astrum

Reproducibility | Nickel Alloy

GD-MS

Application Note **AN75**

Analysis using Astrum GD-MS: Precision and accuracy over 4 years repeat analysis of a Ni alloy reference material

Instrumentation

The Astrum is the latest generation of GD-MS instruments developed to be the benchmark in this category. The instrument was designed in conjunction with the users of the most widely deployed GD-MS, the VG9000. The best design concepts of the VG9000 were combined with advances in sample cell design, control electronics and pumping technology to produce a high performance instrument designed specifically for ultra-trace analysis of impurities with excellent reproducibility of data and ability to run a wide range of matrices.

Method

Certified Reference Material BAS 346A is a widely used and well characterised nickel alloy with more than a dozen certified trace elements as well as information values on all matrix elements, plus a few others. The sample was run on the Astrum in the flat configuration under standard settings (see Table 1). Multiple runs spread over 4 years on different instruments were performed by different users and using different batches of the reference sample.

To mimic daily use of the instrument in a commercial environment, the source and sample holders were changed regularly and cleaned. The surface exposed to the glow discharge was also refreshed by alternate use of three cut pieces of BAS 346A, wet grinding of the surface with SiC grit #60 then #240, followed by de-ionised water and methanol surface cleaning. After each GD source change, the ion counter efficiency was checked by measuring ^{180}Ta and ^{181}Ta on the ion counter and faraday respectively with a high purity Ta sample. Subsequently, the detector cross-calibration factor was adjusted to allow for up to 11 orders of magnitude linear ion counts detection.

For each set of runs, the routine setup consisted in tuning for maximum sensitivity (using ^{58}Ni) and target resolution (~4000 using M/Δm). No calibration of the instrument sensitivity was performed, and results shown use the standard set of published Relative Sensitivity Factors (RSFs)[1].

[1]*Vieth, W. and Huneke, J. C. (1991) Spectrochimica Acta 46B (2), p.137-153.*

Parameter	Value
Discharge voltage	1kV
Discharge current	2mA
Ar gas flow	0.2-0.45 sccm
Acquisition	Four peak-width window, 80 channels, IC: 80ms/chan. Faraday: 80ms/chan.
Repeat measurements	4-8
Max peak intensity (^{58}Ni)	2-3 E-10 A
Resolution	4000

Table 1: Instrument parameters for flat sample analysis

Results

Results using standard RSFs for certified elements are given in Table 2, with a graphical representation of ^{209}Bi in Figure 1.

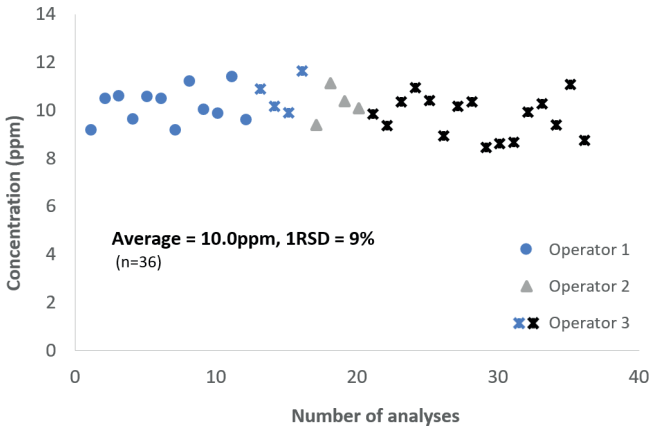

Figure 1: Repeat measurements of BAS 346A for ^{209}Bi using standard set of RSFs. Three different instruments (Blue, Grey, Black data points) were used by three different operators (see legend) over approximately 4 years.

Element	Mean	1SD	RSD%
Mg	119	10	8
Zn	37.1	1.6	4
Ga	66.4	3.4	5
As	31.0	1.2	4
Se	5.2	0.6	12
Ag	30.6	1.4	4
Cd*	0.38	0.13	35

Element	Mean	1SD	RSD%
Sn	77.6	5.0	6
Sb	36.9	2.1	6
Te	6.6	0.6	9
Tl	1.0	0.1	9
Pb	14.3	1.3	9
Bi	10.0	0.9	9

^{111}Cd is used for Cd measurement and has a significant interference close to analyte peak, hindering integration.

Table 2: Long term reproducibility of CRM BAS 346A measurements on three instruments by three operators using standard RSFs (n=36 apart from Se and Te were n=24).

To achieve the additional accuracy of the results, and highlight the robustness of the instrument, data collected by operator 2 (Japan, 4 analysis points) were used to generate a new set of RSFs and applied to all data acquired, including the remaining two instruments and operators. Fully quantitative results are shown in Table 3, including the certified values.

Element	Mean	Certified
Mg	134	130
Zn	31.3	28.8
Ga	51.6	49.6
As	52.0	50.4
Se	6.3	5.7
Ag	43.8	42.5

Element	Mean	Certified
Sn	93	93
Sb	45	45
Te	10.1	9.3
Tl	1.8	1.9
Pb	20.7	22.2
Bi	10.2	10.3

Table 3: Quantitative data (ppm) for BAS 346A runs using operator 2 data for calibration. n=36 apart from Se and Te were n=24.

Discussion

The Astrum GD-MS exhibits excellent reproducibility even when different instruments, users and reference material batches are used. The data acquired over almost 4 years only required tuning for sensitivity and checking resolution, without the need to calibrate the instrument to produce high quality data. Additional accuracy (to ±10%) is achieved by using a single set of data that can be applied universally to all three instruments and operators.