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## MÖSSBAUER $^{237}\text{Np}$ AND CRYSTALLOGRAPHIC STUDIES OF $\text{M}^{\text{II}}\text{NpF}_6 \cdot 3\text{H}_2\text{O}$ ( $\text{M}^{\text{II}} = \text{Mn}, \text{Fe}, \text{Co}$ ) COMPOUNDS

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The compounds  $\text{M}^{\text{II}}\text{NpF}_6 \cdot 3\text{H}_2\text{O}$  with  $\text{M}^{\text{II}} = \text{Mn}, \text{Fe}, \text{Co}$  were prepared as single crystals by hydrothermal synthesis ( $T = 400^\circ\text{C}$ ,  $P = 2000$  bars).  $\text{CoNpF}_6 \cdot 3\text{H}_2\text{O}$  crystallises in a monoclinic system with  $\text{C}_2$  space group. Cell parameters are  $a = 12.143(9)\text{\AA}$ ;  $b = 6.922(5)\text{\AA}$ ;  $c = 7.942(5)\text{\AA}$ ;  $\beta = 92.84^\circ$ .

The Mössbauer measurements were performed in a conventional He Cryostat. The Mössbauer source used in the experiments was a 500 mCi  $^{241}\text{Am}$  metal with a conventional sine mode drive system.

A microbalance magnetometer attached to a varying temperature Cryostat was used for the susceptibility measurements. The maximum applied magnetic field was 14KG.

The Mossbauer spectroscopy of  $^{237}\text{Np}$  shows a magnetically split hyperfine spectrum at 4.2K for all those compounds.

The spectra can be fitted with a magnetic hyperfine field associated to a quadrupole splitting using the linear correlation between  $B_{\text{eff}}$  and  $e^2qQ$ . From isomer shift measurements, we confirm the IV charge state of Np in these 3 compounds.

The magnetic susceptibility shows antiferromagnetic type transition.  $1/\chi = f(T)$  follows a Curie-Weiss law above  $T_N$ .

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## THE PREPARATION OF TECHNETIUM OXYFLUORIDES AND THEIR CHARACTERIZATION BY $^{99}\text{Tc}$ , $^{17}\text{O}$ AND $^{19}\text{F}$ NMR SPECTROSCOPY

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Relatively little is known about the chemistry of technetium, especially in its highest oxidation state. At the same time,  $^{99}\text{Tc}$  ( $I=9/2$ ,  $Q=-0.19 \times 10^{-24}\text{cm}^2$ ) is one of the more sensitive NMR nuclei (sensitivity relative to the proton is 0.275 at 100% abundance). Pertechnetate,  $\text{TcO}_4^-$ , the standard for  $^{99}\text{Tc}$  NMR spectroscopy, also displays primary isotopic shifts for  $^{17}\text{O}$ - and  $^{18}\text{O}$ -enriched samples. Technetium-99 NMR has proven an invaluable structural probe in the study of Tc(VII) oxyfluorides.

Noble gas fluorides ( $\text{KrF}_2$  and  $\text{XeF}_6$ ) have been used to synthesize novel Tc(VII) species from solutions of  $\text{TcO}_3\text{F}$  in anhydrous HF, i.e.  $\text{Tc}_2\text{O}_5\text{F}_4$  and  $\text{TcO}_2\text{F}_3$ . Pertechnetyl fluoride has also been prepared and its fluoride ion donor properties studied in HF solution. Solid  $\text{TcO}_3^+\text{AsF}_6^-$  has been isolated from these solutions and characterized. In addition to  $^{99}\text{Tc}$  NMR, compounds have also been studied by  $^{19}\text{F}$  and  $^{17}\text{O}$  (enriched) NMR spectroscopy.