hydroxide solution which had been standardized against benzoic acid.[1] Each acid was carefully purified. Benzoic acid was recrystallized from alcohol and then sublimed. Anthranilic, citric, picric, salicylic and succinic acids were recrystallized from hot water. Phthalic acid was recrystallized from hot water and also reprecipitated from its sodium salt.

These solubility experiments with the acids were carried out by H. M. Taylor, Senior student in Chemistry.

A Color Reaction.—In working with certain samples of cymene, it was observed that they turned red rather quickly when they contained *p*-anisidine in solution. The amount of *p*-anisidine used was small, about one part in 500. A study of this phenomenon developed the fact that a perfectly pure cymene does not change color, at least within 2 hours, if exposed to a moderate light. Impure cymene will turn pale yellow, deep yellow, pale red or deep red according to the degree of impurity or to the time of action. A strong light, especially sunshine, facilitates the development of the color. Coloration also occurs in the dark. It may be added that solutions of *p*-anisidine in other solvents also develop a color.

CHAPEL HILL, N. C.

[CONTRIBUTIONS FROM BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

CRYSTALLINE CHLOROTETRACETYL FRUCTOSE AND RELATED DERIVATIVES.[2]

BY D. H. BRAUNS.

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The acetates of fructose have been obtained crystalline only by applying a principle which differs materially from the ordinary methods of acetylation. This principle consists in the action of acetylating agents on *finely powdered fructose at a low temperature* (*below* 0°).[3] The present communication deals with the use of these stable crystalline *acetates* as starting material for the preparation in the crystalline state of other derivatives of fructose.

Crystalline β-penta-acetyl or tetra-acetyl fructose in chloroform solution gives with phosphorus pentachloride and aluminum chloride a crystalline substance which has proved to be one of the isomeric forms (probably α-) of chloro-tetra-acetyl fructose. With phosphorus pentachloride alone it gives a good yield of another crystalline substance, supposed to be the β-isomer. It is to be regretted that the necessity of postponing further investigations has prevented the securing of definite proof of the

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[1] Bur. Standards *Bull.* 8, 643 (1912).

[2] The work described in this paper was done in the Carbohydrate Laboratory of the Bureau of Chemistry at the time when Dr. C. S. Hudson was Chemist in charge of that laboratory.

[3] "On a Crystallized *d*-Fructose-tetracetate," *Proc. Roy. Acad. Amsterdam*, 10, 563 (1907–1908).

possibility of preparing *two* isomeric chloro-tetra-acetyl derivatives of a sugar.

In continuation of the investigation of crystalline fructosides[1] crystalline β-ethyl fructoside and its tetra-acetate have been prepared and studied. While all attempts to obtain a crystalline α-form of methyl fructoside, either by enzymatic action or by fractional sublimation from Fischer's syrupy methyl fructoside, have been unsuccessful, it is noteworthy that the sublimation yields a crystalline β-methyl fructoside.

Experimental.

Crystalline β-penta-acetyl fructose was converted into chloro-tetra-acetyl fructose by the method which F. V. Arlt[2] used for preparing chloro-acetyl glucose.

Ten g. of β-penta-acetyl fructose was dissolved in 14 cc. of dry chloroform in an Erlenmeyer flask with glass stopper, cooled with ice-water, and there were added first 2.5 g. of aluminum chloride and then 6.5 g. of phosphorus pentachloride, the mixture being shaken at room temperature for an hour, occasionally warming the solution on the steam-bath. The reaction product was diluted with chloroform, quickly washed in a separatory funnel with small amounts of ice-water with addition of some sodium hydrogen carbonate, dried with calcium chloride and the chloroform quickly evaporated by means of a current of dry air passed over its surface in a large crystallizing dish. The residue was taken up with dry ether and the ether almost completely evaporated in a current of dry air. The chloro-tetra-acetyl fructose separated in long needles, which were filtered by suction and washed with small amounts of ether, or better with a mixture of petroleum ether and ether, as the crystals are very soluble in ether. The crystals may be recrystallized by evaporating the ether solution with a current of dry air. By placing a saturated ether solution in a stoppered bottle in the refrigerator and seeding the solution, large crystals separated overnight. Yield about 6 g., or about 60%. β-Fructose *tetra*-acetate has also proved in this case to be a valuable starting product as the same results are obtained as with the less available β-fructose *penta*-acetate.

