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Fast temperature programming in routine analysis of multiple pesticide residues in food matrices

Kateřina Maštovská, Jana Hajšlová*, Michal Godula, Jitka Křivánková, Vladimír Kocourek

Institute of Chemical Technology, Department of Food Chemistry and Analysis, Technická 3, 166 28 Prague 6, Czech Republic

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Abstract

Flash gas chromatographic (GC) analysis of 15 organophosphorus pesticides commonly occurring in food crops was performed using the Thermedics Detection EZ Flash upgrade kit installed in the oven of a HP 5890 Series II Plus gas chromatograph. The temperature program and splitless time period were the main parameters to be optimized. In the first set of experiments wheat matrix-matched standards were analyzed both by: (i) the flash GC technique (resistive heating of a 5 m capillary column), and (ii) the conventional GC technique (moderate oven temperature programming of a 30 m capillary column). Using the flash GC technique, the analysis time was reduced by a factor of more than 10 compared to the conventional GC technique. Dramatically improved detectability of analytes was achieved due to much narrower peak widths. The flash GC technique was compared with another approach to faster GC analysis employing a 5 m column and fast temperature programming with a conventional GC oven. In comparison with this alternative, in the case of flash GC significantly better retention time repeatability was observed. The other superiority of resistive heating is very rapid cooling down (i.e., equilibration to the initial conditions) which contributes to the increased sample throughput.

Keywords: Temperature programming; Food analysis; Flash chromatography; Pesticides

1. Introduction

Faster gas chromatographic (GC) separation is a generally beneficial option, since the decreased time of analysis results in the increased sample throughput and, consequently, the laboratory operating costs can be reduced significantly.

Reduction of analysis time can be achieved either by changing column parameters (shorter column length, smaller column inner diameter, thinner film of stationary phase) or operational parameters (faster temperature program rate, isothermal analysis, different carrier gas, higher carrier gas flow-rate, vacuum outlet operation) or a combination of both approaches can be applied. Using narrow-bore capillary columns (reduced inner diameter) is a common way of obtaining fast GC separation [1–4]. On the other hand their application in trace analysis is rather impractical, not only because of the low sample capacity (sample clean-up of a crude extract, commonly preceding GC separation, unavoidably leaves some matrix components in purified extract, often at concentration levels higher than analytes), but also due to the difficulties encountered with splitless injection (column clogging). As regards operational

*Corresponding author. Tel./fax: +420-2-2435-3185.
E-mail address: jana.hajslova@vscht.cz (J. Hajšlová).

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parameters, temperature has an essential influence on the gas chromatographic separation. Since isothermal GC is still restricted to the analysis of samples with a relatively narrow boiling point range, fast temperature programming (with conventional GC ovens or by resistive heating) is considered here.

Fast temperature programming with conventional GC ovens usually combines optimized conditions such as narrow-bore capillary columns and thin film of stationary phases with a powerful oven heater. The thermal mass of the GC oven, however, limits the heating and cooling rates. This is a severe limitation to achieving the full theoretical potential for fast GC analysis. Not only fast temperature programming rates during the GC separation, but also minimal cooling-down period between two subsequent runs (i.e., rapid equilibration to initial conditions) can significantly contribute to the increased sample throughput.

Resistive heating techniques eliminate the conventional air bath ovens. Electrical current is employed to heat a conductive material (a metal) located in very close distance from the column. Its temperature can be determined by resistance measurements. The temperature program is converted into a resistance program and the electrical circuit applies the appropriate amount of power to change the resistance per unit time. Thermedics Detection (USA) was the first company to commercialize resistive heating for GC. So called ''flash GC'' is available nowadays as a stand-alone system (Flash-GC instrument) or as an upgrade kit (EZ Flash) which enables a conventional GC system to be converted to a flash GC system [5,6]. The performance of the Flash-GC instrument has been demonstrated for instance on the rapid GC screening for drugs of forensic interest [7]. Several different approaches for obtaining fast GC separation of a saturated hydrocarbon sample such as narrow-bore capillary column GC and flash GC (using a Flash-2D-GC instrument) have been also investigated [8]. The possibilities and limitations of fast temperature programming employing the EZ Flash upgrade kit for the analysis of an alkanes mixture, an industrial glycol mixture and phenol and cresol in diesel oil have been discussed [9]. A study comparing the conventional GC analyses of standard solutions containing *n*-alkanes and polycyclic aromatic hydrocarbons with EZ Flash analyses as well as with fast GC analyses using a short narrow bore capillary column and fast oven temperature programming has been published [10]. Both fast GC techniques have been demonstrated to be capable to reduce significantly the analysis time, however, speeding up the analysis in any of this way decreases the resolution of the peaks. On the other hand, it has been also shown that under some circumstances the loss of resolution does not greatly affect the analysis of examined samples (e.g., single target analyte well separated from matrix components and/or relatively clean extract). Two routes towards a faster GC separation: (i) resistive heating of a short column, and (ii) a short column operated at carrier gas velocities above the optimum have been studied and the advantage of the resistive heating technique documented. The possibility of rapid screening of *n*-alkanes, selected polycyclic aromatic hydrocarbons, triazines and organophosphorus pesticides employing the EZ Flash with flame ionization detection (FID) has been tested, however, only standards dissolved in neat solvents have been used for the experiments [11]. The repeatability of different characteristics of GC analysis, including retention times, over the wide range of programming rates (48°C/min to 1200°C/min) using the EZ Flash system for the analysis of an alkanes mixture has been studied [12]. No statistically significant difference between the precision of the retention times at the lowest and the highest tested temperature programming rates has been observed.

