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On-line chromatography with capillary columns and temperature programming

Determination of traces of 2-butenes in 1-butene and analysis of a reformate

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ABSTRACT

Two examples of automatic on-line chromatographic analysis with capillary columns and temperature programming are presented. The first is the detailed analysis of the effluent from a reforming unit. The sample is a C_4–C_{11} cut, mainly aromatic, which contains more than 200 compounds. It is shown that with automatic injection onto a capillary column, the resolution is as good as with manual syringe injection. Moreover, no discrimination between light and heavy compounds is observed. The second is the determination of ethylene, 1-butene (*ca.* 80%), traces of 2-butenes (less than 50 ppm), iso- and *n*-butane and C_6 olefins. This analysis was effected on a chromatograph equipped with two non-polar capillary columns and two detectors with different sensitivity ranges. The sample is injected simultaneously onto the two columns. On one detector the traces are analysed but the peak of the main component is saturated; on the other detector, the main component is analysed. The reproducibility of the retention times was checked, in both examples, in a 1-month test with automatic on-line injection.

INTRODUCTION

Automatic on-line process chromatographs are intended to operate automatically day and night without any intervention of the operator except for calibration of the apparatus. Up to now, almost all process chromatographs have worked under isothermal conditions and utilized packed columns, because the reproducibility of retention times is better under isothermal conditions than with temperature programming, and consequently the problem of the identification of peaks more easily solved, and injection onto packed columns is easier than onto capillary columns and the use of split injection is not necessary. However, the resolution of packed columns is worse than that of capillary columns. Therefore the analysis of complex mixtures with

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packed-column process chromatographs requires the use of column switching and often multi-oven chromatographs[1].

A few examples of the use of capillary columns have been described previously, mainly under isothermal conditions[2–4]. This paper describes the use of capillary columns and temperature programming for the automatic on-line analysis on two complex real examples: the complete analysis of gasoline and the determination of traces of 2-butenes in 1-butene.

Previously, a laboratory method was developed for the analysis of a reformate (gasoline from a reforming unit) and for the automatic calculation of the research and motor octane numbers (RON and MON, respectively) of this type of sample. The principle of the method is the separation of all the components of the sample on a capillary column with temperature programming, the automatic identification of those components, the calculation of their amount and the calculation of the octane numbers of the sample[5–7]. Such an analysis cannot be performed on packed columns. The transfer of this method to the automatic on-line analysis of reformate is described below.

Another application of on-line automatic capillary chromatography is the determination of traces components in gas samples in which the amount of the main component is greater than 70%. The principle of the analysis is the injection by gas valves of the sample simultaneously onto two capillary columns in the same gas chromatograph and connected to two different detectors. The sensitivity ranges of the two detectors are different: on the first the trace components are detected but the peak of the major compound is saturated, and on the second, the trace components are not detected but the peak of the main component is correct. This analysis is performed with temperature programming with a sub-ambient initial temperature.

Both on-line automatic methods were tested on pilot plants operating continuously.

EXPERIMENTAL

Analysis of a reformate

The samples were effluents from a pilot reforming plant operating continuously. The automatic on-line chromatographic analyses were carried out using a Hewlett-Packard 5890 gas chromatograph equipped with a Valco automatic liquid injection valve, a split injector and a flame ionization detector. The column was a 50 m × 0.22 mm I.D. wall-coated open tubular (WCOT) fused-silica capillary column coated with a non-polar methylsilicone stationary phase (PONA; Hewlett-Packard). The operating conditions were as described previously[5–7].

Integration of the peaks was carried out using a Hewlett-Packard 3392 integrator and data handling using a Hewlett-Packard HP 1000 A400 computer. Calibration, identification, quantification of components and determination of physical properties were performed using a Fortran program developed at the Institut Français du Pétrole.

Determination of traces of 2-butenes in 1-butene

The samples were effluents from a 1-butene pilot plant (Alphabutol IFP process) operating continuously. The automatic on-line chromatographic analyses were carried

out using a Delsi DI700 gas chromatograph equipped with two Valco automatic gas injection valves, two flame ionization detectors and a liquid nitrogen device for cooling the oven to sub-ambient temperature.

The columns were two 50 m × 0.2 mm I.D. WCOT fused-silica capillary columns coated with PONA. The carrier gas was helium and the operating conditions were as follows: −50°C for 2 min, increased from −50 to −15°C at 5°C/min and from −15 to 200°C at 10°C/min; the sensitivity ranges of the detectors were 10^{-11} A/mV (range 11, low-range detector) and 10^{-12} A/mV (range 12, high-range detector).

