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Note

Automatic data collection and analysis using an FM tape recorder

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The work described in this note was carried out with the aim of reducing the time required for the analysis of chromatography data. An essential part of such a scheme is automatic recording of the detector output in a usable form. For routine analysis, the instrument may work unattended, in which case amplifier attenuation must be recorded together with the detector output. However, for work of an exploratory or research nature, attenuation for each component can only be guessed beforehand and the presence of an operator is thus required. No attempt has therefore been made to automate the separation, judicious choice of column length, packing, etc., reducing this to a minimum.

The scheme at first considered was to connect the detector directly to a DEC PDP8 processor. Fully on-line processing has been described by Bartoli *et al.*¹ This would, however, have monopolised the processor unnecessarily, and it was decided to record the output on an FM tape recorder and feed the signal into the processor when this was available.

EXPERIMENTAL

Chromatographic analysis

Quenched gas samples from an arc discharge in hydrocarbon-nitrogen mixtures were analysed on a Pye 104 chromatograph, using a 2 m × 6.2 mm I.D. glass column packed with silica gel maintained at a constant temperature of 180°. Nitrogen was used as carrier gas. The main components of interest were methane, ethane, ethylene and acetylene and these were detected with a flame ionisation detector. No other components were present in sufficient quantity to give a measurable output signal. The analysis was complete within 7 min.

Recording of detector output

The detector output was in the range 0-1 mV and the signal was displayed on a 1-mV chart recorder to provide a visible record. In addition, the signal was amplified to the range 0-IV and fed to an SE 8/4 4-channel FM tape recorder. The tape speed was set to 1⁷/₈ in./sec.

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Processing of the detector output

When a complete tape had been produced, the output from the tape recorder was connected to an analogue-to-digital converter on the PDP8. The tape was then played back at 15 in./sec. The digital data could be used immediately by a programme to compute peak areas and component concentration but it was found more convenient to store the data in a disc file or on paper or magnetic tape for further reference. DEC LAB 8E BASIC was used to sample and store data from the tape recorder. Samples were taken at a rate of 20 per sec.

RESULTS AND DISCUSSION

Table I, showing six samples corresponding to a typical baseline portion of the chromatogram, reveals a regular oscillation corresponding to less than 0.5% full-scale deflection. When the tape recording was played back onto a chart recorder, an oscillation in the baseline was observed with peak to peak magnitude of 1.2% for playback at $1\frac{7}{8}$ in./sec and less than 0.5% for playback at 15 in./sec. The oscillation in the numerical data was of the same order and seems to have arisen from flutter during the recording rather than in the A-Ds. The induced noise was within the flutter specification given by the manufacturer for the tape recorder and was negligible for fast playback.

TABLE I

NUMERICAL DATA CORRESPONDING TO CHROMATOGRAM BASELINE

Full-scale deflection is ± 1.00000000 .

<i>Sample</i>	<i>Deflection</i>
1	+0.195E - 2
2	+0.586E - 2
3	+0.195E - 2
4	+0.586E - 2
5	+0.195E - 2
6	+0.586E - 2

Processing of the numerical data

With the data stored in the PDP8, calculations could be performed using programmes of any desired degree of sophistication, in any suitable language. For the data obtained here, a version of Algol-60 (Rogalgol Mk22) was chosen. A simple programme was written* with no data smoothing apart from spike elimination, since the chromatogram was "well behaved".

Briefly, after setting a baseline, the programme reads each data point until a value is reached that is greater than 1% above baseline. When this trigger point is reached, the next ten points are held. If these ten points are not continuously rising, a subroutine checks for a noise spike or shift in baseline. Reading then continues. If the points are continuously rising, they are accumulated until a value of less than 1% above baseline is found. The peak area is then simply the sum of all the ordinates

* Details may be obtained from the author.

INPUT FILENAME ?PNCHD2
 ENTER TOTAL POINTS, TRIG AND RESET LEVELS 850, .01, .01,
 NEW BASELINE, H= -0.7617179E-02
 AT POINT NO. 56

NEW BASELINE, H= +0.6054679E-02
 AT POINT NO. 129

ENTER ATTENUATION
 50
 PEAK STARTS AT POINT NO. 319
 NUMBER OF POINTS= 26
 AREA= +0.6940423E+01
 SCALED AREA= +0.3470211E+03
 TOTAL SCALED AREA= +0.3470211E+03

NEW BASELINE, H= +0.4601556E-02
 AT POINT NO. 385

ENTER ATTENUATION 850
 PEAK STARTS AT POINT NO. 531
 NUMBER OF POINTS= 55
 SPLIT INTEGRAL, AREA= +0.3797895E+01
 SCALED SPLIT INTEGRAL, AREA= +0.1898947E+03
 ENTER NEXT ATTENUATION 100
 AREA= +0.5055159E+01
 SCALED AREA= +0.5055158E+03
 TOTAL SCALED AREA= +0.6954106E+03

END OF DATA

AREA SUMMARY
 +0.3470211E+03
 +0.6954106E+03
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00
 +0.0000000E+00

END OF ANALYSIS

†

Fig. 1. Output from analysis programme.

accumulated. Trigger and reset values are adjustable to suit any particular chromatogram. The arbitrary areas may be calibrated by running a chromatogram of known composition.

The programme is designed to ask the operator for the attenuation corresponding to each peak. A further subroutine allows the area to be calculated for a "split integral", *i.e.* where attenuation is changed in mid-peak to avoid an off-scale signal. The programme also contains a subroutine to check and compensate for a sloping baseline (Fig. 1).

Performance

The narrowest peak, corresponding to methane, comprised 26 data points. The area of this peak was measured by four methods: (a) peak height \times width at half-height; (b) planimeter; (c) counting squares; (d) digital computer. The values are shown in Table II. The agreement for the last three methods is remarkable; *i.e.*, within $1/2\%$, and shows that no accuracy is lost through the various stages of data processing. Values are also shown for the ethane peak (55 points). It is worth noting that in both cases method (a) gave inconsistent results.

TABLE II
MEASURED PEAK AREAS (cm²)

Compound	Method			
	Peak height \times width at half-height	Planimeter	Counting squares	Digital computer
Methane	3.280	3.453	3.478	3.470
Ethane	6.421	6.960	6.817	6.954

CONCLUSIONS

The use of a tape recorder provides a simple and relatively cheap method of automating data collection and analysis from a chromatograph. An additional advantage is that the data processor need not be available at the same time as the separation is performed. The complexity of chromatograms that can be handled depends only on the sophistication of the programme used to analyse the data and, as this work shows, a very simple programme may be sufficient. There is no loss of accuracy through several stages of signal processing.

REFERENCE

- 1 B. Bartoli, S. Mobilio, N. Spinelli and F. Vanoli, *J. Chromatogr.*, 107 (1975) 51.