In the presented study, EZ Flash GC operation conditions for separation of 15 organophosphorus pesticides were optimized. Wheat matrix-matched standards were analyzed by both flash and conventional GC techniques and the results were compared. Alternative approach to faster GC analysis employing a short capillary column and fast temperature programming with a conventional GC oven was also investigated.

2. Experimental

2.1. Chemicals and materials

Pesticide standards, all 95% or higher purity, were obtained from Dr. Ehrenstorfer (Germany). Standard

Table 1
Concentrations of pesticides in standard solutions A–D (in ethyl acetate)

Pesticide	Concentration level (in μg/ml)			
	A	B	C	D
Chlorpyrifos-ethyl	2.208	0.442	0.221	0.044
Chlorpyrifos-methyl	2.375	0.475	0.238	0.048
Diazinon	2.304	0.461	0.230	0.046
Dichlorvos	2.670	0.534	0.267	0.053
Dimethoate	3.085	0.617	0.309	0.062
Ethion	2.844	0.569	0.284	0.057
Malathion	2.590	0.518	0.259	0.052
Methidathion	2.290	0.458	0.229	0.046
Mevinphos	3.910	0.782	0.391	0.078
Omethoate	4.870	0.974	0.487	0.097
Parathion-ethyl	1.956	0.391	0.196	0.039
Parathion-methyl	2.046	0.409	0.205	0.041
Phosalone	2.655	0.531	0.266	0.053
Phosmet	2.225	0.445	0.223	0.045
Pirimiphos-methyl	2.188	0.438	0.219	0.044

solutions (A–D) were prepared in ethyl acetate, see Table 1. The solvents used (ethyl acetate, cyclohexane) were analytical grade (Merck, Germany). Wheat grains were obtained at a retail market.

2.2. Apparatus

An automated high-performance gel permeation chromatography (HPGPC) system (Gilson, France) equipped with a PL gel (600×7.5 mm, 50 Å) high-performance column (PL Labs., UK) was used for the clean-up of wheat extracts. All solvent reductions were made on a Büchi rotary evaporator.

Flash GC experiments were performed using the Thermedics Detection EZ Flash upgrade kit installed in the oven of a Hewlett-Packard HP 5890 Series II Plus gas chromatograph equipped with electronic pressure control (EPC), a split/splitless injector, a flame ionization detection (FID) system, a flame photometric detection (FPD) system and a HP 7673A autosampler. The data were processed on HP GC Chemstation A.04.05. The EZ Flash upgrade kit consisted of a control module, a Flash-GC column (5 m×0.25 mm, 0.25 μm TDX-RTX 5) and interface heaters for the injector and detector. Since the kit was originally dedicated to the use with FID, a laboratory-made capillary adapter enabling its connection with FPD was made to provide selective detection for organophosphorus pesticides. For conventional GC–FPD analysis a 30 m×0.25 mm, 0.25 μm DB-5 capillary column (J & W Scientific) was used.

Another approach to faster GC analysis employing a short capillary column and fast temperature programming with a conventional GC oven was also investigated. For this purpose a 5 m×0.25 mm, 0.25 μm DB-5MS capillary column (J & W Scientific) was installed into a Hewlett-Packard HP 6890 Plus gas chromatograph equipped with EPC, a split/splitless injector, a nitrogen–phosphorus detection (NPD) system and a HP 7683 autosampler. This instrument enables maximum temperature programming rate of 2°C/s.

2.3. Preparation of matrix-matched standards

A 25-g amount of wheat sample was homogenized with 25 g of anhydrous sodium sulfate (Na_2SO_4) and 100 ml of ethyl acetate for 2 min using a Turrax macerator at 10 000 rpm. The homogenate was filtered through a layer of 20 g anhydrous sodium sulfate and the filter cake was rinsed three times with 25 ml of ethyl acetate. The combined filtrates were rotary evaporated (38°C, 250 mbar) to a volume of 25 ml and the final volume of the crude extract was then adjusted in a volumetric flask to 50 ml with cyclohexane.

A 2-ml volume of crude extract was injected onto a HPGPC column, under conditions as follows: cyclohexane–ethyl acetate (1:1, v/v) mobile phase, flow-rate 1 ml/min, collected fraction 15.5–31 ml. This collected ''pesticide'' fraction was rotary evaporated and any remaining solvent was blown down under a gentle stream of nitrogen.

