Nu TIMS

Total evaporation of Uranium using Nu TIMS Instrument

A Thermal ionisation mass spectrometer (TIMS) is used, as part of the analytical capability of a plant, to measure the uranium and plutonium isotopes in a nuclear production facility to maintain material quality control, environmental control and nuclear material accountancy for safeguards purposes. In this environment a TIMS instrument is expected to run 24/7, to provide continuous data to the plant staff. A change in the isotope ratios can indicate a problem in the operation such as contamination or fault, which requires immediate attention.
The Nu TIMS instrument has been designed with flexibility for the analysis of nuclear materials and the capability of running quickly, robustly and automatically for long periods of time. With rapid pump down times, sample changeover can be achieved in 1 hour, maximising sample analysis time. With the added capability of automatic total evaporation measurements, time spent at the instrument can also be reduced.
This report will demonstrate 24/7 automatic analysis of uranium over one working week, providing data compatible with that shown in the literature.

Experimental

A method was designed so that one turret of 20 samples can be analysed in a 24-hour period using the total evaporation technique. The total evaporation method continuously heats a sample until it is exhausted from the filament (Figure 1), the average isotopic values are taken at the end of the analysis minimizing the effects of fractionation.

Ion Beam Emission

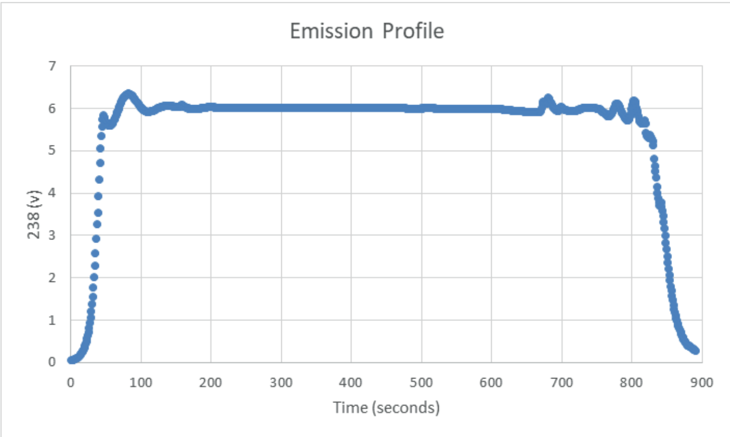

Figure 1: Uranium Total Evaporation beam profile during measurement

Filament Loading

Even with the use of total evaporation loading of the filaments needs to be identical if possible. The tungsten filaments had two 0.5 ul drops of standard solution in 2% nitric placed on them and were dried at 0.5A. This helped keep the sample spot size on the filament to a minimum for increased ionisation efficiency and helped maintain a similar fractionation between filaments. After the second drop of sample had completely dried the filaments were individually raised to 2.5 Amps and held for 10 seconds to oxidise the uranium.

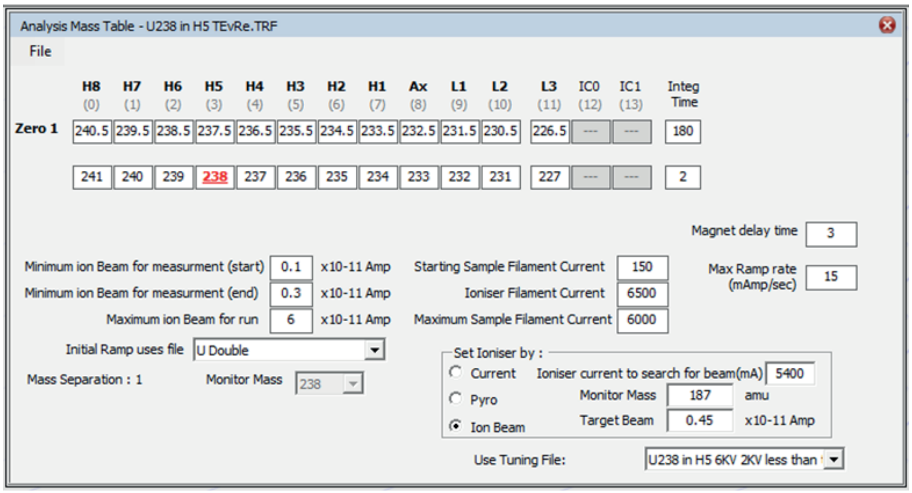

Figure 2: Uranium Total Evaporation method file using rhenium ion beam

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Total Evaporation (TE) Method

In this method (Figure 2), the rhenium ionization filament is set so that it produces a rhenium beam of 0.45 V and pre-tunes the filament prior to finding the Uranium beam, minimizing loss of sample. The evaporation filament with the sample on is then raised for finding and tuning of uranium. At an intensity of 0.1 V on the 238U isotope, the measurement begins and the 238U ion beam is raised to an intensity of 6V and controlled there until the end of the measurement (Figure 1). Parameters are shown in Table 1.

Ionisation Filament	Evaporation Filament	Target Intensity	Baselines	Measurement Duration
Re double Ramp rate: 20 mA/s to 3900 mA, then 5 mA/s	W double Ramp rate: Starting at 15mA/s and decreasing to 4mA/s as the current increases.	6 V on 238U	180 seconds before the filaments are ramped	Approximately 1 hour per sample at a loading of 50-77 ng.

Table 1: Parameters for the total evaporation method with rhenium ion beam

Results

Carousel number	Samples (maximum 20)
1	All U010
2	All U050
3	Positions 1-10 U010, Positions 11-20 U050
4	Positions 1-10 U050, Positions 11-20 U010
5	Random positions 10 x U010, 10 x U050

Table 2: Order of samples on each carousel

Standards were running for 5 consecutive days at the rate of one carousel per day to demonstrate analytical performance and robustness in a nuclear production environment (Table 1) The standards were run on a combination of same standard and mixed standard turrets as shown in Table 1. Tables 3 and 4 below show the results from each standard for each turret and overall.

	U010 77ng							ion/atom
Turret	No of runs	U234/U238	%RSD	U235/U238	%RSD	U236/U238	%RSD	efficiency(%)
1	20	0.0000548	1.87	0.0101425	0.03	0.0000707	1.70	0.20
2								
3	10	0.0000549	1.20	0.0101422	0.02	0.0000700	2.61	0.25
4	10	0.0000549	1.44	0.0101435	0.02	0.0000699	1.91	0.24
5	9	0.0000548	1.99	0.0101419	0.03	0.0000703	0.93	0.28
Average		0.0000548	1.63	0.0101425	0.02	0.0000702	1.79	0.23

Table 3: Summary average of each turrets runs for uranium standard U010

	U050 50 ng							ion/atom
Turret	No of runs	U234/U238	%RSD	U235/U238	%RSD	U236/U238	%RSD	efficiency(%)
1								
2	18	0.000294368	0.50	0.052790	0.02	0.000508791	0.24	0.16
3	10	0.000294596	0.36	0.052784	0.03	0.000508269	0.24	0.19
4	10	0.000294662	0.24	0.052790	0.06	0.000508841	0.25	0.17
5	10	0.000294586	0.31	0.052782	0.02	0.000508675	0.27	0.22
Average		0.000294615	0.30	0.052785	0.04	0.000508595	0.26	0.18

Table 4: Summary average of each turrets runs for uranium standard U050

Conclusion

The Nu TIMS instrument has been designed with flexibility for the analysis of nuclear materials and the capability of running quickly, robustly and automatically for long periods of time. With rapid pump down helping maximize sample analysis time and the capability of automatic total evaporation measurements, minimizing operator time whilst maintaining precision.