The X-Ray Crystal Structure of a Low-spin Pseudo-octahedral Complex of Iron(II)

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Summary The crystal structure of $[\{N(CH_2CH_2N{=}CHC_5H_4N)_3\}Fe](BF_4)_2$ contains Fe^{II} with slightly distorted octahedral co-ordination, and mean Fe–N distance 1·96 Å: the tertiary amine nitrogen atom is at a distance of 3·44 Å from the iron atom.

L. J. WILSON AND N. J. ROSE[1,2] have prepared the Mn^{II}, Fe^{II}, Co^{II}, Ni^{II}, Cu^{II}, and Zn^{II} complexes of the potentially heptadentate ligand (trenpy), $N(CH_2CH_2N{=}CHC_5H_4N)_3$, the Schiff base prepared from (tren), $N(CH_2CH_2NH_2)_3$, and pyridine-2-carboxaldehyde. All of these complexes are high-spin except the iron complex, which is diamagnetic. As part of a structural study of these complexes, we have determined the crystal structure of the compound $[(trenpy)Fe](BF_4)_2$.

Crystal data: $FeC_{24}H_{27}N_7B_2F_8$, M 643·05, space group $P2_1/c$, a = 10·599(2), b = 15·504(3), c = 17·247(3) Å, β = 96·383(9)°, Z = 4, D_m = 1·507, D_c = 1·516 g cm^{-3}.

Complete three-dimensional X-ray intensity data were collected with Mo-K_α radiation out to 2θ *ca.* 50° on a Picker automated diffractometer. A total of 4866 independent reflections were measured. The structure was solved from Patterson and Fourier syntheses and has been refined by least-squares. At the present stage of refinement, with anisotropic thermal parameters, the discrepancy indices are R = 0·115 and weighted R = 0·098. Hydrogen atoms have not yet been introduced and there is some indication of possible rotational disorder of one of the BF_4^- ions.

The complex cation does not deviate markedly from C_3 symmetry about the axis defined by the iron and tertiary amine nitrogen atoms. The tertiary amine nitrogen atom is at a distance of 3·44 Å from the iron atom and the NC_3 group is almost exactly planar, indicating a considerable distortion from the "unstrained" configuration of a tertiary amine nitrogen atom. The other six Fe–N distances are nearly equal: Fe–imine nitrogen, all 1·95 Å; Fe–pyridine nitrogen, 1·96, 1·97, 1·98 Å. The co-ordination polyhedron is pseudo-octahedral, twisted 6° from a trigonal anti-prism (octahedral) toward a trigonal prism. Bond distances and angles within the ligand appear to be normal.

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[1] L. J. Wilson and N. J. Rose, Abstracts, 158th National Meeting of the American Chemical Society, New York, N.Y., September, 1969, INOR. No. 089.

[2] L. J. Wilson and N. J. Rose, *J. Amer. Chem. Soc.*, 1968, **90**, 6041.