Supporting Information

Synthesis and Redetermination of the Crystal Structure of NbF5

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# Vibrational Spectroscopy

The Raman spectra were measured with a Monovista CRS+ confocal Raman microscope (Spectroscopy & Imaging GmbH) using a 532 nm solid-state laser and either a 300 grooves/mm (low-resolution mode, FWHM: <4.62 cm−1) or an 1800 grooves/mm (high-resolution mode, FWHM: <0.368 cm−1) grating. The sample was filled and sealed in a quartz capillary inside the glovebox and was then placed under the Raman microscope for data acquisition.

IR spectra were recorded on a Bruker alpha FT-IR spectrometer using the ATR Diamond module with a resolution of 4 cm−1. The spectrometer was located inside a glovebox (MBraun) under argon atmosphere. The spectra were processed with the OPUS software package.(OPUS V7.2, 2012)



Figure S 1: Infrared (black) and Raman spectrum (red) of NbF5 powder.



Figure S 2: Enlarged section of the Raman spectrum of NbF5. No further bands have been observed in the region of 1000 – 4000 cm–1.

Table S8. Observed vibrational frequencies for the Raman spectrum (>50 cm–1) and the IR spectrum (> 400 cm–1) of NbF5. Approximate band and assignment is given according to literature (Beattie *et al.*, 1969; Preiss & Reich, 1968).

|  |  |
| --- | --- |
| ν(observed) / cm–1 | Assignment |
| IR | Raman |  |
|  | 68 | lattice vibration |
|  | 133 | Nb–F deformation |
|  | 165 |
|  | 179 |
|  | 224 | Ring-deformation |
|  | 236 |
|  | 250 |
|  | 270 |
| 483 |  | Nb–F stretching |
| 656 | 658 |
|  | 669 |
|  | 681 |
| 698 | 718 |
| 718 | 753 |
| 745 | 767 |

# References

Beattie, I. R., Livingston, K. M. S., Ozin, G. A. & Reynolds, D. J. (1969). *J. Chem. Soc., A* 958–965.

OPUS V7.2 (2012). Ettlingen, Germany: Bruker Optik GmbH.

Preiss, H. & Reich, P. (1968). *Z. Anorg. Allg. Chem.* **362**, 19–23.