



QA/QC report for Tellus Border samples analysed at Activation Laboratories Ltd  
using 1C-Expl (QOP-PGE-ICPMS)

Prepared for the Geological Survey of Ireland

**by**

Emmanuel Esemé, Nancy Spoelstra, Owen Rigby and Luis Antonio

October 2013

## Summary on data reports

4142 samples received from the Geological Survey of Ireland were analysed by code 1C-Expl (fire assay with lead button collection and ICP-MS finish) and reported according to batches as summarized in Table 1 below.

**Table 1: Batch code used in reporting data for samples with corresponding batch names, dates received, number of samples in reports, number of reports issued and dates**

Batch Code	Batch Name	Date received	Number of Samples	# of reports issued	Date reported	Revised report date
A13-06036	TELLUS BORDER-1	05/30/2013	418	2	06/17/2013	07/17/2013
A13-06058	TELLUS BORDER-2	05/30/2013	416	2	06/14/2013	07/17/2013
A13-06079	TELLUS BORDER-3	05/30/2013	419	2	06/14/2013	07/17/2013
A13-06093	TELLUS BORDER-4	05/30/2013	422	2	06/19/2013	07/17/2013
A13-06166	TELLUS BORDER-5	05/30/2013	419	2	06/19/2013	07/17/2013
A13-06168	TELLUS BORDER-6	05/30/2013	419	2	06/17/2013	07/17/2013
A13-06171	TELLUS BORDER-7	05/30/2013	416	3	06/20/2013	07/17/2013 07/24/2013
A13-06174	TELLUS BORDER-8	05/30/2013	420	2	06/24/2013	07/17/2013
A13-06178	TELLUS BORDER-9	05/30/2013	420	2	06/20/2013	07/17/2013
A13-06182	TELLUS BORDER-10	05/30/2013	373	2	06/24/2013	07/17/2013

All sample batches were reported initially using the LONMIN template to show dates and times as requested. Following observation that serial dates and times were required instead of those shown in reports using the LONMIN template; revised reports were issued on July 17<sup>th</sup> after creation of the Geological Survey of Ireland (GSI) template for reporting. A third report was issued on July 24<sup>th</sup> for A13-06171 because some blanks in the second report were showing weights in the QC portion of the revised report issued on July 17<sup>th</sup>.

## Sample preparation, analysis and quality control

Samples were received as pulps and sent by the preparation department to fire assay. In the fire assay department, samples were weighed and weights recorded. Samples weights varied from less than 1 g to a maximum of 10 g. All samples including low weight samples were weighed as received in batches of 35. Two certified reference materials (1 standard for every 18 samples) and two blanks were added to each batch. Due to the limited amount of material received, duplicates usually inserted for analyses using this method were not available. The crucibles were numbered sequentially for each batch and samples introduced into the oven for fusion.

Samples were fused initially at 850°C for 30 minutes to avoid contamination, and then followed by fusion at 950°C for 15 minutes and finally 1060°C for 15 minutes. No repeats were performed

indicating there was no problem with the fusion of samples. The slag was separated from the lead buttons and cupels numbered based on crucible numbers. The lead buttons were cupped at 950°C for about 40 minutes and resulting beads transferred into glass tubes numbered according to cupels and sent to the ICP-MS department for instrumental analysis.

In the ICP-MS department, the Ag beads including blanks, samples and standards were digested in hot (95°C) HNO<sub>3</sub> + HCl acids and 4mL of the solution allowed to cool for 2 hours. 0.5 mL of the solution from digestion was then diluted to a final volume of 10 mL using an aqua regia solution. The aqua regia solution consisted of 3.5 mL trace metal grade HCl mixed with 9mL trace metal grade HNO<sub>3</sub> and diluted with distilled water to 2000 mL. The dilution of the solution (0.5 mL) from bead digestion to 10 mL using this aqua regia solution then yielded a 2.4 % aqua regia solution (3:1 v/v HCl:HNO<sub>3</sub>). Diluted samples were then run on a Perkin Elmer Sciex ELAN 6000 or 9000 ICP-MS for Au, Pd and Pt as summarised in Table 2 below.

