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Gas chromatographic separation and automatic identification of complex mixtures of organic solvents in industrial wastes

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ABSTRACT

The analysis of complex mixtures of industrial solvents in chemical wastes was carried out by the simultaneous use of non-polar and polar wide-bore capillary or packed columns. The co-eluting peaks on one of the two phases were resolved on the other phase, and the identification and quantitative analysis of mixtures containing up to 32 compounds was possible. Some integrators and data collectors were tested to allow the automatic identification of the compounds by comparing the chromatograms obtained from a 50:50 split of the sample into two parallel columns connected to identical flame ionization detectors.

INTRODUCTION

The disposal of toxic industrial wastes through landfill, incineration or other procedures is a controversial subject, as toxic chemicals or their decomposition products may contaminate water courses or escape into the atmosphere during the production, conservation and treatment of the wastes. The lack of adequate regulation and official treatment and disposal plants in many countries has led to the illegal collection, transportation and dumping of wastes that, after the introduction of legislation designed to control the problem, must be recovered, identified and properly disposed of.

A significant part of the problem is the analysis of the complex mixtures of industrial solvents that, being in liquid form and often stored in metallic drums which are subject to corrosion, may contaminate the landfill sites and be leached by running water, or escape into the atmosphere as a result of their appreciable vapour pressures. The various analytical procedures for such samples have been tested and certified by official test methods [1–7]. The characterization of the components of these mixtures is also necessary to determine the most convenient and safe disposal or destruction procedure. This characterization may be very difficult owing to the lack of information on the origin of the waste and any prior treatment. This difficulty was recently illustrated by the phenomenon of the so-called "poison ships", *i.e.* freighters which, loaded with industrial waste destined for dumping sites overseas, had to search for unloading ports after changes in local rules forbade the discharge at the original destination.

An example of this situation is the Syrian vessel MV *Zanoobia*, the third of a series of cargo-ships (*Linx, Makiri* and *Zanoobia*) which were involved in a complex sequence of loading, transfer and unloading of poisons. *Linx* was originally chartered

for the carriage of 2100 tons of industrial waste to a dumping site at Djbouti (East Africa). The load was not accepted for discharge at Djbouti, and the cargo was then carried to Puerto Cabello (Venezuela) and transferred from *Linx* to *Makiri* and then to *Zanoobia*, which attempted to call at other Asiatic and European ports (Tartous, Salonika, Cagliari, Carrara) and was eventually moored, unloaded and decontaminated in Genoa harbour. During this long journey, the identification tags on the barrels were cancelled, freight documents were lost, spilt or damaged containers were replaced and their contents mixed together, and the resulting composition of the cargo (10 800 barrels) was completely unknown [8]. Similar problems were found in the decontamination of other freighters (*Jolly Rosso, Karin B, Deep Sea Carrier*) and of many dumping sites and waste repositories.

The routine analysis of this type of sample requires the identification of the components of the mixture and their determination at concentrations ranging from a tenth of a part per million to percentage values. This peculiar application influences the choice of instrumentation suitable for screening these samples: gas chromatography (GC) with flame ionization detection (FID) has to be used, as this technique offers the required sensitivity and linearity over a wide concentration range. The use of narrow-bore capillary columns should be avoided for the preliminary screening of unknown samples owing to their reduced sample capacity and shortened life in the presence of samples contaminated with high-boiling-point compounds (such as oils, polymers, additives, organometallics and surfactants). For the same reason, the use of gas chromatography–mass spectrometry (GC–MS) should be restricted to the final confirmation of some components and cannot be used for a general screening due to the enormous number of samples.

The analysis of complex mixtures on packed or wide-bore capillary columns offers the efficiency and selectivity required for identification purposes when stationary phases of different polarities are used and the retention times or index values of the components are compared with those of previously known standard samples. Simultaneous analysis on different columns is necessary to reduce the analysis time.

Polar and non-polar stationary phases were therefore used for the analysis of samples containing the 32 compounds most frequently found as components of industrial solvents or waste mixtures. The performance of packed and wide-pore capillary columns was evaluated, and the possibility of using commercial integration systems to automatically identify the components of the mixtures was investigated.

EXPERIMENTAL

The analyses were carried out using a Varian (Palo Alto, CA, USA) Model 3400 gas chromatograph equipped with dual flame ionization detector, packed columns, capillary split–splitless injectors and an integration system.

