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An evaluation of the use of laminar C_{18} disks employed as part of the solid phase extraction process. Simultaneous capillary gas chromatography - mass spectrometry determination of triazines, organochlorine pesticides, and polyaromatic hydrocarbons

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Abstract. The combination of solid phase extraction and gas chromatography - mass spectrometry enables fast and reliable analysis of pesticides residues in drinking water. The evaluation of analysis techniques is a priority for laboratories dealing with quality control. The ordinance 89-3 of the 1/3/89 fixes the maximum acceptable concentrations of micro-pollutants in drinking water including pesticides and chemically related substances belonging to highly diversified chemical families. This paper described a multiresidue analysis method of triazines, organochlorine pesticides and polyaromatic hydrocarbons, adapted from the method E.P.A. 525.2. This multiresidue method and especially the solid phase extraction process employing laminar C_{18} disks, is evaluated together with relevant statistical tests as outlined in the NF XP T 90-210 procedure.

Key words. Pesticide analysis – water analysis – solid phase extraction – gas chromatography – mass spectrometry – quality control.

Introduction

The ordinance 89-3 of the 1/3/89 modified by the ordinances 90-330 of the 4/10/90, 91-257 of the 3/7/91 and 95-363 of the 4/5/95 fixes the requirements for quality of drinking water [1]. Pesticides from agricultural origin and similar substances may be researched in agreement with this ordinance. In accordance with the European directive 80/778/CEE of the 07/15/80 revised under directive 98/83/CEE of the 03/11/98 [2], the maximum acceptable levels of each individual compound is set at 0.100 µg/L. The total concentration of all the organic micro-pollutants may not exceed 0.500 µg/L. France goes beyond the European directive by setting acceptable levels at 0.030 µg/L for aldrin, dieldrin, heptachlor and heptachlor epoxide. At present, the number of authorised substances for crop protection is approximatively 3000 and increases continually [3]. It is therefore obviously impossible to monitor all the authorised substances systematically [2]. Hence the World Health Organisation published a priority list of 35 molecules in 1994 [4,5]. This list is based on the physico-chemical properties of the incriminated substances as a first criterion. These properties determine the potential of these substances to contaminate the environment. This list also takes in consideration the toxicity of each pesticide as well as the evaluation of the risks pertaining to a given population.

Since the environment requires stringent controls by employing highly sophisticated analytical techniques, it has become crucial for the controling laboratories to develop quick qualitative and quantitative multiresidue analysis methods for the analysis of micro-organic pollutants [6]. Gas chromatography - mass spectrometry is known as a powerful tool of identification and quantification. It has became a well established technique and many authors described analytical methods using GC-MS [7–12]. In practice, it is necessary to standardise both the methods of sample preparation and analysis. The results obtained will then be reliable and hence enable an increase of environmental supervision.

A very common problem occurs when a laboratory wants to use a technique before having processed the validation of a method completely. Are the experimental conditions of analysis well adapted to the choice of samples to be process? This question is crucial, because it becomes imperative to reduce the experimental error prior processing the samples.

In many instances, it is often necessary to extrapolate a given reference method apply to substances that are not referenced explicitly. It is stated that precautionary measures

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must be taken in such cases [13]. When formalizing a method according to the procedure of quality insurance, a quality plan must take into consideration all the steps of the analytical process, starting from the sample preparation and its preservation. It must include the choice of the exact experimental conditions such as the volume and the percentages of chemicals in a mixture, the temperature and the duration of the extraction, the weight of the test sample and the apparatus tuning and maintenance. The optimisation of all the steps of the process is the most important point of the validation of a method. The analyst must justify all the deviations from the reference method in accordance with quality control procedures, and justify the choice of particular modifications. Furthermore, the analytical material must be in accordance with the requirements of the norm. Some assays are often necessary to test its conformity [14]. In this paper, our method is in compliance with the method E.P.A. 525.2. So we have chosen an empirical approach utilising fewer assays.

Mallinckrodt Baker has developed a solid phase extraction system using laminar disks bonded with C_{18} silica, this material have been described by Pichon [15]. This paper draws the valuation of this material and its use according to the E.P.A. method 525.2 [16]. The tests are based on the comparison between simple reference solutions and synthetic reference solutions fortified by standard addition in a wide range of concentration. The analysis of triazines, chloroacetanilide herbicides, organochlorine pesticides and polyaromatic hydrocarbons is performed simultaneously (Fig. 3). The simultaneous analysis of such different chemical families allows a good evaluation of how reliable of the solid phase extraction system. However, the speed and safety of the technique must also be considered.

