torily in our solvent systems. However, Hyflo Super-Cel due to its larger particle size tended to plug the nozzle of the chromatography spray bottle.

TABLE I

R_F VALUES OF REFERENCE COMPOUNDS AND PRODUCTS FROM ENZYMATIC DIGEST OF MALTOSE

	Adsorbent			
	Filter-Cel	*Hyflo-Super-Cel*	*Hyflo Super-Cel: Filter-Cel (6: 4)*	*Kieselguhr G*
Maltose digest				
Unknown	0.76	—	0.94	—
Glucose	0.67	0.94	0.76	0.96
Maltose	0.48	0.84	0.51	0.83
Isomaltose*	0.25	0.71	0.35	0.71
Panose*	0.15	0.50	0.16	0.55
Unknown	0.06	0.32	—	0.28
Unknown	0.00	0.00	0.00	0.00
Reference compounds				
Xylose	0.85	0.94	0.90	0.95
Glucose	0.67	0.94	0.76	0.95
Maltose	0.48	0.84	0.51	0.83
Development time (min)	80	30	50	50

* Tentative identification.

Conclusions

As a result of our work to date we have found the availability, low cost and high resolving power of ordinary filter aid materials make them excellent adsorbents for thin layer chromatography of carbohydrates. It is hoped similar results can be obtained with other classes of compounds.

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A method for the gas chromatographic separation of estrogens employing a solid injection system*

In recent communications from our laboratory, the gas chromatographic separation of synthetic mixtures of estrogens as well as their isolation from biological material

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have been reported[1,2]. In all of the reported investigations injections were carried out with the compounds dissolved in solvent.

A method employed in our laboratory for the gas chromatographic separation of a synthetic mixture of estrone, 17β-estradiol and estriol in the absence of solvent has been developed. The ability to separate the estrogens in the absence of a solvent would be extremely helpful in handling biological samples.

Experimental

An aliquot of redistilled dioxan is drawn up into a 10 μl microsyringe (Hamilton Syringe Co., Whittier, Calif.). Immediately afterwards an equivalent aliquot of a synthetic mixture of estrone, 17β-estradiol and estriol is drawn up into the same microsyringe. The contents of the microsyringe are then placed on a solid injection syringe (Hamilton Syringe Co., Whittier, Calif.). A domestic hair dryer is employed to facilitate the transfer and initially evaporate the solvent. The syringe is then placed in a 101° drying oven for 20 min to ensure the complete evaporation of the solvent.

The sample is introduced, employing the solid injector, into a Barber-Colman, model 10, gas chromatograph equipped with a ^{90}Sr ionization detector.

A 6 ft. long, "U" shaped, pyrex glass column with an internal diameter of 5 mm was packed with a stationary phase of 3% QF-1 (fluorosilicone) on 100/120 Gas Chrom-P (Applied Science Laboratories, Inc., State College, Pa.). The temperature of the column was maintained at 252°, the detector at 274° and the "flash heater" at 319°. Argon was employed as the carrier gas and a flow rate of 88 ml/min was maintained. The column was previously conditioned, while vented to the air at 255° and at the operating pressure of the carrier gas for 24 h.

Results and discussions

In Fig. 1 may be observed the gas chromatographic separation of the estrogens following their introduction into the instrument in solvent. In Fig. 2 may be seen the gas chromatographic separation of the same compounds following their introduction with a solid injection system. It may be readily observed that the peak representing the solvent has been eliminated.

The elimination of the solvent peak will offer three advantages: first, the operating sensitivity can be increased without introducing the interference of a broad solvent peak; secondly, the retention times of the compounds can be decreased, also increasing sensitivity; a third advantage is the ability to introduce into the chromatograph larger volumes of concentrated samples than would be possible employing an injection with a solvent.

Other methods of introducing solids into a gas chromatograph have been suggested by BOWMAN AND KARMEN[3], RENSHAW AND BIRAN[4] and MCCOMAS AND GOLDFIEN[5]. The methods, in each case, as suggested by the other investigators require interruption of the carrier gas flow, a modification of the injection system or difficulty in injecting the samples. None of the above-mentioned objections were observed employing the method suggested in this paper.

