The "relative viscosities" reported in the table are the relative times of flow from a 5-cc. pipet.

Stability of Cyclohexene Oxide to Alkali.—Ten cc. of cyclohexene oxide was refluxed for six hours with 20 cc. of 10% sodium hydroxide solution, separated, dried and distilled. The cyclohexene oxide was unchanged, b. p. 129° (740 mm.).

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Summary

1. Reduction of 2-chlorocyclohexanone with the cyclohexyl, isopropyl, and *t*-butyl Grignard reagents gives predominantly a new isomer of 2-chlorocyclohexanol, which forms cyclohexanone instead of cyclohexene oxide on treatment with alkali.

2. The isomeric chlorohydrins of cyclohexene differ very slightly in their physical properties, but more than 300-fold in their rate of reaction with alkali.

3. Addition of hypochlorous acid to cyclohexene, or hydrogen chloride to cyclohexene oxide, yields only the oxide-forming chlorohydrin.

4. Indirect evidence is discussed that the oxide-forming chlorohydrin has the *trans* configuration.

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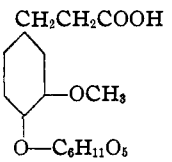
NOTE

Glucosidodihydroferulic Acid[1]

BY NELSON K. RICHTMYER AND RAYMOND M. HANN

A recent study has shown that phenyl glucosides may be cleaved to a considerable extent by hydrogenation in the presence of platinum catalysts.[2] The reduction of an ethylenic linkage in the side chain of a substituted phenyl glucoside, however, may be effected quantitatively with the aid of palladium and hydrogen. Thus, tetraacetylglucosidoferulic acid[3] was converted to the dihydro compound, which in turn was smoothly deacetylated by sodium methylate to produce glucosidodihydroferulic acid



Experimental

Tetraacetyl-β-*d*-glucosidodihydroferulic Acid (β-(3-Methoxy-4-tetraacetyl-β-*d*-glucosidophenyl)-propionic Acid).—A solution of 8.5 g. of tetraacetylglucosidoferulic acid in 140 cc. of glacial acetic acid was shaken with 1.7 g. of palladium black in an atmosphere of hydrogen for three hours; 361 cc. of gas, equivalent to one mole, was absorbed. The solution was filtered from the catalyst, and the solvent allowed to evaporate at room temperature in a vacuum desiccator over potassium hydroxide. The residue crystallized spontaneously and represented the theoretical yield of reduced acid.

Tetraacetyl-β-*d*-glucosidodihydroferulic acid crystallizes from solution in twenty parts of 50% methyl alcohol in brilliant colorless plates. It melts to a clear colorless oil at 155° (corr.) and shows an $[\alpha]_D^{20}$ value of −24.9° (0.2854 g. in 10 cc. of chloroform in a 1-dm. tube rotated 0.71° to the left). Upon recrystallization the $[\alpha]_D^{20}$ value was −25.2° (0.2060 g. in 10 cc. of chloroform in a 1-dm. tube rotated 0.52° to the left), and the average value of −25.0° is accepted for the compound.

Anal. Calcd. for $C_{24}H_{30}O_{13}$: C, 54.73; H, 5.75; OCH_3, 5.90. Found: C, 54.77; H, 5.88; OCH_3, 6.21.

β-*d*-Glucosidodihydroferulic Acid (β-(3-Methoxy-4-β-*d*-glucosidophenyl)-propionic Acid).—Forty and four-tenths cubic centimeters of 0.94 *N* sodium methylate solution was added slowly to a solution of 5.0 g. of the acetylated acid in 100 cc. of dry chloroform at −3°. After one-half hour an equivalent quantity of 5 *N* sulfuric acid was added and the solution was concentrated *in vacuo* to dryness. The glucosido acid was separated from the sodium sulfate by repeated extractions with hot 95% alcohol, and crystallized readily in rosets of short needles upon concentrating and cooling. The crude yield was 3.3 g. (quantitative). It was recrystallized from 3 parts of 95% alcohol.

β-*d*-Glucosidodihydroferulic acid is readily soluble in water and in this medium shows an $[\alpha]_D^{20}$ value of −56.9° (0.2620 g. in 10 cc. in a 1-dm. tube rotated 1.49° to the left). It melts at 179–180° (corr.) to a clear colorless oil.

Anal. Calcd. for $C_{16}H_{22}O_9$: C, 53.60; H, 6.19. Found: C, 53.59; H, 6.26.

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(2) Richtmyer, THIS JOURNAL, **56**, 1633 (1934).

(3) Hann, *ibid.*, **52**, 5049 (1930); **56**, 1631 (1934).