Materials and methods

Solid phase extraction

Ultrapure water system	Millipore, Milli-Q+ 185
Methanol	Merck *Suprasolv*
Dichlormethane	Merck *Suprasolv*
Ethyl acetate	Merck *Suprasolv*
Hydrochloric acid 6N	Merck *Suprapur*
n-Dodecane	Sigma-Aldrich, tested for pesticide analysis
Benfluralin	Riedel-de-Haen Pestanal
Extraction disks	Bakerbond Speedisk C_{18} and six-port extraction station
Concentration workstation	Turbo Vap II, Zymark.

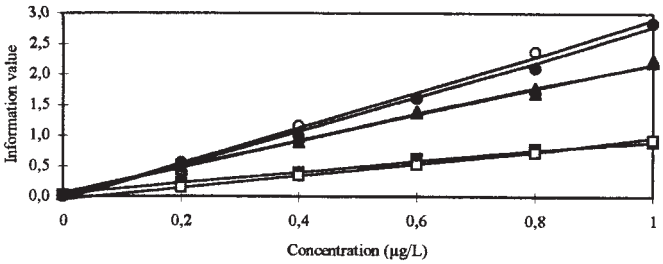

Fig. 1. Comparison of calibration curves obtained from simple and synthetic reference solutions for simazine, atrazine and lindane.

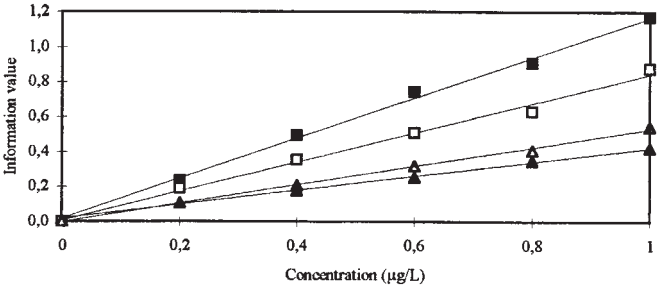

Fig. 2. Comparison of calibration curves obtained from simple and synthetic solutions for hexachlorobenzene and heptachlor epoxide.

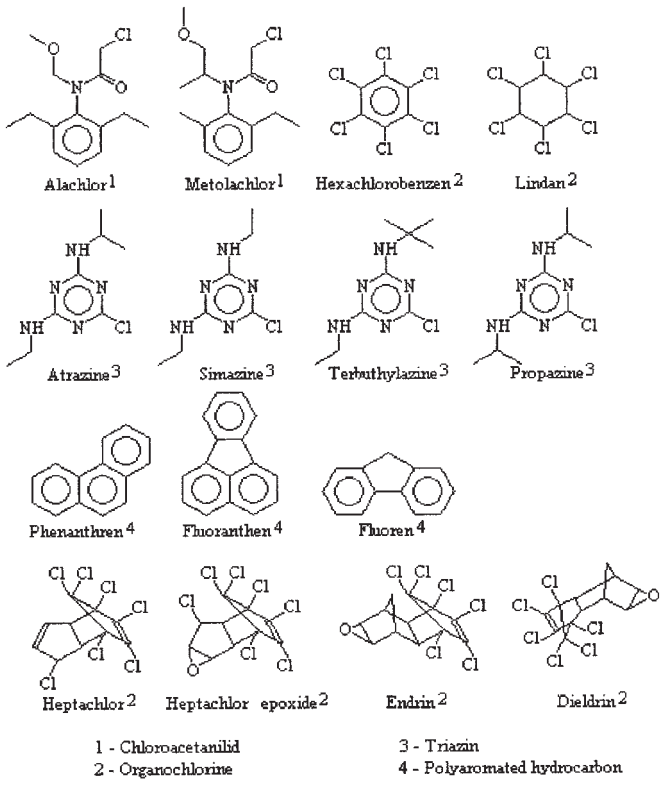

Fig. 3. Chemical structures of extracted compounds.