Integration of the peaks and data handling were carried out on a Hewlett-Packard HP 1000 A600 computer. The calculation of correct amount of each compound from the results given by the two detectors was performed using a Fortran program developed at the Institut Français du Pétrole.

RESULTS AND DISCUSSION

Analysis of a reformate: reproducibility

The automatic identification of the compounds in the reformate and the calculation of the RON and MON is based on the following principle. First, the *n*-alkanes are identified from their retention times; then, the retention indices with temperature programming for each peak are calculated and these calculated retention indices are compared with reference values in order to identify the components in the sample; and finally, using the amount of each compound and its RON and MON, the RON and MON for the sample are calculated.

The retention indices with temperature programming depend on the temperature of elution of the compound. This means that for automatic identification of all the compounds, they have to be eluted at almost the same temperature[5,6]. Consequently, the retention times of the *n*-alkanes should be adjusted so as to be in a previously defined range and they must remain constant from one analysis to another.

TABLE I

REPRODUCIBILITY OF RETENTION TIMES OF *n*-ALKANES DURING 5 WEEKS OF AUTOMATIC OPERATION OF THE REFORMATE OCTANE ANALYSER

n-Alkane	*Retention time (min)*				
	1st week	*2nd week*	*3rd week*	*4th week*	*5th week*
C_5	5.37	5.37	5.37	5.38	5.39
C_6	7.72	7.72	7.72	7.73	7.73
C_7	12.15	12.15	12.16	12.16	12.17
C_8	18.84	18.84	18.85	18.85	18.86
C_9	26.97	26.97	26.98	26.99	26.99
C_{10}	35.56	35.56	35.57	35.58	35.59
C_{11}	43.97	43.98	43.98	43.99	44.01
C_{12}	52.02	52.03	52.03	52.05	52.06
C_{13}	59.60	59.61	59.61	59.62	59.64
C_{14}	66.75	66.76	66.76	66.77	66.79
C_{15}	73.50	73.51	73.51	73.52	73.54

TABLE II

REPRODUCIBILITY OF THE AUTOMATIC ON-LINE ANALYSIS OF THE SAME REFORMATE SAMPLE DURING 5 DAYS AT A FREQUENCY OF 6 ANALYSES PER DAY

NA = *n*-Alkanes; IA = isoalkanes; N = naphthenes; A = aromatics; O = olefins.

Day	*Analysis*	*NA (%)*	*IA (%)*	*N (%)*	*A (%)*	*O (%)*	*Toluene (%)*	*Unknown (%)*	*RON*	*MON*
1	1st	8.98	22.37	1.89	65.34	1.36	14.11	0.08	94.3	84.9
	2nd	8.98	22.38	1.86	65.29	1.39	14.12	0.12	94.3	84.9
	3rd	8.96	22.36	1.90	65.34	1.36	14.14	0.08	94.3	84.9
	4th	8.94	22.29	1.86	65.45	1.35	14.14	0.13	94.3	84.9
	5th	8.90	22.18	1.87	65.57	1.37	14.17	0.12	94.3	84.9
	6th	8.90	22.23	1.93	65.53	1.39	14.16	0.04	94.4	84.9
2	7th	8.89	22.21	1.89	65.55	1.39	14.12	0.09	94.3	84.9
	8th	8.87	22.16	1.94	65.60	1.36	14.12	0.08	94.3	84.9
	9th	8.87	22.12	1.90	65.68	1.36	14.17	0.09	94.3	84.9
	10th	8.84	22.08	1.89	65.74	1.34	14.17	0.12	94.3	84.9
	11th	8.80	21.93	1.88	65.88	1.39	14.15	0.14	94.3	84.9
	12th	8.81	21.98	1.96	65.82	1.36	14.13	0.07	94.4	84.9
3	13th	8.80	21.98	1.92	65.82	1.36	14.12	0.13	94.3	84.9
	14th	8.80	21.97	1.93	65.83	1.36	14.15	0.13	94.3	84.9
	15th	8.83	22.02	2.00	65.76	1.36	14.16	0.04	94.4	84.9
	16th	8.80	21.93	1.98	65.93	1.35	14.17	0.04	94.4	84.9
	17th	8.79	21.94	1.99	65.92	1.35	14.19	0.03	94.4	85.0
	18th	8.79	21.88	1.94	65.98	1.33	14.19	0.08	94.4	84.9
4	19th	8.78	21.87	1.94	65.97	1.34	14.19	0.12	94.3	84.9
	20th	8.76	21.83	1.97	66.00	1.37	14.14	0.09	94.4	84.9
	21st	8.76	21.83	2.01	66.01	1.36	14.13	0.05	94.4	84.9
	22nd	8.76	21.85	2.02	65.99	1.36	14.15	0.04	94.4	84.9
	23rd	8.77	21.79	1.97	66.03	1.35	14.18	0.03	94.3	84.9
	24th	8.75	21.72	1.97	66.10	1.34	14.17	0.12	94.3	84.9
5	25th	8.75	21.71	1.98	66.14	1.34	14.20	0.09	94.4	84.9
	26th	8.73	21.67	2.05	66.13	1.36	14.18	0.09	94.4	84.9
	27th	8.70	21.68	2.05	66.20	1.35	14.17	0.04	94.5	84.8
	28th	8.74	21.68	2.07	66.09	1.39	14.17	0.04	94.4	84.9
	29th	8.72	21.61	2.02	66.19	1.39	14.18	0.09	94.4	84.8
	30th	8.71	21.59	2.01	66.24	1.37	14.17	0.09	94.4	84.9