The following columns were used (Supelco, Bellefonte, PA, USA). (a) Two wide-bore glass capillary columns (60 m × 0.75 mm I.D.), *i.e.* a non-polar dimethylpolysiloxane (SPB-1) column and a polar polyethylene glycol column (Supelcowax-10). Both were operated at a flow-rate (nitrogen) of 7 cm^3/min. (b) Two packed columns (3 m × 2.1 mm I.D.), *i.e.* a non-polar column with 10% SP-2100 methylsilicone and a polar column with 10% SP-1000 (polyglycol substituted terephthalic acid) both on 80–100 mesh Supelcoport and operated at a flow-rate (nitrogen) of 30 cm^3/min.

The analyses used for measuring the absolute and relative retentions of the compounds were carried out under isothermal temperature conditions (60°C). The practical use of the method for routine analyses allowed a decrease of the total run time by proper temperature programming up to 150–180°C. The injector and detector temperatures were 200 and 250°C, respectively. The packed columns were installed on the two injectors and connected to the two flame ionization detectors to allow simultaneous injections onto the polar and non-polar phases. The wide-bore capillary columns were installed alone for the initial calibration and then connected in parallel to the split–splitless injectors by a microsplitting Y-shaped glass connector (press-fit fittings, Varian, Sunnyvale, CA, USA) that allowed the injected samples to be divided between the two columns leading to identical detectors. Splitless injections were made to avoid sample partition due to the different boiling points.

The outputs of the detectors were monitored with

dual-channel integrators or data systems (Varian 4400, Spectra Physics Chromjet, Varian Vista 420, Varian DS 650), the performances of which were evaluated to determine the possibility of achieving automatic identification of the mixture components.

The retention times of individual compounds were measured by injecting samples diluted in carbon disulphide with concentrations between 0.1 and 1 g/l, taking into account the different sensitivity of FID to various compounds, to give peaks with similar areas. Partial or complete mixtures of the components were also injected to check the effective capacity of the columns to separate closely eluting peaks.

RESULTS AND DISCUSSION

Table I lists the components of the mixture used in the order of elution on the non-polar column and their adjusted retention times, retention indices and retention relative to toluene on wide-bore non-polar and polar columns. Toluene was chosen as the reference compound because, as shown in Table III,

TABLE I

RETENTION VALUES ON NON-POLAR AND POLAR WIDE-BORE CAPILLARY COLUMNS

Adjusted retention times (t'_R), retention index with respect to n-alkanes (I) and retention relative to toluene (r) are shown. Column temperature, 60°C; carrier gas, nitrogen; flow-rate, 7 cm³/min.

Compound	SPB-1			Supelcowax-10		
	t'_R (min)	I	r	t'_R (min)	I	r
(1) Ethanol	0.26	388	0.06	3.03	944	0.44
(2) Acetone	0.38	405	0.08	1.40	832	0.20
(3) Propan-2-ol	0.39	412	0.09	2.85	935	0.41
(4) Propan-1-ol	0.52	428	0.11	6.34	1046	0.92
(5) Dichloromethane	0.61	439	0.13	3.09	946	0.45
(6) Butan-2-one	1.01	482	0.22	2.55	919	0.37
(7) Ethyl acetate	1.21	496	0.26	2.26	902	0.33
(8) Trichloromethane	1.27	501	0.28	5.82	1034	0.85
(9) Butan-2-ol	1.38	520	0.30	9.18	1095	1.33
(10) 2-Methoxyethanol	1.46	533	0.32	17.14	1184	2.49
(11) 1,2-Dichloroethane	1.60	552	0.35	8.22	1080	1.19
(12) Isopropyl acetate	1.85	584	0.41	2.41	911	0.35
(13) Butan-1-ol	1.85	584	0.41	13.09	1146	1.90
(14) Benzene	1.97	598	0.44	3.50	964	0.51
(15) 2-Nitropropane	2.23	631	0.49	11.63	1129	1.69
(16) 1,2-Dichloropropane	2.47	658	0.54	6.94	1058	1.01
(17) Trichloroethylene	2.63	675	0.58	4.87	1010	0.71
(18) 2-Ethoxyethanol	2.75	687	0.61	23.22	1231	3.38
(19) Toluene	4.57	757	1.00	6.88	1057	1.00
(20) Isobutyl acetate	4.59	757	1.01	5.45	1025	0.79
(21) n-Butyl acetate	6.29	796	1.38	8.32	1082	1.21
(22) Tetrachloroethylene	6.53	801	1.43	5.97	1038	0.87
(23) 4-Hydroxymethylpentan-2-one	7.20	813	1.58	59.59	1374	8.66
(24) 5-Methylhexan-2-one	8.69	836	1.90	13.53	1150	1.97
(25) Ethylbenzene	9.56	848	2.10	12.37	1138	1.80
(26) p-Xylene	10.23	856	2.25	13.00	1145	1.89
(27) m-Xylene	10.23	856	2.25	13.62	1151	1.98
(28) Cyclohexanone	10.60	861	2.35	35.97	1301	5.23
(29) Isoamylacetate	10.65	862	2.37	11.79	1131	1.71
(30) o-Xylene	12.16	878	2.67	18.24	1193	2.65
(31) 2-Ethoxyethyl acetate	12.64	882	2.80	40.53	1319	5.89
(32) 2-Butoxyethanol	13.18	888	2.93	79.52	1416	11.56