The solid phase extraction is performed using a 50 mm diameter Bakerbond Speedisk bonded with 750 mg of C_{18} silica. The disk is rinsed out with 5 mL of dichlormethane, than conditioned with 10 mL methanol and 10 mL of ultra pure water. A test sample of 1 L is acidified to a pH of 2 by adding 2 mL of hydrochloric acid 6N. 5 mL of methanol are added to improve the extraction of non-polar and slightly polar compounds. This is followed by the addition of 100 µL of benfluralin [5 mg/L methanol] as internal standard. The sample is filtered with a delivery of 200 mL/min. The elution is performed with 10 mL of dichlormethane and 10 mL of a 1/1 V/V ethyl acetate – dichloromethane mixture. 100 µL of n-dodecane are added in order to minimise loss of volatile compounds and the sample is concentrated to 1 mL by evaporation.

Gas chromatography/mass spectrometry

Chromatograph	Varian GC Star 3400
Autosampler	Varian AS 8200 CX
Mass spectrometer	Varian Saturn 2000
Carrier gas	Helium 6.0 AGA, head column pressure 17 psi
Injector	Varian 1078 with high performance insert for splitless on-column injection
Column	HP1, 100 % dimethylpolysiloxan, L 30m, d 0.25 mm, e 0.25 µm
Standards of pesticides	Riedel-de-Haen Pestanal.

The chromatographic conditions like the temperature of the oven and of the injector were optimised using an experimental design methodology. The methodology was described in a previous article [17]. A test sample of 1 µL is injected. The injector is heated from 60 °C to 260 °C at 300 °C/min and held in splitless mode for 1 min. The temperature program of the column is 80 °C to 180 °C at 25 °C/min to limit solvent effect; held at 180 °C for 1 min; 180 °C to 250 °C at 3 °C/min; and held at 250 °C for 10 min. The qualitative analysis is performed using the mass spectra obtained at the retention time specified for each compound. Each detected molecule is quantified by calculating the following ratio; peak area on ion chromatogram of specific *m/z* / peak area of the internal standard. The retention time and the specific *m/z* used for each compounds are listed in Table I.

Results and discussion

Tables II and III summarise the calibration results obtained for the fifteen compounds from simple reference solutions. These solutions were prepared in ethyl acetate with certified standards. Concentration are expressed as mg/L. It allows the quantification of levels of pesticide under µg/L when taking in consideration the concentration factor resulting from the extraction of real samples. The calibration function

Table I. Analytical parameters of the GC - MS method.

Retention time (min)			*m/z ratios used for quantitation*	
Fluoren	12.65	165	166	
Benfluralin	15.01	276	292	
Simazine	15.86	186	201	
Atrazine	16.28	200	202	215
Hexachlorobenzene	16.39	249	284	286
Propazine	16.66	172	214	229
γ-HCH	17.04	181	183	219
Terbuthylazine	17.19	173	214	229
Phenanthren	18.08	178		
Alachlor	21.44	160	188	
Heptachlor	22.01	272	274	337
Métolachlor	23.79	162	238	
Fluoranthen	26.41	202		
Heptachlor epoxide	26.47	253	289	353
Dieldrin	30.74	263	345	380
Endrin	31.94	245	317	345

for each compound is characterised by the following six parameters [18];

- a, the slope and b, the ordinate at the origin which link the information value Y and the concentration $[C]$ by a linear relation, $Y = a\,[C] + b$,
- r^2, the correlation coefficient which evaluate the linearity of the relationship between the information value Y and the concentration $[C]$,
- S_y, the residual standard deviation of the linear function $Y = a\,[C] + b$, it characterises the dispersion of the values Y_i around the regression straight line,
- S_a, the standard deviation of the slope of the function $Y = a\,[C] + b$,
- S_0, the standard deviation of the method, equal to the residual standard deviation Sy divided by the slope a.

The standard deviation of the method allows the evaluation of the statistic error on the GC-MS results. The uncertainty is insignificant when compared with the level of the information value of each compound. Result shows the precision of calibration obtained from simple reference solutions with the GC/MS quantification technique.

The calibration functions obtained using synthetic reference solutions fortified with standard addition are summarised in tables IV and V. To prepare these solutions, 1 L of drinking water considered as the reference matrix, was acidified to a pH of 2 with 2 mL of hydrochloric acid 6N. This was followed by standard additions using pure certified pesticides as listed, dissolved in methanol. Contrary to the simple reference solutions, the synthetic reference solutions allow the evaluation of all the analytical processes, and espe-

Table II. Calibration functions of triazines and PAHs obtained from simple reference solutions.

