THE NU ATTOM HIGH RESOLUTION ICP-MS: Linearity of the Unique Extended Dynamic Range Detector System

INTRODUCTION

High resolution instruments such as the Attom from Nu Instruments are a powerful tool for the fast quantification of a large number of elements at varying concentrations in a variety of sample materials. Generally, samples analysed with such instruments can have very low concentrations of trace and ultra-trace elements but at the same time, some of the major elements may be of interest too. Therefore, it is important to be able to analyse these in the same sample dilution without prior knowledge of the concentrations with a versatile detector system which is able to automatically cope with the varying signal intensities.

The default detector system commonly used is an electron multiplier operating in pulse counting mode. This is able to cope with ion beams of up to 2-3 million counts per second (cps). For larger beam sizes, instead of using a dual-mode multiplier in "analogue" mode, the Attom uses a unique attenuation system. This deflects the ion beam through a grid that attenuates the ion beam by a pre-determined factor so that it can still be detected in pulse counting mode. This yields a dynamic range of the pulse counting electron multiplier of up to 9 orders of magnitude. An optional Faraday is available to further extend the dynamic range by up to 3 orders. A general graph of the three detector stages is shown in Figure 1.

The switchover between these different detector stages is fast and automatic. The switchover is based on the signal intensity exceeding a pre-defined threshold for each stage. Furthermore, the crossover calibration needs to be mass independent, linear between the three different detector stages and very stable over the course of analytical sessions (see AN17).

In this note we show examples of the linearity of the crossover calibration of all three detector stages for a selection of elements across the mass range.

Instrumentation

The Nu Attom is a double-focusing, high-resolution magnetic sector mass spectrometer. The instrument is entirely purpose designed and built to provide the best performance and reliability coupled with flexibility and ease-of-use for precise and accurate elemental and isotope ratio analysis. A unique detector system gives the Nu Attom a large dynamic range, and its electrostatic scanning capability has the widest range in its class (40%). Furthermore, the continuously variable high resolution means that sufficient resolution for isobaric separation can be selected with minimum compromise in sensitivity.

Experiment

An ICP standard solution containing Li, Be, Mg, Sc, Co, Y, In, Cs, Tb, Tm, Pb and U was used at concentrations ranging from 50 fg/g up to 20 µg/g to establish the linearity across the three different detector stages. A blank prepared from ultrapure 2% HNO_3 was subtracted from the standards before calculating the calibration graphs.

Prior to the analysis of the standard series, the cross calibration factor between the pulse counting and attenuated pulse counting mode was determined using a 1 ng/g tuning solution. This calibration establishes the amount that the ion beam is reduced when it is passed through the grid. It is determined by measuring the ion beam in normal (un-attenuated) and attenuated mode for a selection of elements across the mass range using the instrument tuning solution. Similarly, the cross calibration factor between the attenuated pulse counting mode and the Faraday detector mode was determined using a 250 ng/g instrument tuning solution. These two factors are then automatically applied to the data.

The crossover factors derived from the detector cross calibration were as follows:

- *Pulse-counting versus attenuated pulse counting: 420*
- *Attenuated pulse counting versus Faraday: 0.85*

The blank corrected data are shown in Table 1. The colour coding applied to the intensity data is comparable to Figure 1, such that blue data has been acquired in the pulse counting, orange data in attenuated pulse counting and red data in Faraday mode.

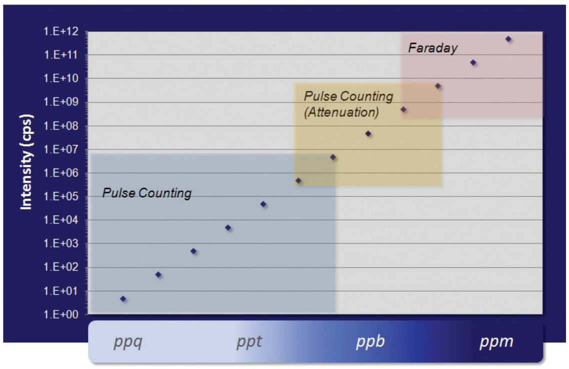

Figure 1: *Detector modes for the Attom HR-ICP-MS*

Discussion

The results are shown for a selection of elements from the above list in graphs of concentration versus signal intensity in linear and logarithmic scale, respectively (Figure 2). The data in the logarithmic scale graph is colour coded in the same way as the different detector modes in Figure 1 and the data in Table 1. This has been done to easily identify the detector modes that were used to acquire the data.