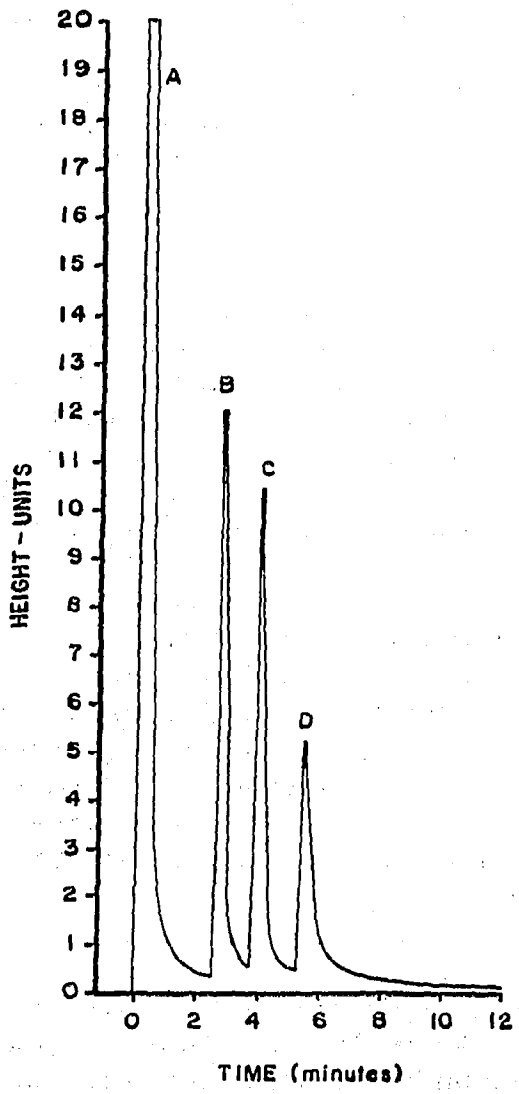

Fig. 1. Estrogens introduced in solvent. Gas chromatographic separation of (A) solvent, (B) 1.0 μg 17β-estradiol, (C) 1.0 μg estrone and (D) 2.0 μg estriol. Conditions: column, 6 ft., 5 mm I.D. pyrex glass column; 3% QF-1 on 100/120 Gas Chrom-P; 252°, 88 ml/min flow rate; detector, 274°; flash heater, 319°.

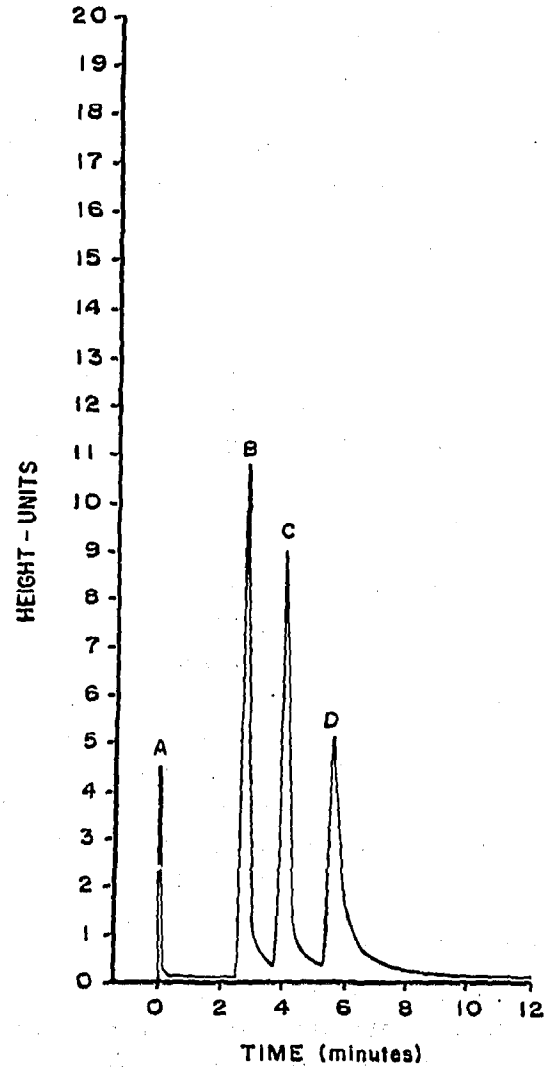

Fig. 2. Estrogens introduced in solid injector. Gas chromatographic separation of (A) air peak, (B) 1.0 μg 17β-estradiol, (C) 1.0 μg estrone and (D) 2.0 μg estriol. Conditions: column, 6 ft., 5 mm I.D. pyrex glass column; 3% QF-1 on 100/120 Gas Chrom-P; 252°, 88 ml/min flow rate; detector, 274°; flash heater, 319°.

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