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Astrum Note **GD07**

Trace level analysis of non-metallic elements using the Astrum GD-MS

Instrumentation

The Nu 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 for both conducting and non-conducting samples.

Image: Astrum ES

It is recognised that the glow discharge source ionises non-metal elements, for example carbon, nitrogen, oxygen, sulfur, phosphorus and even halogens. By coupling a low-flow type GD source (glow gas <1 sccm) with sample and source cryo-cooling capabilities (about -180° C), high purity glow gas (99.9999%) and an online gas purifier (SAES getter), very low gas background levels can be achieved in the ion source chamber which enables trace gaseous elements to be quantified in the µg/g range.

Trace Oxygen in Steel

Samples used for this work are listed in the table below (Table 1) with their respective certified concentrations and information values. Figure 1 shows the data for all samples with oxygen, including information values for the NIST samples, in ion beam ratio concentration (IBR, all RSFs = 1). The correlation is based on the JSS and JSAC samples only.

SRM	O (wt ppm)
JSS (The Iron and Steel Institute of Japan)	
GS-1a	87
GS-1c	46.0 ± 3.8
GS-2b	14.6 ± 3.4
GS-3b	38.5 ± 2.4
GS-5a	136 ± 9
JSAC (The Japan Society for Analytical Chemistry)	
JSAC0111	2.3 ± 0.4
NIST (National Institute of Standards and Technology)	
661	(9)
662	(11)
663	(7)
664	(17)
665	(63)

Table 1: List of reference materials and certified values used for oxygen analysis. Information values are given in brackets.

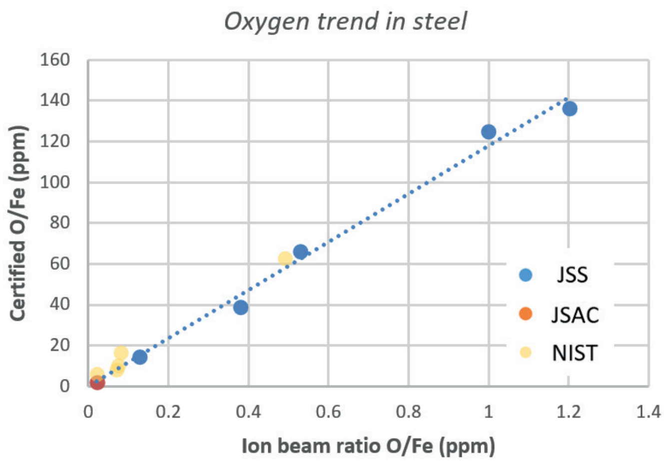

Figure 1: Oxygen trend in steel samples using JSS and JSAC samples. NIST results are information only.

Reproducibility of data was also investigated by preparing three pins of the reference materials listed in Table 1 with identical shape and analysing them twice, about one year apart, on the same instrument. Results for the reproducibility test are shown in Figure 2.

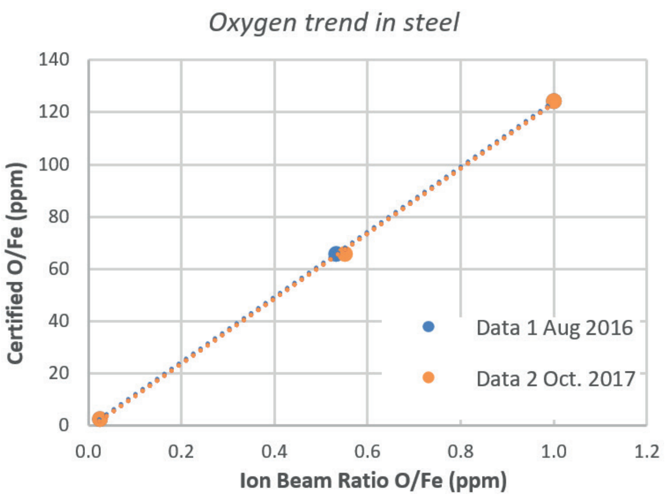

Figure 2: Reproducibility test by running JSAC 0111, GS-1c and GS-1a samples in August 2016 and October 2017.

Results shown in Figure 1 display a good correlation between the certified reference values and instrument response. The RSF for oxygen in steel is about 120 based on this data. Additionally, running the same samples more than one year apart, as shown in Figure 2, displays excellent data overlap, suggesting that minimal calibration is required to obtain reproducible data over time.

In both Figures 1 & 2, the calibration line is close to cross the origin, suggesting trace levels of oxygen can be reached. An estimate of background equivalent concentration (BEC) is measured by using NIST SRM 685w, high purity gold. The IBR on O: Au shows to be around 0.02 ppm, and using the oxygen RSF of 120 and gold RSF of 2.6, the oxygen BEC for is about 1 ppm using the Astrum GD-MS.

Trace Sulphur and Carbon in Steel

A different set of reference materials was used to determine trace levels of C and S in steel samples. The materials used are listed in Table 2, with results shown in Figures 3 to 5.

