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Note

The rapid gas-chromatographic determination of sucrose as its trimethylsilyl ether on an open tubular column

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The determination of sucrose as its trimethylsilyl ether by g.l.c. on packed columns has been reported by several workers[1–8], with analysis times varying from ~4 min[9] to ~40 min[1]. We have shown that an open tubular column (190 m × 0.5 mm) coated with OV17 is suitable for the same purpose[10] and that the analysis time is ~15 min. Trehalose was used as an internal standard, and trimethylsilylimidazole was used as the silylating reagent.

Subsequently, we found that the analysis time may conveniently be reduced to ~1 min by using a 40-m column, as shown in Fig. 1. Under optimum conditions, a relative standard deviation of less than 0.1% may be obtained for a pure solution, as illustrated in Table I, which lists the results for 5 derivatives prepared by silylating the same 50% aqueous solution containing equimolar amounts of sucrose and trehalose. The relative standard deviation is slightly higher for such impure substances as molasses. In order to obtain the highest precision, it is essential that the injection septum and column be in good condition, that the injection block be of sufficiently high temperature, and that the detector be clean. Failure to take these precautions results in a substantially higher value for the relative standard deviation.

The precision shown in Table I appears better than the value obtainable on packed columns, even though it is difficult, from the few reports published[3,4,8], to judge the precision of sucrose analysis on packed columns. The overall precision of analysis, however, is a function of the standard error of the mean $\sigma/\sqrt{n}$, where σ is the standard deviation and n is the number of replicate analyses. Since, in a given time, it is possible to perform a larger number of analyses on an open tubular column than on a packed column, the former should yield the better overall precision, even if the relative standard deviations of the two methods are the same. The nine replicate analyses shown in Table I took ~12 min to perform, and result in a standard error of the mean of <0.01%.

A detailed study of the factors that affect the precision of this analysis will be published elsewhere.

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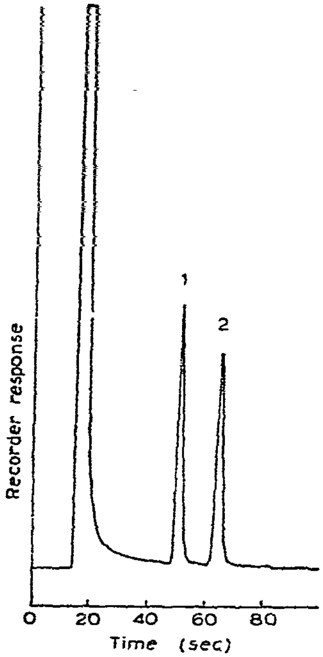

Fig. 1. Gas chromatogram of the *O*-trimethylsilyl derivatives of sucrose (1) and trehalose (2) on a 40-m open tubular column coated with OV17.

TABLE I

ANALYSIS OF FIVE IDENTICAL SAMPLES

Sample	*Weight of sugar solution (mg)*	*Sucrose*[a] *in solution (%)*	*Standard*[b] *deviation (%)*	*Relative*[b] *standard deviation (%)*
1	11.0	24.20	0.020	0.08
2	10.9	24.19	0.015	0.06
3	11.0	24.18	0.017	0.07
4	11.3	24.18	0.018	0.07
5	11.0	24.17	0.007	0.03

[a]Detector response of sucrose and trehalose assumed equal for illustrative purposes. [b]The relative standard deviation is for 9 replicate analyses.

EXPERIMENTAL

Chromatography. — A Perkin–Elmer Model 3920 gas chromatograph fitted with an inlet splitter and flame-ionization detector was used. A stainless-steel open tubular column (40 m × 0.5 mm; Handy & Harman Tube Company) was coated by the plug method with OV17 (Applied Science Laboratories Inc.) using benzyltriphenylphosphonium chloride (Aldrich Chemical Company) as a wetting agent. The inlet temperature was set at 350°, the oven temperature at 255°, and the detector at 300°. Dry, oxygen-free hydrogen was used as carrier. Peak areas were determined by using a Hewlett–Packard 3380 electronic integrator.

Silylation. — The trimethylsilyl derivative was prepared by treating 11 mg of the aqueous sugar solution with 0.6 ml (accurately pipetted) of a reagent consisting of 4 parts by volume of trimethylsilylimidazole (Ohio Valley Speciality Chemical Inc.) and 1 part of dry pyridine. The initial reaction is vigorous for dilute, aqueous solutions. The silylation is conveniently performed in a 1-ml reactivial (Pierce Chemical Company) fitted with a Teflon-faced liner. The derivatives prepared from a 50% aqueous solution may be used after being kept for 10 min at room temperature and may be stored for at least 48 h.

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