To 60 g. of β-fructose tetra-acetate dissolved in 180 cc. of dry chloroform in a glass-stoppered Erlenmeyer flask and cooled with ice-water there were added first 15 g. of dry aluminum chloride and afterwards 38 g. of phosphorus pentachloride. The mixture was kept for half an hour at room temperature then shaken out with ice-water and treated as has been described for the first preparation. The yield was about 40 g. of chloro-tetra-acetyl fructose, or about 65%.

A few grams of chloro-tetra-acetyl fructose was also obtained by the

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[1] THIS JOURNAL, **38,** 1216 (1916).

[2] Arlt, *Monatsh.*, **22,** 144 (1901); see also Skraup and Kremann, *ibid.*, **22,** 375 (1901).

following method, used by E. Fischer for obtaining chloro-tetra-acetyl glucose. 7 g. of β-fructose penta-acetate dissolved in 8 cc. of acetic acid saturated with hydrogen chloride, was made up to a volume of 25 cc. with glacial acetic acid and, after standing for half an hour, the mixture was poured into cold chloroform. The chloro-tetra-acetyl fructose was obtained by working up the chloroform solution in the manner already described.

This chloro-tetra-acetyl fructose is not a stable substance. It decomposes in a day in an open dish, and keeps only a little longer in a desiccator. The best way to preserve it for a longer time is in a tightly closed bottle in benzol solution in the refrigerator. In this way it can be kept for a few weeks. Before using, it was always recrystallized. Combustion (with lead chromate) gave the following data.

Subs., 0.2950: CO_2, 0.4991; H_2O, 0.1395.

Calc. for $C_6H_7O(C_2H_3O_2)_4Cl$: C, 45.83; H, 5.22. Found: C, 46.14; H, 5.29.

The quantitative estimation of acetyl was combined with the quantitative estimation of chlorine by the following method.

0.6000 g. of substance was saponified by shaking with 135.5 cc. of 0.1 *N* sodium hydroxide solution for 5 hours at 0° and titrating with 0.1 *N* sulfuric acid and phenolphthalein. For neutralization 53.39 cc. of 0.1 *N* sulfuric acid was required, hence 82.11 cc. of 0.1 *N* sodium hydroxide had been neutralized. An excess of silver nitrate solution was added to the warm neutral solution and the precipitate filtered on a weighed Gooch filter. 0.2310 g. of silver chloride was obtained, equivalent to 9.52% of chlorine in the substance. The 0.2310 g. of silver chloride had been formed from an amount of hydrochloric acid corresponding to 16.11 cc. of 0.1 *N* hydrochloric acid, hence in 0.6000 g. substance 82.11 — 16.11 = 66.0 cc. of 0.1 *N* solution or 66.0% of acetic acid is found. In a similar experiment of 5.5 hours duration, 9.52% of chlorine and 66.1% of acetic acid were found.

The theoretical quantities for chloro-tetra-acetyl fructose are 9.67% of chlorine and 65.5% of acetic acid.

In the filtrate from the silver chloride precipitate no chlorine could be detected by the copper oxide test.

The molecular weight was determined by the lowering of the freezing-point of benzene.

In 3 experiments 1.962, 4.037 and 6.548 g. of chloro-tetra-acetyl fructose per 100 g. of benzene gave the respective depressions 0.287°, 0.562° and 0.903°, from which the values 342, 360 and 362 are obtained for the molecular weight in comparison with the theoretical value 367.

The specific rotation was measured in chloroform solution.

2.0239 g. of twice-crystallized chloro-tetra-acetyl fructose was made up to 25 cc. with U. S. P. chloroform. The rotation, measured for a one-dcm. tube at 20° with sodium light, was 12.48 circular degrees to the left; hence $[\alpha]_D^{20} = -156.6$.

Beautiful large crystals were obtained once by keeping a saturated ether solution in the ice-box overnight. The rotation produced by 0.4351 g. in a similar solution was 2.80 circular degrees to the left, hence $[\alpha]_D^{20} = -160.9$.

Other values obtained from recrystallized preparations were always between the above specific rotations, therefore, the value $[\alpha]_D^{20} = -160.9$ is taken as the specific rotation of pure chloro-tetra-acetyl fructose.