**Table 2: Range of samples run concurrently on 2 ICP-MS instruments for the Tellus Border samples**

Batch Code	Batch Name	Range of samples run on ELAN 9000	Range of samples run on ELAN 6000
A13-06036	TELLUS BORDER-1	584501C to 584961C	None
A13-06058	TELLUS BORDER-2	584962C to 585119C and 585265C to 585418C	585120C to 585264C
A13-06079	TELLUS BORDER-3	585569C to 585717C	585419C to 585568C and 585718C to 585858C
A13-06093	TELLUS BORDER-4	585859C to 586153C and 586231C to 586305C	586154C to 586230C
A13-06166	TELLUS BORDER-5	586306C to 586527C and 586600C to 586672C	586528C to 586599C and 586673C to 586745C
A13-06168	TELLUS BORDER-6	586819C to 586891C and 587038C to 587111C	586745C to 586818C, 586893C to 587037C and 587112C to 587184C
A13-06171	TELLUS BORDER-7	587411C to 587482C and 587558C to 587630C	587185C to 587410C and 587483C to 587557C
A13-06174	TELLUS BORDER-8	587852C to 587922C	587631C to 587851C and 587923C to 588070C
A13-06178	TELLUS BORDER-9	588071C to 588146C and 588219C to 588440C	588147C to 588218C and 588293C to 588367C
A13-06182	TELLUS BORDER-10	588514C to 588659C and 588734C to 588900C	588660C to 588733C

A six points plus blank calibration was run with a second source calibration check solution. Instruments were recalibrated after every 70 GSI samples (2 racks of samples). Each rack of samples consisted of 42 beads that included 2 blanks, 2 certified reference materials, 3 duplicate samples and 35 samples from the Geological Survey of Ireland. Due to the absence of material for duplicates for samples submitted, duplicates during analysis of samples were empty vials. Blanks were analyzed first, followed by the samples and then the digested standards. Two washes were inserted between

digested standards and same standards were run in succession. Sample weights and volumes were entered into the instrument software for final calculations.

Three certified reference materials were used for quality control. This included CDN PGM 23 produced by CDN Resources Laboratories as well as Oreas 45d and 45e produced by Ore Research and Exploration Pty. CDN PGMS 23 was produced from a mixture of Pd/Pt ore and Au concentrate ([www.cdnlabs.com](http://www.cdnlabs.com)). Oreas 45d was produced from a 50:50 blend of mineralised lateritic soil and barren soil, while Oreas 45e was prepared from a mineralised lateritic soil ([www.ore.com.au](http://www.ore.com.au)). Control charts (shown below) were set up based on historical recoveries of the standards. The y-axis represents measured values of standards run with samples while the x-axis represents consecutive standards from various batches of samples analysed. Normally, any standard that is outside of the mean  $\pm$  3 standard deviations is investigated. The standard is repeated alongside the samples in the batch to verify if it is an issue related to the instrument. If repeat data is similar to original data but still outside  $\pm$  3 standard deviations, a re-fire is requested. As seen from the control charts presented below, no standard showed values outside the upper/lower control limit (ucl/lcl) and hence no re-fires were required (Note that there was no material for re-fires even if re-fires were required).