[C] (mg/L)	*Simazine*	*Atrazine*	*Propazine*	*Terbuthylazine*	*Fluoren*	*Phenanthren*	*Fluoranthen*
0.200	0.145	0.439	0.596	0.569	0.368	0.295	0.405
0.400	0.343	0.975	1.234	1.192	0.785	0.642	0.826
0.600	0.520	1.374	1.845	1.798	1.236	0.997	1.116
0.800	0.722	1.779	2.549	2.452	1.740	1.454	1.408
1.000	0.926	2.195	3.064	2.935	2.209	1.776	1.869
Slope	0.9700	2.1586	3.1261	2.9955	2.3190	1.8870	1.7556
Origin	–0.0508	0.0573	–0.0181	–0.0080	–0.1239	–0.0994	0.0714
Corr. r^2	0.9995	0.9966	0.9983	0.9979	0.9989	0.9973	0.9923
Residual S.D.	0.0072	0.0399	0.0407	0.0432	0.0244	0.0309	0.0489
Slope S.D.	0.0107	0.0595	0.0607	0.0643	0.0364	0.0461	0.0729
Method S.D.	0.007	0.019	0.013	0.014	0.011	0.016	0.028

Given values are the area of the reconstituted chromatographic spike of pesticides using specific *m/z* rapports divided by the area of the spike of the internal standard (benfluralin 500 µg/L).

Table III. Calibration functions of organochlorine pesticides obtained from simple reference solutions.

Concentration (mg/L)	*Hexachloro -benzene*	*γ-HCH*	*Alachlor*	*Heptachlor*	*Metolachlor*	*Heptachlor epoxide*	*Dieldrin*	*Endrin*
0.200	0.231	0.545	0.416	0.154	0.931	0.105	0.052	0.026
0.400	0.494	1.153	0.967	0.298	1.938	0.179	0.142	0.078
0.600	0.744	1.609	1.398	0.442	2.940	0.254	0.236	0.130
0.800	0.906	2.375	1.875	0.606	4.055	0.348	0.355	0.182
1.000	1.173	2.833	2.276	0.724	4.923	0.423	0.460	0.228
Slope	1.1472	2.8990	2.3142	0.7246	5.0498	0.4028	0.5142	0.2540
Origin	0.0212	-0.0365	-0.0021	0.0099	-0.0724	0.0200	-0.0593	-0.0236
Corr. r^2	0.9946	0.9944	0.9972	0.9982	0.9989	0.9981	0.9972	0.9994
Residual S.D.	0.0267	0.0689	0.0385	0.0097	0.0526	0.0056	0.0086	0.0019
Slope S.D.	0.0398	0.1027	0.0574	0.0144	0.0785	0.0083	0.0128	0.0028
Method S.D.	0.023	0.024	0.017	0.013	0.010	0.014	0.017	0.007

Given values are the area of the reconstituted chromatographic spike of pesticides using specific m/z ratios divided by the area of the spike of the internal standard (benfluralin 500 µg/L).

cially the solid phase extraction technique. The results in tables IV and V show clearly that the responses with the disks are linear over all the calibration range. Furthermore, these results confirm that the octadecyl silica phase is suitable for the extraction of compounds belonging to various chemical families over a wide range of polarity. These disks are perfectly adapted to the development of multiresidue analysis. Using this material, the moderately polar herbicides and the non-polar compounds like polyaromated hydrocarbons are being extracted simultaneously.

When comparing the results in tables II, III, IV and V, it appears that the slope standard deviation, the residual and the method standard deviations are higher for calibration curve using synthetic reference solutions than the others. It is due to the uncertainty induced by the extraction process. But the extent of this uncertainty is slight, showing the homogeneity of the disks properties.

Tables VI and VII summarise the results of statistical tests of Snedecor and Student, which are described by the XP T 90-210 procedure [18]. The Snedecor test evaluates the deviation between the variance of the slope of the two calibration functions. The deviation is significant if the *F*-value is superior to the tabulate value of Snedecor. The test is applied with 3 and 4 degrees of liberty corresponding to the number of points of each calibration range minus 2, and at the level of significance $\alpha = 0.01$ ($F_{3;4;1\%} = 24.3$). Finally, the statistical deviations between the variance of the two calibration procedures are insignificant for all the produces. This result confirms that the solid phase extraction process does not induce particular dispersion.

