especially when rapid analysis is desired or small samples must be used. Methanol can be analyzed simultaneously with H_2O_2 by this method, but some interference between CH_2O and H_2O_2 is noted.

University of California, Los Alamos Scientific Laboratory,   N. R. GREINER
Los Alamos, New Mexico 87544 (U.S.A.)

1 W. C. SCHUMB, C. N. SATTERFIELD AND R. L. WENTWORTH, *Hydrogen Peroxide*, Reinhold, New York, 1955.

Errors in peak areas due to irregular travel of recorder charts

In quantitative gas–liquid chromatography (GLC) where concentrations are determined from peak areas it is widely appreciated that a detector–amplifier–recorder system with a linear response is required to give meaningful peak heights, and various methods for the determination of the linearity have been proposed. It is usually tacitly assumed that the rate of chart movement, on which depends the peak width, is perfectly regular, but it has been found that serious short-term variations in the rate of travel can occur with some recorders. AAKER[1] has discussed other aspects of recorder performance.

During precise determinations of detector response factors it was observed that the repeatability of the results was governed by the peak widths in successive chromatograms and that there were variations in width that could not be attributed to the operation of the column but rather to the recorder. The regularity of the chart movements of seven potentiometric recorders was investigated by applying to them a signal pulse from an electronic timer at regular intervals and measuring the distances between consecutive pulses on the charts with a travelling microscope. The charts were run at or near maximum speed to minimise measurement errors. A pulse interval of 15 sec was taken as being comparable with peak elution times in many applications. The pulse interval was derived ultimately from mains frequency but as a check on the regularity it was arranged that the pulses applied to the recorder also interrupted a steady signal supplied to a digital integrator and initiated the print-out mechanism so that a digital measure of the interval was obtained.

The results shown in Table I include the mean pulse intervals as measured on the charts and by the integrator, together with the ranges and standard deviations of both measurements. It may be seen that with recorders 1 to 5 there was considerably more variation in the intervals on the chart than on the integrator, indicating

TABLE I

RECORDER CHART TRAVEL

Recorder	1	2	3	4	5	6	7	7
Recorder chart speed	45 in./h	80 in./h	45 in./h	45 in./h	45 in./h	60 in./h	12 cm/min	3 cm/min
No. of observations	32	22	19	30	27	30	15	10
Pulse interval on chart								
Mean, cm	0.456	0.881	0.471	0.466	0.454	0.620	2.990	0.746
Range, % of mean	9.2	7.4	9.8	4.7	14.5	1.9	1.2	1.9
Standard deviation, % of mean	2.44	2.33	2.48	1.23	3.65	0.556	0.033	0.192
Pulse interval on integrator								
Mean, counts	268.4	223.2	226.8	231.8	176.5	164.6	—	—
Range, % of mean	2.2	4.0	4.8	1.7	1.7	1.8	—	—
Standard deviation, % of mean	0.50	0.83	1.34	0.015	0.43	0.53	—	—

that the chart movement was irregular. Recorders 6 and 7 gave substantially less irregularity than the other five.

Recorders 1 to 5 were similar models from one manufacturer having a chart width of 10 in. Recorder 6 was of different manufacture but similar specification while recorder 7 was a 20 cm width flat-bed model. The most obvious difference between the recorders was that 1–5 included a chain and sprockets in the chart-drive mechanism whereas 6 had worms and pinions and 7 had an entirely spur-gear train, but it has not so far been possible to prove that the chain is the source of the irregularity. The fitting of an improved type of chain supplied by the manufacturer made no significant difference to the chart travel.

The irregularities demonstrated could cause substantial errors in analysis and it is evident that for precise work careful testing and selection of recorders is important.

Benzole Producers Ltd., Technical Department,   G. W. GIRLING
Watford By-Pass, Watford, Herts. (Great Britain)   A. R. GIGG
                                                                         M. R. HELEY

1 D. A. AAKER, *Anal. Chem.*, 37 (1965) 1252.

Analysis of sugar mixtures by gas–liquid chromatography

The separation of sugar derivatives by gas–liquid(GLC) chromatography was actively studied recently[1]. Remarkable results were obtained for the separation of sugars as the trimethylsilyl and permethylated derivatives[2]. The only disadvantage of the method is the complicated pattern of the chromatogram as each sugar yields up to four isomers during methylation or trimethylsilylation. Thus GLC analysis of sugars as trimethylsilyl and methyl derivatives is efficient for simple mixtures only and for the identification of preseparated monosaccharides. GLC of derivatives giving only a single peak on the chromatogram for parent monosaccharide would be preferable for sugar determination. TMS derivatives of aldono-1,4-lactones and diethyl-dithioacetal acetates[3,4] satisfy this demand but are rather difficult to prepare. Acetates of polyols[5,6] have been suggested for GLC and are available for quantitative sugar analysis. However it should be noted that the elution time for the compounds is considerable due to their higher boiling points.

This paper describes the use of permethylated polyols and permethylated methyl glycosides for qualitative and quantitative analysis of sugars by GLC.

Hitherto the absence of a simple and effective procedure for producing permethylated polyols prevented the use of these derivatives for GLC analysis. The new and effective procedure of carbohydrate methylation in the presence of sodium hydride and dimethyl sulphoxide[7] has been recently applied to a number of carbohydrate types[8].