Program in BASIC to combine data from two different selective detectors and its application for screening of pesticides in residue analysis

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Introduction

For the analysis of pesticide residues in food, gas chromatography with selective detectors is established internationally as the most suitable method. The application of electron capture (ECD) and nitrogen-phosphorus (NPD) detectors enables the selective detection of contaminants at trace level in the presence of a multitude of compounds extracted from the matrix, which do not respond to these detectors.

The number of compounds used in agriculture for plant protection and the variety of pollutants in the environment has increased to such a level that it is impossible to separate them all in a single chromatogram, despite using high-performance capillary columns. These capillary columns allow the retention times of compounds to be determined with a very high accuracy and good reproducibility. The high resolution facilitates the differentiation of substances belonging to the same structural class as organophosphate pesticides (PP) or chlorinated pesticides (CP).

When splitting the effluent of the capillary column to both selective detectors, additional information about the identity of the individual compounds can be obtained from the chromatograms by calculating the response ratios.

A concept has recently been developed for automated pesticide residue analysis—realizing it by means of a gas chromatograph with options for BASIC programming, dual-channel operation and automatic liquid sampling [1 and 2].

The BASIC program, which controls the automated analysis, calculates the residue concentrations and evaluates the degree of certainty of the pesticides found is presented here.

Materials and methods

The pesticide residue analysis was performed with a gas chromatograph (HP 5880 A, Hewlett Packard, Palo Alto, California, USA) using capillary columns (BP 1, SGE-Scientific Glass Engineering, Australia) and effluent splitting to the two selective detectors (NPD and ECD). The signals from detectors are processed in a dual-channel integrator connected to two terminals, allowing both chromatograms to be recorded in parallel. The manufacturer's operating system and chromatography programs are used for data collection, immediate recording of the chromatograms, recognition of calibrated peaks and quantitation. The internal standard method is generally used. The instrument is also equipped with an autosampler, HP 7671 A, and the microprocessor's memory is extended by a cartridge tape unit.

The procedure of the complete pesticide analysis has already been described [1 and 2].

The program

The HP 5880 A was launched as the first gas chromatograph which could be programmed in BASIC; this allows it to execute individual calculations, to format reports and to control the autosampler. The authors' program was developed to fulfil the following requirements:

- (1) The ability to run a food sample with parallel signal processing in two channels and storage of the data.
- (2) Calculation of peaks found by means of several calibration tables.
- (3) Evaluation of the results by comparing the peaks identified in the two channels.
- (4) Summarizing final results in a clearly arranged report.

The HP 5880 A was designed to store one calibration table in each channel. However, the large number of pesticides included in the authors' analytical method requires at least three calibration mixtures with the three corresponding calibration tables in each channel. Therefore the calibration tables have to be saved on an external memory: in this case a tape. The calibration tables are included in the three 'Analysis files'. Each file contains two calibration tables generated by dual-channel recording of one calibration mixture. Additionally, the analysis files contain the parameter settings of the instrument to run the gas chromatographic analysis. A special problem arises from the fact that communication between the two channels is limited—calculations can easily be performed, but processing in one channel cannot be controlled by the other one. Therefore two separate programs have been created, these are synchronized by means of waiting loops.

The programs are documented in figures 1 and 2; the REM statements should make them almost self-explanatory.

For readers unfamiliar with the HP 5880 A, a list of some of the commands for activating special procedures and functions integrated in the program might be helpful:

START AUTO SEQ X, X: Starts the automatic sampler. The two numbers define the first and last bottle.

RECALIB: The areas from the calibration mixtures found in the most recent run and the amounts from the original calibration run of the same mixture are used to calculate new response factors.

RECALIB RUN TIME: The retention times in the calibration table are replaced by real times found in the most recent run.

SAMPLE \$: Returns the number of the sampler bottle in the tray.

ID \$: Returns the sample names from the sample table.

DETECTOR A O: NPD is switched off.

AMT (I): Returns the concentration of a specified peak calibrated.

HEAD \$: Returns the title from the calibration table.

