II.—ESTIMATION OF PENTOSES AND PENTOSANS BY DIFFERENT METHODS.

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In the foregoing investigation, the author made a comparative study of different methods commonly used for estimating pentoses and pentosans in straw. There is no method at present for direct estimation; those in use depend on the quantitative transformation of the carbohydrates, by distilling with acid, into furfural which can be estimated in various ways. Since small amounts of furfural are formed also from other carbohydrates, none of the methods can be regarded as specific for pentoses and pentosans.

Distillation of furfural.—Distillation with 12 per cent. hydrochloric acid, directly according to Tollens (Browne, *Handbook of Sugar Analysis*, 1912, 450) and with steam according to Jolles (*Sitzungsber. Akad. Wiss. Wien* 1905, **114 (IIB)**, 1191) respectively were tried. Steam distillation using phosphoric acid (Youngberg, *Biol. Chem.*, 1927, **73**; 599) was also attempted. It was observed that furfural was not completely removed by steam even after eight hours of distillation. The collected distillates, which were never less than three litres, rendered the adoption of gravimetric methods impracticable,

Methods involving the use of (1) phloroglucinol (Kröber, *J. Landw.* 1900, **48**, 357), (2) barbituric acid (Jäger and Unger, *Ber.*, 1902, **35**, 4440; 1903, **36**, 1222), (3) bisulphite-iodine (Jolles, *loc. cit.*), (4) electrical titration (Pervier and Gartner, *J. Ind. Eng. Chem.* 1923, **15**, 1167, 1255), (5) excess of bromine (Powell and Whittekar, *J. S. C. I.*, 1924, **43**, 35T), (6) phenylhydrazine (Ling and Nanji, *Biochem. J.*; **15**, 466), (7) aniline acetate (Youngberg, *J. Biol. Chem.*, 1927, **73**, 599) and (8) benzidine reagent (McCance, *Biochem. J.*, 1926, **20**, 1111) were tried.

The first two were gravimetric, the last two colorimetric and the rest volumetric. The colorimetric methods could not be applied to the hydrochloric acid distillates owing to difficulties in matching colours.

Furfural.—Known quantities of pure furfural were estimated according to the different methods (Table I).

TABLE I.

Method	Furfural		Deviation per cent.
	Actual, grams	As estimated, grams	
Phloroglucinol	0·1202	0 1245	+ 3·6
Barbituric acid	,,	0·1205	+ 0·3
Bisulphite-iodine	,,	0·1195	+ 0·7
Electrical titration...	0·0992	0·0996	+ 0·4
Excess of bromine	0·1290	0·1285	+ 0·3
Phenylhydrazine	0·2362	0·2369	+ 0·3

It was found difficult to determine the end-point by the electrical titration method. The bromine method was easy to conduct and gave quite consistent results.

Samples of pure xylose were treated with 12 per cent. hydrochloric acid and distilled directly according to Tollens (*loc. cit.*) and with steam according to Jolles (*loc. cit.*), the results being given in Tables II and III, respectively.

TABLE II.

Method	Phloroglucinol	Barbituric acid	Bisulphite	Bromine	Phenyl-hydrazine
Deviation per cent. from the actual	+ 2·8	—2·4	2·5	—0·1	—1·4

TABLE III.

Method	Bisulphite	Bromine
Deviation per cent. from the actual ...	4·5	—0·2

Similar specimens steam distilled with 85 per cent. phosphoric acid and estimated colorimetrically gave results differing on an average by 0·4 per cent. from the actual.

The following results (Table IV) were obtained for pure arabinose distilled with 12 per cent. hydrochloric acid according to Tollens.

TABLE IV.

Method	Phloroglucinol	Barbituric acid	Bisulphite	Bromine	Aniline acetate
Deviation per cent. from the actual.	−1·1	−2·0	+2·5	+0·2	Very large

It was inferred from the above that the bromine method of Powell and Whittekar gave the most accurate results.

It was found, on further trial, that the experimental error arising through the use of the same method could be reduced to a minimum by (*a*) using ground glass joints in place of rubber or cork stoppers for distillation of furfural and (*b*) titrating, after completion of reaction, against N/50 thiosulphate. The deviations being small and on either side of the mean, it was possible to reduce the error, still farther, by repetition.

SUMMARY.

Distillation with 12 per cent. hydrochloric acid from an all glass apparatus followed by estimation of furfural in the distillate by the bromine method of Powell and Whittekar was found to be the most satisfactory technique for estimating pentoses and pentosans.

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