The residue remaining after solvent evaporation was redissolved in 1 ml of standard solution (standard solutions A–D, see Table 1) to obtain the matrix-matched standards simulating real samples. Blank samples were prepared by redissolving the residue in 1 ml of ethyl acetate.

2.4. Gas chromatographic conditions

The following conditions were the same for both conventional and flash GC experiments: helium as a carrier gas (constant flow 1 ml/min), inlet tempera-

ture 250°C, injection volume 1 μl (splitless); detection: FID (300°C; air 430 ml/min, hydrogen 30 ml/min, make-up (helium) 20 ml/min; data acquisition rate 20 Hz) and FPD (270°C; air 120 ml/min, hydrogen 100 ml/min, make-up (helium) 20 ml/min; data acquisition rate 20 Hz). However, different columns, splitless time periods and temperature programs were used. In comparison with conventional GC a shorter splitless time period in the case of flash GC was applied (1.9 min for conventional GC and 0.5 min for flash GC). The optimization of this parameter will be discussed later. The conventional GC temperature program was set as follows: 60°C for 2 min, 10°C/min to 180°C, 2°C/min to 240°C, 15°C/min to 325°C (held for 3 min). The flash GC temperature program was: 60°C for 0.5 min, 360°C/min to 90°C, 63.5°C/min to 180°C, 82.9°C/min to 325°C (held for 1.25 min).

Fast GC experiments employing a short capillary column and fast temperature programming with a conventional GC oven were performed under the following conditions: helium as a carrier gas (constant flow 1 ml/min), inlet temperature 250°C, injection volume 1 μl (splitless), splitless time period 0.5 min; detection: NPD (300°C; air 60 ml/min, hydrogen 3 ml/min, make-up (nitrogen) 10 ml/min; data acquisition rate 20 Hz); temperature program: 60°C for 0.5 min, 120°C/min to 90°C, 63.5°C/min to 180°C, 82.9°C/min to 325°C (held for 1.25 min).

3. Results and discussion

Since the EZ Flash upgrade kit we had available was originally dedicated for coupling with a HP FID system, the first set of experiments was performed with this detector. The temperature program and splitless time period were the main parameters to be optimized. Since the total run times were short (about 5 min), optimization of the temperature program could be performed quickly. Using an RTX-5 capillary column it was not, however, possible to obtain good separation of all analytes (RTX-5 stationary phase seemed to be less selective for the analyzed compounds than, for example, DB-5MS – see below). Some compounds contained in the test mixture of 15 organophosphorus pesticides remained unresolved, see Fig. 1. The first critical pair was chlorpyrifos-methyl and parathion-methyl (coelution

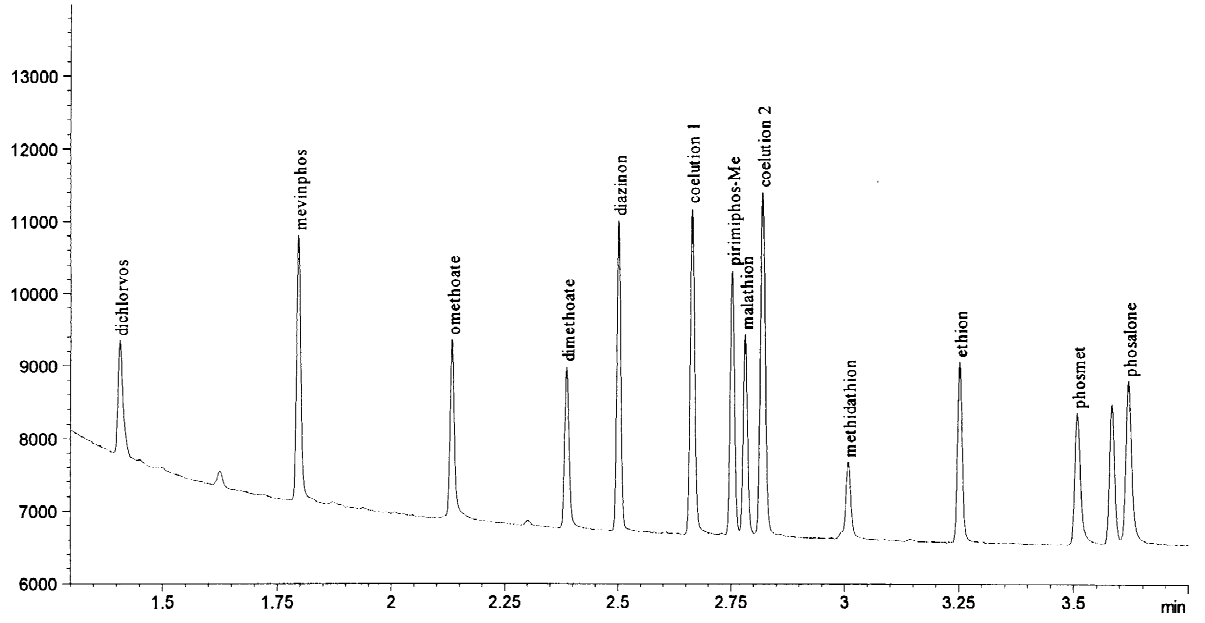

Fig. 1. Chromatogram of a flash GC–FID analysis of the standard solution at concentration level A, representing the optimized conditions (see the flash GC conditions in Section 2.4).

