The Structure 1,4-Bis(dicarbonylcyclopentadienyliron)-*trans*,*trans*-buta-1,3-diene

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THE reaction of the sodium salt of dicarbonylcyclopentadienyliron with *cis*-3,4-dichlorocyclobutene has yielded a product whose n.m.r. spectrum was inconsistent with that of the expected product, carbonylcyclobutadienecyclopentadienyl iron carbonyl.[1] The structure of this compound† has now been determined by single crystal *X*-ray diffraction methods.‡

Crystal data: $C_{18}H_{14}O_4Fe_2$, monoclinic, $a = 6{\cdot}79$, $b = 11{\cdot}44$, $c = 11{\cdot}76$ Å, $\beta = 113{\cdot}13°$; $Z = 2$, space group $P2_1/c$. By use of a General Electric XRD-5 diffractometer equipped with single-crystal orienter and a balanced Ni–Co filter pair, three-dimensional single-crystal intensity data were collected by the stationary crystal–stationary counter method to the limit $2\theta = 160°$ (Cu-K_α);

† We thank Professor R. Pettit for supplying crystals of this compound.

‡ While this manuscript was in preparation, the independent crystallographic work of Dr. M. R. Churchill and his co-workers came to our attention (see preceding Communication).

within this limit, 1282 of the 1835 possible independent reflections had intensity observably higher than background.

An elemental analysis was not yet available, but very rough density measurements indicated a formula weight of the asymmetric unit in the range 202—218. The structure, which was solved by the heavy-atom method after obtaining the iron atom position from a sharpened Patterson map, is shown to be (I). This structure has been refined to a final R value of 0·085 (observed reflections) using the block-diagonal approximation to the least-squares method, with individual anisotropic temperature factors.

A stereoscopic drawing[2] of the molecule is shown in Figure 1. The cyclobutene ring has opened, and an iron atom is σ-bonded to each end of the *trans,trans*-butadiene chain. The molecule is centrosymmetric, occupying a crystallographic inversion centre; the formula weight of the symmetric unit is thus 202·9. The main interatomic distances and angles are shown in Figure 2.

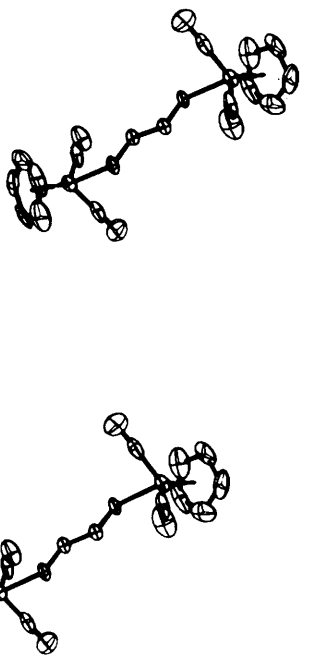

FIGURE 1

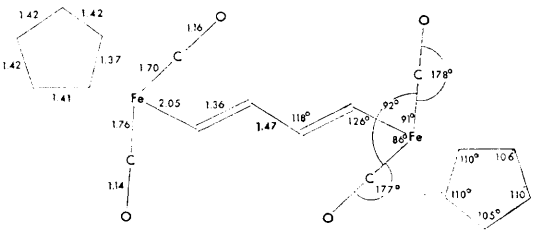

FIGURE 2

The mean cyclopentadienyl ring C–C distance of 1·41 Å and the mean Fe–C(ring) distance of 2·10 Å agree well with values for similar complexes, *e.g.* tetracarbonylcyclopentadienyldi-iron,[3] 1·41 and 2·12 Å, respectively. The ring is planar to within 0·02 Å and the distance from the metal to the ring plane is 1·72 Å. The distances and angles of the Fe–CO(terminal) groups are similar to those usually observed, *e.g.* butadienetricarbonyliron[4] and a phosphine derivative of 2-σ-tetracarbonyl-iron–π-allyltricarbonyliron.[5] The Fe–C σ-bond distance of 2·03 Å agrees with that in the latter.

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[1] R. Pettit, personal communication.
[2] C. K. Johnson, ORTEP, ORNL-3794 Revised, Oak Ridge National Laboratory, Oak Ridge, Tennessee, 1965.
[3] O. S. Mills, *Acta Cryst.*, 1958, 11, 620.
[4] O. S. Mills and G. Robinson, *Acta Cryst.*, 1963, **16**, 758.
[5] R. E. Davis, *Chem. Comm.*, 1968, 248.