The aqueous residue from the steam distillation was evaporated to dryness, after filtration, the product dried overnight at 100° and then sublimed, m. p. 184.5° (corr.). A mixed melting point with pure succinic acid showed no depression.

Anthracene–Maleic Anhydride.—This product was prepared according to the method of Diels, Alder and Beckmann.[5] The decomposition was carried out as described above except that no appreciable distillation took place. The sublimate which adhered to the upper portion of the decomposition apparatus was resublimed, m. p. 58–59°. Microscopic examination confirmed the existence of maleic anhydride.

The residue from the decomposition was digested with an excess of 10% sodium hydroxide solution and the insoluble portion washed twice by decantation. This material, after drying, was sublimed, m. p. 215.1° (corr.). A mixed melting point with anthracene showed no depression.

Anal. Calcd. for $C_{14}H_{10}$: C, 94.40; H, 5.60. Found: C, 94.20, 94.33; H, 5.27, 5.55.

α-Terpinene.—This terpene was synthesized from linaloöl by the method of Bertram and Walbaum.[6]

α-Terpinene–Maleic Anhydride.[7]—To 0.3 mole of terpene there was added 0.2 mole of maleic anhydride. The mixture was refluxed for three hours and then distilled with steam to remove the unused terpinene and extract some of the unused maleic anhydride. The residual oil was taken up in ether, dried and distilled twice under reduced pressure; yield, 16 g. of product which boiled at 152–154° at 1 mm.; $n^{67}D$ 1.4913; d^{67}_4 1.100; MR calcd. for $C_{14}H_{18}O_3^{(-)}$, 61.47; found, 61.64. *Anal.* Calcd. for $C_{14}H_{18}O_3$: C, 71.77; H, 7.75. Found: C, 71.37; H, 7.56. The decomposition was carried out as described for the phellandrene–maleic anhydride. The products were also isolated by the same procedure. The oil was found to have the following characteristics: b. p. 173–175° (corr.); $n^{20}D$ 1.4820; d^{20}_4 0.852; MR calcd. for $C_{10}H_{14}^{(-)}$, 44.8; found, 44.8. On oxidation with chromic acid the only acid obtained was terephthalic, which was identified through its methyl ester in the usual way. The acidic portion of the decomposition products was proved to be succinic anhydride by the same procedure previously described. The analytical data were: subs., 0.0703 g. NaOH (0.518 *N*), 23.25 cc.; n. e., calcd. for $C_4H_6O_2$, 59.0; found, 58.5; m. p. 184° (corr.). A mixed melting point with succinic acid of known purity showed no depression.

(5) Diels, Alder and Beckmann, *Ann.*, **486**, 191 (1931).

(6) Bertram and Walbaum, *J. prakt. Chem.*, **45**, 601 (1892).

(7) Koch, Dissertation, Christian Albrecht University, Kiel, 1932.

HERCULES EXPERIMENT STATION
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New Method of Preparation of Methylmaltoside Heptaacetate with Orthoester Structure

BY EUGENE PACSU AND FRANK V. RICH

In our attempts to prepare the "γ"-methylmaltoside heptaacetate of Freudenberg,[1] we have found that his method of preparation of the necessary chloroacetylmaltose[1,2] with ortho ester structure is very laborious, and frequently gives negative results. We have succeeded in discovering a new method by which it is possible to obtain the former compound in one step, and in fairly good yield. Our procedure consists in the treatment of β-maltose octaacetate in chloroform solution with aluminum chloride at room temperature. Freudenberg's chloroacetylmaltose so formed is not isolated in crystalline form. Its methyl alcoholic solution, in presence of pyridine, gives rise to the "γ"-methylmaltoside heptaacetate which crystallizes from the solution in a very pure state.

The formation of Freudenberg's chloroacetylmaltose under the above experimental conditions is remarkable. Kunz and Hudson[3] have shown that both octaacetyl lactose and cellobiose suffer double epimerization in the presence of aluminum chloride in boiling chloroform solution, the two sugars being converted into two new disaccharides, namely, neolactose and celtrobiose. We have attempted to apply our method to both lactose and cellobiose octaacetates, but, in contrast to the maltose acetate, no appreciable reaction could be detected; the starting materials were recovered virtually unchanged. Haworth and co-workers[4] have found that maltose, cellobiose and lactose differ only in that maltose is a 4-α-glucosidoglucose, while cellobiose and lactose are, respectively, 4-β-glucosido-glucose, and 4-β-galactosido-glucose. Such a conclusion, while in best accord with many properties of these sugars and their derivatives, fails to explain the sharp distinction observed between the behavior of these sugar acetates toward aluminum chloride at low temperature. At the present time the reason for this difference remains obscure.