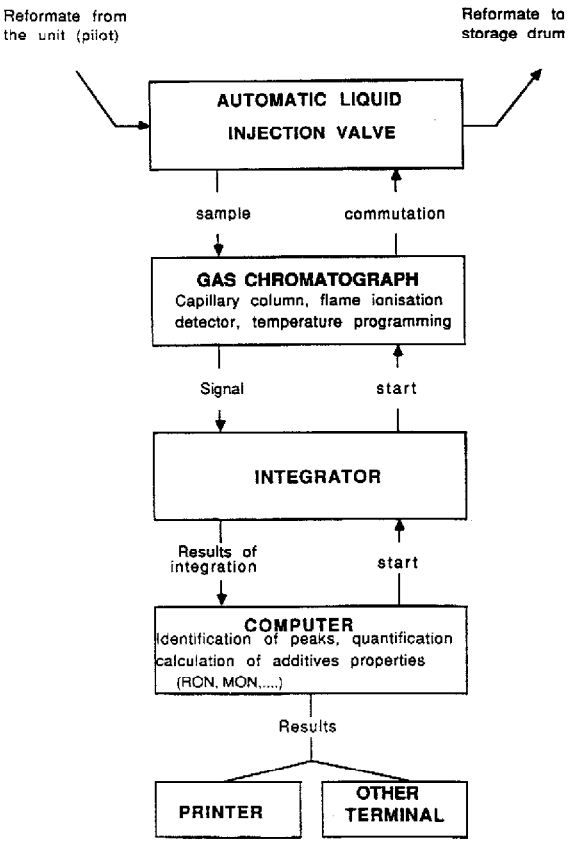

Fig. 1. Principle of the automatic on-line analysis of a reformate.

Table I gives the retention times of the *n*-alkanes in a calibration sample injected once a week when the apparatus is operating automatically with a frequency of eight analysis every 24 h. It can be seen that the main shift in retention time is less than 0.4%. This means that the *n*-alkanes in the sample are automatically identified from their retention times and the other compounds by their retention indices with temperature programming.

This was confirmed by the automatic on-line analysis during five days of the same sample circulating in a loop; the frequency of analysis was six every 24 h. The results are given in Table II. The small shift in the amounts of alkanes and aromatics is due to evaporation of a portion of the lighter compounds.

Fig. 1 shows the principle of the automatic analysis of a reformate.

Analysis of a reformate: on-line injection

A reformate is a complex mixture of hydrocarbons from C_4 to C_{11} (mainly aromatics), which contain more than 200 different components. The separation is performed on a non-polar capillary column; the operating conditions were previously optimized[5,6]. Owing to the complexity of the sample, it is very important to obtain the

