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### Title

PREPARATION AND STRUCTURAL CHARACTERIZATION OF  $\text{Fa-NpF}_5$

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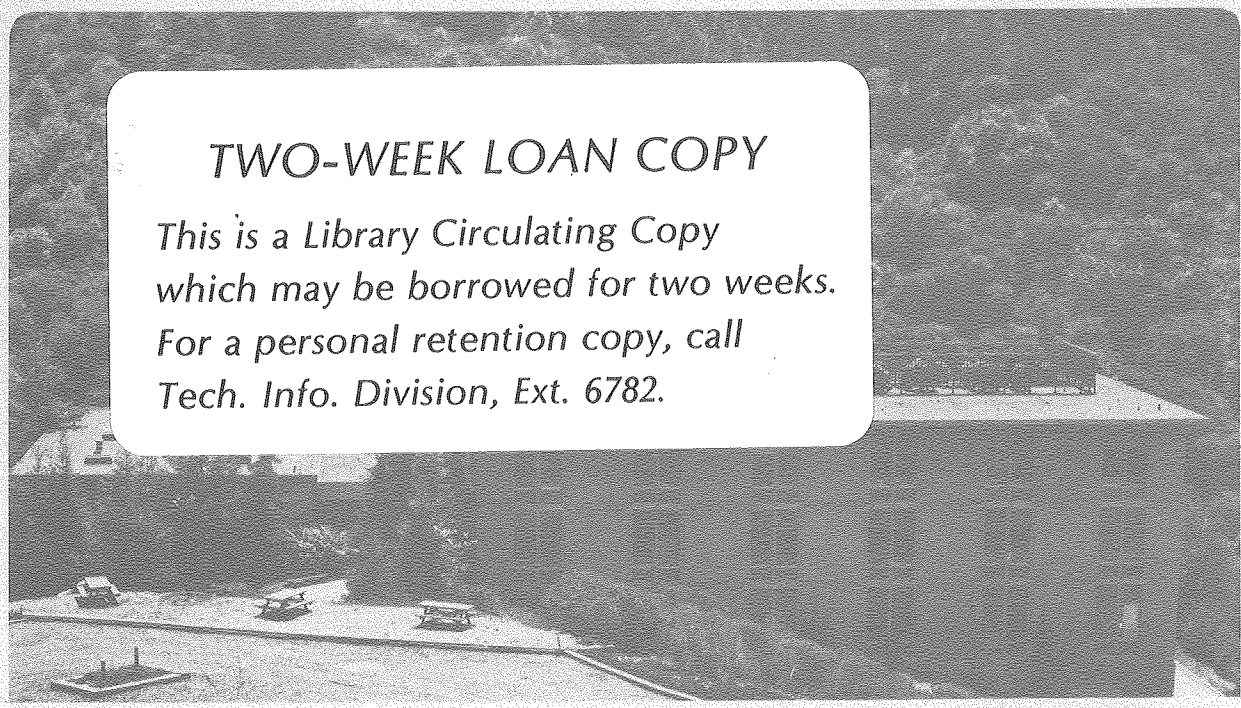
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PREPARATION AND STRUCTURAL CHARACTERIZATION OF  $\alpha$ -NpF<sub>5</sub>\*

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Introduction

Until recently, only the pentafluorides of uranium and protactinium had been prepared, although thermodynamic calculations suggested NpF<sub>5</sub> and possibly PuF<sub>5</sub> should be stable (1). Russian workers (2) have recently reported the synthesis of NpF<sub>5</sub> by the oxidation of NpF<sub>4</sub> in anhydrous hydrogen fluoride with KrF<sub>2</sub> at room temperature. Analysis of this material by spectroscopic and analytical methods showed it to be NpF<sub>5</sub>. We have recently developed a new and relatively simple synthesis for UF<sub>5</sub> (3) by the reaction of UF<sub>6</sub> dissolved in anhydrous hydrogen fluoride with excess PF<sub>3</sub>. A similar reaction has now been carried out with NpF<sub>6</sub> with the resultant product being  $\alpha$ -NpF<sub>5</sub>.

Experimental

NpF<sub>6</sub> was dissolved in anhydrous hydrogen fluoride at room temperature in a Kel-F tube. This solution was frozen at 77K and an excess amount of PF<sub>3</sub> was condensed onto the frozen solution. The liquid N<sub>2</sub> bath was removed and the solution was allowed to warm to room temperature. A bluish-white ppt formed during the warming process. After warming to room temperature the anhydrous hydrogen fluoride, PF<sub>3</sub> and PF<sub>5</sub>, were removed by distillation. Samples for x-ray analysis were prepared in an argon atmosphere box.

X-ray powder patterns were obtained with a 114 mm Debye-Scherrer camera using copper K $\alpha$  radiation. All the lines in the powder pattern could be assigned on the basis of the  $\alpha$ -UF<sub>5</sub> structure (4). The observed lines were fitted to the calculated pattern using the least squares program LCR-2 with the Nelson-Riley correction (5). The measured d-spacings, lattice parameters, and intensities are shown in Table 1.

TABLE 1  
Powder Diffraction Data for  $\alpha$ -NpF<sub>5</sub> at Room Temperature

Intensity <sup>a</sup>	hkl	Observed d spacings (Å)	Observed 2 $\theta$ <sup>b</sup> (deg)	Calculated 2 $\theta$ <sup>b,c</sup> (deg)
S+	110	4.611	19.25	19.26
S	101	3.678	24.20	24.23
M+	200	3.258	27.37	27.35
M+	211	2.440	36.83	36.84
M	220	2.308	39.03	39.04
W	002	2.222	40.60	40.54
S-	310	2.062	43.90	43.87
W+	112	2.004	45.24	45.24
M-	301	1.955	46.45	46.46
W+	202	1.840	49.55	49.58
W	321	1.676	54.77	54.74
W	400	1.633	56.35	56.38
W	222	1.604	57.47	57.53
W+	330	1.537	60.19	60.14
M-	312	1.514	61.22	61.24
M-	411	1.492	62.22	62.23
M-	420	1.459	63.77	63.77

<sup>a</sup> S = strong, M = medium, W = weak.

<sup>b</sup> Cu radiation -  $\lambda = 1.5418$  Å.

<sup>c</sup> Tetragonal lattice  $a = 6.53 \pm 0.03$  Å,  $b = 4.45 \pm 0.03$  Å.

### Results

Although the reaction utilized for the synthesis of  $\text{NpF}_5$  was a low temperature one, the structure of  $\alpha\text{-NpF}_5$  determined is the same as  $\alpha\text{-UF}_5$ , the high temperature form. The  $\alpha\text{-NpF}_5$  structure is consistent with the infrared and Raman spectra reported by the Russian workers (2).

The same reaction was run with  $\text{PuF}_6$  but the product obtained was amorphous and had the same tan color as  $\text{PuF}_4$ .

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