It can be seen that the linear fit applied to each full data set yields R^2 of >0.999. Furthermore, in the logarithmic scale plot, no prominent non-linearity can be observed even at the crossover areas of the different detector modes.

Conclusions

The Nu Attom is a high resolution ICP-MS that is ideal for the most demanding ICP-MS requirements where sensitivity, precision, and speed of analysis are paramount. The results of this experiment highlight that the unique extended dynamic range detector system is suitable for the analysis of samples with varying concentrations in a range of materials.

Concentration	Signal Intensity (counts per second [cps], Blank corrected)											
µg/g	Li(7) cps	Be(9) cps	Mg(24) cps	Sc(45) cps	Co(59) cps	Y(89) cps	In(115) cps	Cs(133) cps	Tb(159) cps	Tm(169) cps	Pb(208) cps	U(238) cps
0.00005	25097	6495		45037	53906	54944	60352	51056	72737	80575	28332	61915
0.0001	52421	13221	29324	95115	109410	112051	123292	107725	146823	160627	61601	124991
0.0002	70896	25524	68740	178682	214049	219173	240826	208133	292277	321816	126464	246160
0.0005	197700	65327	265638	461622	548795	565030	615000	544000	746392	816992	331392	631372
0.001	390548	133934	607013	945661	1134911	1148864	1285179	1136599	1554863	1708011	664092	1292988
0.002	931296	293140	1337815	2106372	2536982	2606879	2822637	2521745	3438191	3764881	1533764	2833831
0.005	2066064	657590	2949898	4512947	5462894	5621475	6013013	5464700	7337246	8108145	3242683	6271388
0.01	4203057	1363537	6140664	9516985	11250283	11655646	12425790	11230567	15428533	16929513	6782770	12860344
0.02	8270318	2686690	11867461	18564846	22085469	22594932	24417321	21937151	29621607	32419802	13084639	25222035
0.05	20700749	6692166	30423171	47191283	55778546	56481549	61848192	56620340	75781499	71260579	33869983	63246300
0.1	43229746	14163503	63645799	98453160	124397683	123296877	137933287	121393094	164002735	182032082	68336945	142197085
0.5	234452948	67524398	293215746	469151798	589103259	596191176	666928293	589016897	809496909	905002535	382545378	712577859
1	421697018	126802968	517123362	826059829	1037272214	1058509112	1180005696	1044614067	1468131949	1636858549	687526651	1286672741
2	826746956	253440764	1006324361	1624327620	2024959480	2097928588	2349949516	2081911196	2926980263	3282473405	1361743256	2600670868
5	2025873048	614093854	2466499142	4011702776	4944513163	5154108363	5708563749	5067788225	7204836068	8032551433	3262570347	6395692716
10	4162811250	1233076376	4997398018	8212888793	9990393313	10414876153	11436247893	10271517938	14548156917	16139917225	6408949872	12988233290
20	8350467305	2406557442	9831043462	16436983674	19487896434	20236037202	22140835908	20296954891	28560953422	31227151932	12218874966	25709051018

Table 1:
Blank corrected intensity data for the standard series. Data shown in blue = pulse counting mode, orange = attenuated pulse counting mode, red = Faraday mode.