The substance melts at 83°, is colorless and odorless, has a bitter taste and is easily soluble in the usual solvents excepting water and petroleum ether. It is decomposed by the air, even in a desiccator. When exposed to the air a pure preparation still gave the next morning after washing with an ether-chloroform mixture a weak test for chlorine, melted at 124° (unsharp) and had a specific rotation of —113°. After recrystallization from ether pure β-tetra-acetyl fructose was separated, identified by the melting-point and specific rotation. A nearly quantitative conversion into β-tetra-acetyl fructose was obtained by shaking 10 g. of *chloro-tetra*-acetyl fructose in ether solution with 10 g. of silver oxide and 5 cc. of water for a day. Eight g. of pure β-tetra-acetyl fructose was separated and the product was identified by melting-point and specific rotation. This reaction identified the substance as a fructose derivative. The following experiment seems to indicate that this chloro-tetra-acetyl fructose is an α-derivative.

Two g. of chloro-tetra-acetyl fructose was dissolved in 60 cc. of methyl alcohol and 4 g. of silver carbonate and 2 g. of silver oxide were added. The mixture was shaken for 1.5 days, filtered with the addition of some decolorizing carbon and gave on evaporation a colorless syrup which did not crystallize. Seeding with β-methyl-tetra-acetyl fructose was not effective in starting the crystallization. The syrup was dried in a vacuum desiccator in order to determine the specific rotation.

0.5423 g. of this dry syrup was dissolved in 25 cc. of chloroform and showed in a one-dcm. tube a rotation of 0.09 circular degrees to the left, hence $[\alpha]_D^{20} = -4.1$. Crystalline methyl-tetra-acetyl fructose has a specific rotation of —124.6.

E. O. v. Lippmann, in his book "Die Chemie der Zuckerarten," Vol. 1, p. 870, states that Kolli and Vachovié mention chloroacetyl fructose;[1] it is said to be entirely analogous to chloro-aceto glucose. The translation of the original reference,[2] by the courtesy of Dr. E. Yanovsky, disclosed that the note made by v. Lippmann comprises all that could be found in the original literature regarding the preparation and properties of this compound. It is very probable that Kolli and Vachovié, in the year 1880, in which Colley's "aceto chlor*glukose*" was known only as a syrup, have simply applied the method of Colley to fructose and have considered the syrupy reaction product as chloro-aceto fructose.

In one of the experiments for preparing chloro-tetra-acetyl fructose a small amount of large crystals separated on evaporation of the ether solution, which had an appearance and melting-point differing from those

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[1] *Chem. Zentr.*, 80, 613 must be *Chem. Zentr.*, 11, 613 (1880).

[2] *Proc. Russ. Phys. Chem. Soc.*, 12, 4–6 (1880); see also *Ber.*, 13, 2389 (1880).

of the chloro-tetra-acetyl fructose. Several experiments under different conditions were made in order to obtain this substance again, but were fruitless. At last it was found that if aluminum chloride is omitted the reaction produces a good yield of the new substance which is entirely different from the chloro-tetra-acetyl fructose which has been described. The explanation why this new compound has been formed the first time is obvious. Phosphorus pentachloride had been first added and after a short time (in which the new compound was formed) aluminum chloride.

Preparation of the Supposed β-Chloro-tetra-acetyl Fructose.

Sixty g. of β-tetra-acetyl fructose was dissolved in 180 cc. of dry chloroform in a glass-stoppered Erlenmeyer flask and cooled with ice-water. Forty g. of phosphorus pentachloride was added and the mixture was shaken occasionally, lifting the stopper now and then to release the excess pressure. After 15 minutes the phosphorus pentachloride had nearly disappeared, and the reaction product was shaken in a separatory funnel with ice-water with addition of small pieces of ice and afterwards of ice-water to which sodium hydrogen carbonate had been added. The chloroform solution was dried with calcium chloride and filtered, the chloroform evaporated and the residue mixed with absolute alcohol. Crystals separated directly and were filtered by suction and recrystallized from boiling absolute alcohol. In this way beautiful large crystals were obtained which resembled fructose tetra-acetate. The yield was about 35 g. or nearly 60% of the tetra-acetate used. The new compound is a stable substance, and analysis gives figures which agree with a chloro-tetra-acetyl hexose. Combustion (with lead chromate) gave the following data.

Subs., 0.3123: CO_2, 0.5275; H_2O, 0.1417.

