

09.3-4 STRUCTURE OF BIS(DIPHENYLPHOSPHINO)ETHYL-AMINOTRICARBONYLIRON, $\text{Fe}(\text{CO})_3(\text{C}_6\text{H}_5)_2\text{PN}(\text{C}_2\text{H}_5)_2\text{P}(\text{C}_6\text{H}_5)_2$. By E. C. Horsfield, D. W. Engel and K. G. Moodley, Physics and Chemistry Departments, University of Durban-Westville, Private Bag X54001, 4000 Durban.

$M_r=553.32$, monoclinic, $C2/c$, $a=35.741(8)$, $b=10.008(2)$, $c=15.049(2)$ Å, $\beta=95.72(1)$, $V=5356.2$ Å³, $Z=8$, $D_m=1.368$, $D_x=1.372$ Mg/m³, $\lambda(\text{MoK}\alpha)=0.7107$ Å, $\mu=0.706$ mm⁻¹, $F(000)=2288$, $R=0.083$ ($R_w=0.045$) for 4696 unique reflections (none omitted) and 335 parameters. Reaction of the ligand $(\text{C}_6\text{H}_5)_2\text{PN}(\text{C}_2\text{H}_5)_2\text{P}(\text{C}_6\text{H}_5)_2$, abbreviated L, and $\text{Fe}_2(\text{CO})_9$ under photochemical conditions gave a red complex $\text{Fe}_2(\mu\text{-CO})(\text{CO})_6(\mu\text{-L})$. However, recrystallisation from a toluene solution yielded yellow crystals of the mononuclear title compound $\text{Fe}(\text{CO})_3\text{-L}$.

The coordination geometry around the metal centre is intermediate between trigonal bipyramidal and square pyramidal. The structure can be compared with an analogous structure containing a bis(diphenylphosphino)methane ligand (Cotton, Hardcastle & Rusholme, 1973, *J. Coord. Chem.* **2**, 217-223). While both complexes deviate far from either idealised geometry, the complex containing our ligand based on ethylamine tends distinctly more towards the trigonal bipyramidal geometry.

09.3-5 THE STRUCTURE OF THE LOW TEMPERATURE CRYSTALLINE PHASE OF $\text{Cu}[\text{O}_2(\text{C}_6\text{H}_4)_2\text{CH}_2]_2$, A COMPOUND WHICH EXHIBITS DISCOTIC MESOMORPHISM AT SLIGHTLY ELEVATED TEMPERATURES. By Elizabeth M. Larson and P.G. Eller, Los Alamos National Laboratory, University of California, Los Alamos, NM 87545 (USA) and Bassam Freiha, M.L. Horng and Michael P. Eastman, Department of Chemistry, University of Texas, El Paso, TX 79968 (USA).

bis[1,3-di(p-n-octyloxyphenyl)propane-1,3-dionato]copper(II), $M_r=1028.89$, triclinic, $P\bar{1}$, $a=11.311(8)$, $b=16.059(7)$, $c=17.006(7)$ Å, $\alpha=82.25(4)^\circ$, $\beta=75.03(3)^\circ$, $\gamma=77.40(4)^\circ$, $Z=2$, $V=2902.7$ Å³, $D_x=1.177$ g/cm³, $\text{MoK}\alpha(0.70926$ Å), $F(000)=1114$, $T=230$ K, $R=7.2\%$ and $R_w=8.0\%$ for 2335 unique reflections having $I \geq 3\sigma I$. EPR studies of the discotic phase of this compound (octyloxy) and the octyl analogue show the octyl compound to behave as a one dimensional Heisenberg antiferromagnet, while the octyloxy compound does not exhibit such evidence. This structure shows the overlap of a phenyl ring on an adjacent molecule with the copper center. This overlap, an avenue for π electron donation to the copper, could explain the differences in the magnetic behavior of the two species.

09.3-6 THE NEUTRON DIFFRACTION ANALYSIS OF $[(\text{C}_5\text{Me}_5)\text{Ir}(\mu\text{-H})_3\text{Ir}(\text{C}_5\text{Me}_5)]^+[\text{C}_2\text{O}_4]^{2-} \cdot \text{C}_6\text{H}_6$. By Robert Bau*, Raymond C. Stevens, Malcolm McLean, Department of Chemistry, University of Southern California, Los Angeles, Ca. 90089; and Thomas F. Koetzle*, Department of Chemistry, Brookhaven National Laboratory, Upton, N.Y. 11973, U.S.A.

The structure of the $[(\text{C}_5\text{Me}_5)\text{Ir}(\mu\text{-H})_3\text{Ir}(\text{C}_5\text{Me}_5)]^+$ cation has been determined via single-crystal neutron diffraction techniques.

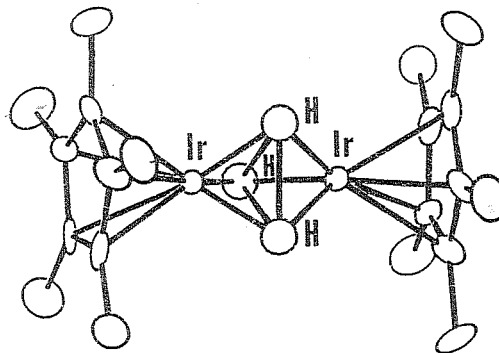


Fig. 1. Structure of the $[(\text{C}_5\text{Me}_5)\text{Ir}(\mu\text{-H})_3\text{Ir}(\text{C}_5\text{Me}_5)]^+$ cation, with H atoms of the methyl groups removed for clarity.

$[(\text{C}_5\text{Me}_5)\text{Ir}(\mu\text{-H})_3\text{Ir}(\text{C}_5\text{Me}_5)]^+[\text{C}_2\text{O}_4]^{2-} \cdot \text{C}_6\text{H}_6$ crystallizes in the space group $P2_12_12$, with $a = 13.261(3)$ Å, $b = 13.625(3)$ Å, $c = 8.612(2)$ Å, $V = 1556.0(8)$ Å³, $d(\text{calc}) = 1.783$ g cm⁻³. Data on a specimen of dimensions $3.0 \times 1.5 \times 1.0$ mm were collected at 20 K at the Brookhaven High-Flux Beam Reactor, and the structure has been refined to a present R factor of 0.041 for 1781 reflections. This work represents the first accurate structure determination on a metal-metal bond bridged by three hydride ligands, with the following average parameters: Ir-H = 1.741 Å, Ir-Ir = 2.467 Å, H...H = 2.125 Å, Ir-H-Ir = 90.19°, H-Ir-H = 75.23°. Comparisons of this result with other bridged systems [e.g., $M(\mu\text{-H})M$, $M(\mu\text{-H})_2M$, $M(\mu\text{-H})_4M$] will be presented. We thank the U.S. National Science Foundation (R.B.) and the U.S. Department of Energy (T.F.K.) for financial support.