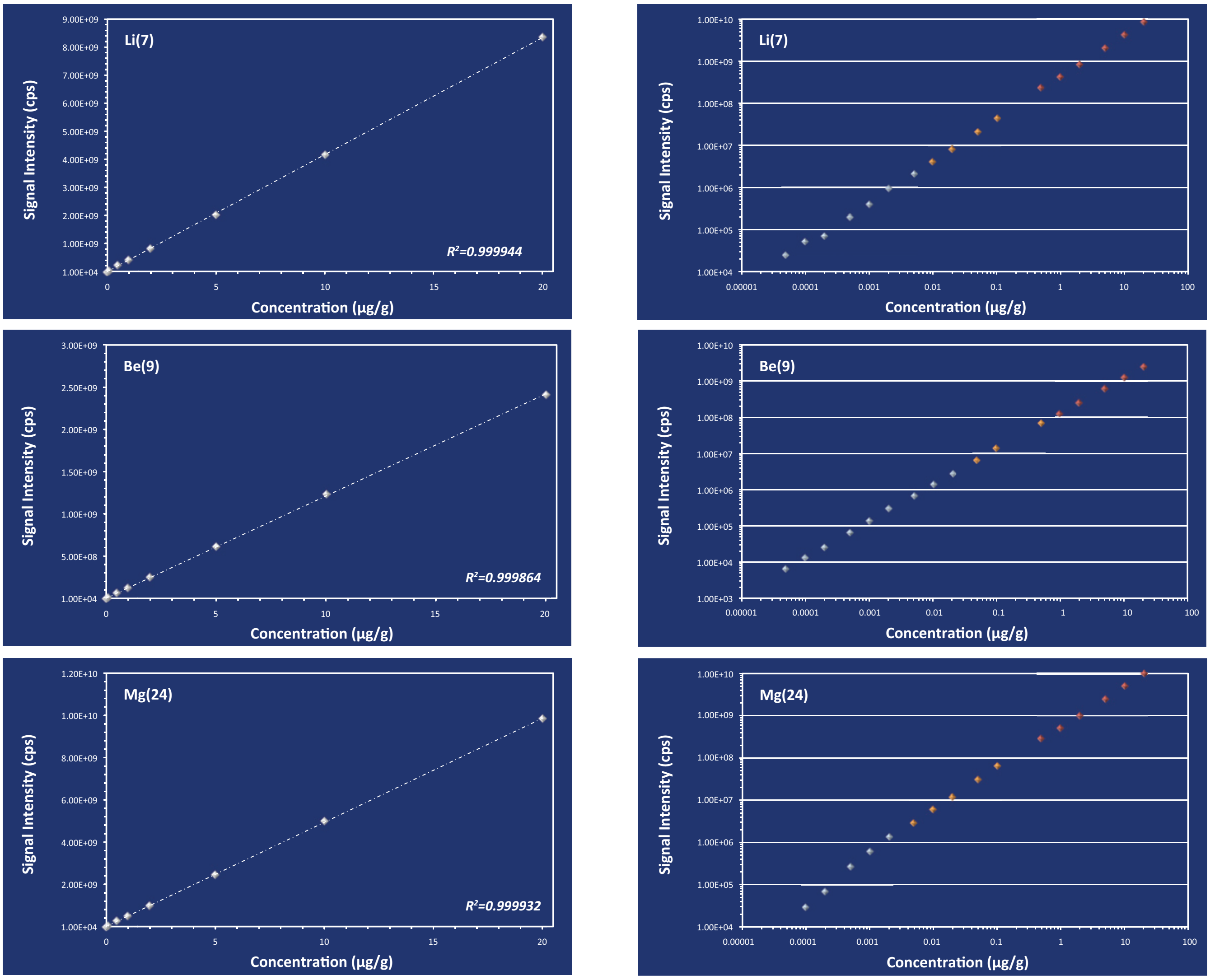

Figure 2: *Calibration graphs for blank corrected data of each element in linear (with linear regression line, left) and logarithmic scale (right).*

