

tion, a quantity sufficient to dissolve more than half the water originally present.

Separation of 2,3-Dimethyl- α -methylglucoside and 2,3,4-Trimethyl- β -methylglucoside.—A solution was made up containing 1.330 g. of dimethylmethylglucoside and 1.058 g. of trimethylmethylglucoside in 100 cc. of water; 50 cc. of this was extracted five times, using 50 cc. of chloroform for each extraction, and the combined chloroform extracts were washed with 50 cc. of water. The two solutions were evaporated, the residues weighed, made up to 20 cc. with water and their rotations measured. From the "apparent molecular rotation" thus found, the composition of each extract was calculated, and thus the yield of each sugar.

The table shows the results of this first extraction. The "theoretical" values are those calculated from the partition coefficients recorded above.

Init. compn.	Cpd.	Dimethyl-Trimethyl-		
		methyl- glucoside	methyl- glucoside	
Grams		1.330	1.058	
	%	55.7	44.3	
Extracted H ₂ O soln.	Yield	Calcd., g.	1.120	0.0005
		Found, g.	1.100	.022
	Purity	Calcd., %	99.9
		Found, %	98.2
Washed CHCl ₃ extr.	Yield	Calcd., g.	0.032	0.999
		Found, g.	.023	.928
	Purity	Calcd., %	...	97.5
		Found, %	...	97.6

The water used to wash the chloroform extract contained about 15% of the original sugars. This was extracted and the extract washed exactly as the original solution was treated. When the two aqueous solutions and the two chloroform solutions have been combined, the final result is:

Compound	Dimethyl- methylglucose	Trimethyl- methylglucose	
Purity, %	Calcd.	99.9	97.4
	Found	98.3	97.4
Yield, %	Calcd.	95.5	99.6
	Found	97.4	97.6

The final wash water, which should contain only 1% of the original sugars (0.55% found), may be rejected.

It should be noted that the scheme here followed was devised partly because it approximated to that which has been usual in these Laboratories and partly because it was expected to give reasonable yields of both sugars in a fairly pure state,

without undue expenditure of time. But it is obvious that in special cases it may be modified to produce even greater purity of one component at the expense of the yield, without increasing the time spent on the process, or to obtain any desired degree of separation by the expenditure of sufficient labor. The results substantially confirm the effectiveness of the methods used here to separate mixtures of methylated sugars, and thereby to determine their relative proportions, and add weight to the arguments of Irvine and Stiller [THIS JOURNAL, 54, 1486 (1932)] in discussing the methylation of sucrose.

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RECEIVED OCTOBER 6, 1934

Electrolytic Reduction of Imido Ethers

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It has been shown that the reduction of imido ethers in acid solution with sodium amalgam yields aldehydes.² When, however, this class of compounds are subjected to electrolytic reduction, it was found that primary amines are formed. Since imido ethers, in form of their hydrochlorides, are available in quantitative yield from nitriles,³ the method offers an alternative way for the reduction of nitriles to primary amines.

Experimental

The reduction is carried out at 0°, in the usual manner using lead electrodes, 2 *N* aqueous sulfuric acid and the hydrochlorides of the imido ethers. Six typical reductions gave the following yields: benzylamine, 76%; 3-tolubenzylamine, 70%; 4-tolubenzylamine, 94%; ethylamine, 16%; phenylethylamine, 14%; 4-ethoxybenzylamine, 66%.

4-Ethoxybenzylamine.—Since this compound has not been described hitherto, its picrate and hydrochloride were prepared and the latter analyzed. The picrate (from water) melts at 191° and the hydrochloride (from ethanol) melts at 234°.

Anal. Calcd. for C₉H₁₄ONCl: Cl, 18.9. Found: Cl, 18.9.

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RECEIVED DECEMBER 13, 1934

(1) Abstracted from Doctoral Dissertation, "Die Elektrolytische Reduction der Imidoether, der Nitrostyrole und des β -Oxyppyridins." Freiburg, Germany, 1920.

(2) Henle, *Ber.*, **35**, 3039 (1902).

(3) Pinner, *ibid.*, **16**, 353 (1883).