SRM	C (wt ppm)	S (wt ppm)
NIST (National Institute of Standards and Technology)		
NIST1765	60 ± 10	38 ± 3
NIST1766	150 ± 10	24 ± 2
NIST1767	520 ± 3	90 ± 3
NIST1768	10 ± 2	3 ± 1
NIST1264a	8710 ± 50	250 ± 10

Table 2: SRM used for carbon and sulphur analysis.

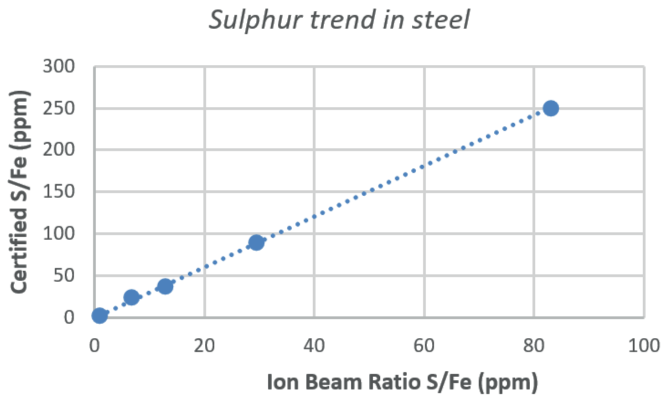

Figure 3: Sulphur trend in steel sample.

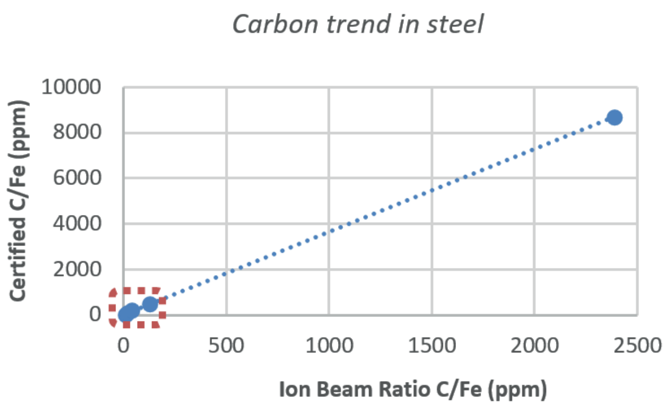

Figure 4: Carbon trend in steel sample. Area highlighted in red is expanded in Figure 5.

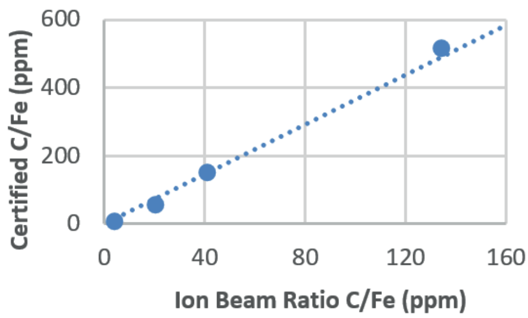

Figure 5: Closed-in view of carbon calibration curve for the lower calibration values near the origin.

The data for both sulphur and carbon display excellent correlation over the calibration range, and RSFs are ~3.0 and ~3.65 for S and C, respectively.

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Trace Hydrogen in Titanium

A similar approach was taken for the analysis of hydrogen in titanium matrix samples. A list of reference materials and certified values used are given in Table 3. Figure 6 shows the correlation between the certified and measured data.

SRM	H (wt %)
JTS (The Japan Titanium Society)	
Pure-Ti (TO-6)	0.0019
Pure-Ti (TO-3)	0.00055
6 Al-4 V (TO-5)	0.0039

Table 3: List of standard reference materials used to measure hydrogen in titanium samples.

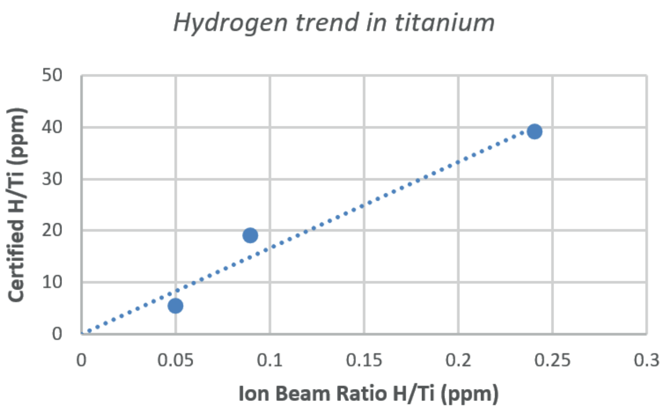

Figure 6: Correlation between certified and IBR data for hydrogen in titanium sample.

Discussion

The Nu Astrum GD-MS shows potential to measure trace levels of oxygen, carbon, sulphur and even hydrogen in metal matrices. This is a combination of low-flow source technology, together with effective cryo-cooling and high purity glow gas introduction system used. For all calibration curves, the intercept can be forced through the origin and with minimal effect to the data quality, highlighting very low background equivalent concentrations for the elements of interest in this note.