1) and the second one chlorpyrifos-ethyl and parathion-ethyl (coelution 2).

A parameter that is essential to set up in splitless injection is the splitless time period, i.e., the time for which the split vent is closed during the injection. Considering the length of the column and the demand for keeping the total run time as short as possible, it was obvious that the splitless time period duration had to be shorter compared to a conventional GC analysis. The effect of splitless time period duration (0.1–0.9 min) on responses of analytes and their eluting band shapes was studied. For this purpose appropriate temperature programs were created using the same temperature programming rates (as in the flash GC temperature program in Section 2.4), but different initial times corresponding to the splitless time period used. As expected, the longer the splitless time period, the more sample was transferred onto the GC column. However, with increased time of split vent closed, the expanding flooded zone started to cause some distortion of peaks of early eluting analytes. As a compromise, a 0.5 min splitless time period was set.

Considering pesticide analysis, flash GC–FID is undoubtedly suitable for a rapid analysis of pesticide formulations or a check of standards purity. However, for analysis of residues in complex samples such as plant extracts, specific detectors are needed. For the purpose of determination of organophosphorus pesticides in wheat the EZ Flash upgrade kit was connected to a HP FPD system by means of a laboratory-made capillary adapter whereby the selective detection of target analytes was enabled. Wheat matrix-matched standards were analyzed by both flash and conventional GC–FPD and the results were compared. Flash GC analyses were performed under the conditions optimized during the experiments with FID. In the case of the conventional GC, the laboratory-validated procedure was employed. A six times longer (30 m) capillary column was used with the inner diameter and the film thickness the same for both conventional and flash GC columns (the stationary phases were similar). The splitless time period applied in conventional GC experiments was approximately four times longer (1.9 min), and under these conditions a larger amount of sample was transferred onto the column compared to flash GC. In spite of this, with flash GC significantly lower detection limits of all analytes were achieved. Fig. 2 shows chromatograms of the lowest matrix-matched standard (concentration level D) obtained by both GC techniques.

As can be seen, using the conventional GC system, at the lowest concentration level some of the pesticides were not detected. Owing to much narrower peak widths obtained by flash GC, improved detectability of analytes (higher signal-to-noise ratio) could be achieved, even if the amount of analytes transferred onto the column was higher in the case of conventional analysis (due to the longer splitless time period). This is demonstrated in Fig. 3, where peak areas, peak heights and peak widths of three selected pesticides (diazinon, pirimiphos-methyl and ethion) in the wheat matrix-matched standard at concentration level D obtained by conventional and flash GC are compared. As it is evident, using the flash GC technique, approximately ten times narrower peaks were obtained which resulted in higher peaks (approx. 3–4 times) and a higher signal-to-noise ratio, even if absolute peak areas were about half of those obtained by the conventional GC technique. The use of flash GC reduced the analysis time by a factor of more than 10 (5 min total run time instead of 53 min), although some resolution had to be sacrificed.

To assess whether fast temperature programming realized by a modern gas chromatograph represented by a HP 6890 Plus (equipped with a 5 m long capillary column) could be used in the routine practice substituted by resistive heating, experiments employing both techniques under practically identical conditions were realized. As given in Experimental, almost identical temperature programs were set for both column heating techniques, with exception of the initial phase: the programming rate 360°C/min applied during the first 5 s in flash GC exceeded the maximum temperature programming rate of a HP 6890 Plus gas chromatograph which is 120°C/min. Since qualitative GC employing conventional detectors heavily relies on the precision of retention times, repeatability of this parameter is critical for unbiased identification of analytes. Ten repeated injections of standard solutions containing organophosphorus pesticides at concentration levels A–C were made. While retention time relative standard deviations (RSDs) in the range of 0.027 to 0.057% were

