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 a-FeF$_3$ ($R_3C$ (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$^{3+}$ ($\mu = 4.07(8)\mu_B$) 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.

Using hydrothermal conditions (380°C, 200 MPa), a new form of Nd$_5$MnFeF$_9$ is obtained (Leblanc, Ferey, Calage, De Pape, J. Solid State Chem. (1983) 47, 24). The 3D structure, built from $MnFeF_3$ octahedra (Figure) is related to that of BaNb$_2$O$_6$ (Galasso, Layden, Ganung, Mater. Res. Bull. (1968) 3, 397). The compound is antiferromagnetic ($T_N = 117.7 \pm 0.8K$) with a parasitic superimposed ferromagnetism ($\mu = 0.005 \mu_B$ mol$^{-1}$ at 4.2K).

Neutron powder diffraction patterns were recorded at 130K and 4.2K (Pnca, $Z = 4$, $a = 10.5280(4)$ Å, $b = 7.7973(3)$ Å, $c = 12.8158(5)$ Å, $\lambda = 1.909$ 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 ($12^\circ < 2\theta < 115.5^\circ$, 422 hkl triplets) and deuterium atoms were localized from Fourier maps. As a consequence of the complexity of the structure (29 independents 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 ($R_{Nuc} = 0.050$, $R_{Mag} = 0.040$, $R_{Prof} = 0.073$) between observed and calculated intensities was obtained when the magnetic moments ($\mu = 4.51(5)\mu_B$) lie along $b$ with spins alternatively up and down (Figure). A slight canting apart this direction may occur but could not be refined ($C_{2}F_{3}^{0}$ mode). According to these results, all the magnetic interactions are antiferromagnetic, particularly inside the MnFeF$_3$ 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$_3$ octahedra is clearly confirmed.

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure.png}
\caption{Diagram of the magnetic structure.}
\end{figure}