The Olefin Metathesis Reaction applied to Tricarbonylcyclo-octatetraeneiron and the Crystal Structure of a Dimeric Product

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Summary Reaction of $C_8H_8Fe(CO)_3$ with WCl_6–EtOH–$EtAlCl_2$ catalyst leads to a new dimeric product, the structure of which has been determined by X-ray crystal analysis.

THE olefin metathesis reaction has been known for more than 10 years but it has mainly been applied to mono-olefins.[1] We report a new application, the reaction of a metathesis catalyst with an organometallic complex. Using tricarbonylcyclo-octatetraeneiron as the substrate, the reaction was carried out in toluene at room temperature with WCl_6–EtOH–$EtAlCl_2$ as catalyst.[2] Chromatography of the reaction mixture gives several products, among which is a pale yellow crystalline compound, whose analysis and mass spectrum indicate the composition $C_{16}H_{16}Fe_2(CO)_6$. The spectroscopic data do not allow an unambiguous assignment and so an X-ray structure determination was carried out.

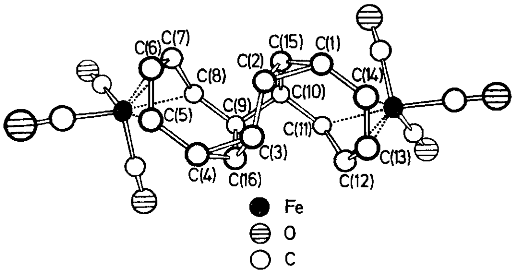

FIGURE The molecule viewed down the idealised 2-fold axis. Distances averaged over the pseudo symmetry are: Fe---C(i) (mean for i = 6,7,12,13) 2·06(2); Fe---C(i) (mean for i = 5,8,11,14) 2·14(2); C(1)–C(2) 1·54; C(1)–C(15) 1·50; C(2)–C(15) 1·53; C(2)–C(3) 1·49; C(9)–C(10) 1·56; C(9)–C(16) 1·53; C(4)–C(5) 1·49; C(5)–C(6) 1·44; C(6)–C(7) 1·40; C(7)–C(8) 1·45; C(8)–C(9) 1·52 Å. The mean e.s.d. in a C–C bond is 0·02 Å and the mean difference between pseudo-symmetry equivalent pairs is 0·03 Å.

Crystal data: $C_{22}H_{16}Fe_2O_6$, $M = 488$, orthorhombic, space group $Pbc2_1$ (non-standard setting of $Pca2_1$); $a = 6{\cdot}368(1)$, $b = 22{\cdot}888(4)$, $c = 13{\cdot}185(2)$ Å, ($\lambda = 0{\cdot}7107$ Å for Mo–K_α); $U = 1922$ Å^3; $Z = 4$, $D_c = 1{\cdot}69$ g cm^{-3}, $\mu = 15{\cdot}89$ cm^{-1} for Mo–K_α.

Crystals were obtained from toluene–methanol at 0 °C. The structure was solved by conventional heavy atom methods and all hydrogen atoms were located. Least-squares refinement converged at $R = 0{\cdot}053$ using 1538 independent reflections [$2\theta \leqslant 54°$ and $|F|^2 \geqslant 3\sigma(|F|^2)$] which were measured on a four-circle diffractometer with Mo–K_α radiation.

The molecular structure is illustrated in the Figure which is a view down the idealised 2-fold axis and shows the co-ordination of each iron atom to four adjacent carbon atoms in a new $C_{16}H_{16}$ skeleton containing five fused rings. This arrangement of the $C_{16}H_{16}$ skeleton has not been reported previously, either as a hydrocarbon[3] or as a metal complex.[4] From our results it is not possible to say whether the product is obtained by self-addition of two rearranged $C_8H_8Fe(CO)_3$ molecules, or whether the catalyst acts as a template. We hope that the characterisation of other products from the reaction and its extension to other substrates now under investigation will help to explain the mechanism. Recently the preparation of other polycyclic $C_{16}H_{16}$ isomers have been reported.[5]

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