Single-crystal XRD and solid-state NMR structural resolution of a layered fluorinated gallium phosphate: RbGa$_4$(PO$_4$)$_3$F$_4$·C$_8$N$_2$H$_{14}$·2H$_2$O (MIL-145)

Charlotte Martineau,$^a$ Thierry Loiseau,$^{a,b}$ Lionel Beitone,$^a$ Gérard Férey,$^a$ Boris Bouchevreau and Francis Taulelle.$^a$

$a$ Institut Lavoisier de Versailles (ILV), UMR CNRS 8180, Université de Versailles Saint Quentin, 45, avenue des Etats-Unis, 78035 Versailles cedex, France.

$a$ present address: Unité de Catalyse et Chimie du Solide (UCCS), UMR CNRS 8181, Université de Lille Nord de France – ENSCL, Bât. C7, BP 90108, 59652 Villeneuve d’Ascq, France.

Electronic Supplementary Information

To be submitted to *Dalton trans.*

Version July 4, 2012

Revised August 29, 2012
Fig. S1 Thermogravimetric curve of RbGa₄(PO₄)₃F₄·C₅N₂H₁₄·2H₂O or MIL-145 (under O₂, 2 °C.min⁻¹).
**Fig S2** Experimental and reconstructed $^1\text{H} \to ^{15}\text{N}$ CPMAS NMR spectra of RbGa$_4$(PO$_4$)$_3$F$_4$·C$_5$N$_2$H$_{14}$·2H$_2$O (MIL-145) showing the presence of four inequivalent nitrogen atoms.
Fig. S3. $^{71}$Ga NMR spectra of RbGa$_3$(PO$_4$)$_2$(HPO$_4$)F$_4$C$_5$N$_2$H$_{16}$.2H$_2$O (MIL-145). (a) Static WURST-QCPMG spikelet spectrum recorded at $B_0 = 4.7$ T. Single pulse NMR spectra recorded at (b) $B_0 = 11.7$ T and $\nu_{\text{MAS}} = 34$ kHz and (c) $B_0 = 17.6$ T and $\nu_{\text{MAS}} = 30$ kHz. The reconstructed MAS NMR spectra are show below. The star represents an unidentified impurity.
**Fig. S4.** 2D $^{19}$F→$^{19}$F DQ-SQ NMR spectrum of RbGa$_4$(PO$_4$)$_3$F$_4$·C$_3$N$_2$H$_{14}$·2H$_2$O (MIL-145). The top spectrum, on which lines are labeled, is the full projection on the horizontal dimension. The thick line indicates the diagonal of slope 2. Dash lines indicate F-F cross-correlations.
Some additional comments on the data collection:

*The completeness is reported as 0.913 is quite low but if one slightly reduces the theta range, the completeness is greater than 0.99 for the coverage up to 28.0° for instance.*