F-elements Solubility in Molten Fluorides - Studies by Reflectance Spectroscopy

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Preparation of CeF₃ pellets

Ce(NO₃)₃·6 H₂O → Dissolution → Precipitation of CeF₃ → Washing of CeF₃ → Filtration and drying of CeF₃ (90-100 °C) → Powder of CeF₃
Preparation of PuF₃ pellets

10M HNO₃ + 0.05M HF

PuO₂ → Dissolution → Pu(NO₃)₄ solution → Dilution to 5M HNO₃

H₂O

Diluted HF → Acetone → Sulfamic acid → Ascorbic acid

Reduction Pu (IV) to Pu (III)

Concentrated HF

NH₄HF₂ → Washing of PuF₃ × nH₂O

Precipitation of PuF₃ × nH₂O

He + 5.6% H₂ → Dehydration PuF₃ × nH₂O → Powder PuF₃
Preparation of AmF₃ pellets

1. Dissolution of ²⁴³AmO₂ with 6M HNO₃
2. Precipitation with HF Concentrated Acid
3. Washing
4. Centrifugation of pulp, drying (90-100°C) and dehydration (Ar, 450°C)
5. Powder AmF₃
Isothermal saturation method

Leaktight optical cell (Pt crucible + He atmosph.) with sapphire windows and Pt/Ir mirrors in complex of Spectrometer “SOLAR-TII”™

Reflectance spectroscopy method

Ni Cell

Sampler with Ni filter 40 μm

Loading device
Solubility of f-element fluorides vs temperature in FLINAK

- AmF$_3$, this work
- CeF$_3$, this work
- PuF$_3$, this work
- Benes
- Seregin

Solubility, mol%

T, K
Reflectance Spectra of CeF$_3$ in fluoride melts

- LiF-CeF$_3$ (7.7 mol%), $T=1275K$
- NaF- CeF$_3$ (8.06 mol%), $T=1325K$
- KF- CeF$_3$ (7.21 mol%), $T=1275K$
- LiF- NaF-KF-CeF$_3$ (11.1 mol%), $T=875K$
- BeF$_2$-CeF$_3$ (2.8 mol%), $T=1325K$
Maxima of the reflection bands for the molten alkaline metal fluorides and BeF$_2$
containing cerium (III) ions in 10$^3$cm$^{-1}$

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Reflection band maxima</th>
<th>Center of gravity of T$_{2g}$ state</th>
</tr>
</thead>
<tbody>
<tr>
<td>LiF</td>
<td>42,02</td>
<td>38,04</td>
</tr>
<tr>
<td>NaF</td>
<td>41,67</td>
<td>39,87</td>
</tr>
<tr>
<td>KF</td>
<td>41,74</td>
<td>40,25</td>
</tr>
<tr>
<td>BeF$_2$</td>
<td>42,17</td>
<td>39,68</td>
</tr>
<tr>
<td>LiNaKF</td>
<td>41,81</td>
<td>41,05</td>
</tr>
</tbody>
</table>
Dependence of reflection peak (265nm) on CeF$_3$ content in FLINAK

\[ N = 0.095 \cdot T - 63,852 \]
Dependence of CeF$_3$ solubility on temperature in FLINAK

![Graph showing the dependence of CeF$_3$ solubility on temperature in FLINAK.](image)

2. Spectroscopy, this work
3. Isothermal saturation, this work
Conclusion

• Solubility of f-elements in molten LiF-NaF-KF (FLINAK) was studied in the temperature range from 550 to 700°C by isothermal saturation of the melt involving sampling through a 40 mkm nickel filter to reduce the suspension capture.

• Solubility of CeF3 in molten LiF-NaF-KF (FLINAK) was studied in the temperature range from 600 to 700°C by the reflectance spectroscopy using a leaktight cell with sapphire windows and platinum/iridium mirrors.

• Experimental data which are in good agreement.

• Reflectance spectroscopy will be used to study solubility of actinide trifluorides in molten fluorides.