Biochemical Studies on Pityrol, VIII Distillation of Sucrose

By

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Sucrose purified by recrystallisation of commercial cane sugar, was found to melt at $170^{\circ}-171^{\circ}$ and showed $[a]_{\rm D}=64^{\circ}$.

30 grm. of the sugar were distilled at ordinary pressure in an iron retort of 500 c.c. capacity, the reaction began to take place vigorously at about 240° , and then ceased after 2 hours. The highest temperature of the retort during the reaction was about 610° . 100 grm. of the sample, thus, yield 25 grm. of tar and water liquor, 45 grm. of charcoal and 20 litres of gas.

The distillate, which contains tarry matter, was a dark reddish-brown acidic liquor of a density of 1.06, the colour on standing, changing gradually to a dark brown, forming a dark brown solid deposit at the bottom of the vessel.

The distillate separated from the brown deposit, was treated with ether for 5 hours to extract furfurals and some organic acids formed from the sugars by distillation, and distilling off the solvent, there was found an oily residue which consisted of acidic and neutral substances. The solid substance in the aqueous distillate, which was insoluble in ether was ascertained from its chemical nature to be humus. The percentage of these substances in the distillate was as follows:

Water	75
Ncutral substance	4.5
Acidic substance	1.5
Humus	8.5
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1. NEUTRAL SUBSTANCE

11.5 grm. of the neutral substance were subjected to fractional distillation under 7 mm. pressure, and the following fractions of the constants resulted :

	Fraction	Yield	d ²⁵ 4,	${}^{n_D^{25}}$
I	34°42°	0,6 grm.	1.1449	1.5188
2	42°-47°	0,I "	-	1.5179
3	47°-65°	1.8 "	1.1219	1.5147
4	65°—140°	0.3 "		1.5118
5	140°-142°	0.6 "	1.2065	1.5 3 66
6	Residue	4. "	-	-
	l		J	

A. Furfura!.

The first fraction was confirmed to be composed of furfural by its analysis and also by its physical constants.

C=62.5; H=4.5, theory requires C=62.5; H=4.2 for $C_3H_4O_2$ R Methyl Eurfurgi

B. Methyl Furfural.

The third fraction was analysed, and the results agree with the theoretical value for methylfurfural.

C=63.2; 65.8: H=5.2; 5.1: theory requires C=65.5;

 $H \approx 5.4$ for $C_6 H_6 O_2$.

C. Oxymethyl Furfural.

The fifth fraction was assumed to consist mostly of oxymethyl furfural by analysis of the results.

C=59.7; 59.3; H=5.6; 5.7; theory C=57.1; H=4.8 for C₆H₆O₃.

2. ACIDIC SUBSTANCE

The acidic substance consists of volatile and non-volatile acids and the former was found by its chemical properties to be formic acid and the latter laevulinic acid.

3. HUMUS SUBSTANCE

The brown solid substance which occurs in the distillate and is insoluble in ether, was analysed with the following results

C = 62.65; H = 4.55.

4. CHARCOAL

The non volatile substance which remained in the retort was analysed with the following results :

C = 70.45; 67.56; H = 4.59; 4.92.

5. QUANTITATIVE ESTIMATION OF THE SUBSTANCES IN THE DISTILLATE

The quantity of furfural and methyl furfural substance in the distillate was determined by means of phloroglucin, and the content of the oxy-methyl furfural was also determined in the non-volatile neutral substance.

The quantitative estimation of the formic acid and the laevulinic acid in the distillate was performed by determining the total volatile acids by titration with a dilute caustic soda solution and the results are as follows:

1 %
2,6 "
2.5 "
3.1 ,,
0.4 "

The further researches on Pityrol are now being studies in Professor Matsumoto and my laboratories and the result will be communicated in a near future.

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