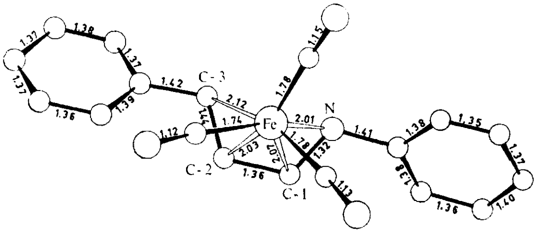
# Crystal Structure of Tricarbonyl-*N*-cinnamylideneanilineiron

By A. DE CIAN and R. WEISS*

(*Laboratoire de Cristallochimie Associé au C.N.R.S., Institut de chimie de Strasbourg, B.P.* 296-R8, 67-*Strasbourg, France*)

THE preparation of iron carbonyl complexes containing azomethine analogues of 1,3-dienes, such as PhCH : CH·CH : NPh,$Fe(CO)_3$ (I), and PhN : CMe-CMe : NPh,$Fe(CO)_3$ (II) has been reported.[1] From the n.m.r. and i.r. spectra it was inferred that $\pi$-1,3-co-ordination intervenes in (I), and $\sigma$-chelation through the nitrogen lone-pairs in (II).

We have now determined the *X*-ray crystal structure of (I). The results (Figure) show that the bonding of the unsaturated imine ligand with the $Fe(CO)_3$ group is similar to that found in the conjugated diolefins co-ordinated to the same group[2] and that the nitrogen lone-pair does not play an important part in the bonding with the metal. The nitrogen atom is only 0·10 Å ($\sigma$ = 0·023 Å) away from the plane of the three carbon atoms C-1, C-2, and C-3; the deformation of the bond angle C-2–C-1–N is larger: 113·26° ($\sigma$ = 1·59°). The C-1–N distance of 1·320 Å ($\sigma$ = 0·021 Å) shows that the C-1–N bond order is still far greater than 1. The rest of the unsaturated ligand presents a similar geometry to that found in the butadiene fragments in a number of conjugated diolefins co-ordinated to the $Fe(CO)_3$ group. The C-1–C-2 bond length of 1·362 Å ($\sigma$ = 0·023 Å) is however significantly different from that of 1·45 Å found in $C_4H_6Fe(CO)_3$ [3] and is nearer to that of 1·374 Å found in $C_6F_8Fe(CO)_3$.[4]. The bond angles OC–Fe–CO of 89·28, 97·22, and 101·83° ($\sigma$ = 1·1°) show the same distortion as in $C_4H_6Fe(CO)_3$,[3] $C_8H_8[Fe(CO)_3]_2$,[5] and $C_6F_8Fe(CO)_3$,[4] the 89·28° bond angle being the one in *trans* position to the nitrogen and C-3 carbon atoms.



FIGURE

Crystal data: $M = 348$, monoclinic; $a = 10{\cdot}289(12)$, $b = 6{\cdot}845(09)$, $c = 22{\cdot}563(20)$ Å, $\beta = 105{\cdot}30 \pm 0{\cdot}20°$, $U = 1532$ Å$^3$, $D_m = 1{\cdot}49$, $Z = 4$, $D_c = 1{\cdot}51$; space group $P\,2_1/c$ (No. 14). Intensities of 1613 independant non-zero reflexions have been measured with a Pailred automatic diffractometer using Mo-$K_\alpha$ radiation. No absorption corrections were applied.

The structure has been resolved by the symbolic addition procedure[6] and Fourier methods. The refinement to an $R$ value of 0·09 was carried out by isotropic full-matrix least-squares analysis and difference synthesis.

(*Received, February 8th*, 1968; *Com.* 153.)

---

[1] Sei Otsuka, T. Yoshida, and A. Nakamura, *Inorg. Chem.*, 1967, **6**, 20.

[2] M. R. Churchill and R. Mason, *Adv. Organometallic Chem.*, 1967, **5**, 93.

[3] O. S. Mills and G. Robinson, *Acta Cryst.*, 1963, **16**, 758.

[4] M. R. Churchill and R. Mason, *Proc. Chem. Soc.*, 1964, 226.

[5] B. Dickens and W. N. Lipscomb, *J. Chem. Phys.*, 1962, **37**, 2084.

[6] I. L. Karle and J. Karle, *Acta Cryst.*, 1966, **21**, 849.

**Printed by W Heffer & Sons Ltd Cambridge England**