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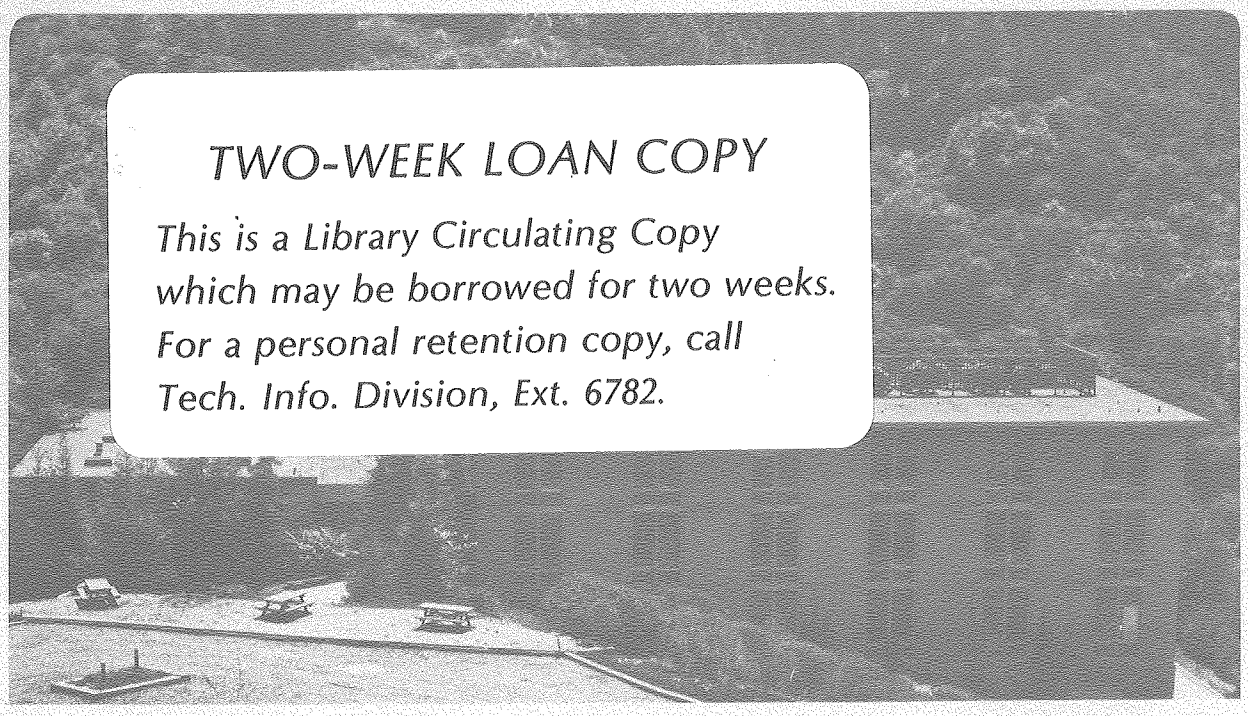



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# PREPARATION AND STRUCTURAL CHARACTERIZATION OF α-$NpF_5$*

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## Introduction

Until recently, only the pentafluorides of uranium and protactinium had been prepared, although thermodynamic calculations suggested $NpF_5$ and possibly $PuF_5$ should be stable (1). Russian workers (2) have recently reported the synthesis of $NpF_5$ by the oxidation of $NpF_4$ in anhydrous hydrogen fluoride with $KrF_2$ at room temperature. Analysis of this material by spectroscopic and analytical methods showed it to be $NpF_5$. We have recently developed a new and relatively simple synthesis for $UF_5$ (3) by the reaction of $UF_6$ dissolved in anhydrous hydrogen fluoride with excess $PF_3$. A similar reaction has now been carried out with $NpF_6$ with the resultant product being α-$NpF_5$.

## Experimental

$NpF_6$ 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 $PF_3$ was condensed onto the frozen solution. The liquid $N_2$ 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, $PF_3$ and $PF_5$, 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 Kα radiation. All the lines in the powder pattern could be assigned on the basis of the α-$UF_5$ 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 $\alpha$-$NpF_5$ at Room Temperature

| Intensity[a] | hkl | Observed d spacings (Å) | Observed 2$\theta$[b] (deg) | Calculated 2$\theta$[b,c] (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 |

[a] S = strong, M = medium, W = weak.

[b] Cu radiation - $\lambda$ = 1.5418 Å.

[c] Tetragonal lattice a = 6.53 ± 0.03 Å, b = 4.45 ± 0.03 Å.

## Results

Although the reaction utilized for the synthesis of $NpF_5$ was a low temperature one, the structure of $\alpha$-$NpF_5$ determined is the same as $\alpha$-$UF_5$, the high temperature form. The $\alpha$-$NpF_5$ structure is consistent with the infrared and Raman spectra reported by the Russian workers (2).

The same reaction was run with $PuF_6$ but the product obtained was amorphous and had the same tan color as $PuF_4$.

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