hexanes was likewise demonstrated by dehydrohalogenation to the same pair of Δ^1 and Δ^2 perfluoropropylcyclohexenes in each instance. C gave a 15/85 mixture and A gave a 60/40 mixture of Δ^1/Δ^2 olefins according to gas chromatography, n.m.r. vinyl proton resonance areas and infrared analysis. D gave pure Δ^1 olefin. A study of related chemistry of these substances is being carried on.

CONTRIBUTION NO. 325 FROM THE
PIONEERING RESEARCH SECTION
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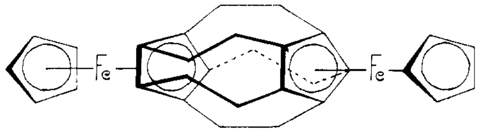
PENTAETHANODIFERROCENE

Sir:

Experimental results, obtained in this laboratory, provide evidence in serious contradiction to the recent assignment of Structure I to material obtained by prolonged treatment of ferrocene with aluminum chloride in the presence of ethylene dichloride.[1] The substance in question, whose formation is viewed as resulting from multi-alkylation of two ferrocene nuclei by five molecules of ethylene dichloride, possesses the name, "pentaethanodiferrocene";[1] and, the present experimental basis for rejection of I lies in the observation that material identical with "pentaethanodiferrocene" is produced by treatment of ferrocene with aluminum chloride *in the absence of ethylene dichloride.*



Structure I.

Careful repetition of the procedure reported by the original workers[1] gave rise to a yellow-colored solid possessing properties [dec., near 130°; mol. wt., 527 (benzene); d^{22}_4 1.37 ± 0.14 (methanol)] strikingly close to those reported for pentaethanodiferrocene [dec., near 130°; mol. wt., 545, 568 (benzene); d^{20}_4 1.461 (methanol)].[1] A yellow-colored solid (dec., near 130°), then obtained from similar treatment of ferrocene—except that benzene was used in place of ethylene dichloride—was found to be identical with the material produced in ethylene dichloride. The identity was demonstrated by means of superimposable infrared spectra (chloroform) and, more definitively, by the congruence of X-ray powder patterns determined from the two purified solids.[2]

These results clearly show that the aliphatic carbons present in structural association with the two ferrocene nuclei of "pentaethanodiferrocene" must have as their source a third ferrocene nucleus or its equivalent; and, that the sequence of bonding for all of these units is different from that depicted by I. Since ferrocene is known to undergo oxidation to ferricinium cation in the presence of strong electrophile and air,[3,4,5] and decomposition of the unstable ferricinium species in the presence of a proton source[6] may be expected to result in generation of cyclopentadiene, it would appear likely that the aliphatic carbons exist as substituted cyclopentane rings either as such or in a modified form. Experiments designed to provide information to allow a choice among several structural possibilities are now in progress, and all results will be reported in a full account of this work.

[1] A. N. Nesmeyanov and N. S. Kochetkova, *Doklady Akad. Nauk, S.S.S.R.*, **126**, 307 (1959).

[2] The author is indebted to Prof. Richard Layton of this Department for determination of these X-ray powder patterns.

[3] T. J. Kealy and P. L. Pauson, *Nature*, **168**, 1039 (1951).

[4] V. Weinmayr, *J. Am. Chem. Soc.*, **77**, 3009 (1955).

[5] M. Rosenblum and J. O. Santer, *ibid.*, **81**, 5517 (1959).

[6] Under the experimental conditions used,[1] it is not unreasonable to expect appreciable amounts of hydrogen chloride formed by the action of atmospheric moisture on aluminum chloride.

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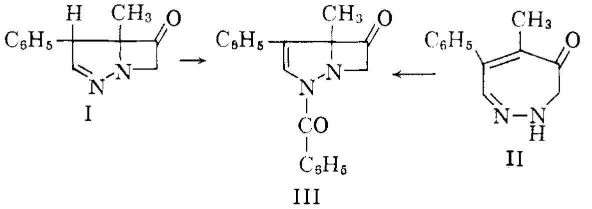
HETEROCYCLIC STUDIES. IX. SOME TRANSFORMATIONS OF THE 1,2-DIAZABICYCLO[3.2.0]HEPTANE SYSTEM[1]

Sir:

Current interest in the chemistry of strained bicyclic systems prompts us to report very briefly the formation and some of the reactions of 2-benzoyl-5-methyl-4-phenyl-1,2-diazabicyclo[3.2.0]-3-hepten-6-one (III). This compound is produced by benzoylation of the bicyclic ketone (I)[2] with benzoyl chloride in pyridine; m.p. 126–127° (dec.), (Found: C, 75.07; H, 5.33; N, 9.15). The structure follows from the method of preparation and the physical properties (λ_{KBr} 5.56, 6.13 μ; n.m.r. (60Mc.): 3 proton CH_3 peak at 95 c.p.s., 2 proton quartet for CH_2 at 277 c.p.s., J_{AB} 17 c.p.s., 11 proton aryl and vinyl peak at 450 c.p.s. max.).

A more practical preparation of III is provided by a facile bridging reaction that occurs on treatment *of the diazepinone* II[3] with benzoyl chloride in pyridine or dimethylaniline; acetyl chloride furnishes the corresponding 2-acetylbicyclic ketone.



In contrast to the parent bicyclic ketone I, which is tautomeric with II and is rapidly converted to II or derivatives thereof under most reaction conditions, III displays a remarkably broad

[1] Supported in part by a grant from the Geschickter Fund for Medical Research.

[2] J. A. Moore and R. W. Medeiros, *J. Am. Chem. Soc.*, **81**, 6026 (1959).

[3] J. A. Moore and J. Binkert, *ibid.*, **81**, 6029 (1959).