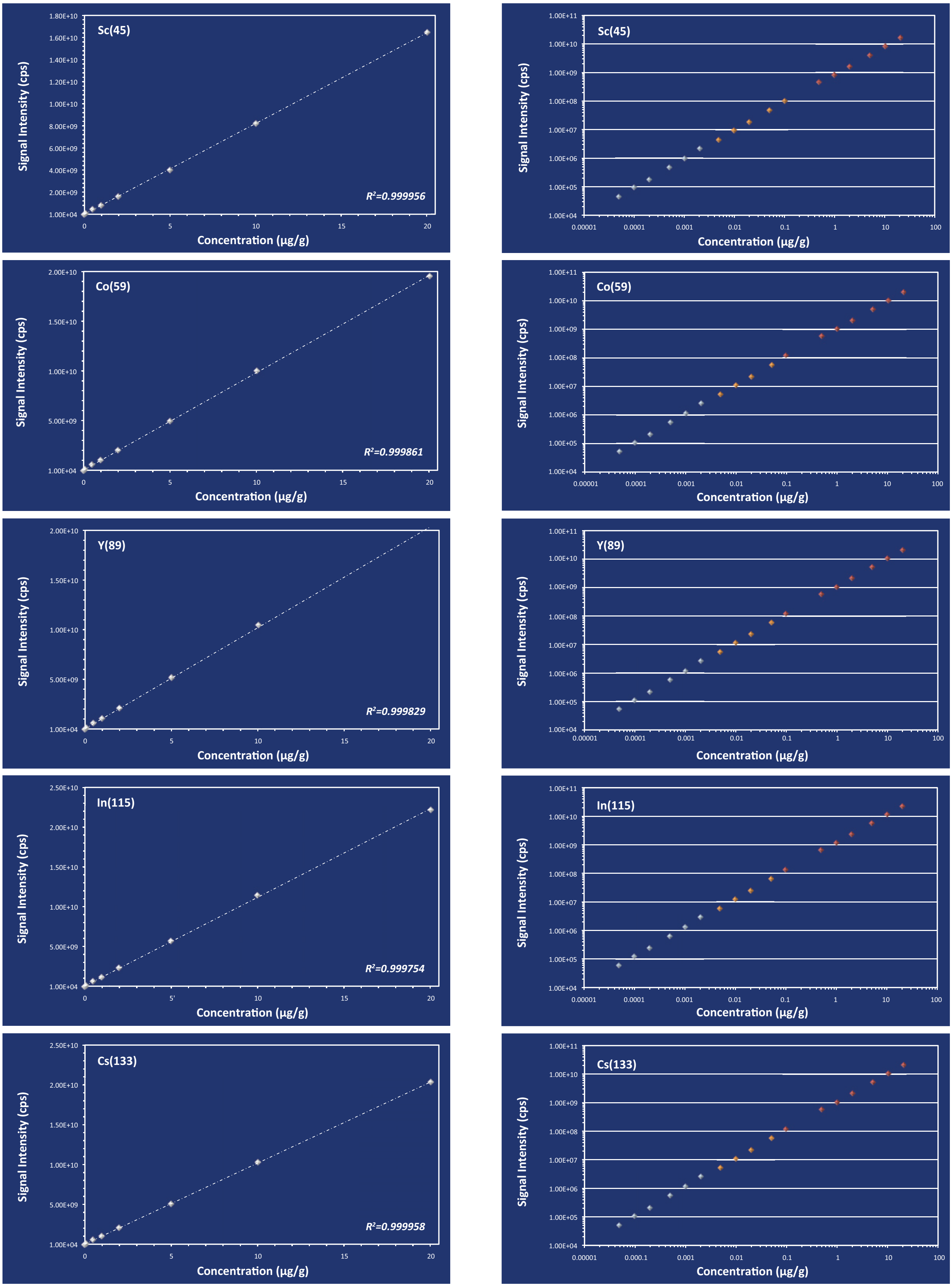

Figure 2 (continued): *Calibration graphs for blank corrected data of each element in linear (with linear regression line, left) and logarithmic scale (right).*

