ture.[10] The mixture was then thoroughly blended by further grinding together, placed in a Perkin-Elmer potassium bromide evacuable die, and pressed under approximately 25,000 p.s.i. (gauge) for 3 min. Spectral measurements were made using the disk holder described by Waggoner[11] with a similarly prepared 400 mg. potassium bromide disk for a blank.

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(10) The final compositions of the disks were: 0.05 mg. of *p*-bromoacetophenone, 0.3 mg. of nitrosobenzene, 0.2 mg. of 3-phenacylideneoxindole, each in 400 mg. of potassium bromide.

(11) W. H. Waggoner, *Chemist-Analyst*, **48**, 80 (1959).

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## Cyclic Ether Formation by Bis[(1-hydroxyethyl)cyclopentadienyl]iron

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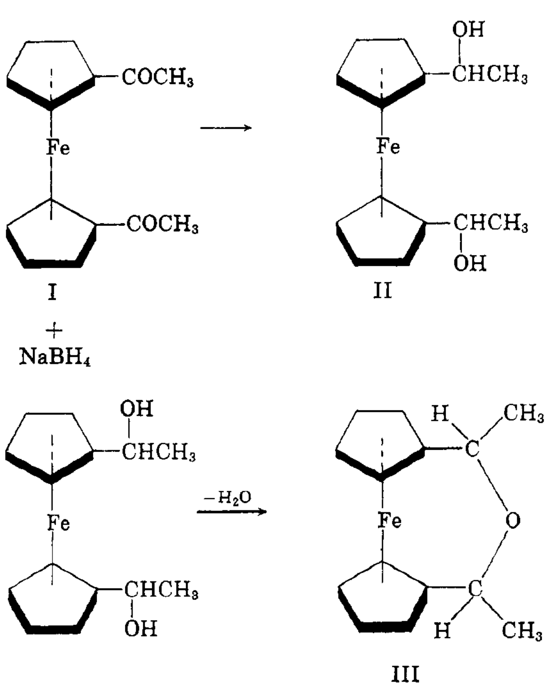
The preparation of divinylferrocene was attempted in this laboratory by the use of diacetylferrocene (I) as a starting material. This was reduced to form bis [(1-hydroxyethyl) cyclopentadienyl]iron (II). This compound was previously reported as melting at 69–71°.[1] Infrared data and other evidence indicate that the compound produced by reduction of diacetylferrocene with sodium borohydride in this laboratory is in fact bis[(1-hydroxyethyl)cyclopentadienyl]iron II and has a melting point of 105–106°.

The attempted dehydration of this dialcohol (II) produced a cyclic ether (III) between the side chains of the two cyclopentadiene rings of ferrocene rather than the desired divinyl ferrocene.

The infrared spectrum of II shows a strong hydroxy bond at 3.10 μ. The carbonyl bond is absent in the spectrum. Additional significant bonds appears at 7.35, 7.60, 9.10, and 12.41 μ. The infrared spectrum of the cyclic ether III shows a disappearance of the hydroxyl bonds. An ether bond at 8.8 μ can be cited as evidence for a cyclic ether. Other bonds appear at 7.68, 9.36, 9.88, 11.95, and 12.35 μ.



EXPERIMENTAL

Diacetylferrocene was prepared by following the procedure of Woodward (2).

*Preparation of bis[(1-hydroxyethyl)cyclopentadienyl]iron.* In a liter one-neck flask, fitted with a reflux condenser, was placed 27.0 g. (0.0985 mole) of diacetyl ferrocene, 7.56 g. (0.2 mole) of sodium borohydride, and 200 ml. of isopropyl alcohol. The mixture was refluxed for 5 hr. on a steam bath.

The solution was then evaporated to dryness by means of an air stream and water was added to the residue to hydrolyze the salt formed in the reaction. The product was then extracted with ether. The ether was removed and a yellow crystalline solid was obtained, which was recrystallized from methanol The yield was 14.4 g. (53.4%), m.p. 105–106°.

*Anal.* Calcd. for $C_{14}H_{18}O_2$ Fe: C, 61.31; H, 6.57. Found: C, 61.36; H, 6.72.

*Preparation of 1,1′-diethyl-α,α′-epoxybiscyclopentadienyliron (II)(III).* In a 1-l. flask, fitted with a reflux condenser, was placed 27.0 g. (0.0985 mole) of diacetylferrocene, 7.56 g. (0.2 mole) of sodium borohydride, and 200 ml. of isopropyl alcohol. The mixture was refluxed for 5 hr. on a steam bath.

The solution was then evaporated to dryness by means of an air stream and 100 ml. of water containing 5 ml. of glacial acetic acid was added to the residue to hydrolyze the salt formed in the reaction. The product was extracted with ether. The ether was evaporated and a yellow crystalline solid was obtained. The product was recrystallized from methanol. The yield was 11.2 g. (44.5%), m.p. 95–96°.

*Anal.* Calcd. for $C_{14}H_{16}O$ Fe: C, 65.52; H, 6.25. Found: C, 65.53; H, 6.42.

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(1) R. J. Graham *et al.*, *J. Am. Chem. Soc.*, **79**, 3416 (1957).

(2) R. B. Woodward, M. Rosenblum, and M. C. Whiting, *J. Am. Chem. Soc.*, **74**, 3458 (1952).

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## Preparation of 2,6-Dioxocyclohexanepropionic Acid and δ-Oxoazelaic Acid from Glutaric Anhydride

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The preparation of δ-oxoazelaic acid (5-oxononanedioic acid) from disodioacetone dicarboxylic