



# Thermal decomposition data of uranium containing microspheres produced via internal gelation and ammonium diuranate powder

Authors: Christian Schreinemachers , Gregory Leinders 

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## Description

A combination of simultaneous thermal analysis, evolved gas analysis and non-ambient XRD techniques was used to characterise and investigate the thermal decomposition behavior in the  $\text{NH}_3 - \text{UO}_3 - \text{H}_2\text{O}$  class of materials.

One compound was prepared according to a typical ammonium diuranate precipitation reaction, and could be identified as  $3\text{UO}_3 \cdot \text{NH}_3 \cdot 5\text{H}_2\text{O}$ . Microspheres prepared by the sol-gel method via internal gelation were associated to the composition  $3\text{UO}_3 \cdot 2\text{NH}_3 \cdot 4\text{H}_2\text{O}$  under the specified conditions.

The products were analysed using the techniques listed below, the resulting data are part of this dataset.

- TGA, combined with EGA-MS ( $T_{\text{max}} = 1300\text{ }^\circ\text{C}$ , heating rate =  $2\text{ }^\circ\text{C}/\text{min}$ )
- TG-DSC, combined with EGA-MS ( $T_{\text{max}} = 1300\text{ }^\circ\text{C}$ , heating rate =  $10\text{ }^\circ\text{C}/\text{min}$ )
- ambient XRD (dried products after synthesis)
- in-situ* high temperature XRD (including initial and final scans, taken at  $35\text{ }^\circ\text{C}$ )
  - $T_{\text{max}}$  for  $3\text{UO}_3 \cdot \text{NH}_3 \cdot 5\text{H}_2\text{O} = 1300\text{ }^\circ\text{C}$ ;  $T_{\text{max}}$  for  $3\text{UO}_3 \cdot 2\text{NH}_3 \cdot 4\text{H}_2\text{O} = 650\text{ }^\circ\text{C}$
  - Samples measured directly on a Pt/Rh heating strip (Pt/Rh phase visible in patterns, blank scan included)

## Devices used for data aquisition

The data included in this dataset were measured using the devices listed underneath.

### Laboratory balance

- Manufacturer: Mettler-Toledo
- Model: AG204
- Uncertainty:  $0.02\text{ mg}$  ( $1\sigma$ )

### Thermobalance

- Manufacturer: Netzsch
- Model: STA 449 F1 Jupiter
- Uncertainty:  $0.014\text{ mg}$  ( $1\sigma$ )

### Mass spectrometer

- Manufacturer: Netzsch
- Model: QMS 403 C Aeolos

### X-ray diffractometer

- Manufacturer: PANanalytical
- Model: X'Pert Pro
- Detector: PANalytical X'Celerator (position-sensitive)
- High temperature chamber:
  - Manufacturer: Edmund Bühler,
  - Model: Hochtemperaturkammer HDK 2.4

## Content of this dataset

### Structure

The data are organised in archives. There exists one archive for each sample:

- `3UO3·1NH3·5H2O_(ADU-powder).zip`
- `3UO3·2NH3·4H2O_(IG-particles).zip`

If a sample archive is unzipped, a sample folder containing subfolders for the applied analyses methods is extracted.

The subfolders are named based on an analysis technique and contain the files belonging to the individual technique and sample.

The folder structure after extraction of both sample archives can be summarised as follows:

```
├──3UO3·1NH3·5H2O_(ADU-powder)
│   ├──TG-DSC
│   ├──TGA
│   ├──XRD_(ambient)
│   └──XRD_(high-temperature)
└──3UO3·2NH3·4H2O_(IG-particles)
    ├──TG-DSC
    ├──TGA
    ├──XRD_(ambient)
    └──XRD_(high-temperature)
```

### Files

All files of this dataset are listed in the scheme underneath (extraction of sample archives necessary).

The individual files are briefly described below the scheme.

```
|   license.txt
|   readme.md
|   readme.pdf
|
├──3UO3·1NH3·5H2O_(ADU-powder)
│   ├──TG-DSC
│   │   ├──ADU_TGDSC.csv
│   │   └──ADU_TGDSC_MS.csv
│   ├──TGA
│   │   ├──ADU_TGA.csv
│   │   └──ADU_TGA_MS.csv
│   ├──XRD_(ambient)
│   │   └──ADU_XRD.xy
│   └──XRD_(high-temperature)
│       ├──ADU_HTXRD.gp
│       ├──ADU_HTXRD.pdf
│       ├──ADU_HTXRD_1000C.xy
│       ├──ADU_HTXRD_1100C.xy
│       ├──ADU_HTXRD_1200C.xy
│       ├──ADU_HTXRD_1300C.xy
│       ├──ADU_HTXRD_150C.xy
│       ├──ADU_HTXRD_250C.xy
│       ├──ADU_HTXRD_350C.xy
│       ├──ADU_HTXRD_35C_final.xy
│       ├──ADU_HTXRD_35C_initial.xy
│       ├──ADU_HTXRD_450C.xy
│       ├──ADU_HTXRD_550C.xy
│       ├──ADU_HTXRD_600C.xy
│       ├──ADU_HTXRD_650C.xy
│       ├──ADU_HTXRD_700C.xy
│       ├──ADU_HTXRD_800C.xy
│       ├──ADU_HTXRD_900C.xy
│       └──PtRh_heating-strip_blank.xy
└──3UO3·2NH3·4H2O_(IG-particles)
    ├──TG-DSC
    │   ├──IG_TGDSC.csv
    │   └──IG_TGDSC_MS.csv
    ├──TGA
    │   ├──IG_TGA.csv
    │   └──IG_TGA_MS.csv
    ├──XRD_(ambient)
    │   └──IG_XRD.xy
    └──XRD_(high-temperature)
        ├──IG_HTXRD.gp
        ├──IG_HTXRD.pdf
        ├──IG_HTXRD_150C.xy
        ├──IG_HTXRD_250C.xy
        ├──IG_HTXRD_350C.xy
        ├──IG_HTXRD_35C_final.xy
        ├──IG_HTXRD_35C_initial.xy
        ├──IG_HTXRD_450C.xy
        ├──IG_HTXRD_550C.xy
        ├──IG_HTXRD_600C.xy
        ├──IG_HTXRD_650C.xy
        └──PtRh_heating-strip_blank.xy
```

