then rinsed to a pH of 9.5 with deionized water. Solutions of N potassium chloride and of N potassium sulphate have been used as starting materials for the preparation of caustic solutions. The potassium salts were selected in preference to the corresponding sodium salts because they contained smaller amounts of silica. The salt solutions were passed through the resin at the rate of 0.15 volume/ volume resin/min. A total of one volume of salt solution per volume of resin was treated in each cycle. Regeneration of the column between cycles was carried out with sodium bicarbonate and sodium hydroxide by the same method as the original regeneration. In each cycle the hydroxide effluent was collected in five equal fractions and analysed for silica content and for their hydroxide content. Typical results are given in Table 1.

Table 1. CHARACTERISTICS OF HYDROXIDE SOLUTIONS PREPARED VIA

ION EXCHANGE				
Cycle	1	2	3	4
Column size	2 in.*	$2 \text{ in.} \dagger$	2 in.	2 in.
Influent	1 N pot.	1 N pot.	1 N pot.	1 N pot.
	chloride	chloride	sulphate	sulphate
N Hydroxide			-	
Cut 1		0.369		0.413
Cut 2		0.501		0.502
Cut 3	0.106	0.501		0.455
Cut 4	0.311	0.437		0.402
Cut 5	0.451	0.379		0.343
p.p.m. Silica				
Cut 1	5		0.78	0.20
Cut 2	5	$2 \cdot 2$	0.67	0.15
Cut 3	5	$2 \cdot 0$	0.31	0.11
Cut 4	5 5 5 5 5		0.29	0.10
Cut 5	5	~~ ~	0.24	0.10

* Glass column. † Only sodium hydroxide used as regenerant.

It may be seen that the quality of the effluent hydroxide increases with increasing number of regeneration cycles. The solution prepared in cycle 4, when used for solubilizing silica at a ratio of 1 ml./50 ml. of water sample, contributes only 0.002 p.p.m. to the final solution being analysed. This is below the present limits of detection of silica by colorimetric methods.

The above procedure is now being employed in a study of the concentration of soluble and total silica in ion-exchange resin deionization systems, and the results of this study will be submitted for publication in the near future.

SALLIE FISHER ROBERT KUNIN

Rohm and Haas Co.,

Philadelphia, Penn. Jan. 11.

¹ Straub, F. G., and Grabowski, H. A., Indust. Eng. Chem., Anal. Ed., 16, 574 (1944).

Use of the Orcinol - Sulphuric Acid Reaction in the Positive Identification of Certain Monosaccharides from a Salivary Mucoid

An approximate identification of sugars separated from hydrolysates of biological material can usually be made by paper chromatography. Positive identification is usually achieved by the measurement of some specific physical property such as optical rotation, or the preparation of derivatives possessing characteristic melting points. It frequently happens that sugars separated from hydrolysates are present in insufficient amounts to enable such methods of identification to be used.

The modified orcinol-sulphuric acid reaction described by Bruckner¹ is eminently suitable for the identification of small amounts of various hexoses, which give characteristic absorption spectra readily distinguishable from one another. The different aldopentoses exhibit the same absorption curve which, however, is different from those of the individual hexoses. As little as 0.1 mgm. of the sugar is necessary to obtain a satisfactory absorption curve.

In the course of our investigations into the composition of mucoids occurring in human saliva, a fraction was obtained which was thought to be identical with the 'blood group A substance' described by Aminoff, Morgan and Watkins². Less than 100 mgm. of the substance was hydrolysed by refluxing with water in the presence of 1 gm. of 'Amberlite IR120' ion-exchange resin at 100° C. for 16 hr. The resin was filtered off and washed with water. The combined filtrate and washings were evaporated to dryness in vacuo and the residue taken up in 2 ml. of water.

One-dimensional chromatograms were run on Whatman No. 1 paper in methyl ethyl ketone/acetic acid/water (6:1:1), a very rapidly running solvent introduced in this laboratory (Leaver, A. G., and Irwin, M., unpublished work). Two of these gave R_F values approximating to those found for galactose and fucose. When the hydrolysate was again run with spots of these two sugars run adjacently, the tentative identification was confirmed.

The total hydrolysate was applied to three wider strips of the very thick paper Whatman No. 3 MM, application being in the 'ribbon' form across the width of each paper. Development was continued for 18 hr., after which the chromatograms were dried, narrow strips cut from each edge and sprayed with aniline hydrogen phthalate. The areas corresponding to the two principal bands obtained were cut out of the main strips and eluted with distilled water. The two eluates were evaporated to dryness in vacuo, the solids being weighed and taken up in distilled water to give a concentration of 0.1 mgm. per ml. Both solutions were then treated with orcinol and sulphuric acid, according to the method of Bruckner¹, absorption curves being measured on the 'Unicam' S.P.600 spectrophotometer. The first solution gave the typical absorption curve for galactose, whereas the latter exhibited the highly characteristic spectrum of fucose, which shows maxima at 390 mµ and 500 mµ and is totally different from all other spectra examined (Bruckner, private communication).

Thus galactose and fucose were identified as neutral sugars present in the hydrolysate of the salivary mucoid. It is of interest that Aminoff, Morgan and Watkins² found these two sugars in hydrolysates of

human 'blood group A substance'. Use of the orcinol – sulphuric acid reaction for the positive identification of monosaccharides affords a rapid and simple way of making definite confirmation of results obtained by paper chromatography.

We are indebted to Dr. J. Bruckner for his valuable assistance and for making available the unpublished absorption curve for fucose.

M. IRWIN A. G. LEAVER

Medical Research Council (N.Z.), University of Otago Dental School, Dunedin, New Zealand. Feb. 2.

¹ Bruckner, J., Biochem. J., 60, 200 (1955).

² Aminoff, D., Morgan, W. T. J., and Watkins, W. M., Biochem. J., 46, 426 (1950).