Calc. for $C_6H_7O(C_2H_3O_2)_4Cl$: C, 45.83; H, 5.22. Found: C, 46.07; H, 5.08.

The quantitative estimation of acetyl showed that only very small amounts of chlorine are split off by alkali at 0°.

For this estimation 0.250 g. was saponified by shaking with 75 cc. of 0.1 *N* sodium hydroxide solution for 3.5 hours and titrated with 0.1 *N* sulfuric acid and phenolphthalein. The amount of acetic acid produced was found to be 68.8%. In a similar experiment of 4.5 hours duration the value 70.2% was found. The theoretical quantity of acetic acid in chloro-tetra-acetyl hexose is 65.5%.

The neutral liquid resulting from the saponification gave a slight opalescence with silver nitrate solution and the same liquid evaporated to a syrup gave a strong chlorine test with copper oxide.

A chlorine determination in the original substance gave the following data.

Subs., 0.2083: AgCl (Carius), 0.0806.

Calc. for $C_6H_7O(C_2H_3O_2)_4Cl$: Cl, 9.67. Found: 9.57.

The molecular weight was determined by the lowering of the freezing-point of benzene.

In 2 experiments 1.3115 g. and 2.6036 g. of chloro-tetra-acetyl hexose per 100 g. of benzene gave the respective depressions 0.190° and 0.360°, from which the values 345 and 361 are calculated for the molecular weight in comparison with the theoretical value 367. The specific rotation was measured in chloroform solution. 0.9900 g. of twice-recrystallized substance was made up to 25 cc. with chloroform and the solution gave a rotation in a one-dcm. tube at 20° with sodium light of 1.79 circular degrees to the right; hence $[\alpha]_D^{20} = +45.2$. After a third recrystallization from absolute alcohol 0.4274 g. of substance produced a rotation of 0.775 circular degrees to the right, hence $[\alpha]_D^{20} = +45.3$, which is taken as the specific rotation of the pure substance.

The melting-point of the pure substance is 108°. It is colorless, odorless, has no taste (or is very slightly bitter) and is less soluble in the usual solvents than the chloro-tetra-acetyl fructose described before. Though it is very probable that this new compound represents an isomeric chloro-tetra-acetyl fructose, the one of specific rotation—160.9 being the α- and the other of specific rotation +45.3 the β-form, only a few experiments for confirming this view could be performed on account of lack of time. The following experiment shows, however, that the new compound is very probably a fructose derivative and that no rearrangement of the fructose molecule has taken place. Seventy g. of crystalline barium hydroxide ($Ba(OH)_2 + 8H_2O$) was dissolved in one liter of water on the steam-bath and filtered into a bottle with a glass stopper. The solution was cooled with ice to about 5° and 20 g. of the new chloro derivative in powder form was added. After 1.5 hours shaking the substance had dissolved. The liquid was kept 0.5 hour longer in ice, was then saturated with carbon dioxide, filtered, shaken with silver carbonate and again filtered. The amount of sulfuric acid necessary to precipitate the dissolved barium acetate was calculated to be 218.1 cc. of N solution, since this amount is equivalent to the acetic acid that is obtained from 4 acetyl groups in chloro-tetra-acetyl fructose. After filtration and concentration *in vacuo* the solution could be filtered clear, and by carefully adding 0.1 N sulfuric acid to the filtrate, it was brought to the stage where it gave only a slight precipitate with sulfuric acid. On evaporating this solution in a vacuum desiccator a yellowish syrup was obtained which became gradually light brown. It was not possible to obtain crystals from this syrup. It reduced Fehling's solution readily, contained chlorine and gave a strong *Seliwanoff reaction* (a red color with hydrochloric acid and resorcin) *indicating the presence of a ketose.*

0.2394 g. of syrup was dissolved in 25 cc. of water and polarized in a one-dcm. tube, but no rotation could be detected. One g. of syrup gave with phenylhydrazine and sodium acetate about one g. of osazone. It was re-crystallized from methyl alcohol, the crystals giving a melting-point of 173°. The quantity was too small to recrystallize again.

The experiments indicate that the chlorine is much more strongly attached *in this compound than in the chloro-tetra-acetyl fructose which was first described.*[1]

Ethylation of β-Fructose Tetra-acetate.

