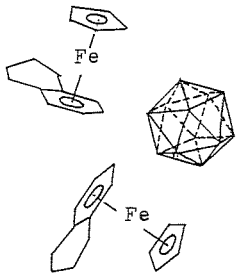
09.3–8 CRYSTAL STRUCTURES OF (1) BIS(η^5-CYCLOPENTADIENYL-η^6-TETRAHYDRONAPTHALENE-IRON) DODECAHYDRO-DODECABORATE AND (2) BIS(η^5-CYCLOPENTADIENYL-η^6-ISOPROPYLBENZENE-IRON)DODECAHYDRO-DODECABORATE. By Chen Liqing & Pan Kezhen, Fujian Institute of Research on the Structure of Matter, Academia Sinica, Fuzhou, China and Zhang Lun & Hu Peizhi, Department of Chemistry, Wuhan University, Wuhan, China.

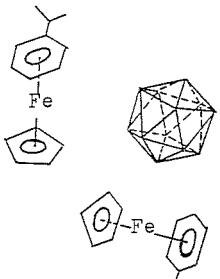
Compound 1 (see Title and Figure) Crystal data are : Orthorhombic s.g. Pbca, a=20.854(5), b=14.629(4), c=21.051(5) Å, Z=8. R=0.080 for 1899 reflections with $I \geq 3\sigma$. The cation $(C_5H_5Fe\text{-}C_{10}H_{12})^+$ has a sandwich conformation. The dihedral angle between the Cp ring and the conjugate six-member ring (Ph) in tetrahydronapthalene is 2.6°. The anion $B_{12}H_{12}^{2-}$ is a slightly distorted icosahedrom. The more important distance and angle averages are Fe-C(Cp)=2.031, Fe-C(Ph)=2.085, C-C(Cp)=1.382, C-C(Ph)=1.416, C-C(single bomd)=1.540, B-B=1.776 Å, B-B-B=60.0 (trigon) & 108.0°(pemtagon). The distance between Fe and Cp ring is 1.658 Å, and that between Fe and Ph ring is 1.530 Å.

Compound 2 Crystal data are : Monoclinic s.g. C2/c, a=18.703(9), b=25.120(7), c=15.190(7) Å, β=94.70(4)°, Z=8. R=0.070 for 2931 reflections with $I \geq 3\sigma$. It has a structure similar to that of Compound 1. The average bond lengths : Fe-C(Cp)=2.035, Fe-C(Ph)=2.075, C-C(Cp)=1.377, C-C(Ph)=1.394, B-B=1.777 Å. The distance from Fe to the Cp ring is 1.664 Å, and that to the Ph rimg is 1.537 Å. The dihedral angle between Cp and Ph rings is 0.8°.

In both compounds, the cation and amiom are of the distorted square and hexahedron coordinatioms, respectively.



(1)
$(C_{15}H_{17}Fe^+)_2 \cdot B_{12}H_{12}^{2-}$



(2)
$(C_{14}H_{17}Fe^+)_2 \cdot B_{12}H_{12}^{2-}$

09.3–9 CRYSTAL AND MOLECULAR STRUCTURES OF $(\eta^5\text{-}C_5H_5)_2Mo(SO_4)$ and $|(\eta^5\text{-}C_5H_5)_2MoI(CH_3CN)|(PF_6)$. By M. A.A.F. de C.T. Carrondo, A.M.T.S. Domingos* and M.T.Leal, Centro de Química Estrutural, Complexo I, I.S.T., Lisboa, Portugal. *Sector de Química, LNETI, Sacavém, Portugal.

Following our structural studies on Mo and Ti bis-cyclopentadienyl complexes (Carrondo and Domingos, J. Organomet. Chem. (1983), 253, 53) we now report the crystal structure determinations of the title compounds.

Crystal data for $(\eta^5\text{-}C_5H_5)_2Mo(SO_4)$ (I)-Mr=322,11, orthorhombic Ama2, a=12.979(4), b=8.547(2), c=9.356(5) Å, V=1037.9 Å^3, Z=4, MoKα radiation, 475 reflections with $F>3\sigma(F)$.

Crystal data for $|(\eta^5\text{-}C_5H_5)_2MoI(CH_3CN)|(PF_6)$ (II) - Mr=538,94, triclinic P1, a=7.7989(6), b=10.3044(8), c=10.5565(5) Å, α=96.218(4), β=94.466(4), γ=102.697(5)°, V=818.2 Å^3, Z=2, MoKα radiation, 4187 reflections with $F>3\sigma(F)$.

The structures were solved by Patterson and difference electron density syntheses using Shelx and refined for compound (I) by full-matrix least-squares to R=0.025 and for compound (II) by blocked matrix least-squares (R=0.086, still in progress).

Molecules of (I) have the bent metallocene structure in which the Mo atom is attached to two oxygen atoms of the sulphate ligand and two η^5-C_5H_5 groups in a distorted tetrahedron coordination. The Mo atom is at an average distance of 1.967(7) Å from the Cp rings and the angle between the ring normals is 134.8(4)°. The Mo-O(1) and Mo-O(2) bond lengths are respectively 2.057(9) and 2.162(8) and the O(1)-Mo-O(2) bond angle is 65.9(2)°. For the related compound $[(C_5H_5)_2MoO_2PO_2Mo(C_5H_5)_2](PF_6)_2$ (Prout, Couldwell and Forder, Acta Cryst. (1977), B 33,218) a mean Mo-O bond length of 2.126(3) Å and O-Mo-O bond angle of 67.8(1)° have been reported. The structure of compound (II) consists of two discrete organometallic cations and two hexafluorophosphate anions per unit cell. The Mo atom coordinates two η^5-C_5H_5 groups, one iodide and the nitrogen atom of one acetonitrile ligand. In each molecule the coordination geometry about the Mo atom is a distorted tetrahedron. At the present stage of refinement the Mo-I and Mo-N bond lengths and the N-Mo-I bond angle are respectively 2.840(4) Å, 2.172(23) Å and 80.8(6)° for one molecule and 2.822(4) Å, 2.096(21) Å and 81.4(5)° for the second.

Details of the molecular geometry will be given and comparison to similar compounds presented.