Application Note: 30017

Total Sample Evaporation of 1 ng SRM U350

Precision and Accuracy by Static Faraday Cup Multicollection

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Key Words

- TRITON
- Atom %
- Nuclear Application
- Static Mode
- TI-MS
- Uranium

Analysts are aiming to analyze smaller and smaller sample amounts while not compromising the precision and accuracy of the measured isotope ratios. It is even more challenging in case of real samples with varying sample concentrations and different matrices.

This is in particular true for nuclear applications, including safeguards, environmental control and reprocessing of nuclear fuels, where official regulations have to be fulfilled. There is a requirement for precise and accurate analysis of uranium and plutonium samples in the low nanogram range (sub-nano Curie activity).

One of the fundamental problems in Thermal Ionization Mass Spectrometry (TI-MS) is the fact of time-dependent isotopic fractionation that occurs during the evaporation of the sample. As samples are heated and ionized in a TI source, the lighter isotopes are evaporated more rapidly and the measured isotope compositions will generally change from light to heavy during the course of analysis.

The analysis of elements that cannot be normalized to an internal isotope system requires a highly reproducible loading technique and ionization conditions and/or external correction procedures to achieve reproducible results of high precision.

One solution to this problem has been the development of "Total Sample Evaporation".

During total sample evaporation, a sample is continuously and completely evaporated from the filament. During the run, the filament current is continuously adjusted in order to follow a predefined and reproducible evaporation profile.

At the same time, all isotopes of interest are collected simultaneously during the entire period of evaporation.

The final results are represented by the averaged isotopic mean values at the end of the analysis, when no measurable amount of sample is left on the filament.

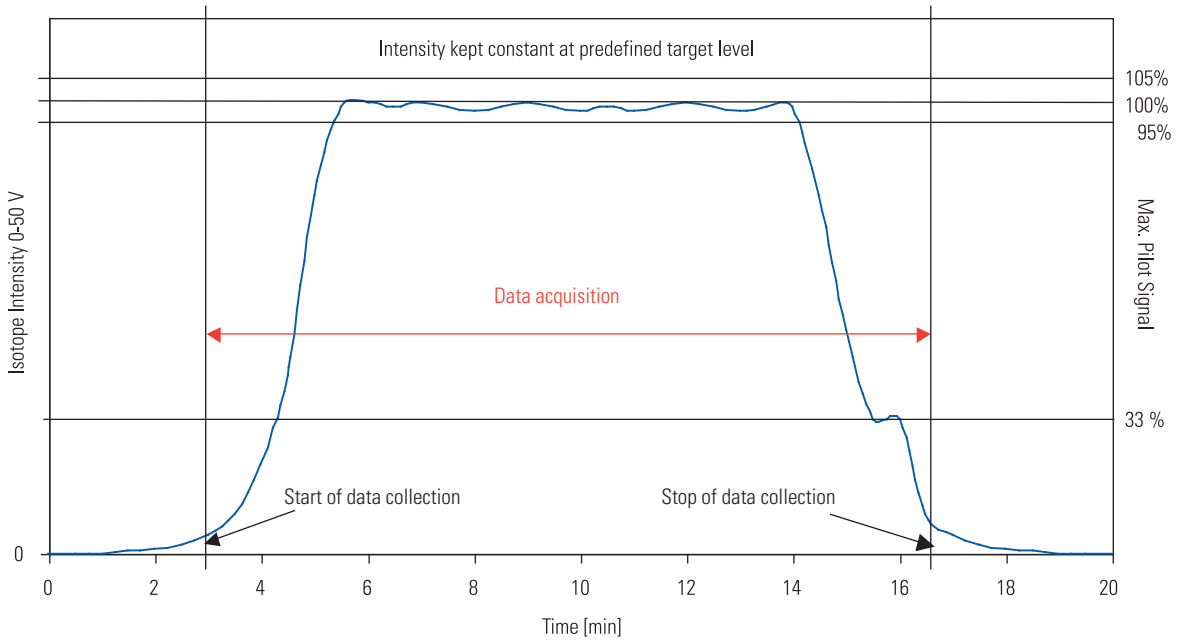

Figure 1. Schematic Acquisition Procedure of the Total Sample Evaporation Process.