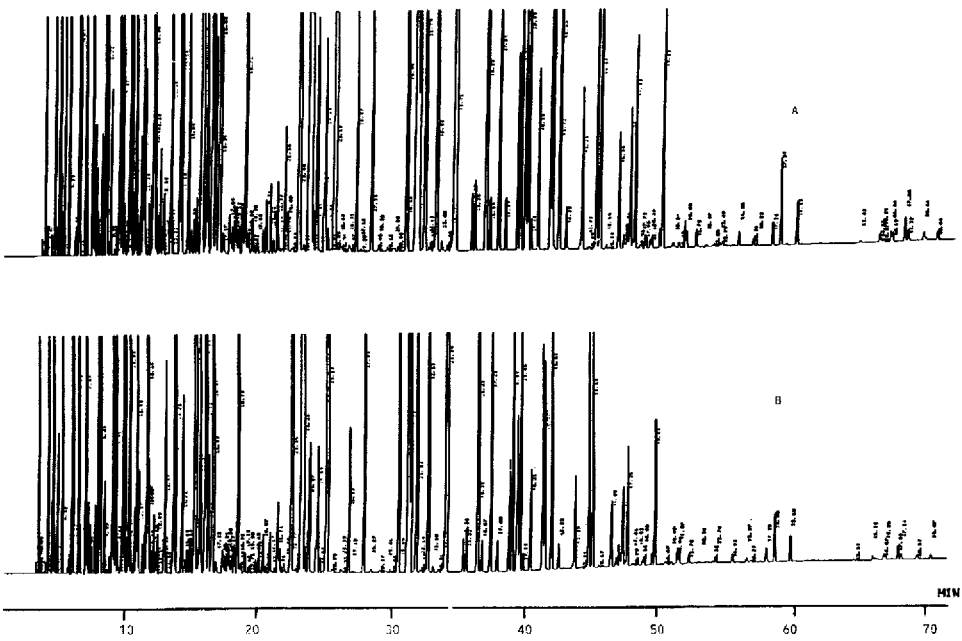

Fig. 2. Comparison of (A) syringe manual injection and (B) liquid valve injection capillary column chromatograms of a reformate.

same efficiency of the separation by valve injection and by syringe injection. The sample is injected in the liquid phase with a 0.5-μl injection valve in series with the split injector where it is vaporized. Fig. 2 shows that the separation is equally effective using automatic valve injection and manual injection.

The discrimination between light and heavy compounds during split injection is another known problem. With manual injection, no discrimination occurs over a large boiling point range, generally around 200°C. However, with automatic valve injection, the way in which the sample is vaporized is different, and the range without discrimination could be much smaller.

Consequently, it was checked that there was no discrimination due to the split injection. Table III shows the result of the analysis with automatic valve injection of a standard sample of *n*-alkanes in carbon disulphide. It can be seen that with valve injection there is no discrimination from C_5 to C_{13} but the amount of heavier compounds is subject to error. This means that the range without discrimination is shorter with automatic valve injection than syringe injection. However, the upper limit is higher than the final boiling point of the reformate. Consequently, the automatic analysis of a reformate can be performed by this method. This was confirmed by comparison of the analyses of the same sample using manual and automatic injection (Table IV).

Determination of a trace component in a major compound: principle of the method

On most commercially available gas chromatographs, the analysis of samples containing a major component and traces of other compounds is a problem because at

TABLE III

VALVE AUTOMATIC INJECTION OF A CALIBRATION SAMPLE CONSISTING OF THE SAME AMOUNTS OF *n*-ALKANES FROM C_5 TO C_{18} IN CARBON DISULPHIDE

Up to C_{13}, no discrimination occurs.

n-Alkane	*Concentration (%, w/w)*	*n-Alkane*	*Concentration (%, w/w)*
C_5	8.43	C_{12}	8.11
C_6	8.42	C_{13}	8.02
C_7	8.21	C_{14}	6.97
C_8	8.23	C_{15}	5.95
C_9	8.32	C_{16}	4.92
C_{10}	8.19	C_{17}	4.31
C_{11}	8.23	C_{18}	3.68

a high range of the detector the peak of the major component is saturated (Fig. 3) and at a lower range the trace components are not detected. Also, if the trace components are eluted close to the major component, it is not possible to program an automatic change of range.

