A new variety of hydrated iron trifluoride (H$_2$O)$_{0.33}$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$_3$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, 6351). 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$^{3+}$ ($\mu = 4.07(8)$μB) 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.

Using hydrothermal conditions (380°C, 200 MPa), a new form of Nd$_2$MnFeF$_6$ is obtained (Leblanc, Ferey, Calage, De Pape, J. Solid State Chem. (1983) 47, 24). The 3D structure, built from MnFeF$_6$ octahedra (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 ± 0.3K) with a parasitic superimposed ferromagnetism ($\mu = 0.005$ μB.mole$^{-1}$ at 4.2K).

Neutron powder diffraction patterns were recorded at 130K and 4.2K (Pnma, Z = 4, a = 10.5280(4) Å, b = 7.7973(3) Å, c = 12.8158(5) Å, μ = 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 independent sublattices. The Rietveld-Hewatt profile refinement method (Rietveld, J. Appl. Cryst. (1969) 2, 65; Hewatt, Harwell Report AERE-R7350 (1973)) was used (12°<2θ<15.5°, 422 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 $H_{Mn}^{2+} = H_{Fe}^{3+}$. The best fit ($R_{\text{Nuc}}$ = 0.050, $R_{\text{mag}}$ = 0.040, $R_{\text{prof}}$ = 0.073) between observed and calculated intensities was obtained when the magnetic moments ($\mu = 4.51(5)$μB) lie along b with spins alternatively up and down (Figure). A slight canting appert 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$_6$ 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$_6$ octahedra is clearly confirmed.