Table IV. Calibration functions of four triazines and three PAHs obtained from synthetic reference solutions of drinking water extracted with C_{18} laminar speedisks.

Standard addition (µg/L)	*Simazine*	*Atrazine*	*Propazine*	*Terbuthylazine*	*Fluoren*	*Phenanthren*	*Fluoranthen*
0.000	0.023	0.038	0.000	0.000	0.129	0.189	0.070
0.200	0.223	0.484	0.617	0.604	0.621	0.602	0.507
0.400	0.396	0.889	1.258	1.161	1.070	1.028	0.915
0.600	0.616	1.394	1.806	1.720	1.512	1.317	1.281
0.800	0.782	1.690	2.359	2.131	1.901	1.630	1.624
1.000	0.896	2.242	3.151	2.946	2.437	2.053	1.973
Slope	0.8945	2.1637	3.0756	2.8387	2.2607	1.8131	1.8903
Origin	0.0423	0.0412	-0.0060	0.0077	0.1480	0.2300	0.1166
Corr. r^2	0.9930	0.9965	0.9973	0.9942	0.9986	0.9958	0.9975
Residual S.D.	0.0315	0.0538	0.0672	0.0908	0.0354	0.0493	0.0394
Slope S.D.	0.0376	0.0643	0.0803	0.1085	0.0423	0.0590	0.0471
Method S.D.	0.035	0.025	0.022	0.032	0.016	0.027	0.021

Given values are the area of the reconstituted chromatographic spike of pesticides using specific *m/z* ratios divided by the area of the spike of the internal standard (benfluralin 500 ng/L).

Table V. Calibration functions of eight organochlorine pesticides obtained from synthetic reference solutions of drinking water extracted with C_{18} laminar speedisks.

Standard addition (µg/L)	*Hexachloro -benzene*	*γ-HCH epoxide*	*Alachlor*	*Heptachlor*	*Metolachlor*	*Heptachlor*	*Dieldrin*	*Endrin*
0.000	0.000	0.000	0.016	0.007	0.000	0.004	0.000	0.004
0.200	0.188	0.550	0.571	0.170	1.180	0.106	0.136	0.033
0.400	0.352	0.992	1.036	0.286	2.267	0.208	0.189	0.076
0.600	0.509	1.601	1.509	0.484	3.459	0.318	0.297	0.122
0.800	0.629	2.100	1.933	0.692	4.276	0.408	0.385	0.173
1.000	0.875	2.847	2.513	0.799	5.280	0.544	0.463	0.228
Slope	0.8361	2.7849	2.4352	0.8187	5.2688	0.5306	0.4527	0.2263
Origin	0.0076	-0.0442	0.0453	-0.0029	0.1092	-0.0008	0.0188	-0.0072
Corr. r^2	0.9928	0.9948	0.9982	0.9925	0.9968	0.9976	0.9907	0.9904
Residual S.D.	0.0298	0.0842	0.0428	0.0298	0.1246	0.0109	0.0183	0.0093
Slope S.D.	0.0356	0.1006	0.0511	0.0357	0.1489	0.0131	0.0219	0.0111
Method S.D.	0.036	0.030	0.018	0.036	0.024	0.021	0.040	0.041

Given values are the area of the reconstituted chromatographic spike of pesticides using specific m/z ratios divided by the area of the spike of the internal standard (benfluralin 500 ng/L).

The comparison between the values of the slope of the calibration curves is performed using the Student test. The slope are significantly different if the *t*-value is superior to the tabulate value of Student with 7 degrees of liberty, (5 – 2 + 6 – 2 = 7), and a level of significance $\alpha = 0.001$ ($t_{7;0,999} = 5.41$). The results contained in Tables V and VI shows that only hexachlorobenzene and heptachlor epoxide fail the Student test. For the thirteen other molecules, it is possible to calibrate the method only from simple reference solution without extracting systematically a complete range of calibration. But the calibrations and the quality controls of heptachlor epoxide and hexachlorobenzene must imperatively be performed from synthetic reference solutions, fortified by standard additions and extracted by the same method than the real samples. The figures 1 and 2 draw the comparison between simple and synthetic reference solutions for five pesticides. The calibration results obtained from extracted solutions for simazine, atrazine and lindane

Table VI. Comparison among the calibration functions of simple reference solutions and synthetic reference solutions using the *F*-test of Snedecor and the *t*-test of Student, triazines and PAHs.