This problem can be solved by analysing the sample simultaneously on two similar columns, connected to two different detectors, in the same chromatograph. One of the detectors is at a high range in order to detect the trace components and the other is at a low range in order to avoid the saturation of the peak of the major component. To calculate the exact amount of each component, the sample must also contain a compound present in a medium amount, the peak of which is not saturated in the high-range detector and which is detected by the low-range detector. This

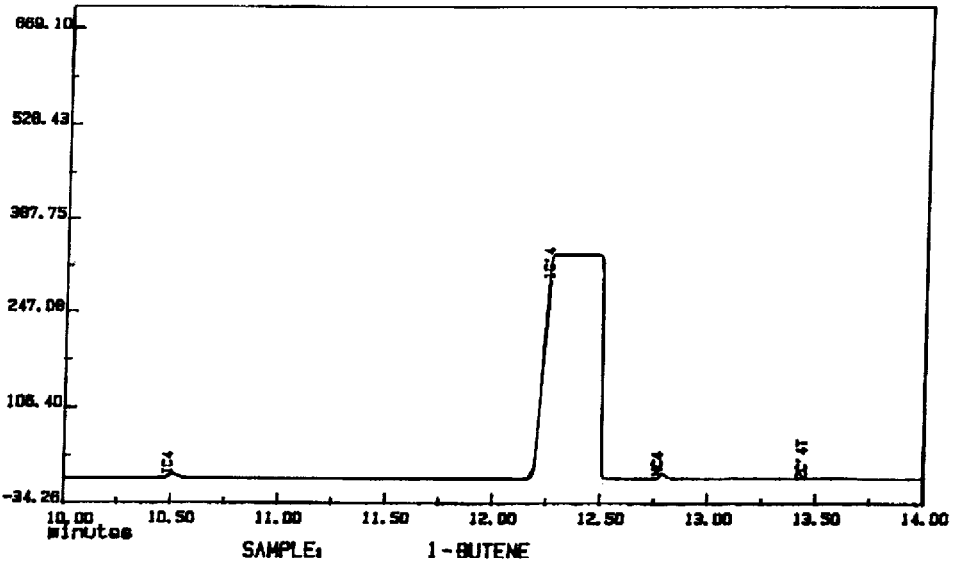

Fig. 3. Chromatogram of the saturated peak of 1-butene.

TABLE IV

COMPARISON OF ANALYSES OF THE SAME REFORMATE WITH ON-LINE AUTOMATIC INJECTION AND NORMAL SYRINGE INJECTION

Compounds	*Automatic valve injection*											*Manual syringe injection*										
	C_3	C_4	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	*Total*	C_3	C_4	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	*Total*
n-Alkanes	0.01	0.48	2.48	2.60	2.08	0.54	0.06	0.02	0.00	0.00	8.28	0.01	0.48	2.47	2.58	2.07	0.54	0.06	0.02	0.00	0.01	8.23
Isoalkanes	0.00	0.00	3.38	6.28	7.60	2.55	0.32	0.00	0.00	0.00	20.14	0.00	0.00	3.37	6.26	7.56	2.54	0.31	0.00	0.00	0.00	20.05
Naphthenes	0.00	0.00	0.00	0.29	0.30	0.13	0.00	0.00	0.00	0.00	0.73	0.00	0.00	0.00	0.28	0.30	0.15	0.00	0.00	0.00	0.00	0.74
Aromatics	0.00	0.00	0.00	4.17	19.69	27.48	16.09	2.47	0.16	0.09	70.15	0.00	0.00	0.00	4.14	19.59	27.46	16.20	2.51	0.17	0.15	70.22
Olefins	0.00	0.01	0.10	0.15	0.24	0.13	0.01	0.00	0.00	0.00	0.64	0.00	0.01	0.10	0.15	0.24	0.16	0.01	0.00	0.00	0.00	0.68
Unknown	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.01	0.02	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.03	002	0.04	0.09
RON						97.99											98.03					
MON						88.7											88.7					

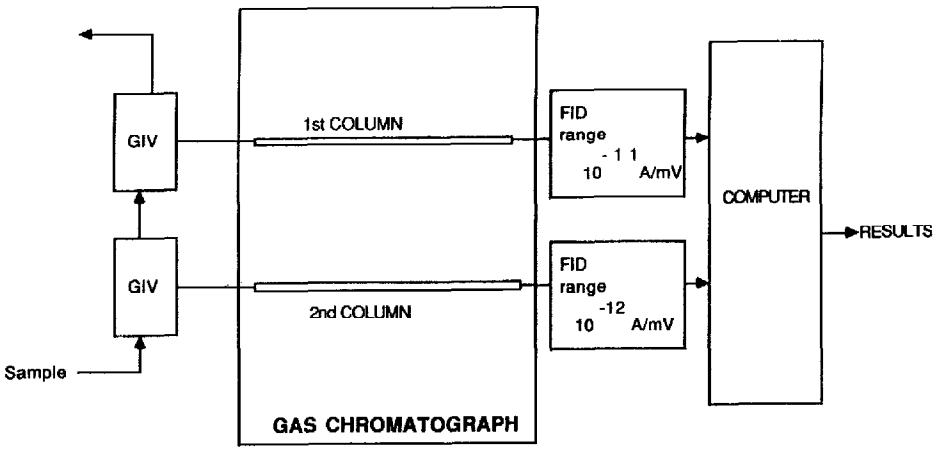

Fig. 4. Scheme of the procedure for the analysis of trace components in a major compound. GIV = Gas injection valve.

compound is used as an internal standard to correct the results from the high-range detector.

