A Di-iron–Anthracene Complex *via* Ultrasonics

Michael J. Begley, Shakher G. Puntambekar, and Anthony H. Wright*

Department of Chemistry, University of Nottingham, Nottingham NG7 2RD, U.K.

Ultrasonics can be used to make the novel di-iron–anthracene complex, $Fe_2(\mu,\eta^3,\eta^3\text{-anthracene})(CO)_6$, the crystal structure of which reveals a new mode of activation for the anthracene ligand.

Efforts to make polymetallic–polyaromatic compounds are frequently frustrated by the difficulty of introducing the polyaromatic component into the co-ordination sphere of the metal. Extending our investigations of polymetallic aromatic systems[1] to polyaromatic compounds, we are examining new synthetic routes to such molecules. We report the synthesis and structure of a new di-iron–anthracene complex, made using ultrasound. This is a rare example of an ultrasonic reaction leading to a product unobtainable *via* traditional chemistry.

Low-temperature ultrasound creates reaction conditions dramatically different from those created thermally or photochemically.[2] Thus, when $Fe_2(CO)_9$ and an excess of anthracene are suspended in n-hexane at −20 °C and subjected to ultrasound[†] for about 12 h, two products can be readily isolated (Scheme 1), $Fe_3(CO)_{12}$ and an orange complex, $(C_{14}H_{10})Fe_2(CO)_6$ (**1**).

This compound is easily distinguished[‡] from the mononuclear complex $(C_{14}H_{10})Fe(CO)_3$ (**2**)[3] which is the sole product of the thermal reaction of anthracene with iron carbonyl substrates. A range of sources of the $Fe(CO)_3$ moiety under thermal conditions including $Fe(CO)_3(\text{cyclo-octene})_2$[4] have been tested and no sign of (**1**) has been detected. Further, attempts to generate (**2**) from (**1**) have been unsuccessful. This suggests that the mechanism of the

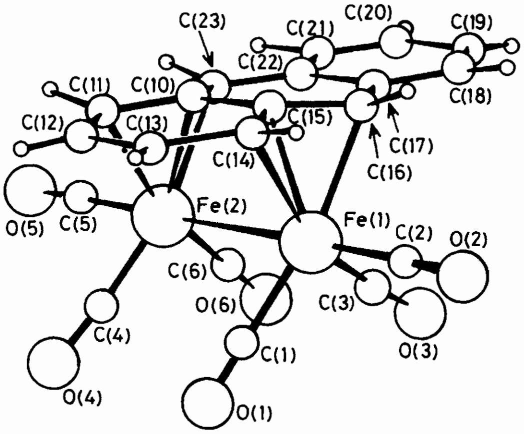


Figure 1. The structure of $Fe_2(\text{anthracene})(CO)_6$. Distances and angles include: Fe–Fe 2.872(1), Fe(1)–C(1) 1.786(3), Fe(1)–C(2) 1.785(3), Fe(1)–C(15) 2.059(2), Fe(1)–C(14) 2.222(3), Fe(1)–C(16) 2.185(2), C(1)–O(1) 1.137(3), C(2)–O(2) 1.133(3) Å, C(2)–Fe(1)–C(1) 91.5(1), C(2)–Fe(1)–C(3) 93.1(1), C(1)–Fe(1)–C(3) 102.1(1), C(15)–Fe(1)–C(1) 126.3(1), C(15)–Fe(1)–C(2) 110.0(1), C(15–Fe(1)–C(3) 124.1(1)°.

[†] The preparation was carried out using a Vibracell VC500 ultrasonic generator made by Sonics Materials.

[‡] Spectroscopic data for (**1**): ν_{CO} 2052, 2012, 1992, 1977, 1964 cm^{-1} (cyclohexane); ^1H n.m.r. ($CDCl_3$) δ 4.53 (s, 2H), 4.60 (m, 2H), 6.27 (m, 2H), 7.16 (m, 2H); ^{13}C n.m.r. ($CDCl_3$) δ 53.9, 58.2, 125.6, 126.6, 126.8, 131.0 (14C, $C_{14}H_{10}$); 211.1, 214.0, 214.5 (6C, CO).