	Simazine	*Atrazine*	*Propazine*	*Terbuthylazine*	*Fluoren*	*Phenanthren*	*Fluoranthen*
F (Snedecor*)	12.34	1.17	1.75	2.84	1.36	1.64	2.39
T (Student**)	2.58	0.08	0.70	1.70	1.46	1.37	2.26

* : The standard deviations of the slopes are not significantly different if $F < 24.3$. ** : The slopes of the calibration functions are not significantly different if $T < 5.41$.

Table VII. Comparison among the calibration functions of simple reference solutions and synthetic reference solutions using the F-test of Snedecor and the t-test of Student. organochlorine pesticides.

	Hexachloro -benzene	*g-HCH*	*Alachlor*	*Heptachlor*	*Metolachlor*	*Heptachlor epoxide*	*Dieldrin*	*Endrin*
F (Snedecor*)	1.25	1.04	1.26	6.09	3.60	2.45	2.94	15.43
T (Student**)	**8.31**	1.12	2.25	3.29	1.77	**11.32**	3.32	3.22

* : The standard deviations of the slopes are not significantly different if $F < 24.3$. ** : The slopes of the calibration functions are not significantly different if $T < 5.41$.

Table VIII. Recoveries of triazines and PAHs in drinking water using C_{18} laminar Speedisks (3 trials).

Standard addition (ng/L)	*Simazine* %	*Atrazine* %	*Propazine* %	*Terbuthylazine* %	*Fluoren* %	*Phenanthren* %	*Fluoranthen*
200	103	103	99	101	106	109	124%
400	96	99	101	97	101	111	120%
600	102	105	96	96	99	100	115%
800	98	96	94	89	96	95	111%
1000	90	102	101	98	100	99	108%
Mean recovery	98	101	98	96	100	103	116%
S. D.	5	4	3	4	4	7	7%

Table IX. Recoveries of organochlorine pesticides in drinking water using C_{18} laminar Speedisks (3 trials).

Standard addition (ng/L)	*Hexachloro -benzene* %	*g-HCH* %	*Alachlor* %	*Heptachlor* %	*Metolachlor* %	*Heptachlor epoxide* %	*Dieldrin* %	*Endrin* %
200	82	95	120	113	117	126	133	56
400	77	86	110	96	112	126	92	70
600	74	92	108	110	114	130	96	77
800	69	91	104	118	106	125	94	83
1000	76	98	108	109	105	134	90	88
Mean recovery	76	92	110	109	111	128	101	75
S. D.	5	5	6	8	5	4	12	11

are perfectly superimposable to the results obtained from simple reference solutions. These three examples illustrate the ten other substances, which provide perfect results. But it is clear in figure 2, that the two calibration curves do not correspond for heptachlor epoxide and hexachlorobenzene.

Finally, tables VIII and IX summarise the recoveries of extraction obtained for each compound over the range of calibration. The recoveries are calculated from three trials at each level, the means recoveries take into consideration all the trials and all the levels. The results are about 100% for atrazine, simazine, propazine, terbuthylazine, lindane, alachlor and metolachlor. These pesticides are often detected in surface and drinking water samples, and constitute some good indicators to point out pesticide contamination on the environment. But the recoveries of hexachlorobenzene and heptachlor epoxide confirm the results of Table VII. The hexachlorobenzene is underestimated when simple reference solutions are used for calibration. Losses are probably due to concentration by evaporation. Effectively, some assays without dodecane show that the only addition of 100 µL of this reagent improves the recoveries of hexachlorobenzene of about 50%. At the opposite, the heptachlor epoxide is overestimated. The hypothesis of a co-eluted molecule, which increases the signal measured, and the suspicion of external contamination in the laboratory, must be rejected when taken in consideration the perfect linearity of the response. It is probably the addition of reagents in the synthetic reference solutions that influence the interactions between the solvent and the solute. It is well known that a difference concerning the polarity of the solvent does not influence the retention time during a chromatographic separation. But this polarity can perhaps influence the conditions of the ionisation of the molecules in the mass detector and improves the response signal.