The factor K is calculated as K = %M on low-range detector/%m on low-range detector, where: M is the major component the peak of which is saturated on the high-range detector and m is the compound present in a medium amount which is detected on the low-range detector and the peak of which is not saturated on the high-range detector. Then, the correct amount of the major compound is calculated by the equation $\%M = K \cdot \%m$ on high-range detector. Finally, on the analytical report from the high-range detector, the incorrect value of the amount of M is replaced by the correct value and a normalization to 100 is performed.

One of the advantages of this method over, *e.g.*, injection of an external standard, is that it can be easily automated. The sample is injected simultaneously onto the two columns by automatic injection valves. The integration of the peaks from the two detectors is performed on the same mini- or personal computer, which can automatically calculate the correct amount of each compound immediately after the integrations. The scheme of the procedure is shown in Fig. 4.

Determination of traces of 2-butenes in 1-butene

An example of the application of the method is the automatic analysis of effluent from a unit that produces pure 1-butene from the dimerization of ethylene. At the outlet of the reactor, the sample to be analysed contains 1-butene, the major component (*ca.* 80%), remaining ethylene (a few percent) and by-products of the reaction, which are traces of 2-butenes (less than 50 ppm), *n*-butane (around 1000 ppm) and C_6 olefins. The main interest in this process for 1-butene production compared with other types of processes is the very low small amount of 2-butenes obtained as by products. Therefore, it is very important to determine the correct amount of 2-butenes and to monitor it continuously.

A correct separation of 80% of 1-butene and traces of 2-butenes and *n*-butane cannot be easily performed on packed columns. Consequently, this analysis is carried