Application to a real food sample

The automated pesticide residue analysis controlled by the BASIC program described has been designed as a screening procedure. As already mentioned, a complete residue analysis in food of unknown origin cannot be performed on a single capillary column: so the aim of automated screening is to provide the analyst with information about contaminants that

may be present in the specified sample. All of the suspected pesticides listed in the final report are analysed using an independent method of confirmation [1 and 2]. Examples of final reports from the two channels are shown in figures 3 and 4. All peaks identified by means of the data stored for each calibration mixture are printed, and those responding to both detectors are compared by applying their specific response ratios. The deviation from the value calculated from the calibration data is reported as a percentage. A difference of more than 30% is usually an indication that the peak found does not correspond to the substance calibrated. Also, all compounds identified with only one detector are indicated, as well as those belonging to 'critical pairs' in chromatography and those responding to both detectors.

LIST PRGM

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PROGRAM: (ANNOTATION OFF)
10 PRINT "LEADING PROGRAM (CHANNEL 1) FOR RESIDUE ANALYSIS"
20 PRINT "PARALLEL IN NPD AND ECD"
30 REM
40 REM ***** NPD (SIGNAL A): CHANNEL 1
50 REM ***** ECD (SIGNAL D): CHANNEL 2
60 REM ***** COLUMN: METHYLSILICONE- BP1
70 REM ***** INTERNAL STANDARDS FOR NPD: NT (PT)
80 REM ***** FOR ECD: ALDRIN (1,2,3- TCB)
90 REM ***** CALIBRATION TABLES AND INSTRUMENT SETPOINTS
100 REM ***** IN ANALYSIS FILES 1,2 AND 3
110 PRINT
120 PRINT "PUSH Y IF LOADING THE THREE STANDARD MIXTURES INTO THE TRAY"
130 INPUT "AS FOLLOWS: PPI, PPII, CPI AND STARTING CHANNEL 2", AS
140 IF AS < > "Y" THEN 120
150 PRINT
160 PRINT "CREATION OF SAMPLE TABLE"
170 PRINT
180 PRINT "START WITH 4 AND CONCLUDE BY PUSHING EXIT"
190 PRINT
200 SAMPLE TBL
210 INPUT "HOW MANY BOTTLES ARE IN THE TRAY?",N
220 LET C=N-3
230 GET ANALYSIS 1 DEVICE# 6
240 OVEN TEMP ANNOTATION OFF
250 LIST CLOCK TIME
260 START AUTO SEQ 1,1
270 RECALIB
280 RECALIB RUN TIME
290 WAIT 0.2
300 DELETE ANALYSIS 1 DEVICE# 6
310 SAVE ANALYSIS 1 DEVICE# 6
320 GOSUB 2030
330 GET ANALYSIS 2 DEVICE# 6
340 OVEN TEMP ANNOTATION OFF
350 START AUTO SEQ 2,2
360 RECALIB
370 RECALIB RUN TIME
380 WAIT 0.2
390 DELETE ANALYSIS 2 DEVICE# 6
400 SAVE ANALYSIS 2 DEVICE# 6
410 GOSUB 2030
420 GET ANALYSIS 3 DEVICE# 6
430 OVEN TEMP ANNOTATION OFF
440 START AUTO SEQ 3,3
450 RECALIB
460 RECALIB RUN TIME
470 WAIT 0.2
480 DELETE ANALYSIS 3 DEVICE# 6
490 SAVE ANALYSIS 3 DEVICE# 6
500 GOSUB 2030
510 WAIT 1
520 GET ANALYSIS 1 DEVICE# 6
530 REM
540 REM ***** INJECTION OF C SAMPLES AND STORING THE DATA ON TAPE
550 REM
560 FOR N1=4 TO N
570 OVEN TEMP ANNOTATION OFF
580 ATTN 2↑2
590 THRESHOLD 1
600 START AUTO SEQ N1,N1