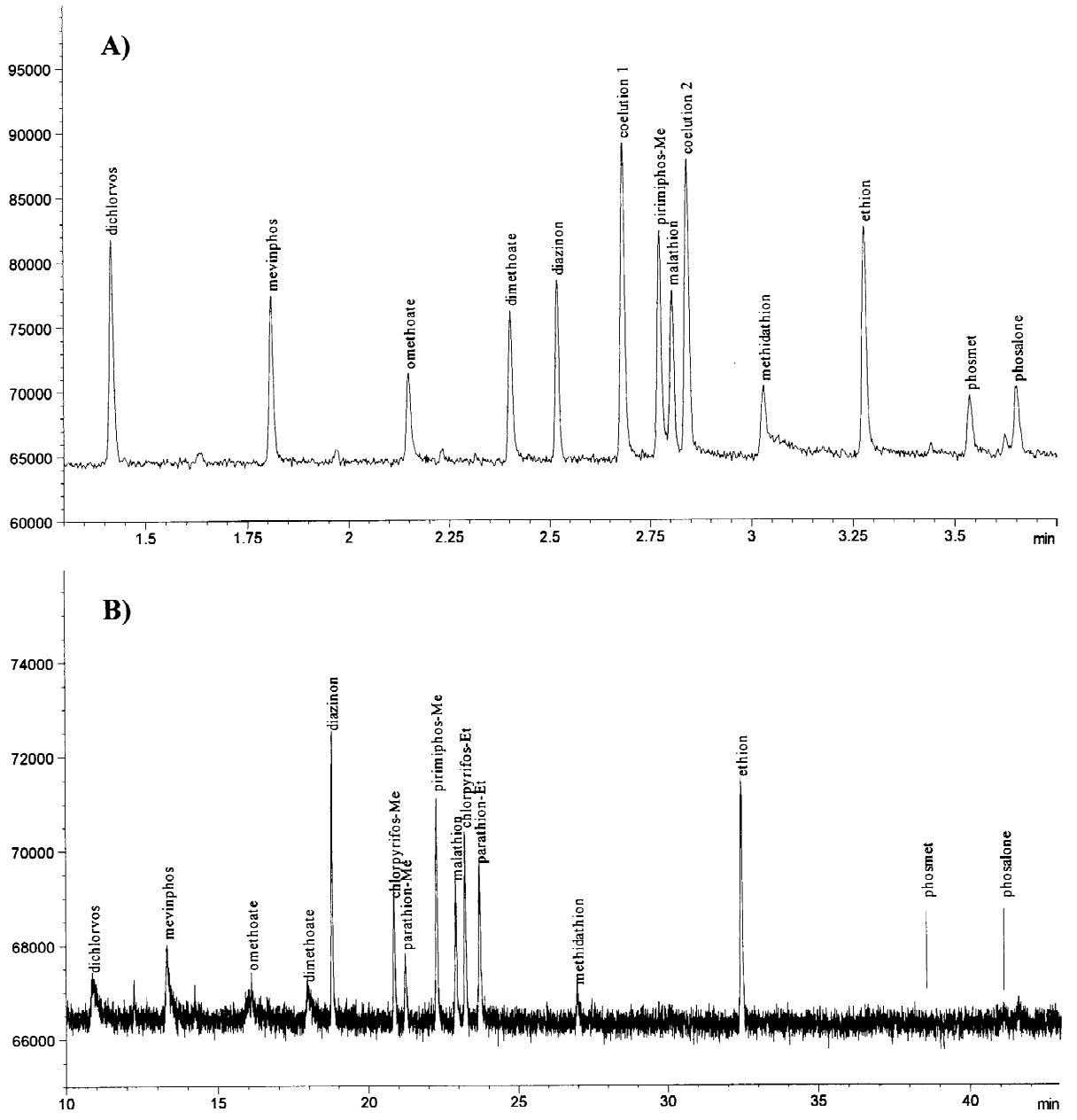

Fig. 2. Chromatograms of a flash (A) and a conventional (B) GC–FPD analysis of the wheat matrix-matched standard at concentration level D.

achieved for flash GC (see Table 2), RSDs ranging from 0.017 to 0.650% were recorded in experiments employing fast oven temperature programming (see Table 3). Retention time repeatability was in the case of fast oven heating (except for very early eluting organophosphates) relatively poor, RSDs generally increased with retention time of analytes. To compare the variance of retention times obtained by both techniques, the *F*-test at the 95% confidence level was used. At all tested concentration levels, retention

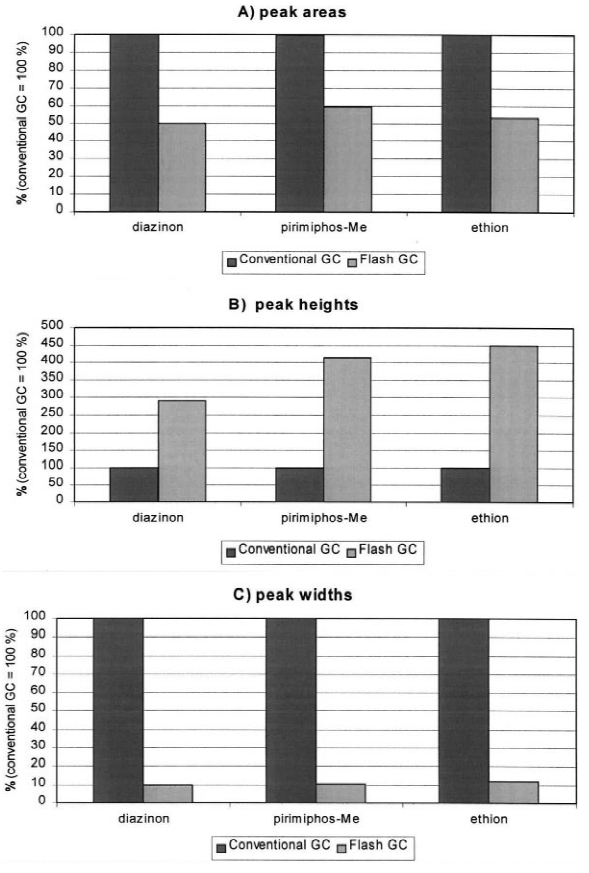

Fig. 3. Comparison of peak areas (A), peak heights (B) and peak widths (C) of diazinon, pirimiphos-methyl and ethion in the wheat matrix-matched standard (concentration level D) obtained by conventional and flash GC (normalized to conventional GC values).

