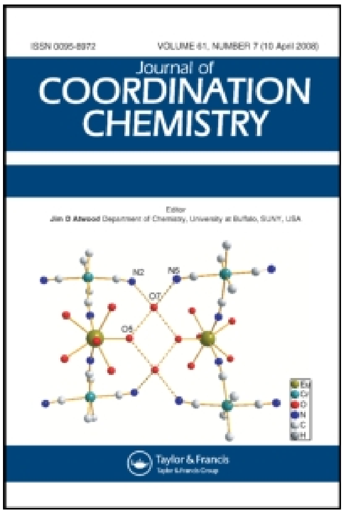

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Ferrocenyl Oxalylmonoximes

Ma Yong-Xiang[a]; Ma Yun[a]; Huang Duo-Sheng[a]; Zhu Ying[a]
[a] Department of Chemistry, Lanzhou University, Lanzhou, China

To cite this Article Yong-Xiang, Ma , Yun, Ma , Duo-Sheng, Huang and Ying, Zhu(1992) 'Ferrocenyl Oxalylmonoximes', Journal of Coordination Chemistry, 26: 3, 229 — 230
To link to this Article: DOI: 10.1080/00958979209409217
URL: http://dx.doi.org/10.1080/00958979209409217

J. Coord. Chem. 1992, Vol. 26. pp. 229–230
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NOTE

FERROCENYL OXALYLMONOXIMES

MA YONG-XIANG,* MA YUN, HUANG DUO-SHENG and ZHU YING

Department of Chemistry, Lanzhou University, Lanzhou 730000, China

(*Received 18 February 1991*)

Keywords: Oxalylmonoximes, X-ray structure

Diferrocenylglyoxime was synthesized by Ertas by the reaction between monolithioferrocene and *anti*—dichloroglyoxime.[1] Preparations and structures of ferrocenyl oxalylmonoximes have not been reported. In the present note, we report the synthesis of diferrocenyl oxalylmonoxime (**1**) and ferrocenyl phenyloxalylmonoxime (**2**), together with the crystal structure of the latter.

Compounds **1** and **2** were prepared by the reaction of the appropriate 1,2-diketone with $NH_2OH.HCl$ in a pyridine-EtOH solution. Compound **1** formed reddish orange crystals, yield 76%, m.p. 174 −175°. Anal.: Found (Calc. for $C_{22}H_{19}NO_2Fe$): C, 60.50 (59.90); H, 4.71 (4.34); N, 3.00 (3.17)%. ^1H n.m.r. ($CDCl_3$, δ, ppm): 4.1–4.8 (m, 18H). I.r. (KBr, cm^{-1}): ν(O—H), 3311m,br; ν(C═N), 1785w; ν(C═O), 1638vs; π(C—H), 822s. Compound **2** is an orange solid, yield 80%, m.p. 157.5–158°. Anal.: Found (Calc. for $C_{18}H_{15}NO_2Fe$): C, 65.15 (64.86); H, 4.58 (4.50); N, 4.43 (4.20)%. ^1H n.m.r. ($CDCl_3$, δ, ppm): 4.4–4.8 (m, 9H); 7.1–7.5 (m, 5H). Solution of **2** in Et_2O-petroleum ether gave reddish orange crystals suitable for X-ray diffraction analysis after several days.

The structure of **2** (the molecule is shown in Figure 1) is orthorhombic, space group of $Pca2_1$, $a = 13.167(1)$, $b = 10.227(1)$, $c = 11.250(3)$ Å, $V = 1514.9$ Å^3, $Z = 4$,

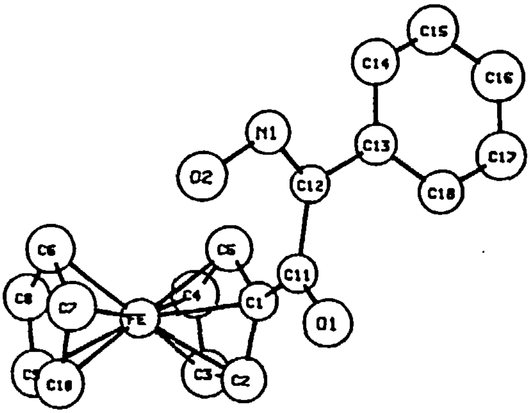

FIGURE 1 Molecular structure of FcCOC(═NOH)Ph.

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* Author for correspondence.

$D_c = 1.401\,\mathrm{g\,cm^{-3}}$. Final R was 0.063 for 1256 independent reflections with $|F_o| \geqslant 3\sigma(|F_o|)$.

The C(1)—C(11) bond (1.46(2) Å) is shorter than the usual C—C bond distance (1.48 Å) link in sp^2 carbons.[2] The average C—C distance in the substituted cyclopentadienyl ring (1.43 Å) is longer than in the unsubstituted ring (1.392 Å). C(11—O(1) is 1.241(9) Å, and the substituted cyclopentadienyl ring is coplanar with it; the sum of the bond angles at C(11) equals to 359.8°. It is thus shown the carbonyl group is conjugated with that cyclopentadienyl ring. C(12)—N(1) (1.32(2) Å) is longer than usual (1.28 Å),[3] and C(11)—C(12) (1.46(2) Å) is a little shortened. The sum of the bond angles at C(12) is 359.9°, and the C(11)—C(12)—N(1)—C(13) fragment is planar.

It is concluded that the spacial arrangement of the atoms in these monoximes is so compact that they can not rotate freely. The two oxygens in the carbonyl and oximino groups are remote, and the monoxime would be expected to form metal complexes with difficulty. Attempts to form complexes with **1** and **2** have, indeed, so far been unsuccessful.

ACKNOWLEDGEMENTS

This work was supported by the National Science Foundation of China.

SUPPLEMENTARY MATERIAL

Full details of data collection and refinement parameters, together with lists of atomic coordinates, bondlengths, angles, thermal parameters, and observed and calculated structure factors are available from M.Y.-X.

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