Figure 4 shows the total chromatogram of fortified groundwater. The concentration of 0.500 µg/L allows a good illustration because spikes are well visible. For very low concentration, the spikes can be visible only on the reconstituted chromatogram from the specific *m/z* ratios. Figures 5 and 7 show the chromatographic spikes of atrazin and metolachlor at very low concentrations in a real groundwater extracted and quantitatively analysed by our method. Figures 6 and 8 shows the mass spectra corresponding to the chromatograms of the Figures 5 and 7. The specific *m/z* ratios of atrazine and metolachlor can be perfectly identified.

Table X presents the limits of quantification of the method for each compound. These limits have been estimated by experimental approach. Five synthetic reference solutions in pure water have been spiked with 0.005 – 0.01 – 0.02 – 0.05 and 0.10 µg/L of each pesticide. The quantitation limit corresponds to the lowest concentration detected and quantified correctly when the extraction and the analysis were performed. Figures 7 and 8 illustrate the case of the metolachlor, these figures are corresponding to a concentration of 0.01 µg/L in a real groundwater. The ratios 162 and 238 are perfectly identifiable and allows a qualitative recog-

Table X. Quantitation limits of organic compounds given in µg/L.

Fluoren	0.01	Phenanthren	0.01
Benfluralin	Internal std.	Alachlor	0.01
Simazine	0.02	Heptachlor	0.02
Atrazine	0.01	Métolachlor	0.01
Hexachlorobenzene	0.02	Fluoranthen	0.01
Propazine	0.01	Heptachlor epoxide	0.02
g-HCH	0.01	Dieldrin	0.02
Terbuthylazine	0.01	Endrin	0.05

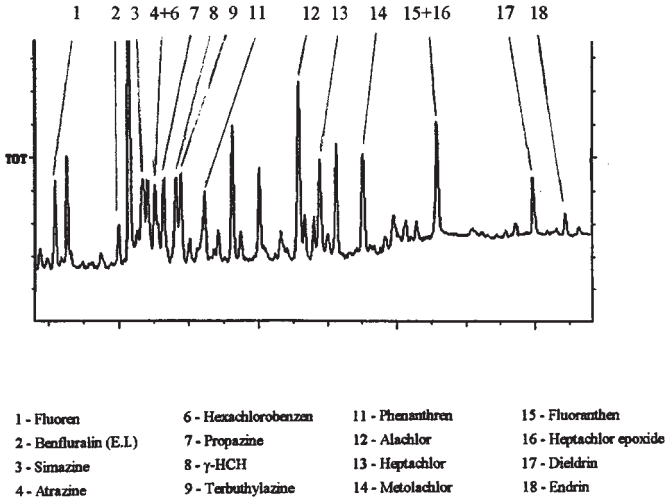

Fig. 4. Total chromatogram of a fortified groundwater (0,500 µg/L).

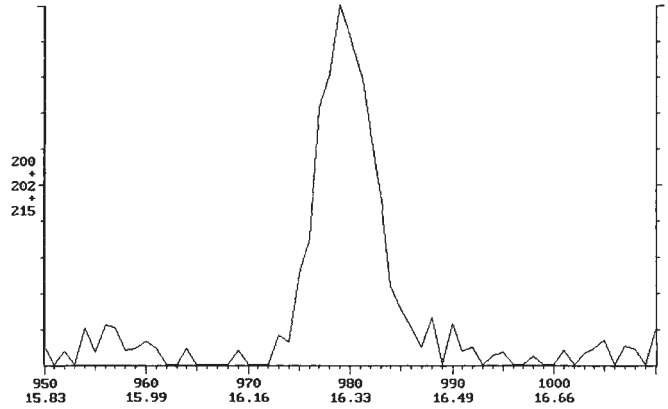

Fig. 5. Reconstituted chromatographic spike of atrazine using specific m/z rapports 200, 202 and 215 in a real groundwater at 0,02 µg/L.