it is present in most of the samples. Table II shows the retention times and relative retention measured on packed columns. The choice of the mixture compounds was made on the basis of available data on the typical composition of industrial solvents, MS identification of the components of various wastes and statistical evaluation of the probability of finding some compounds in different kinds of process by-products. As an example, Table III shows the distribution of the main components in the cargo of MV *Zanoobia*. The percentage value of samples (barrels) containing the listed compounds as main components or at a concentration greater than

0.5% is given. Other compounds listed in Tables I and II but not in Table III were also found in some samples in various concentrations.

Table IV shows the compounds listed in order of elution on the non-polar and polar wide-bore capillary columns, and the compounds that are not resolved on each column are bracketed. The elution order on the packed non-polar and polar columns (SP-2100 and SP-1000) was similar to that observed on the corresponding wide-bore capillary column. A greater number of interfering groups was observed, as shown in Table V.

The different polarities of the stationary phases

TABLE II

RETENTION VALUES ON NON-POLAR AND POLAR PACKED COLUMNS

Adjusted retention times (t'_R) and retention relative to toluene (r) are shown. Column temperature, 60°C; carrier gas, nitrogen; flow-rate, 30 cm^3/min.

Compound	SP-2100		SP-1000	
	t'_R (min)	r	t'_R (min)	r
(1) Ethanol	0.20	0.04	6.12	0.48
(2) Acetone	0.51	0.10	2.71	0.21
(3) Propan-2-ol	0.51	0.10	5.55	0.44
(4) Propan-1-ol	0.66	0.13	12.17	0.96
(5) Dichloromethane	0.66	0.13	5.61	0.44
(6) Butan-2-one	1.20	0.24	4.94	0.39
(7) Ethyl acetate	1.41	0.28	4.34	0.34
(8) Trichloromethane	1.41	0.28	10.65	0.84
(9) Butan-2-ol	1.55	0.31	17.39	1.38
(10) 2-Methoxyethanol	1.76	0.35	34.41	2.72
(11) 1,2-Dichloroethane	1.76	0.35	15.11	1.20
(12) Isopropryl acetate	2.15	0.42	4.56	0.36
(13) Butan-1-ol	2.15	0.42	25.93	2.05
(14) Benzene	2.15	0.42	6.41	0.51
(15) 2-Nitropropane	2.48	0.49	21.77	1.72
(16) 1,2-Dichloropropane	2.71	0.54	12.86	1.02
(17) Trichloroethylene	2.88	0.57	8.91	0.71
(18) 2-Ethoxyethanol	3.50	0.69	45.77	3.62
(19) Toluene	5.06	1.00	12.63	1.00
(20) Isobutyl acetate	5.06	1.00	10.44	0.83
(21) *n*-Butyl acetate	7.14	1.41	15.95	1.26
(22) Tetrachloroethylene	7.14	1.41	11.08	0.88
(23) 4-Hydroxymethylpentan-2-one	8.22	1.62	112.87	8.94
(24) 5-Methylhexan-2-one	9.77	1.93	25.77	2.04
(25) Ethylbenzene	10.49	2.07	22.89	1.81
(26) *p*-Xylene	11.25	2.22	23.43	1.86
(27) *m*-Xylene	11.25	2.22	25.10	1.99
(28) Cyclohexanone	11.84	2.34	68.23	5.40
(29) Isoamyl acetate	11.84	2.34	22.44	1.78
(30) *o*-Xylene	13.34	2.64	33.61	2.66
(31) 2-Ethoxyethyl acetate	14.44	2.85	77.29	6.12
(32) 2-Butoxyethanol	16.68	3.30	153.32	12.14