Experimental

Preparation of "γ"-Methylmaltoside Heptaacetate.—Thirteen grams of β-octaacetylmaltose was dissolved in ice-cold absolute chloroform; to the solution 6 g. of aluminum chloride (Merck) was added, and the mixture was kept at room temperature for an hour, with occasional shaking. The resulting almost colorless, thick mass was poured into a separatory funnel containing cracked ice. After addition of more chloroform, the colorless chloro-

(1) Freudenberg, v. Hochstetter and Engels, *Ber.*, **58**, 666 (1925); Freudenberg and Scholz, *ibid.*, **63**, 1969 (1930).

(2) Freudenberg and Ivers, *ibid.*, **55**, 929 (1922).

(3) Kunz and Hudson, THIS JOURNAL, **48**, 1978, 2435 (1926); Hudson, *ibid.*, **48**, 2002 (1926).

(4) Haworth and Peat, *J. Chem. Soc.*, 3094 (1926); Haworth and Long, *ibid.*, 544 (1927); Haworth, Long and Plant, *ibid.*, 2809 (1927).

form layer was shaken twice with ice water, then dried with calcium chloride, filtered and evaporated under reduced pressure to a colorless sirup. The latter was dissolved in absolute ether, and the solution, after filtration, was evaporated *in vacuo* to a half-solid foamy mass. To the 25 cc. of absolute methyl alcohol solution of the residue, 3 cc. of absolute pyridine was added. Crystallization of the "γ"-methylmaltoside heptaacetate soon started in the solution. After standing for an hour in the ice-box, the solution deposited 4 g. of long needles. The substance did not reduce Fehling's solution, and melted at 164°. It showed $[\alpha]^{20}_{D}$ 98.8°, $[\alpha]^{20}_{C}$ 79.0°, $[\alpha]^{20}_{Hg_1}$ 116.5° (0.3967 g. of subst., 10 cc. of chloroform solution, 2-dm. semi-micro tube; rotations, 7.84, 6.27, 9.24° to the right, respectively). Recrystallization from methyl alcohol did not change the specific rotation. Freudenberg gives m. p. 163–164°, and $[\alpha]_{578}$ 101.6° in acetylene tetrachloride for pure "γ"-heptaacetylmethylmaltoside. The analyses and the behavior of the substance toward alkali and very dilute acid were in accord with Freudenberg's data.

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Viscosity of Deuterium[1]

BY I. AMDUR

In conjunction with measurements on the rate of recombination of atomic deuterium it was necessary to evaluate the number of moles of deuterium circulated each second through the apparatus which consisted, essentially, of a reservoir bulb communicating through a capillary leak to a Wood discharge tube and a circulating pump which served to pump atomic–molecular deuterium mixtures along a recombination tube. The determination of the rate of flow of deuterium gave, in addition, data from which the viscosity of deuterium relative to hydrogen could be calculated.

About three liters of deuterium at 700 mm. and 30° prepared by decomposing 99.9% D_2O with sodium as described by Lewis and Hanson[2] were used as a reservoir in the circulating system employing a Hypervac mechanical pump whose construction made it possible to render the higher pressure (exit) side gas tight and thus return the deuterium, through a liquid air trap, to the reservoir bulb. A capillary flowmeter using Apiezon "B" oil as a manometric fluid was thermostated with the reservoir bulb at 30° and served to check the constancy of flow of deuterium to the discharge tube.

The rate of flow of deuterium, dn/dt, was determined after all recombination rate measurements had been taken. Although the final measurements were completed in about two weeks, the deuterium had been circulated through the apparatus and Hypervac pump intermittently during a six-month period. All precautions were taken to minimize exchange as, for example, prolonged pumping to remove water from the phosphoric acid, which poisoned the recombination tube walls, as well as passage of discharges through the apparatus to heat the walls and further dry the phosphoric acid. Dr. Edward S. Gilfillan, of this Laboratory, very generously analyzed the gas at the conclusion of experiments and found a molecular weight of 3.80. The method employed was the comparison of the deuterium with ordinary hydrogen in a gas density balance. The gas, therefore, contained 90% deuterium and 10% hydrogen on a density basis or 81% D_2, 18% DH and 1% H_2 assuming the usual equilibrium ratio for the molecular species.

Since the rapid pumping system maintained a pressure on the high vacuum side of the capillary leak leading to the Wood tube which was negligible (1 mm. or less) in comparison with that on the high pressure side (700 mm.), the rate of change of pressure in the reservoir bulb when the gas was sent to storage bulbs instead of being returned to the reservoir is

$$dP/dt = kP^2 \quad (1)$$

where k is evaluated from the integrated equation

$$(1/P_2 - 1/P_1) = -k(t_2 - t_1) \quad (2)$$

(1) Presented at a Meeting of the Harvard–Technology Chemical Club, Massachusetts Institute of Technology, January 15, 1935. In the Bulletin of the American Physical Society, February 5, 1935, there is an abstract of a paper, "The Viscosity of Deuterium" to be presented by H. C. Torrey at the New York meeting of the American Physical Society and the Optical Society of America on February 23, 1935. The results contained therein are in accord with those of the present author.

(2) Lewis and Hanson, THIS JOURNAL, 56, 1687 (1934).