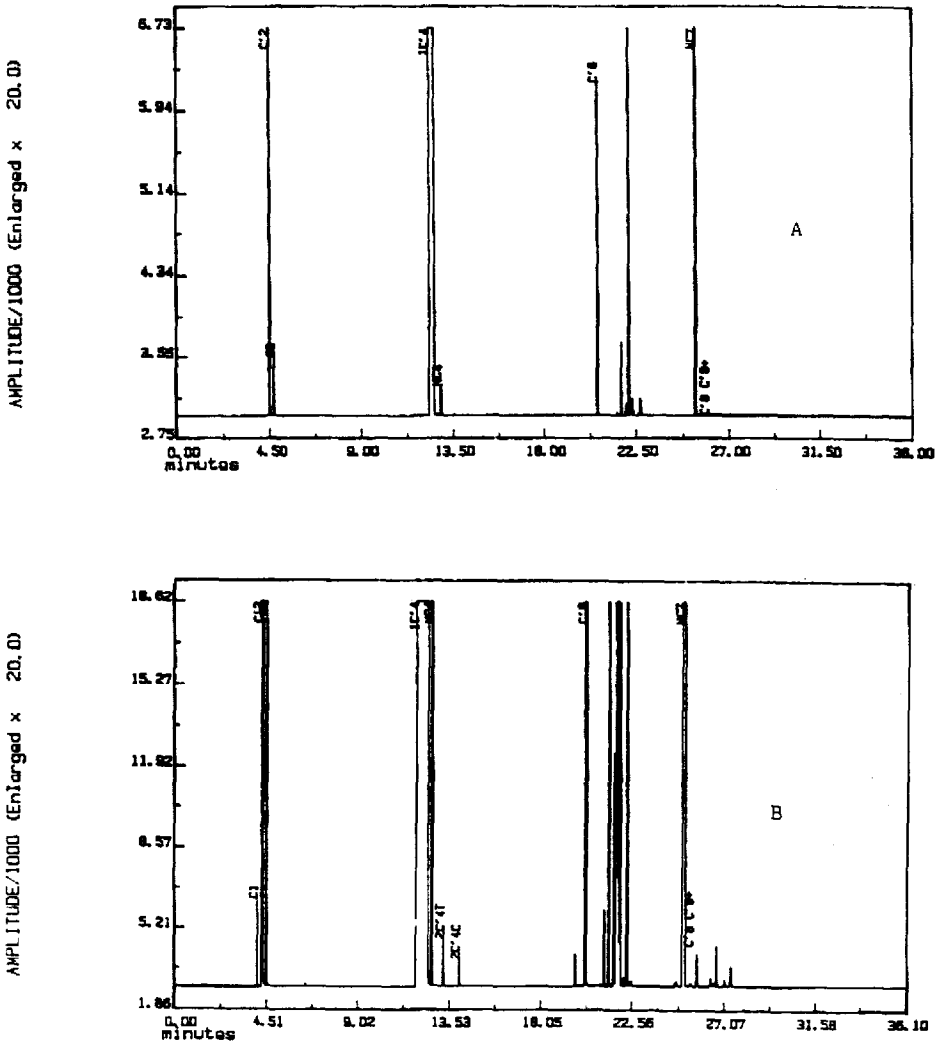

Fig. 5. Determination of traces of 2-butenes in 1-butene: (A) chromatograms of a 1-butene sample with 10^{-11} A/mV range and (B) 10^{-12} A/mV range flame ionization detector.

out on non-polar methylsilicone capillary columns (Fig. 5). The detectors which are used are flame ionization detectors, which are more sensitive than the thermal conductivity type.

The results of the analysis of a sample on a high-range detector, a low-range detector and the final result are shown in Table V. The effect of the saturation of the detector, which gives the amount of minor compounds too high, can be seen.

With a capillary column with a non-polar stationary phase, the separation of all C_4 hydrocarbons has to be done at sub-ambient temperature. Cooling the oven of the chromatograph is effected by liquid nitrogen. The use of a cryogenic cooling oven system could have produced poor reproducibility of the oven programming. During

TABLE V

ANALYSIS OF TRACES OF 2-BUTENES IN 1-BUTENE

Results with 10^{-11} and 10^{-12} A/mV range detectors and correct results after calculation.

Compound[a]	*% on 10^{-11} A/mV range detector*	*K = %M/%m on 10^{-11} A/mV range detector*	*% on 10^{-12} A/mV range detector*	*K · %M on on 10^{-12} A/mV range detector*	*Correct result (%)*
C_1	–		0.0251		0.0067
C'_2, M_1 saturated peak on 10^{-12} A/mV range detector	14.3173	13.5299	9.2730	53.7014	14.3617
C_2	0.1036		0.3933		0.1052
$1C'_4$, M_2 saturated peak on 10^{-12} A/mV range detector	79.0250	74.6787	66.9160	296.4072	79.2703
n-C_4	0.1057		0.4021		0.1075
$2C'_4$ *trans*	–		0.0277		0.0074
$2C'_4$ *cis*	–		0.0163		0.0044
3-Methyl-1-pentene internal standard (m)	1.0582		3.9691		1.0615
Other C'_6	1.8576		6.9950		1.8707
n-C_7	3.5170		11.9045		3.1837
Heavier compounds	0.0156		0.0779		0.028
Total	100		100	373.9196	100

[a] C_1 = methane; C'_2 = ethylene; C_2 = ethane; $1C'_4$ = 1-butene; n-C_4 = n-butane; $2C'_4$ *trans* = *trans*-2-butene; $2C'_4$ *cis* = *cis*-2-butene; C'_6 = hexene; n-C_7 = n-heptane.

5 weeks, the analysis of the sample was carried out automatically every 2 h; once a day, the retention times on the two columns of the main components were recorded (Table VI). The very low shift means that the reproducibility of the retention time is good enough for the automatic identification of the compounds by their retention times without modification of the calibration table. During this 5-week test, the automatic identification of the peaks by their retention times was performed and no error of identification was noticed.

CONCLUSION

It has been demonstrated that automatic on-line capillary column chromatography with temperature programming is possible even for very complex mixtures. For the two examples described, it would be very difficult and perhaps impossible to perform the same analysis with packed columns and/or under isothermal conditions; for a complete separation of a reformate there is no alternative to the use of capillary columns and temperature programming, and the separation of traces of 2-butenes in 1-butene is much easier on capillary columns. This means that the recent improvements in laboratory gas chromatography which involve the wide use of capillary columns and temperature programming will probably be widely used in the future for process chromatography.

TABLE VI

DETERMINATION OF TRACES OF 2-BUTENES IN 1-BUTENE

Reproducibility of retention times of main peaks with sub-ambient temperature programming during 5 weeks of automatic operation of the chromatograph (retention times in min).