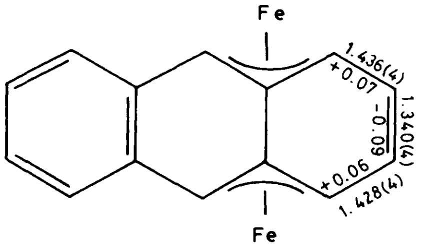


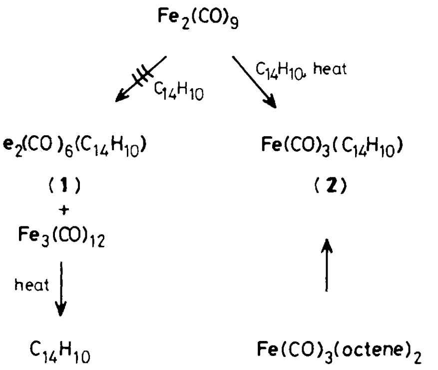
Figure 2. The effect of co-ordination on the anthracene ligand. The resultant bond lengths (Å) are given together with the change from values for free anthracene. Only the major changes in bond lengths are included.

ultrasonic reaction may proceed *via* some $Fe_2(CO)_n$ fragment, rather than a sequential building of (**1**) *via* (**2**).

Thermolysis of (**1**) in a range of solvents leads to rapid decomposition at about 80 °C and no iron-containing species could be identified. Monitoring the solutions using n.m.r. spectroscopy does not reveal any sign of (**2**) but only free anthracene, indicating that (**2**) does not lie on the decomposition pathway for (**1**).

The compound (**1**), an air-stable, orange crystalline solid, has been structurally characterised to establish the disposition of the $Fe(CO)_3$ groups.§ Figure 1 shows that the 'sawhorse' $Fe_2(CO)_6$ fragment is μ,η^3,η^3-bound to the anthracene.

The anthracene is modified significantly upon co-ordination. The molecule may be regarded as being divided into two with an isolated carbon–carbon double bond at one end and a benzene ring at the other (Figure 2). We are currently investigating the implications for the reactivity of the anthracene.



Scheme 1

This bis-allyl mode of bonding has been proposed for the naphthacene complex, $(C_{18}H_{12})Fe_2(CO)_6$, on the basis of n.m.r. spectroscopic evidence[5] and preliminary structural data have been reported for nonaromatic systems.[6] The iron–iron distance is 2.872(1) Å, shorter than the 2.97 Å in $(C_{18}H_{28})Fe_2(CO)_6$[6] but substantially longer than in (acenaphthene)$Fe_2(CO)_5$[7] (2.768 Å).

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§ *Crystal Data:* $C_{20}H_{10}Fe_2O_6$, M = 457.97, space group $C2/c$, a = 16.709(5), b = 12.873(5), c = 16.899(7) Å. β = 91.60(5)°, U = 3633.57 Å^3, D_c = 1.67 g cm^{-3}, D_c = 1.67 g cm^{-3}, $F(000)$ = 1840 for Z = 8. The intensity data were collected on a Hilger and Watts Y290 diffractometer within the limits $1 < \theta < 25°$. $\mu(\text{Mo-}K_\alpha)$ = 16.7 cm^{-1}, no absorption correction was considered necessary. The structure was solved by Patterson and Fourier methods on the basis of 2760 significant $[I > 3\sigma(I)]$ reflections. Refinement by full-matrix least-squares led to final R and R_w values of 0.038 and 0.035 respectively, where the weighting scheme was based on a Chebyshev polynomial. Hydrogen atoms were located in a difference Fourier and refined isotropically. Atomic co-ordinates, bond lengths and angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. See Notice to Authors, Issue No. 1.