time repeatability for all compounds eluting later than dimethoate was better for the flash GC technique. In the case of the early eluting analytes (dichlorvos, mevinphos, omethoate and dimethoate), there was no significant difference between both techniques proved. The superiority of flash GC employing resistive heating over conventional heating as regards excellent repeatability of retention times is well documented in Fig. 4 (dichlorvos, pirimiphos-methyl and phosalone represent an early, medium and late eluting analyte, respectively).

In Fig. 5, a fast GC–NPD analysis of a real sample (wheat matrix-matched standard, concentration level D) carried out by the conventional column heating technique is shown. In this particular case the DB-5MS stationary phase resolved pesticides coeluted on an RTX-5 column. Although both stationary phases are declared as 5% phenyl–95% dimethylpolysiloxane, their structures are rather different: the DB-5MS column contains the phenyl not only as groups pendant on the chain, but also has the phenyl inserted into the polysiloxane chain as an aryl inclusion [13]. Because of this fact, some selectivity difference can be encountered. Some tailing of analytes in Fig. 5 was caused by the typically poor performance of a HP NPD ceramic bead.

It should be noted that while less than 0.5 min was needed for resetting the system employing resistive heating, the cooling down of a HP 6890 Plus GC oven took approximately 6 min. Rapid equilibration of EZ flash GC is thus a distinct advantage as regards sample throughput. In Table 4, approximate time requirements to be anticipated for the analysis of the batch of 18 samples using all GC techniques tested in this study are shown. The number of samples used for this consideration reflects the real-life situation in our laboratory, where the automated HPGPC system is able to clean-up overnight a batch consisting in maximum of 18 samples. In connection, the sequence of 27 injections is considered in the GC step that corresponds to the GC analysis of 18 samples (extracts purified by HPGPC) and nine calibration standards (three calibration levels injected at the beginning of the sequence and then again after each set of nine samples). Contrary to the procedure employing the conventional GC technique, the total time needed for the processing of 18 samples during the working hours can be reduced by a factor of 4.4 in the case of the resistive heating technique, or by a factor of 3.3 in the case of the fast oven temperature programming. It should be noted that generally two benefits may result from the application of fast GC: not only the increased laboratory throughput, but alternatively also potential improvement of the precision of generated data due to the capability of running replicate samples as well as more injections of standards in the same time period. For that reason, the reduction of GC analysis itself is also important. As can be seen in Table 4, the GC step employing resistive heating is approximately 10.7 times faster than the conventional GC analysis as long as the same sequence of samples is considered. In the case of the fast oven temperature programming technique,

Table 2
Flash GC–FID, retention time relative standard deviations (RSDs), $n=10$

Pesticide[a]	Concentration level					
	A		B		C	
	Mean (min)	RSD (%)	Mean (min)	RSD (%)	Mean (min)	RSD (%)
Dichlorvos	1.4070	0.055	1.4086	0.049	1.4063	0.052
Mevinphos	1.7962	0.041	1.7975	0.041	1.7957	0.038
Omethoate	2.1338	0.036	2.1347	0.040	2.1336	0.057
Dimethoate	2.3871	0.032	2.3878	0.039	2.3859	0.034
Diazinon	2.5018	0.039	2.5021	0.039	2.5001	0.030
Pirimiphos-methyl	2.7536	0.034	2.7536	0.035	2.7519	0.037
Malathion	2.7823	0.031	2.7824	0.035	2.7804	0.032
Methidathion	3.0079	0.036	3.0090	0.029	3.0063	0.031
Ethion	3.2520	0.031	3.2527	0.028	3.2502	0.039
Phosmet	3.5081	0.027	3.5084	0.035	3.5059	0.046
Phosalone	3.6196	0.032	3.6206	0.031	3.6190	0.050

[a] The coeluted analytes are not listed.

the total GC analysis time is reduced approximately only by a factor of 5.5.

