1.293	1.315			
	1.301	1.325			
	1.303	1.332			
		1.343			

Table 2 (*continued*)

Cyproconazole	0.962	1.023	*0.962*	1.036	TSD
Cyromazine	ND	ND	0.547	*0.641*	TSD
o'p'-DDT	0.946	*0.942*	ND	ND	ECD
	1	1			
p'p'-DDT	1	1	ND	ND	ECD
Deltamethrin	*1.434*	*1.519*	ND	ND	ECD
	1.459	*1.567*			
Demethon-S-methyl	ND	ND	*0.694*	*0.903*	TSD
Diazinon	0.602	*0.612*	0.584	*0.590*	TSD
Diclobutrazol	0.945	0.983	0.944	*0.977*	ECD
Dichlofluanid	*0.736*	0.822	*0.719*	0.810	ECD
Dichlorvos	0.186	0.238	0.213	0.261	TSD
Dicloran	0.538	0.655	ND	ND	ECD
Dicofol	0.768	0.801	ND	ND	ECD
Dieldrin	*0.931*	0.923	ND	ND	ECD
Dimethoate	0.538	*0.708*	0.532	0.685	TSD
Dimethomorf	*1.490*	ND	ND	ND	ECD
Diphenilammine	0.457	0.239	0.452	*0.498*	TSD
	1.112	1.044			
Endosulfan	*0.885*	0.872	ND	ND	ECD
	0.978	1.004			
Endosulfan-sulphate	*0.494*	*0.469*	ND	ND	ECD
	0.643	0.658			
	1.039	1.082			
Endrin	0.965	0.949	ND	ND	ECD
	1.008	*0.997*			
	1.087	1.049			
	1.100	*1.126*			
Esfenvalerate	1.372	1.428	ND	ND	ECD
	1.394	*1.456*			
Etaconazole	*0.989*	1.007	*0.990*	*1.008*	TSD
	0.995	0.996	*1.012*		
Ethiofencarb	ND	ND	0.627	ND	TSD
Ethion	1	1	1	1	TSD
Ethoprophos	*0.467*	*0.507*	*0.459*	*0.493*	TSD
Fenamiphos	1.139	1.076	*0.896*	*0.944*	TSD
Fenarimol	1.180	*1.193*	ND	ND	ECD
Fenclorfos	*0.702*	*0.726*	0.687	*0.705*	TSD
Fenitrothion	0.723	0.822	0.705	0.808	TSD
Fenoxycarb	ND	ND	1.121	1.161	TSD
Fenpropatrin	*1.116*	1.088	*1.139*	ND	ECD
Fenpropimorf	1.142	1.077	ND	ND	ECD
Fenson	0.780	*0.865*	ND	ND	ECD
Fenthion	*0.756*	0.807	0.738	*0.792*	TSD
	0.760	0.823			
		0.850			
		1.017			
		1.080			
Fenvalerate	1.371	1.426	*1.661*	ND	ECD
	1.393	*1.460*	*1.716*		

(*Continued on p. 20*)

Table 2 (*continued*)

Fluazifop-buthyl	ND	ND	*0.967*	*0.939*	TSD
Flucythrinate	1.303	*1.363*	ND	ND	ECD
	1.320	*1.391*			
Flufenoxuron	*0.608*	ND	*0.613*	0.665	TSD
	0.631	0.688			
Flusilazol	ND	ND	0.944	*0.989*	TSD
Flutriafol	*0.897*	0.851	*0.888*	*0.956*	TSD
		0.956			
		0.380			
Fluvalinate	1.393	*1.497*	ND	ND	ECD
	1.399				
Folpet	0.329	*0.460*	ND	ND	ECD
	0.842	0.910			
Fonofos	0.587	*0.622*	0.575	*0.606*	TSD
Formothion	*0.639*	0.802	0.624	0.785	TSD
Furalaxyl	ND	ND	*0.826*	*0.885*	TSD
Furathiocarb	1.140	1.108	*1.175*	*1.197*	TSD
Heptachlor	0.312	0.591	*0.499*	0.668	ECD
	0.458	0.691			
	0.505				
Heptenofos	0.157	0.203	0.420	0.472	TSD
	0.422	0.488			
Hexaconazole	*0.910*	0.946	*0.901*	*0.933*	
Hexaflumuron	0.311	0.590	0.450	ND	ECD
	0.456				
Imazalil	*0.920*	0.959	ND	ND	ECD
Iprodione	*1.094*	1.116	ND	ND	ECD
		1.109			
Isofenphos	0.835	0.875	0.814	0.860	TSD
λ-cyhalothrin	*1.158*	1.145	*1.227*	1.325	ECD
	1.170	*1.163*			
Lindane	0.572	*0.639*	ND	ND	ECD
Linuron	*0.225*	*0.671*	*0.275*	*0.297*	TSD
	0.556	0.857	*0.712*	0.844	
	0.724				
Malathion	0.742	*0.812*	*0.722*	0.796	TSD
	0.746				
Metalaxyl	ND	ND	*0.681*	*0.750*	TSD
Methamidophos	ND	ND	*0.202*	*0.316*	TSD
Methidathion	*0.863*	0.931	0.846	0.917	TSD
Methiocarb	ND	ND	0.705	0.796	TSD
Metribuzin	0.662	*0.764*	0.648	*0.742*	TSD
Mevinphos	0.306	*0.386*	0.322	0.382	TSD
		0.396		*0.391*	
Molinate	ND	ND	*0.397*	*0.406*	TSD
Monocrotophos	0.457	0.380	*0.510*	0.670	TSD
Myclobutanil	*0.939*	1.023	*0.935*	1.033	TSD
Nuarimol	1.061	1.067	1.066	*1.112*	TSD
Omethoate	*0.431*	*0.603*	*0.429*	*0.586*	TSD
Oxadixil	ND	ND	0.997	*1.145*	TSD

