fractionally precipitated into three gummy fractions. It is important to precipitate the compound with alcohol in order to separate it from the small excess of sulfuric acid. If the solution is left even slightly acidic, the compound will completely hydrolyze to fructose.

The precipitate from each fraction was dissolved in a minimum of water and dried in a vacuum oven at 60°. These fractions appeared to be identical; yield 42%.

Anal. Calcd. for $(C_6H_{10}O_5)_x$: C, 44.4; H, 6.22. Found: C, 43.9; H, 6.27.

Preparation of the Acetate.—The acetylation was carried out by slightly modifying the directions of Haworth.[2] Three grams of the dried fructosan was added to 35 ml. of pyridine and shaken at 30° until dissolved. Thirty ml. of acetic anhydride was added slowly with stirring. The white precipitate which formed was easily redissolved. The mixture was allowed to stand at room temperature for eighteen hours and the straw colored mixture then poured into 500 ml. of ice water. The white precipitate was filtered, washed, dried over phosphorus pentoxide, and again acetylated. The acetyl content according to the method of Armstrong and Arup[3] was found to be 45.4%.

Anal. Calcd. for $(C_6H_7O_5 \cdot 3CH_3CO)_x$: C, 50.0; H, 5.5; acetyl, 44.8. Found: C, 49.8; H, 5.61; acetyl, 45.4.

Preparation of the Barium Salt.—The barium salt was prepared free from barium carbonate in the following manner. Pure fructosan was dissolved in a small amount of water and an excess of saturated barium hydroxide was added. The slightly turbid solution was filtered by suction, taking precaution against the passage of air through the filter. Alcohol was added to the now clear aqueous filtrate, and the snow-white granular precipitate was filtered immediately by suction. The precipitate was washed first with 50% ethanol, and finally with 95% ethanol. The barium salt was then allowed to air dry and was finally dried completely in a vacuum oven at 50°.

Two different samples prepared in a like manner contained 22.4 and 23.5% of barium. The barium salt of the fructosan is soluble in water.

Discussion

Although sufficient data are not on hand to suggest a possible structure for this compound, a few facts have been established.

The fructosan exists in considerable quantities in the stem of the *Yucca mohavensis*. It is very readily hydrolyzed, which suggests the possibility of a fructofuranose.

The compound appears to be similar to the graminin isolated from rye flour.

Although this fructosan was first obtained from the 70% alcohol extract, in later work it was isolated from the hot aqueous extraction of the ground and dried stem.

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[2] Haworth and Percival, *J. Chem. Soc.*, 2277 (1932).

[3] Armstrong and Arup, *ibid.*, **85**, 1043 (1904).

Benzylidene Aminomorpholine Compounds

BY LEROY DUGAN, JR.,[1] AND HELMUT M. HAENDLER

The structure of 4-aminomorpholine suggested its possible use as a reagent for organic qualitative analysis. However, the only one of its reactions which appeared satisfactory was the condensation with aromatic aldehydes to form, in general, colorless, crystalline compounds, which may be of some value for the identification of certain specific aldehydes. 4-(*p*-Aminophenyl)-morpholine undergoes similar reactions, and also reacts with *o*-hydroxyacetophenone, but the resulting compounds will probably be useful only in isolated instances.

4-Aminomorpholine was prepared by the method of Knorr and Brownsdon[2] and was kept as the hydrochloride, m. p. 164–165°. Attempts to use reducing agents other than zinc and acetic acid resulted in the formation of morpholinium chloride and ammonium chloride.

The benzylidene compounds were prepared by reaction of ether or alcohol solutions of the free amine, formed from the hydrochloride by treatment with sodium hydroxide, with ether solutions of the aldehydes.

4-(*p*-Aminophenyl)-morpholine was prepared by the general methods of Kremer, Meltsner and Greenstein,[3] and Lubs,[4] m. p. 130–131°, from the nitro compound, prepared according to Harradence and Lions.[5] The condensation products were prepared by refluxing equivalent quantities of the reactants in alcohol, followed by crystallization.

The quantitative data for the compounds formed by both of these substances are listed in Table I.

TABLE I

4-AMINOMORPHOLINE AND 4-(*p*-AMINOPHENYL)-MORPHOLINE COMPOUNDS

Carbonyl compound	Color	M. p., °C.	Nitrogen analyses, % Calcd.	Found
With 4-aminomorpholine				
o-Hydroxybenzaldehyde	Colorless	75– 76.5	13.60	13.90
m-Hydroxybenzaldehyde	Colorless	145–147.5	13.60	13.85
p-Hydroxybenzaldehyde	Tan	167–168	13.60	13.85
o-Nitrobenzaldehyde	Orange	99–101	17.89	17.84
m-Nitrobenzaldehyde	Yellow	114–114.5	17.89	17.85
Vanillin	Colorless	153–154.5	11.86	11.68
Piperonal	Colorless	76– 77	11.91	12.19
With 4-(*p*-aminophenyl)-morpholine				
Salicylaldehyde	Orange	161–162	9.94	9.95
Piperonal	Tan	167.5–169	9.04	9.02
Vanillin	Yellow	205–207	8.98	9.03
Furfural	Brown	208–209	10.91	10.49
o-Hydroxyacetophenone	Yellow	206–207	9.43	9.52

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[1] Present address: Chemical Warfare Service.

[2] Knorr and Brownsdon, *Ber.*, **35**, 4474 (1902).

[3] Kremer, Meltsner and Greenstein, THIS JOURNAL, **61**, 2552 (1939).

[4] Lubs, U. S. Patent 2,004,763, June 11, 1935.

[5] Harradence and Lions, *J. Proc. Roy. Soc.*, *N. S. Wales*, **70**, 406 (1937).