TABLE III

RELATIVE ABUNDANCE OF SAMPLES CONTAINING VARIOUS SOLVENTS IN THE CARGO OF THE MV *ZANOOBIA*

Compound	Percentage of samples containing compound
Toluene	54
Xylene	53
Benzene	23
Tetrachloroethylene	21
Trichloroethylene	19
Trichloromethane	18
Dichloromethane	12
Acetone	11
Ethyl acetate	7
Butan-2-one	7
1,2-Dichloroethane	6
2-Butoxyethanol	3
1,2-Dichloropropane	3
2-Ethoxyethylacetate	1
2-Methoxyethanol	1
Ethanol	1

result in a very different distribution of peaks in the chromatograms and therefore no couple of compounds shows the same interference on both columns. Capillary columns are much more efficient than packed columns and therefore less interfering peaks are observed.

The data in Table I and II refer to isothermal analysis to allow a correct determination of the retention index values and the relative retention. These values can also be applied to programmed temperature analysis by correction factors or by using interpolation programmes.

For routine analysis, the total time needed for the complete elution of all of the listed compounds can be reduced by temperature programming, without an appreciable reduction in the resolution; the peaks separated during isothermal runs show the same behaviour during programmed analysis. In some instances, the separation of partially co-eluted peaks (see Tables IV and V) slightly increases in programmed runs, probably due to a different slope of the vapour pressure *versus* temperature plot.

Programming the temperature from the initial 60°C at a rate of 5°C/min decreased the retention time of the last eluting peaks on polar columns by about 50%. By decreasing the initial temperature at about 35°C and by programming the temperature of non-polar columns, the resolution of the fast eluting peaks increased and the total run length decreased by about 20%.

Quantitative determination of co-eluted peaks

When two or more compounds are co-eluted on a column, the confirmation of the identity and the quantitation have to be carried out using the chromatogram obtained on the other column. Table IV shows that on the Supelcowax-10 five couples and a triplet of compounds are co-eluted, whereas eight couples show interference on SPB-1. Some of the couples observed on the Supelcowax-10 column are formed by a chlorinated compound and an alcohol (peaks 1 and 5), an aromatic (peaks 20 and 16) and an acetate (peaks 11 and 21), interferences which are not common in real samples. It is therefore convenient to use the polar column for the analysis and the non-polar column for confirmation.

If all the listed compounds are simultaneously contained in the mixture, as a result of the mixing of industrial by-products and wastes of various origins, the complete identification and quantitation are carried out by taking into account both columns. Some typical examples of the procedure, which can also be used as a track for the compilation of computer programs for automatic identification, are given below.

1. Compounds co-eluted on the Supelcowax-10 column are separated on the SPB-1 column (peaks 11 and 21); quantitative analysis is made by using the results obtained on the non-polar column.

2. Compounds co-eluted on the Supelcowax-10 column (couples 1 and 5, 8 and 22, 20 and 16, 15 and 29) are well separated on the SPB-1 column (peaks 1, 15, 16, 22), whereas other peaks show interference with other substances. Comparison of the corrected peak areas allow their amounts to be calculated. As an example, if ethanol is the only component of the couple 1 and 5 to be present, it can be identified on the SPB-1 column and the amounts of compound on both columns is equal. If only dichloromethane is present in the sample, no peak will be observed on the SPB-1 column at the ethanol retention time, and the amount of dichloromethane is calculated from the area of the peak on the polar column. If both compounds are present, the amount of ethanol is measured on the SPB-1

column and dichloromethane by the difference between the two columns. The single peak of propan-1-ol on the Supelcowax-10 column can assist in confirming the composition of the couple 4 and 5 and the SPB-1 column.

3. When a triplet of compounds is co-eluted (peaks 13, 24 and 27) a similar procedure can be followed. If only 5-methyl-2-hexanone (compound 24) is present in the sample, the amount on the SPB-1 column should be equal to the amount on the Supelcowax column. On the SPB-1 column butan-1-ol is co-eluted with isopropyl acetate. The latter compound shows no interference on the Supel-

cowax-10 column and can therefore be quantitated on this column and used to calculated the amount of butan-1-ol by difference on the SPB-1 column. The same procedure can be followed for the third component of the triplet (*m*-xylene), which is co-eluted on the SPB-1 column with *p*-xylene, but as this compound shows no interference on the Supelcowax-10 column, quantitation by difference is possible.