Twenty g. of powdered fructose tetra-acetate, 75 g. of freshly prepared silver oxide and 128 g. of ethyl iodide were heated to 50° in a 300-cc. Erlenmeyer flask fitted with a reflux condenser. When the mixture heated up to boiling by the heat of the reaction it was cooled in cold water and the heating to 50° and cooling were repeated until heating by the reaction no longer took place. It was boiled afterwards for 2 hours under a reflux condenser on the water-bath, the ethyl iodide was distilled off and the residue extracted with ether. By evaporation of the ether, ethyl tetra-acetyl fructose was obtained in crystalline form. Yield, 16 g. of a nearly pure product. It was recrystallized from petroleum ether.

The specific rotation was measured in a chloroform solution. 0.5976 g. of once-recrystallized ethyl tetra-acetyl fructose was made up to 25 cc. with chloroform and the solution was found to read in a one-dcm. tube at 20° with sodium light 3.00 circular degrees to the left; hence $[\alpha]_D^{20} = -125.5$. After a second recrystallization from petroleum ether, 0.6016 g. of substance showed a rotation of 3.0 circular degrees to the left in a similar solution; hence $[\alpha]_D^{20} = -125.6$; and after a third recrystallization from petroleum ether 1.9968 g. of substance caused a rotation of 10.19 circular degrees to the left; hence $[\alpha]_D^{20} = -127.6$, which is taken as the specific rotation of pure β-ethyl tetra-acetyl fructose.

The substance melts at 83°. Combustion, ethoxyl and acetyl determinations gave the following results.

Subs., 0,2705, 0.2253: CO_2, 0.5082, 0.4225; H_2O, 0.1548, 0.1280.

Calc. for $C_6H_7O.OC_2H_5(C_2H_3O_2)_4$: C, 51.04; H, 6.43. Found: C, 51.24, 51.13; H, 6.40, 6.35.

An ethoxyl determination with 0.2489 g. of substance gave 0.1541 g. of silver iodide corresponding to 11.88% of OC_2H_5 group, whereas the theoretical amount is 11.97%. For the quantitative estimation of the acetyl group the substance was saponified by shaking 0.3000 g. with 75 cc. of 0.1 *N* sodium hydroxide solution at 0° for one hour and titrating with 0.1 *N* sulfuric acid, using phenolphthalein as an indicator. The amount of acetic acid was found to be 64.05%. In a similar experiment of 2 hours' duration, the value 63.99% was found, and in a third lasting 2.5 hours, the same value, 63.99% was obtained. The theoretical quantity for a tetra-acetyl ethyl fructose is 63.83%. Evidently the saponification is complete after one hour.

Preparation of β-Ethyl Fructoside.

The method of saponification is the same as that described for the preparation of β-methyl fructoside.[2] The barium hydroxide used was not pure and the ethyl fructoside obtained was not easily purified, as it was hard to obtain ash-free. It was recrystallized first from boiling alcohol and afterwards from water. In this way an ash-free prepara-

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[1] If we consider the theory of secondary valence it is probable that in the β-compound more chlorine valence is used for binding it to the second carbon atom than in the α-compound.

[2] THIS JOURNAL, 38, 1220 (1916).

tion was obtained. The specific rotation was measured in water solution.

1.0302 g. of β-ethyl fructoside, recrystallized from boiling alcohol, was made up to 25 cc. with water. A reading of 6.36 circular degrees to the left was obtained in a one-dcm. tube at 20° with sodium light; hence $[\alpha]_D^{20} = -154.3$. After a further crystallization from water, 0.7225 g. of substance made up in the same manner produced a rotation of 4.50 circular degrees to the left; hence $[\alpha]_D^{20} = -155.7$; and after a further recrystallization from water 0.4082 g. of substance made up in the same manner gave a rotation of 2.545 circular degrees to the left; hence $[\alpha]_D^{20} = -155.3$. This figure was taken as the specific rotation of pure β-ethyl fructoside.

β-Ethyl fructoside melts at 151°. The crystals are colorless, odorless and have a slightly bitter taste or no taste at all. The fructoside is easily soluble in water and hot alcohol. Its aqueous solution is not reduced by boiling with Fehling's solution. After hydrolyzing by boiling the aqueous solution with a mineral acid and neutralizing the cooled solution, it readily reduces Fehling's solution on boiling. This reaction proves, in connection with the analysis recorded below, that the prepared substance is an ethyl fructoside.