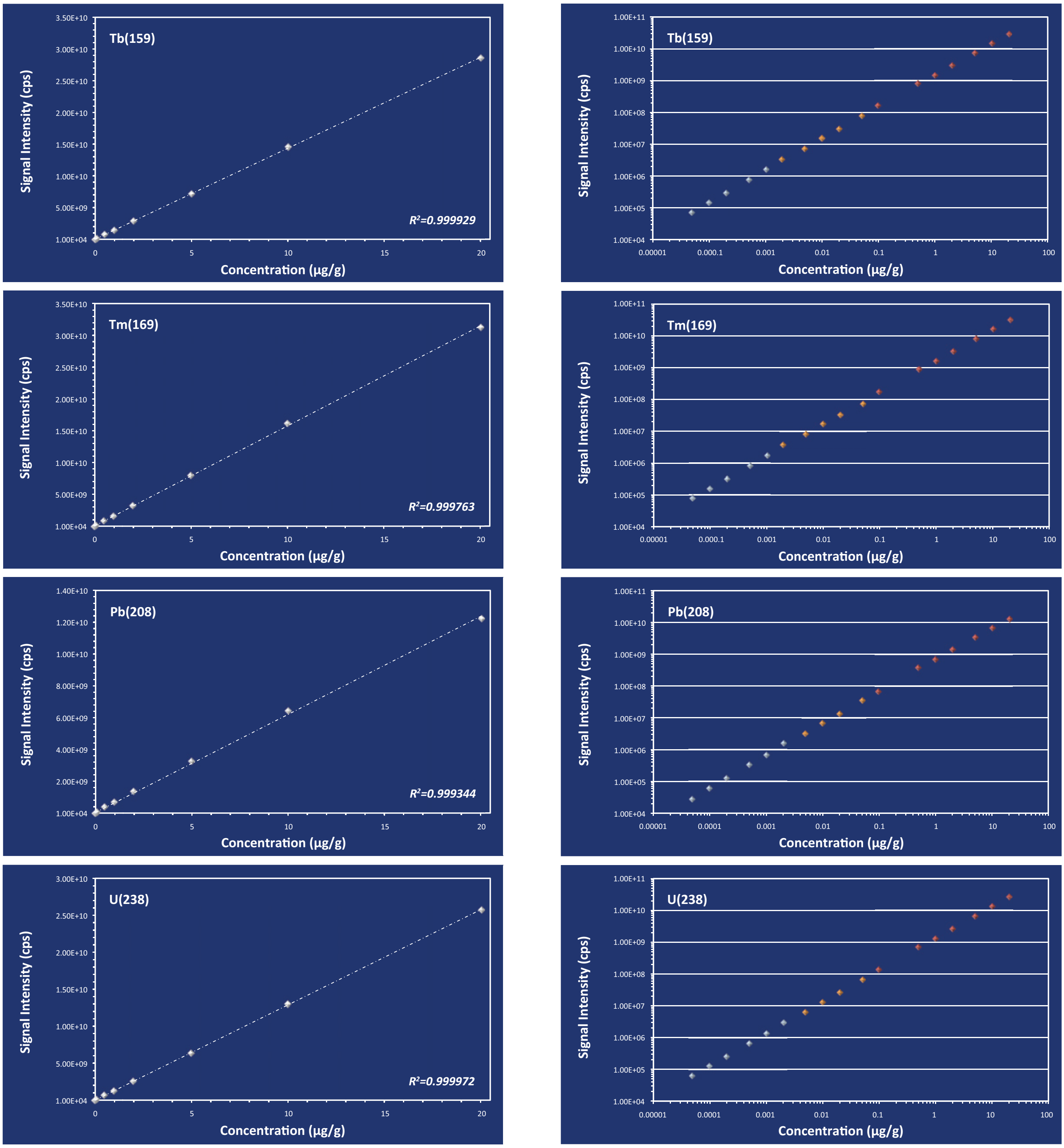

Figure 2 (continued): *Calibration graphs for blank corrected data of each element in linear (with linear regression line, left) and logarithmic scale (right).*