Day	*10^{-11} A/mV detector*					*10^{-12} A/mV detector*				
	C'_2	$1C'_4$	n-C_4	C'_6	n-C_7	C'_2	$1C'_4$	n-C_4	C'_6	n-C_7
1	4.44	12.59	12.96	20.67	25.40	4.23	12.02	12.72	20.33	25.09
2	4.42	12.52	12.89	20.59	25.34	4.21	12.00	12.75	20.32	25.06
3	4.43	12.57	12.94	20.62	25.36	4.22	12.05	12.81	20.34	25.07
4	4.44	12.56	12.94	20.62	25.36	4.23	12.02	12.71	20.31	25.07
5	4.44	12.54	12.91	20.62	25.36	4.23	11.99	12.66	20.29	25.07
6	4.44	12.51	12.90	20.59	25.33	4.23	11.95	12.64	20.27	25.05
7	4.44	12.56	12.95	20.63	25.37	4.23	12.01	12.70	20.31	25.08
8	4.43	12.53	12.91	20.65	25.41	4.23	11.98	12.69	20.31	25.09
9	4.44	12.53	12.91	20.61	25.34	4.23	11.96	12.67	20.28	25.05
10	4.43	12.54	12.87	20.56	25.32	4.22	11.95	12.63	20.27	25.04
11	4.43	12.54	12.84	20.54	25.28	4.22	11.95	12.63	20.26	25.03
12	4.44	12.51	12.85	20.56	25.31	4.23	11.95	12.61	20.27	25.04
13	4.44	12.52	12.92	20.61	25.35	4.23	12.01	12.68	20.32	25.08
14	4.44	12.52	12.88	20.52	25.33	4.23	11.97	12.63	20.28	25.05
15	4.44	12.51	12.82	20.55	25.30	4.23	11.50	12.58	20.24	25.03
16	4.44	12.52	12.83	20.55	25.30	4.24	11.91	12.58	20.24	25.03
17	4.44	12.52	12.80	20.53	25.28	4.23	11.90	12.57	20.23	25.01
18	4.45	12.54	12.85	20.58	25.33	4.24	11.91	12.59	20.27	25.05
19	4.43	12.51	12.78	20.52	25.27	4.23	11.85	12.52	20.18	24.98
20	4.45	12.52	12.93	20.65	25.42	4.23	11.98	12.69	20.30	25.08
21	4.44	12.53	12.81	20.56	25.29	4.23	11.87	12.53	20.21	25.00
22	4.44	12.53	12.93	20.63	25.39	4.23	11.98	12.69	20.29	25.07
23	4.47	12.56	12.96	20.65	25.39	4.23	11.95	12.60	20.26	25.01
24	4.48	12.58	12.95	20.68	25.43	4.22	11.93	12.58	20.20	24.99
25	4.47	12.58	12.97	20.69	25.43	4.22	11.92	12.58	20.21	25.00
26	4.47	12.56	12.92	20.65	25.39	4.20	11.90	12.56	20.20	24.99
27	4.48	12.57	12.90	20.62	25.38	4.21	11.91	12.57	20.25	25.00
28	4.47	12.58	12.91	20.62	25.36	4.21	11.92	12.57	20.24	24.98
29	4.45	12.56	12.87	20.60	25.35	4.20	11.89	12.57	20.24	25.02
30	4.45	12.55	12.82	20.56	25.31	4.21	11.84	12.53	20.20	25.00
31	4.45	12.54	12.81	20.54	25.29	4.22	11.83	12.52	20.18	24.98
32	4.47	12.56	12.82	20.55	25.30	4.23	11.84	12.55	20.19	24.99
33	4.46	12.56	12.80	20.54	25.29	4.22	11.82	12.51	20.18	24.97
34	4.45	12.54	12.78	20.53	25.28	4.21	11.81	12.50	20.18	24.97
Higher gap[a]	0.05	0.08	0.19	0.17	0.16	0.04	0.24	0.31	0.16	0.12
Higher gap[b] (%)	1.1	0.65	1.5	0.8	0.6	0.95	2	2.4	0.8	0.5

[a] Difference between the higher and the lower value.

[b] Higher gap/medium value · 100.

It should be noted that the analyses described in this paper were carried out on laboratory equipment that is not explosion proof. However, equipment including a pressurized shelter to put inside the laboratory apparatus is now in progress and on-line tests in the refinery will take place in the near future.

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