Data integration and automatic identification

The performance of different integrators and data systems for the automatic identification of the

TABLE IV

ELUTION ORDER OF ANALYSED COMPOUNDS

Non-polar (SPB-1) and polar (Supelcowax-10) wide-bore capillary columns were used at 60°C. Compounds not resolved are bracketed. Numbers as in Tables I and II.

SPB-1	Supelcowax-10
(1) Ethanol	(2) Acetone
[(2) Acetone	(7) Ethyl acetate
(3) Propan-2-ol	(12) Isopropyl acetate
[(4) Propan-1-ol	(6) Butan-2-one
(5) Dichloromethane	(3) Propan-2-ol
[(6) 2-Butan-2-one	[(1) Ethanol
(7) Ethyl acetate	(5) Dichloromethane
(8) Trichloromethane	(14) Benzene
[(9) Butan-2-ol	(17) Trichloroethylene
(10) 2-Methoxyethanol	(20) Isobutyl acetate
(11) 1,2-Dichloroethane	[(8) Trichloromethane
[(12) Isopropyl acetate	(22) Tetrachloroethylene
(13) Butan-1-ol	(4) Propan-1-ol
(14) Benzene	[(19) Toluene
(15) 2-Nitropropane	(16) 1,2-Dichloropropane
(16) 1,2-Dichloropropane	[(11) 1,2-Dichloroethane
(17) Trichloroethylene	(21) *n*-Butyl acetate
(18) 2-Ethoxyethanol	(9) Butan-2-ol
[(19) Toluene	[(15) 2-Nitropropane
(20) Isobutyl acetate	(29) Isoamyl acetate
(21) *n*-Butyl acetate	(25) Ethylbenzene
(22) Tetrachloroethylene	(26) *p*-Xylene
(23) 4-Hydroxymethylpentan-2-one	┌ (13) Butan-1-ol
(24) 5-Methylhexan-2-one	│ (24) 5-Methyl-hexan-2-one
(25) Ethylbenzene	└ (27) *m*-Xylene
[(26) *p*-Xylene	(10) 2-Methoxyethanol
(27) *m*-Xylene	(30) *o*-Xylene
[(28) Cyclohexanone	(18) 2-Ethoxyethanol
(29) Isoamyl acetate	(28) Cyclohexanone
(30) *o*-Xylene	(31) 2-Ethoxyethyl acetate
(31) 2-Ethoxyethyl acetate	(23) 4-Hydroxymethyl-pentan-2-one
(32) 2-Butoxy ethanol	(32) 2-Butoxyethanol

separated compounds was evaluated. The following three commercially available devices were tested: Varian 4400 with memory module and replot option (equivalent to Spectra Physics Chromjet), Varian Vista 420 and Varian DS 650, all equipped with dual-channel input.

The aim was to investigate if the standard features of the integrators were suitable for identification of the compounds without the need for special programming.

Fig. 1 shows a flow-chart for the procedure in which the integrators compare the results of two chromatographic runs on the basis of the identified peaks, independent of their retention on the two columns (Varian data systems).

The outputs of the two flame ionization detectors are integrated by the data systems and names are attributed to the peaks on the basis of the retention times within a fixed tolerance and the retention times of individual peaks corrected by linear interpolariton between the retention times of "reference peaks" (generally the largest peaks in a given portion of the chromatogram), identified with variable tolerance of the retention times (window).

After the identification, correction factors are applied and the quantitative report for each column is

TABLE V

ELUTION ORDER OF ANALYSED COMPOUNDS

Non-polar (SP-2100) and polar (SP-1000) packed columns were used at 60°C. Compounds not resolved are bracketed. Numbers as in Tables I and II.