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610 PRINT SAMPLE$,IDS
620 LIST SIGNAL
630 WAIT 0.5
640 EXECUTE E, "SAVE REPORT "&VAL$(N1)&" DEVICE# 6"
650 NEXT N1
660 PRINT
670 DETECTOR A 0
680 PRINT
690 PRINT TAB(15); "REGISTER OF PESTICIDE RESIDUES OF"; C; "SAMPLES"
700 PRINT
710 PRINT
720 IMAGE X,2A,4X,12A,4X,12A,4X,DD.DD,4X,DD.DD,4X,DD.DD,4X,DDDDDD
730 IMAGE X,2A,4X,12A,4X,12A,4X,5A,4X,5A,4X,6A,4X,6A
740 PRINT USING 730; "NO", "SAMPLE", "NAME", "PPM", "RT", "EXP.RT", "AREA"
750 PRINT
760 PRINT
770 FOR N1=4 TO N
780 PRINT "CALCULATED FROM STANDARD MIXTURE I FOR P-COMPOUNDS"
790 PRINT
800 WAIT 0.5
810 REM
820 REM ***** LOADING THE REPORTS INTO MEMORY AND COMPARISON WITH
830 REM ***** THREE STANDARD MIXTURES
840 REM
850 EXECUTE E, "GET REPORT "&VAL$(N1)&" DEVICE# 6"
860 GOSUB 1040
870 WAIT 1
880 GET ANALYSIS 2 DEVICE# 6
890 PRINT
900 PRINT "CALCULATED FROM STANDARD MIXTURE II FOR P-COMPOUNDS"
910 PRINT
920 GOSUB 1040
930 WAIT 1
940 GET ANALYSIS 3 DEVICE# 6
950 PRINT
960 PRINT "CALCULATED FROM STANDARD MIXTURE FOR N-COMPOUNDS"
970 PRINT
980 GOSUB 1040
990 GOTO 1320
1000 REM
1010 REM ***** PRINTING REPORTS AND COMPARISON OF RESULTS
1020 REM ***** BETWEEN ECD AND NPD
1030 REM
1040 FOR I=1 TO #PEAKS
1050 IF AMT(I)>=0.01 THEN 1070
1060 GOTO 1080
1070 PRINT USING 720; SAMPLE$, IDS, NAMES(I),AMT(I),RT(I),EXPRT(I),AREA(I)
1080 NEXT I
1090 PRINT
1100 FOR I=1 TO #PEAKS
1110 LET U$=NAMES(I)
1120 LET F=AMT(I)
1130 FOR J=1 TO #PEAKS(2)
1140 IF AMT(I)<0.01 THEN 1290
1150 IF NAMES(J,2)=U$ THEN 1170
1160 GOTO 1280
1170 IF AMT(J,2)>=0.01 THEN 1190
1180 GOTO 1280
1190 LET E=AMT(J,2)
1200 IF F>=E THEN 1220
1210 IF E>=F THEN 1240
1220 LET Y=(F-E)*100/F
1230 GOTO 1250
1240 LET Y=(E-F)*100/E
1250 IMAGE "THE DIFFERENCE BETWEEN THE DETECTORS AMOUNTS TO",DDD,"%"
1260 PRINT USING 1250;Y
1270 PRINT "FOR",NAMES(J,2)
1280 NEXT J
1290 NEXT I
1300 PRINT
1310 RETURN
1320 IMAGE 72("&")
1330 PRINT USING 1320
1340 PRINT
1350 GOSUB 1580
1355 WAIT 1
1360 GET ANALYSIS 2 DEVICE# 6
1370 GOSUB 1680
1380 GET ANALYSIS 1 DEVICE# 6
1390 GOSUB 1680
1400 PRINT
1410 PRINT USING 1320