Subs., 0.2060: CO_2, 0.3487; H_2O, 0.1413.

Calc. for $C_6H_{11}O_5(OC_2H_5)$: C, 46.12; H, 7.75. Found: C, 46.16; H, 7.67.

An ethoxyl determination with 0.1669 g. of substance gave 0.1821 g. of AgI, corresponding to 20.93 of OC_2H_5. Calc., 21.16.

Preparation of β-Tetra-acetyl Fructose from α-Penta-acetyl Fructose.

α-Penta-acetyl fructose was prepared from β-tetra-acetyl fructose by the action of zinc chloride and acetic anhydride,[1] but the α-penta-acetyl fructose could not be converted into the β-penta-acetyl fructose. Recently, however, a method has been found for converting α-penta-acetyl fructose into β-tetra-acetyl fructose, and, therefore, with β-tetra-acetyl fructose as an intermediate product α-penta-acetyl fructose can be converted into the β-form. 5 g. of α-penta-acetyl fructose was dissolved in 500 cc. of hot water and the solution was heated for one hour in an autoclave at 106°. The solution was neutralized and shaken out with chloroform, the latter yielding on evaporation a crystalline substance which was recrystallized from ether. In this way 0.28 g. of β-tetra-acetyl fructose was obtained which was identified by melting-point and specific rotation.

Efforts to Prepare α-Methyl Fructoside Using a Biochemical Method.

Experiments were made to prepare the α-form of methyl fructoside biochemically. Bourquelot, Herissey, Bridel and Aubry[2] have prepared α-methyl glucoside and α-methyl galactoside in a pure crystalline condition in this way by means of bottom yeast (brewery yeast). As nothing was found in the literature regarding the application of this method to fruc-

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[1] THIS JOURNAL, **37**, 2738 (1915).

[2] *Compt. rend.*, **156**, 168, 491 (1913); *J. pharm. chim.*, [7] **9**, 225, and **10**, 202 (1914).

tose, according to the prescription of Aubry,[1] a macerate of bottom yeast was first prepared by washing and drying brewery yeast and macerating this dry yeast for 24 hours with 10 times the amount of water. Using this material, the results of the above investigation yielding α-methyl glucoside and α-methyl galactoside were confirmed.

To apply the same procedure to fructose 360 g. of methyl alcohol and 100 g. of fructose dissolved in 800 cc. of water were mixed in a bottle, 600 cc. of macerate of yeast was added and the volume made up to 2 liters. The rotation was determined at the beginning of the experiment and after one and three months.

The initial rotation in one-dcm. tube was found to be —4.2; after one month the rotation was —4.1, and after 3 months —4.1. As the change of rotation in within the limits of error it was concluded that bottom yeast does not act under the conditions of this experiment.

Other Efforts to Prepare α-Methyl Fructoside.

In order to obtain an equilibrium mixture of α- and β-methyl fructosides, β-methyl fructoside was treated with a 1% hydrochloric acid solution in methyl alcohol by dissolving in a 50 cc. volumetric flask one g. of β-methyl fructoside in 20 cc. of methyl alcohol, adding 4.2 cc. of 3.32 *N* hydrochloric acid in methyl alcohol and filling up to 50 cc. with methyl alcohol. The rotation was determined in circular degrees with a one-dcm. tube. The following negative rotations were obtained.

After 5 minutes —3.10, 0.5 hour; —3.03, 1 hour; —2.90, 18 hours; —1.60, 2 days; —1.32, 4 days; —1.10, 6 days; —1.02, 8 days; —1.02. At the end of the reaction the liquid had a yellowish color.

In a similar experiment in which 3.6% hydrochloric acid was used the time in which equilibrium was reached was diminished to about 2.5 days, and the resulting solution was colored dark brown. The solutions were worked up separately according to the direction of Fischer,[2] but no crystals could be separated from the resulting syrups.

By subliming Fischer's sirupy mixture of methyl fructosides in a high vacuum (cathode rays) a sublimate could be obtained in a crystalline state. By its chemical behavior and by its melting-point (120°) and specific rotation (—172.0°) it was identified as β-methyl fructoside. No other crystalline substance could be separated by this method.

WASHINGTON, D. C.

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[1] Aubry, *ibid.*, [7] 10, 202 (1914).

[2] *Ber.*, 28, 1160 (1895).