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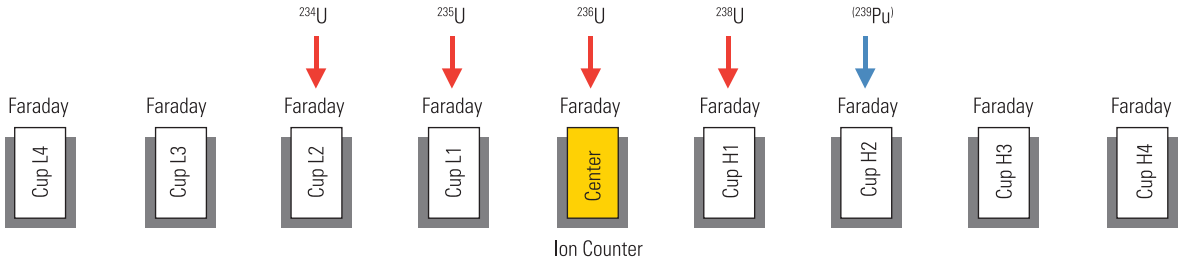
Schematic of the Multi-Collector Configuration



	RATIO			ATOM %			
RUN #	**$^{234}U/^{238}U$**	**$^{235}U/^{238}U$**	**$^{236}U/^{238}U$**	**^{234}U**	**^{235}U**	**^{236}U**	**^{238}U**
1	0.0041415	0.5465275	0.0025227	0.2666	35.1874	0.1624	64.3836
2	0.0039345	0.5465337	0.0026784	0.2533	35.1888	0.1725	64.3854
3	0.0039067	0.5461105	0.0027085	0.2516	35.1711	0.1744	64.4029
4	0.0038790	0.5466193	0.0025322	0.2498	35.1969	0.1630	64.3902
5	0.0039102	0.5466133	0.0024973	0.2518	35.1968	0.1608	64.3906
6	0.0040146	0.5469418	0.0024464	0.2584	35.2093	0.1575	64.3748
7	0.0038969	0.5461063	0.0024487	0.2510	35.1770	0.1577	64.4142
8	0.0036091	0.5468670	0.0025562	0.2324	35.2129	0.1646	64.3902
9	0.0039858	0.5466242	0.0024713	0.2566	35.1961	0.1591	64.3881
10	0.0039341	0.5464046	0.0027161	0.2533	35.1826	0.1749	64.3892
11	0.0038804	0.5461045	0.0026687	0.2499	35.1723	0.1719	64.4059
MEAN	0.0039175	0.5464957	0.0025679	0.2522	35.1901	0.1653	64.3923
SD	0.0001278	0.0002909	0.0001053	0.0082	0.0139	0.0068	0.0112
RSD %	3.3	0.053	4.1	3.3	0.039	4.1	0.017
		NBS Certified Standard Values:		0.2498 (6)	35.1900 (350)	0.1673 (5)	64.3930 (360)

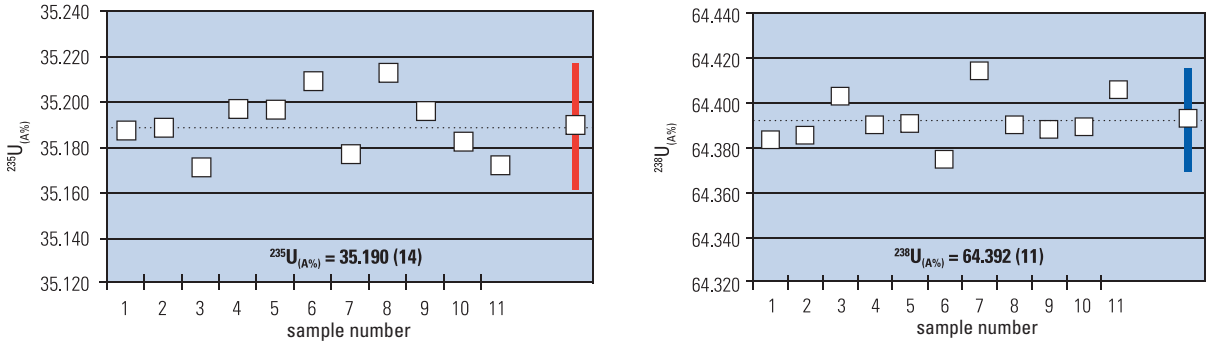


Figure 2. U isotope abundances in SRM U350. 11 runs and their means are plotted, together with the 2SD uncertainties on the means. NBS certified values are shown for reference as dotted lines.

Parameters:

Samples and Loading:
11 loadings of SRM U350, 1 ng each, samples diluted in nitric acid, analyses performed during acceptance tests in a user's laboratory.
Rhenium double filament units used.

Sample Warm-up and Acquisition:
Initial heating slope 40 mA/cycle, different heating slopes at different intensity levels, integration time 4 x 1 s/cycle, analyses finished when intensity dropped below threshold. About 100 data per sample acquired.

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