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1420 PRINT
1430 NEXT N1
1440 REM
1450 REM ***** FOOT NOTES TO THE FINAL REPORT
1460 REM
1470 PRINT TAB(10); " * BELONGS TO A CRITICAL PESTICIDE THAT INDICATES"
1480 PRINT TAB(13); "SIMILAR RETENTION TIME TO A COMPOUND OF ANOTHER"
1490 PRINT TAB (27); "STANDARD MIXTURE"
1500 PRINT
1510 PRINT TAB(10); "+ BELONGS TO A COMPOUND RESPONDING TO ECD AND NPD"
1520 PRINT
1530 GET REPORT 4 DEVICE# 6
1540 STOP
1550 REM
1560 REM ***** FINAL EVALUATIONS
1570 REM
1580 FOR J=1 TO *PEAKS(2)
1590 LET Y$=NAME$(J,2)
1600 FOR I=1 TO *PEAKS
1610 IF NAME$(I)=Y$ THEN 1660
1620 IF NAME$(J,2)="ALDRIN" THEN 1660
1630 IF AMT (J,2)<0.01 THEN 1660
1640 NEXT I
1650 PRINT NAME$(J,2),"IS IDENTIFIED WITH ECD IN";IDS
1660 NEXT J
1670 GOTO 1780
1680 FOR I=1 TO *PEAKS
1690 LET X$=NAME$(I)
1700 FOR J=1 TO *PEAKS(2)
1710 IF NAME$(J,2)=X$ THEN 1770
1720 IF NAME$(I)="PT" THEN 1770
1730 IF NAME$(I)="NT" THEN 1770
1740 IF AMT(I)<0.01 THEN 1770
1750 NEXT J
1760 PRINT NAME$(I), "IS IDENTIFIED WITH NPD IN";IDS
1770 NEXT I
1780 FOR I=1 TO *PEAKS
1790 LET U$=NAME$(I)
1800 LET F=AMT(I)
1810 FOR J=1 TO *PEAKS(2)
1820 IF NAME$(J,2)=U$ THEN 1840
1830 GOTO 1960
1840 IF AMT(J,2)>=0.01 THEN 1860
1850 GOTO 1960
1860 LET E=AMT(J,2)
1870 IF F>=E THEN 1890
1880 IF E>=F THEN 1910
1890 LET Y=(F-E)*100/F
1900 GOTO 1920
1910 LET Y=(E-F)*100/E
1920 IF Y<=30 THEN 1950
1930 GOTO 1970
1940 PRINT
1950 PRINT NAME$(J,2), "IS SUSPECTED IN";IDS
1960 NEXT J
1970 NEXT I
1980 RETURN
1990 REM
2000 REM ***** PRINTING CALIBRATION REPORTS OF STANDARD MIXTURES
2010 REM ***** INCLUDING RELATIVE RETENTION TIMES
2020 REM
2030 FOR I=1 TO *PEAKS
2040 IF NAME$(I)="NT" THEN 2060
2050 GOTO 2070
2060 LET A=RT(I)
2070 NEXT I
2080 PRINT HEAD$
2090 PRINT
2100 PRINT "CAL", "NAME", "PPM", "RT", "REL.RT"
2110 PRINT
2120 FOR I=1 TO *PEAKS
2130 PRINT CAL*(I),NAME$(I),AMT(I),RT(I),RT(I)/A
2140 NEXT I
2150 RETURN

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Figure 1. The program for the NPD channel (leading program).

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LIST PRGM
PROGRAM:          (ANNOTATION OFF)
10  PRINT "PROGRAM (CHANNEL 2) FOR RESIDUE ANALYSIS"
20  PRINT "PARALLEL IN ECD AND NPD"
30  REM
40  REM ***** NPD (SIGNAL A): CHANNEL 1
50  REM ***** ECD (SIGNAL D): CHANNEL 2
60  PRINT
70  INPUT "HOW MANY BOTTLES ARE IN THE TRAY?";N
80  START
90  RECALIB
100 RECALIB RUN TIME
110 GOSUB 1070
120 START
130 RECALIB
140 RECALIB RUN TIME
150 GOSUB 1070
160 START
170 RECALIB
180 RECALIB RUN TIME
190 GOSUB 1070
200 REM
210 REM ***** INJECTION OF SAMPLES AND STORING THE DATA ON TAPE
220 REM
230 FOR B1=104 TO N+100
240 ATTN 2↑9
250 THRESHOLD 9
260 START
270 LIST CLOCK TIME
280 PRINT SAMPLE$,IDS
290 PRINT "SIGNAL D"
300 EXECUTE E, "SAVE REPORT "&VAL$(B1)&" DEVICE # 16"
310 NEXT B1
320 PRINT
330 PRINT
340 PRINT TAB(15); "REGISTER OF PESTICIDE RESIDUE OF"; N-3; "SAMPLES"
350 PRINT
360 PRINT
370 IMAGE X,2A,4X,12A,4X,12A,4X,DD.DD,4X,DD.DD,4X,DD.DD,4X,DDDDDD
380 IMAGE X,2A,4X,12A,4X,12A,4X,5A,4X,5A,4X,6A,4X,6A
390 PRINT USING 380; "NO", "SAMPLE", "NAME", "PPM", "RT", "EXP.RT", "AREA"
400 PRINT
410 PRINT
420 FOR B1=104 TO N+100
430 PRINT
440 PRINT
450 PRINT "CALCULATED FROM STANDARD MIXTURE I FOR P-COMPOUNDS"
460 REM
470 REM ***** LOADING THE REPORTS INTO MEMORY AND COMPARISON WITH
480 REM ***** THREE STANDARD MIXTURES
490 REM
500 EXECUTE E, "GET REPORT "&VAL$(B1)&" DEVICE# 16"
510 REM
520 REM ***** WAITING LOOP FOR SYNCHRONISATION
530 REM ***** WITH THE MAIN CHANNEL 1
540 REM
550 LET W$=SAMPLE$(2)
560 IF W$=SAMPLE$(1) THEN 590
570 WAIT 0.2
580 GOTO 550
590 GOSUB 870
600 REM
610 REM ***** WAITING LOOP FOR SYNCHRONISATION
620 REM ***** WITH THE MAIN CHANNEL 1
630 REM
640 LET A$=HEAD$
650 IF A$="STANDARD MIXTURE II IN ECD" THEN 680
660 WAIT 0.2
670 GOTO 640
680 PRINT
690 PRINT "CALCULATED FROM STANDARD MIXTURE II FOR P-COMPOUNDS"
700 GOSUB 870
710 REM
720 REM ***** WAITING LOOP FOR SYNCHRONISATION
730 REM ***** WITH THE MAIN CHANNEL 1
740 REM
750 LET B$=HEAD$
760 IF B$="STANDARD MIXTURE FOR CHLORINATED COMPOUNDS IN ECD" THEN 790
770 WAIT 0.2
780 GOTO 750
790 PRINT