4. Conclusions

Using the flash GC technique (resistive heating of a short capillary column) for the analysis of 15 organophosphorus pesticides, the GC analysis time was reduced by a factor of more than 10 compared to the conventional GC technique (moderate oven temperature programming of a six times longer high resolution capillary column). Due to much narrower peak widths, improved detectability of analytes

Table 3
Fast GC–NPD, retention time relative standard deviations (RSDs), $n=10$

Pesticide	Concentration level					
	A		B		C	
	Mean (min)	RSD (%)	Mean (min)	RSD (%)	Mean (min)	RSD (%)
Dichlorvos	1.6068	0.061	1.6067	0.024	1.6070	0.032
Mevinphos	1.9605	0.049	1.9603	0.019	1.9606	0.017
Omethoate	2.2653	0.051	2.2644	0.023	2.2645	0.024
Dimethoate	2.4873	0.058	2.4855	0.048	2.4860	0.042
Diazinon	2.5763	0.108	2.5737	0.076	2.5743	0.071
Chlorpyrifos-methyl	2.7156	0.175	2.7116	0.093	2.7125	0.100
Parathion-methyl	2.7345	0.196	2.7299	0.113	2.7309	0.102
Pirimiphos-methyl	2.7934	0.221	2.7883	0.123	2.7892	0.118
Malathion	2.8269	0.260	2.8207	0.137	2.8216	0.133
Chlorpyrifos-ethyl	2.8477	0.256	2.8415	0.140	2.8426	0.133
Parathion-ethyl	2.8709	0.260	2.8640	0.141	2.8654	0.144
Methidathion	3.0298	0.349	3.0209	0.187	3.0221	0.189
Ethion	3.2492	0.481	3.2357	0.247	3.2378	0.271
Phosmet	3.4928	0.588	3.4754	0.297	3.4778	0.322
Phosalone	3.5973	0.650	3.5776	0.318	3.5802	0.361

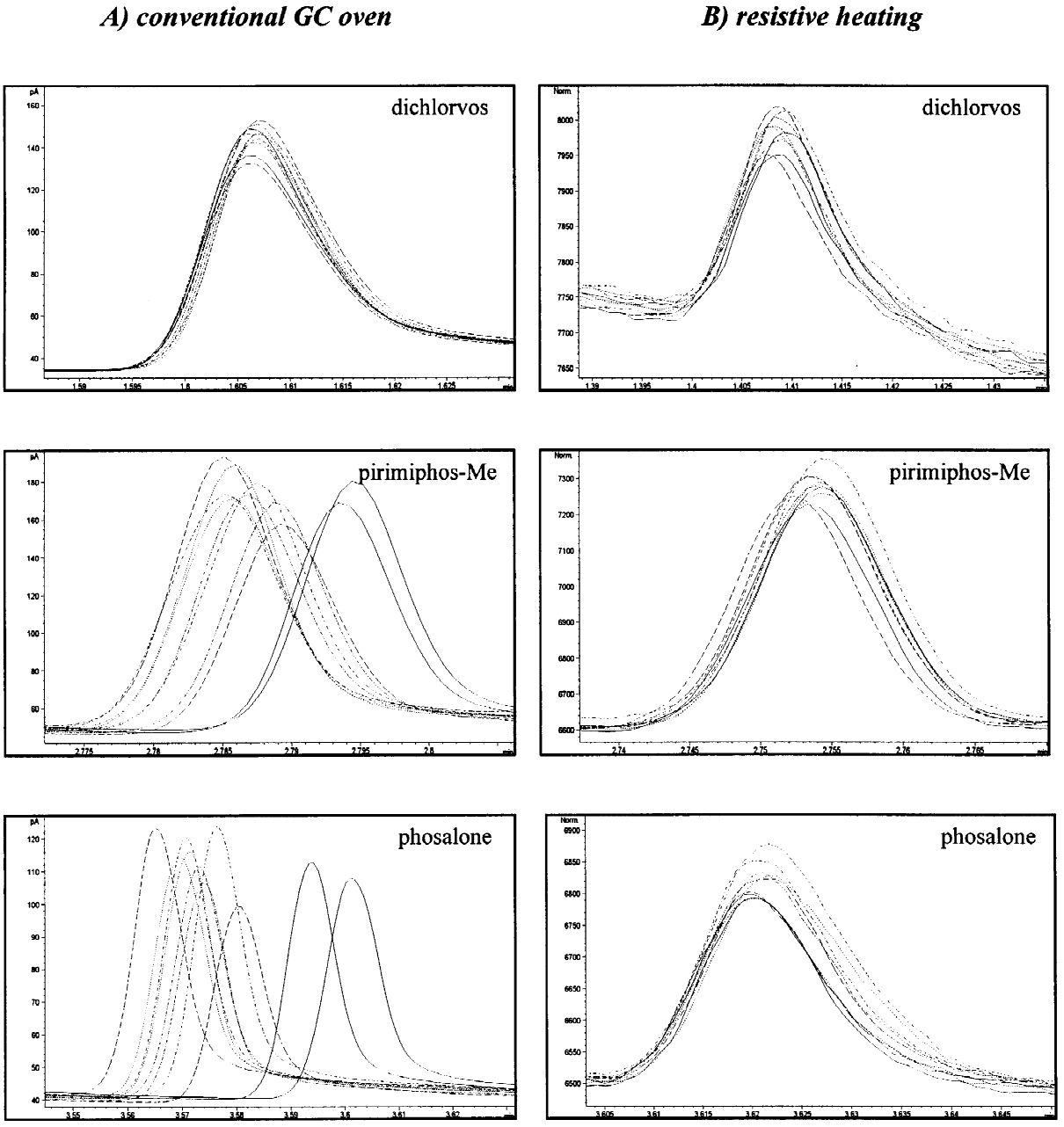

Fig. 4. Overlay of 10 repeated injections obtained by analyses of the standard solution at concentration level C, fast temperature programming realized by: (A) a conventional GC oven, (B) resistive heating (flash GC); – comparison of retention time repeatability of three selected analytes.