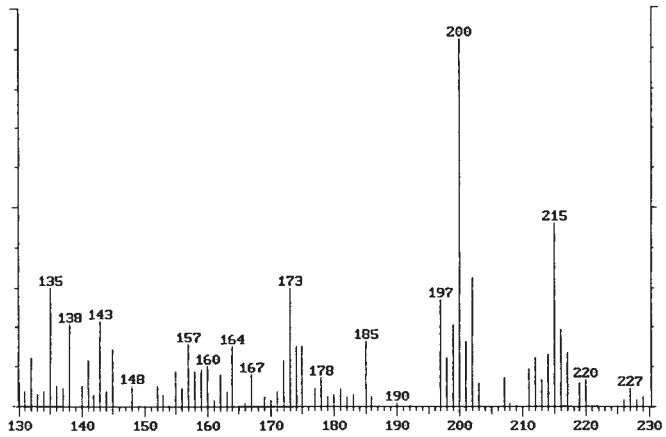

Fig. 6. Mass spectrum of atrazine in a real groundwater at 0,02 µg/L.

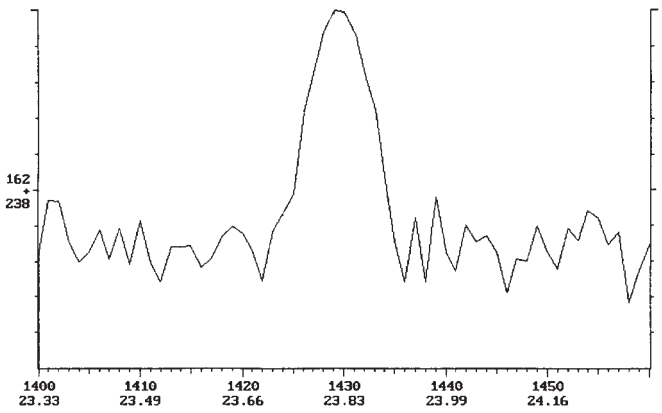

Fig. 7. Reconstituted chromatographic spike of metolachlor using specific m/z rapports 162 and 238.

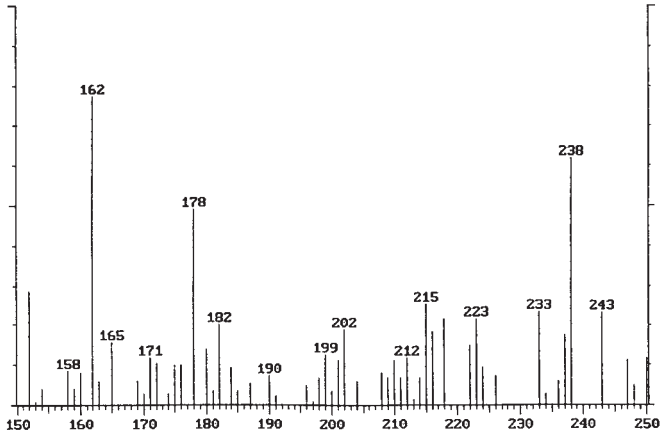

Fig. 8. Mass spectrum of metolachlor in a real groundwater at 0,01 µg/L.

nition of the compound without dubiousness. The chromatographic spike is well distinguishable from the residual noise, this spike is easily integrable to perform the quantitative analysis.

Conclusion

This work shows clearly the suitability of the disks in the field of application of the method E.P.A. 525.2. However, it underlines the necessity for the analyst to remain vigilant and critical, because strong results can occur even when applying a referenced method.

Nevertheless, several advantages derive from this solid phase extraction system [19];

- This system allows used small quantity of solvent for the extraction, which reduces the cost of the analyses and increases the safety of sample handling.
- This solid phase extraction method is faster than the classic liquid/liquid extraction. The use of two six-port stations allows 12 samples to be simultaneously extracted and increases considerably the productivity of the laboratory.
- The homogeneity of the disks and the slight dispersion observed ensure both repeatability and accuracy of the analysis.
- The samples can be filtered with an important delivery, it is then possible to use high volume and to improve the sensitivity of the method.
- No particular losses and no saturation of the silica phase were observed in a wide range of concentration.

This article points out the validity of the solid phase extraction by a step of quality control. The development of referenced methods is a daily problem for many laboratories because it is necessary to control the quality, the cost and the duration of the analysis. But the optimisation of an analytical procedure is subordinate to a perfect knowledge of the phenomena that rule the analytical conditions. This knowledge is essential to ensure the analyst against all the difficulties even in the precise field of a normalised method.

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