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800 PRINT "CALCULATED FROM STANDARD MIXTURE FOR CHLORINATED COMPOUNDS"
810 GOSUB 870
820 GOTO 930
830 REM
840 REM ***** PRINTING REPORTS AND COMPARISON OF RESULTS
850 REM ***** BETWEEN ECD AND NPD
860 REM
870 FOR I=1 TO #PEAKS
880 IF AMT(I)>=0.01 THEN 900
890 GOTO 910
900 PRINT USING 370; SAMPLES, IDS, NAMES$(I), AMT(I), RT(I), EXPRT(I), AREA(I)
910 NEXT I
920 RETURN
930 WAIT 0.2
940 REM
950 REM ***** WAITING LOOP FOR SYNCHRONISATION
969 REM ***** WITH THE MAIN CHANNEL 1
970 REM
980 LET V$=SAMPLES(2)
990 IF V$<>SAMPLES(1) THEN 1010
1000 GOTO 930
1010 NEXT B1
1020 STOP
1030 REM
1040 REM ***** PRINTING CALIBRATION REPORTS OF THE STANDARD MIXTURES
1050 REM ***** INCLUDING RELATIVE RETENTION TIMES
1060 REM
1070 FOR I=1 TO #PEAKS
1080 IF NAMES$(I)="ALDRIN" THEN 1100
1090 GOTO 1110
1100 LET A=RT(I)
1110 NEXT I
1120 PRINT HEAD$
1130 PRINT
1140 PRINT "CAL", "NAME", "PPM", "RT", "REL.RT"
1150 PRINT
1160 FOR I=1 TO #PEAKS
1170 PRINT CAL$(I), NAMES$(I), AMT(I), RT(I), RT(I)/A
1180 NEXT I
1190 RETURN

```

Figure 2. The program for the ECD channel.

REGISTER OF PESTICIDE RESIDUE OF 1 SAMPLES

NO	SAMPLE	NAME	PPM	RT	EXP.RT	AREA
CALCULATED FROM STANDARD MIXTURE I FOR P-COMPOUNDS						
4	PEACHES	PHOSPHAMI+	.54	13.97	13.86	1763
4	PEACHES	PARATH-ME+*	1.48	14.21	14.20	49932
4	PEACHES	MALATHION+	2.69	16.49	16.57	16716
4	PEACHES	ALDRIN	2.00	16.85	16.85	100997
4	PEACHES	PARATHION+*	.20	17.21	17.14	2577
CALCULATED FROM STANDARD MIXTURE II FOR P-COMPOUNDS						
4	PEACHES	DICHLUFENT+*	3.37	14.21	14.18	49932
4	PEACHES	PARAOXON+*	.59	14.98	15.05	5607
4	PEACHES	ALDRIN	2.00	16.85	16.85	100997
4	PEACHES	DURSBAN+*	.10	17.21	17.18	2577
CALCULATED FROM STANDARD MIXTURE FOR CHLORINATED COMPOUNDS						
4	PEACHES	CHLORFPROP-M	.79	7.67	7.60	2719
4	PEACHES	HEPTACHLOR*	.30	14.98	14.97	5607
4	PEACHES	ALDRIN	2.00	16.85	16.85	100997

Figure 3. Print-out from the ECD channel for a screening run of pesticides in peaches.

REGISTER OF PESTICIDE RESIDUES OF 1 SAMPLES

NO	SAMPLE	NAME	PPM	RT	EXP.RT	AREA
CALCULATED FROM STANDARD MIXTURE I FOR P-COMPOUNDS						
4	PEACHES	PARATH-ME+ *	1.60	14.20	14.32	1088
4	PEACHES	PARATHION+ *	.10	17.20	17.28	27
4	PEACHES	NT	2.00	18.19	18.19	1055
THE DIFFERENCE BETWEEN THE DETECTORS AMOUNTS TO 8% FOR PARATH-ME+ *						
THE DIFFERENCE BETWEEN THE DETECTORS AMOUNTS TO 52% FOR PARATHION+ *						
CALCULATED FROM STANDARD MIXTURE II FOR P-COMPOUNDS						
4	PEACHES	DICHLUFENT+ *	5.11	14.20	14.28	1088
4	PEACHES	DURSBAN+ *	.07	17.20	17.31	27
4	PEACHES	NT	2.00	18.19	18.19	1055
THE DIFFERENCE BETWEEN THE DETECTORS AMOUNTS TO 34% FOR DICHLUFENT+ *						
THE DIFFERENCE BETWEEN THE DETECTORS AMOUNTS TO 35% FOR DURSBAN+ *						
CALCULATED FROM STANDARD MIXTURE FOR N-COMPOUNDS						
4	PEACHES	NT	2.00	18.19	18.19	1055

CHLORFPROP-M		IS IDENTIFIED WITH ECD IN PEACHES				
HEPTACHLOR*		IS IDENTIFIED WITH ECD IN PEACHES				