(higher signal-to-noise ratio) was achieved. In comparison with the alternative fast temperature programming technique realized by a conventional GC oven, significantly better retention time repeatability was observed. The other superiority of the flash GC technique is very rapid re-equilibration (i.e., cooling

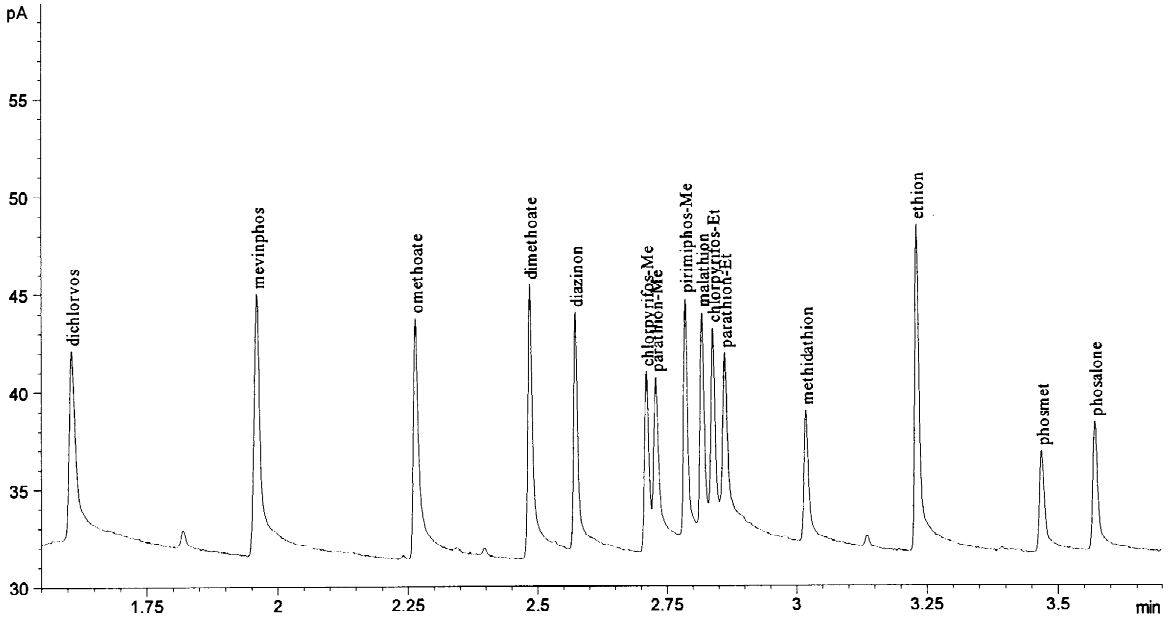

Fig. 5. Chromatogram of a fast GC–NPD analysis of the wheat matrix-matched standard at concentration level D.

Table 4
Time requirements for the analysis of the batch of 18 samples using the conventional GC technique and the two tested fast GC techniques (A – sample preparation, extraction and clean-up step; B – GC step; C – total sample analysis)

Analytical step	Time		
(A)			
Sample preparation	4.50 h		
Extraction	(18×15 min)		
Clean-up	10.50 h		
(overnight)	(18×35 min)		
(B)	Time		
	Conventional GC	Fast GC	
		Oven heating	Resistive heating
GC run	23.85 h (27×53 min)	2.25 h (27×5 min)	2.25 h (27×5 min)
Equilibration of the GC system	2.60 h (26×6 min)	2.60 (26×6 min)	0.22 h (26×0.5 min)
Total GC run	26.45 h	4.85 h	2.47 h
(C)			
Total analysis	41.45 h	19.85 h	17.47 h
Total analysis during the working hours[a]	30.95 h	9.35 h	6.97 h

[a] Time required for the automated HPGPC clean-up not taken into consideration.

down) to the initial conditions which results in higher sample throughput compared to the fast oven temperature programming technique.

The EZ Flash upgrade kit brings flash GC capabilities to existing conventional GCs with relatively low investment. Although this study demonstrates the utilization for only one group of pesticides, representatives of other groups can be also analyzed, since the kits compatible with some other GC detection methods used in pesticide residues analysis (for example with ECD or NPD) are commercially available at the present. The flash GC technique seems to be highly promising for rapid screening of pesticide residues even at very low concentration levels. The limitation insisting in lower obtainable chromatographic resolution, which may be needed for complex mixtures, can be circumvented by its connection to a mass-selective detector, which can be used to spectrometrically resolve peaks [14].

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