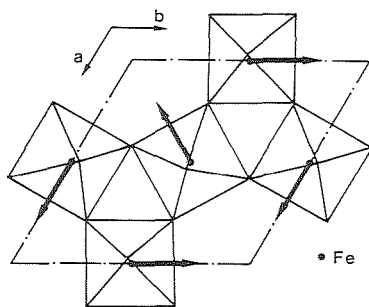**08.5—5   FRUSTRATED MAGNETIC STRUCTURE OF THE HEXAGONAL BRONZOID FeF$_3$.** By M. Leblanc, G. Ferey, J. Pannetier* and R. De Pape, ERA 609, Faculté des Sciences, 72017 Le Mans Cédex, France and *Institut Laue Langevin 38042 Grenoble Cédex, France.

A new variety of hydrated iron trifluoride $(H_2O)_{0.33}FeF_3$ with HTB structure was recently grown by the hydrothermal method (Leblanc, Ferey, Chevallier, Calage, De Pape, J. Solid State Chem. (1983) 47, 53). It dehydrates at 122°C and leads to the HTB new form of FeF$_3$. However, the small quantity of crystals obtained by this way lead us to imagine another synthesis yielding larger amounts of material for neutron powder diffraction experiments : the flash evaporation of a concentrated solution of FeF$_3$ in 49 % HF was followed by heating at 150°C under vacuum of the resulting powder. Then, chemical analysis, within the accuracy of the methods, are consistent with the FeF$_3$ formulation. Neutron diffraction patterns reveal the presence of α-FeF$_3$ R$\overline{3}$C (20 % molar) together with HTB-FeF$_3$. So, their analysis was performed using a multipattern profile refinement program (Thomas, Bendall, Acta Cryst. (1978) A34, S351). At room-temperature the structure of HTB-FeF$_3$ (a = 7.413(2) Å, c = 3.795(1) Å) is related to that of ideal tungsten bronze with empty tunnels. At 4.2K, the magnetic order and the tilting of the octahedra imply the doubling of the c parameter (a = 7.402(2) Å , c = 7.569(1) Å).

The refined magnetic structure can be described with three antiferromagnetic sublattices. The spins of Fe^{3+} (μ = 4.07(8)μ_B) lie in the (a, b) plane at 120° from each other (figure). The magnetic interactions are antiferromagnetic between successive planes along c. This disposition of the spins is due to the 2D-frustration which occurs in the triangles of metallic ions.

08.5—6   MAGNETIC AND CRYSTAL STRUCTURES OF ND$_4$MnFeF$_6$. By M. Leblanc, G. Ferey, J. Pannetier* and R. De Pape, ERA 609, Faculté des Sciences, 72017 Le Mans Cédex, France and *Institut Laue Langevin 38042 Grenoble Cédex, France.

Using hydrothermal conditions (380°C, 200 MPa), a new form of ND$_4$MnFeF$_6$ is obtained (Leblanc, Ferey, Calage, De Pape, J. Solid State Chem. (1983) 47, 24). The 3D structure, built from MnFeF$_{10}$ bioctahedra (Figure) is related to that of BaNb$_2$O$_6$ (Galasso, Layden, Ganung, Mater. Res. Bull. (1968) 3, 397). The compound is antiferromagnetic (T_N = 117.7 ± 0.5K) with a parasitic superimposed ferromagnetism (σ_r = 0.005 μ_B.mole^{-1} at 4.2K).

Neutron powder diffraction patterns were recorded at 130K and 4.2K (Pnc2 , Z = 4, a = 10.5280(4) Å, b = 7.7973(3) Å, c = 12.8158(5) Å, λ = 1.909 Å). The magnetic and nuclear cells are identical, so the Bertaut's theory was applied (Bertaut, Magnetism III, Rado and Shull Ed. (1963) 149). The metallic atoms form 4 magnetic independant sublattices.

The Rietveld-Hewatt profile refinement method (Rietveld, J. Appl. Cryst. (1969) 2, 65 ; Hewatt, Harwell Report AERE-R7350 (1973)) was used (12°<2θ<115.5°, 452 hkl triplets) and deuterium atoms were localized from Fourier maps. As a consequence of the complexity of the structure (29 independants positions) simplifying hypothesis were applied : isotropic thermal motion identical for each type of atoms, pure antiferromagnetism with $\mu_{Mn}{}^{2+}$ = $\mu_{Fe}{}^{3+}$. The best fit (R$_{Nucl}$ = 0.050, R$_{Mag}$ = 0.040, R$_{Prof}$ = 0.073) between observed and calculated intensities was obtained when the magnetic moments (μ = 4.51(5)μB) lie along b with spins alternatively up and down (Figure). A slight canting appart this direction may occur but could not be refined (C$_x$F$_y$G$_z$ mode).

According to these results, all the magnetic interactions are antiferromagnetic, particularly inside the MnFeF$_{10}$ unit where 90° superexchange occurs ; the double correlation superexchange mechanism is excluded. The previously described cationic order between Mn^{2+} and Fe^{3+} inside the MnFeF$_{10}$ bioctahedra is clearly confirmed.