PARATH-ME+ *		IS SUSPECTED IN PEACHES				

* BELONGS TO A CRITICAL PESTICIDE THAT INDICATES SIMILAR RETENTION TIME TO A COMPOUND ANOTHER STANDARD MIXTURE						
+ BELONGS TO A COMPOUND RESPONDING TO ECD AND NPD						

Figure 4. Print-out from the NPD channel for the same screening run as in figure 3, including final report.

Discussion

Figures 3 and 4 show the parallel print-outs from the two terminals reporting a screening run for pesticide residues in peaches. By means of this example, the utility and the limits of our program for evaluating food samples are briefly discussed.

After the clean-up, a number of substances responding to the ECD are generally found in a chromatogram and several are recognized as calibrated pesticides in the screening run. In figure 3 a total of nine pesticides are indicated, together with the internal standard (aldrin). Only these nine compounds out of about 80 pesticides incorporated in three calibration mixtures may be present in the sample. Processing of the NPD signals recorded simultaneously in the other channel results in four organophosphorous pesticides and no nitrogen-containing pesticide (figure 4). For all of the compounds responding to both detectors a calculation of the response ratios and comparison with their calibrated values leads to the discrimination of 'parathion', dichlofenthion' and 'dursban', whereas the response ratio of 'parathion methyl' is close to the calibrated one. Therefore parathion methyl is announced in the final report as suspected. This screening run permits no further decision about 'chlorfenprop methyl' and 'heptachlor'.

All pesticides marked with a cross in figure 3 are organophosphorous compounds and so they also have to respond to

the NPD. As there are no corresponding signals for 'phosphamidon', 'malathion' and 'paraaxon' in the NPD report of this example, all three substances are eliminated and do not appear in the final report. This discrimination procedure results in the final proposal of chlorfenprop methyl, heptachlor and parathion methyl as possibly being present in this sample.

The program proved to be a great help in routine analysis for selecting the positive food samples after screening. A major drawback is the time-consuming data transfer between the tape and the gas chromatograph's memory.

The analyst has to inspect the chromatograms of both detectors in order to evaluate the quality of the separation and the performance of the chromatographic system. After this, the computer supports him by handling the huge amount of information produced by a series of screening runs.

References

1. GOEBEL, H. and STAN, H.-J. in J. Rijks (ed.), *Proceedings of the Fifth International Symposium on Capillary Chromatography* (Elsevier, Amsterdam, 1983), 557.
2. STAN, H.-J. and GOEBEL, H., *Journal of Chromatography* (in press).