the two columns of different polarities on the same detectors as well as by comparing the pesticides RRT values between different detectors. Quantitative analyses were obtained by exploiting the detector more suitable for the pesticide analysed.

Reliable extraction method coupled with effective

Table 2 (*continued*)

Oxyfluorfen	0.947	0.985	ND	ND	ECD
Parathion-ethyl	0.762	0.846	0.742	*0.838*	TSD
Parathion-methyl	0.676	*0.787*	0.660	*0.768*	TSD
Penconazole	0.820	0.888	ND	ND	ECD
Pendimethalin	0.819	0.855	*0.798*	0.844	ECD
Permethrin	1.218	1.181	ND	ND	ECD
	1.226				
Phenthoate	0.841	0.883	0.820	0.871	TSD
Phorate	0515	*0.548*	*0.504*	0.533	TSD
Phosalone	1.147	*1.173*	1.188	1.333	TSD
Phosphamidon	0.601	*0.718*	0.584	*0.693*	TSD
	0.661	*0.795*	0.645	*0.775*	
Pirimicarb	ND	ND	0.624	0.666	TSD
Pirimiphos-ethyl	*0.802*	0.806	*0.779*	0.788	TSD
	1.140	0.850			
Pirimiphos-methyl	*0.728*		*0.708*	0.725	TSD
Prochloraz	*1.242*	*1.295*	ND	ND	ECD
Procymidone	*0.850*	0.927	ND	ND	ECD
Promethrin	ND	ND	0.685	*0.718*	ECD
Propham	ND	ND	*0.340*	0.383	ECD
Profenofos	*0.924*	0.934	*0.918*	0.920	TSD
Propamocarb	ND	ND	*0.297*	*0.324*	TSD
Propiconazole	1.038	*1.037*	*1.039*	*1.058*	TSD
	1.046	1.042	*1.049*	*1.068*	
Propoxur	ND	ND	*0.441*	*0.528*	TSD
Protoathe	*0.671*	*0.783*	*0.655*	*0.763*	ECD
Propyzamide	0.587	0.687	0.572	0.663	TSD
Pyrazophos	1.185	1.184	1.256	*1.366*	TSD
Pyridaphenthion	1.098	1.117	*1.113*	*1.215*	TSD
Quinalphos	0.835	0.875	0.819	0.861	TSD
Simazine	*0.549*	0.648	0.535	0.628	TSD
	0.948	0.985			
	1.139	1.075			
Tebuconazolo	1.060	*1.096*	1.065	1.161	TSD
Tefluthrin	0.620	*0.618*	ND	ND	ECD
Tetraclorvinphos	*0.515*	*0.569*	*0.873*	0.911	TSD
	0.889	0.926			
Tetradifon	1.137	*1.139*	ND	ND	ECD
Tetrametrina	1.099	1.082	*1.126*	*1.151*	ECD
	1.107	1.088			
Thiabendazole	ND	ND	*0.801*	0.912	TSD
Tolclofos-methyl	*0.684*	*0.731*	*0.669*	*0.709*	TSD
Triadimefon	0.767	0.846	*0.746*	*0.832*	TSD
Triadimenol	0.841	0.931	ND	ND	ECD
Triazophos	*1.017*	N.R.	*1.017*	*1.078*	ECD
Trichlorfon	0.186	0.242	*0.138*	*0.094*	TSD
	0.190	*0.262*	0.214	*0.182*	
	0.329	*0.327*	*0.356*	0.261	
Triflumuron	0.326	*0.456*	ND	ND	ECD
Trifluralin	0.504	*0.541*	ND	ND	ECD
Vamidathion	ND	ND	*0.858*	1.005	TSD
Vinclozolin	0.679	*0.757*	ND	ND	ECD

The peaks resolved on the column considered are shown in italics.
ND=not detectable.

clean-up procedure can be matched to the GC determination in order to develop a multiresidual approach which minimise the risk of false positive.

The HRGC configurations proposed may be a useful tool for the multiresidue determinations and confirmation of pesticide residues present in environmental samples.

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