| SPB-2100	SP-1000
(1) Ethanol	(2) Acetone
[(2)Acetone	(7) Ethyl acetate
(3) Propan-2-ol	(12) Isopropyl acetate
[(4) Propan-1-ol	(6) Butan-2-one
(5) Dichloromethane	[(3) Propan-2-ol
(6) Butan-2-one	(5) Dichloromethane
[(7) Ethyl acetate	(1) Ethanol
(8) Trichloromethane	(14) Benzene
(9) Butan-2-ol	(17) Trichloroethylene
[(10) 2-Methoxyethanol	[(20) Isobutyl acetate
(11) 1,2-Dichloroethane	(8) Trichloromethane
[(12) Isopropyl acetate	L (22) Tetrachloroethylene
[(13) Butan-1-ol	[(4) Propan-1-ol
L (14) Benzene	(19) Toluene
(15) 2-Nitropropane	L (16) 1,2-Dichloropropane
(16) 1,2-Dichloropropane	(11) 1,2-Dichloroethane
(17) Trichloroethylene	(21) n-Butyl acetate
(18) 2-Ethoxyethanol	(9) Butan-2-ol
[(19) Toluene	[(15) 2-Nitropropane
(20) Isobutyl acetate	(29) Isoamyl acetate
[(21) n-Butyl acetate	L (25) Ethylbenzene
(22) Tetrachloroethylene	(26) p-Xylene
(23) 4-Hydroxymethylpentan-2-one	[(27) m-Xylene
(24) 5-Methylhexan-2-one	(24) 5-Methyl-hexan-2-one
(25) Ethylbenzene	L (13) Butan-1-ol
[(26) p-Xylene	[(30) o-Xylene
(27) m-Xylene	(10) 2-Methoxyethanol
[(28) Cyclohexanone	(18) 2-Ethoxyethanol
(29) Isoamyl acetate	(28) Cyclohexanone
(30) o-Xylene	(31) 2-Ethoxyethyl acetate
(31) 2-Ethoxyethyl acetate	(23) 4-Hydroxymethyl-pentan-2-one
(32) 2-Butoxy ethanol	(32) 2-Butoxyethanol
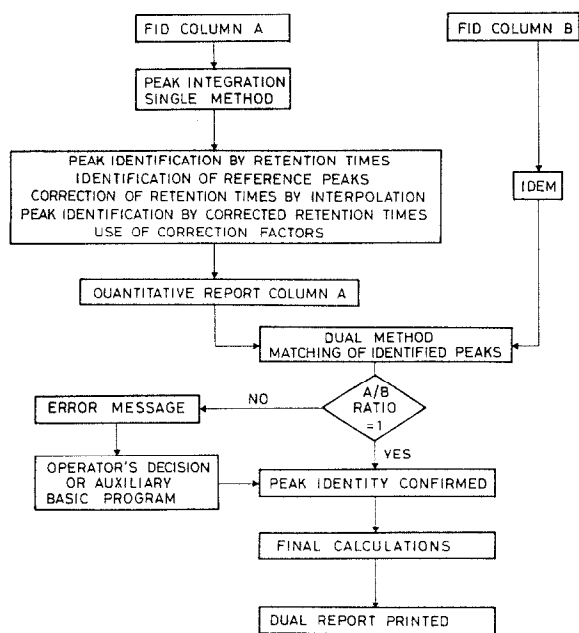
Fig. 1. Flow-chart of the program used by the integrators or data system to identify peaks by simultaneous analysis on columns of various polarities.

printed. By further elaboration, the ratio between the areas or the corrected amounts of the peaks identified on the two columns can be determined automatically. If the ratio for a given compound is equal or nearly equal to unity, this peak can be considered as free of interferences on both columns and, as a result of the different polarities of the stationary phases, its identity is confirmed. If the ratio is not unity, an error message appears and the chromatogram should be interpreted by the operator as described earlier to determine what kind of interference is present and to identify the peaks correctly.

A BASIC program can also be written for the Varian 4400 or Spectra Physics integrators equipped with this option, or for the standard Varian DS 650 data system, which, by using simple IF/AND/OR statements and comparing the calculated corrected peak areas, can determine whether (a) peaks mixed on column A are fully separated on column B and (b) peaks mixed on column A show interference with other peaks on column B.

The first situation, identical to example (1) of the preceding section, is relatively easy to solve because the total amount calculated on column A should correspond to the sum of amounts calculated for the two peaks on column B (after the application of correction factors), whereas complex consideration, and sophisticated programming may be necessary for solving situation (b). The logical flow-sheet of the program can follow the procedure outlined in examples (2) and (3) of the preceding section. Complex programs can identify all the compounds listed in Tables I and II automatically. Relatively simple programs are often sufficient, because the occurrence of situation (b) is infrequent in real samples as waste from different industries has a typical composition, is generally composed of only few substances and never contains all the compounds in this text mixture.

For quantitative analysis, it should be taken into account that sample distribution between the two columns connected in parallel, the different elution order on polar and non-polar columns and the changing peak shapes (due to the polarity of the compounds or to column overloading by highly concentrated samples) may lead to appreciable differences between the correction factors for the same compound on the two detectors. These must therefore be experimentally measured and checked periodically by injecting known samples.

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