There are three files in the *root folder* (not part of the archives), containing information about the dataset itself:

- `license.txt` (license of this dataset, text file)
- `readme.md` (this readme file, markdown file)
- `readme.pdf` (this readme file, pdf document)

### TG data

The *TG-DSC* and *TGA* folders contain the TG data and the corresponding EGA-MS data:

- `*TGA.csv` (TGA data file)
- `*TGA_MS.csv` (EGA-MS data file)
- `*TGDSC.csv` (TG-DSC data file)
- `*TGDSC_MS.csv` (EGA-MS data file)

All files in those folders contain comma separated values.

The content of the TGA/TG-DSC and EGA-MS files, as well as the unit of each column is described underneath.

**TGA/TG-DSC data file** (DSC signal only in TG-DSC files present)

- Time (min)** - Time in minutes, converted from seconds.
- Temperature (°C)** - Temperature in  $^\circ\text{C}$ , directly measured data.
- Mass loss (mg)** - Netto mass loss in mg, corrected by blank mass loss.
- DSC (mW)** - Netto DSC signal in mW, corrected by blank DSC signal.
- Segment** - Segment of temperature program.
- Mass (mg)** - Netto mass in mg, calculated by addition of final *ex-situ* mass to netto mass loss.
- Mass\_std (%)** - Netto mass in %, standardised to standardisation mass.
- DSC\_std (mW/mg)** - Netto DSC signal in mW/mg, standardised to standardisation mass.

For the TGA measurements, the mass present at  $650\text{ }^\circ\text{C}$  was used as standardisation mass, while the mass present at  $665\text{ }^\circ\text{C}$  was used as standardisation mass for the TG-DSC measurements.

### EGA-MS data file

- Time (min)** - Time in minutes, converted from seconds. Additionally, the offset between the MS and TG log files was added.
- Temperature (°C)** - Temperature in  $^\circ\text{C}$ , interpolated using the determined time and the temperature/time relation from the TG data.
- Segment** - Segment of the temperature program, taken from TG data based on the time.
- m/z=...** - Ion current in ampere for the specific m/z signal, directly measured data.

### XRD data

The *XRD\_(ambient)* and *XRD\_(high-temperature)* folders contain the XRD data. Each scan is saved as individual `*.xy` file. Those files contain data, which are separated by whitespaces. The first column (left) corresponds to  $2\theta$  in degree and the second column (right) to the measured intensity.

The last part of the filename of the non-ambient data indicates the measurement temperature in  $^\circ\text{C}$  (string between `*HTXRD_` and `C.xy`). At a temperature of  $35\text{ }^\circ\text{C}$ , an initial scan was taken (`*HTXRD_35C_initial.xy`) and additionally a final scan after completing the temperature program (`*HTXRD_35C_final.xy`).

To point out the contribution of the Pt/Rh heating strip to the patterns, the non-ambient data contain a scan of an Pt/Rh heating strip without sample material (`PtRh_heating-strip_blank.xy`), recorded at room temperature. The data are in agreement to pattern published for a Pt/Rh phase (ICSD: 40356).

Moreover, there exists a pdf file (`*HTXRD.pdf`) containing an overview plot of all non-ambient sample scans and a gnuplot script to generate this overview plot (`*HTXRD.gp`). The reflections of the Pt/Rh heating strip are indicated in the plots.

## ex-situ masses

The mass of the sample was measured prior to and after the TG analysis on a laboratory balance. Those *ex-situ* masses are listed in the table underneath and were used to calculate absolute mass differences and quantify the losses occurred during the evacuation of the TG set-up.

	TGA	TG-DSC	TGA	TG-DSC
	ADU powder	ADU powder	IG particles	IG particles
$m(\text{initial})_{\text{ex-situ}}$	200.80 mg	33.91 mg	214.84 mg	35.99 mg
$m(\text{final})_{\text{ex-situ}}$	170.26 mg	28.58 mg	173.62 mg	30.77 mg