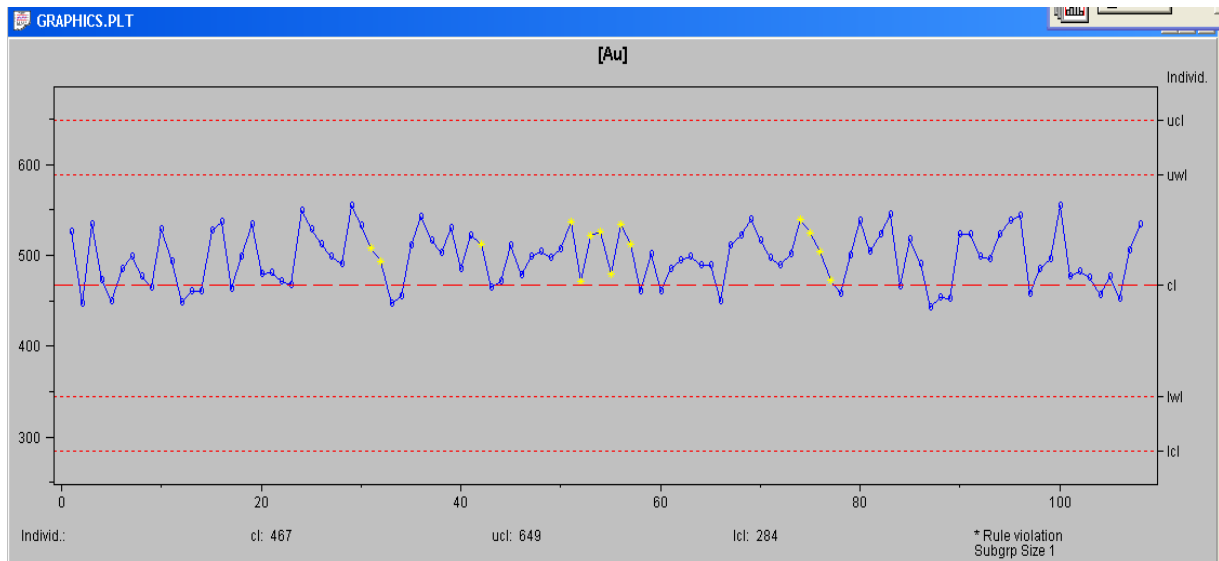


Fig.1 a: Variation in Au for CDN-PGMS 23 reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

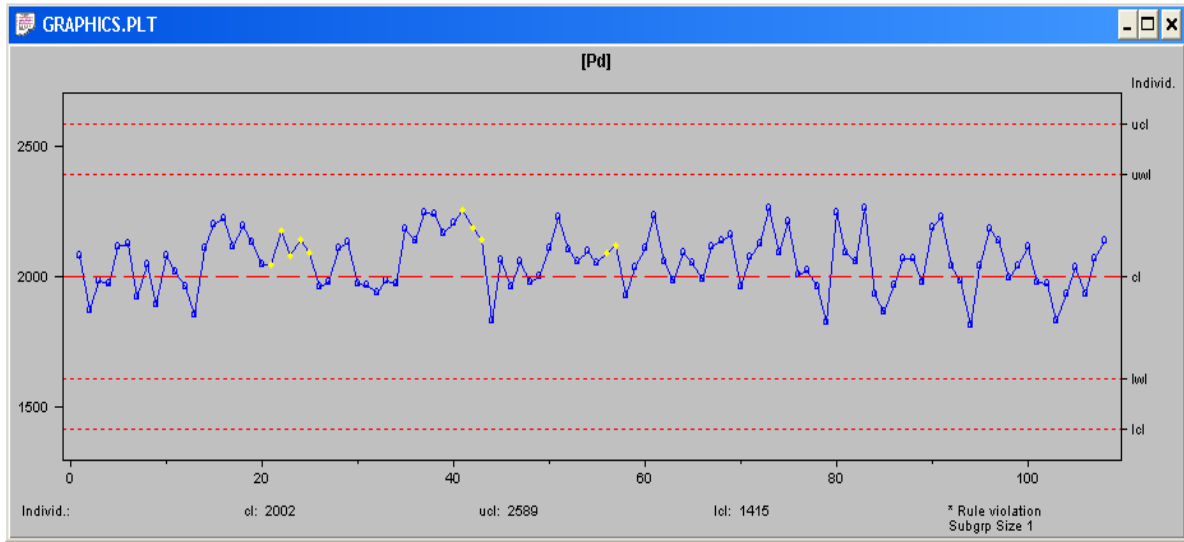


Fig.1 b: Variation in Pd for CDN-PGMS 23 reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

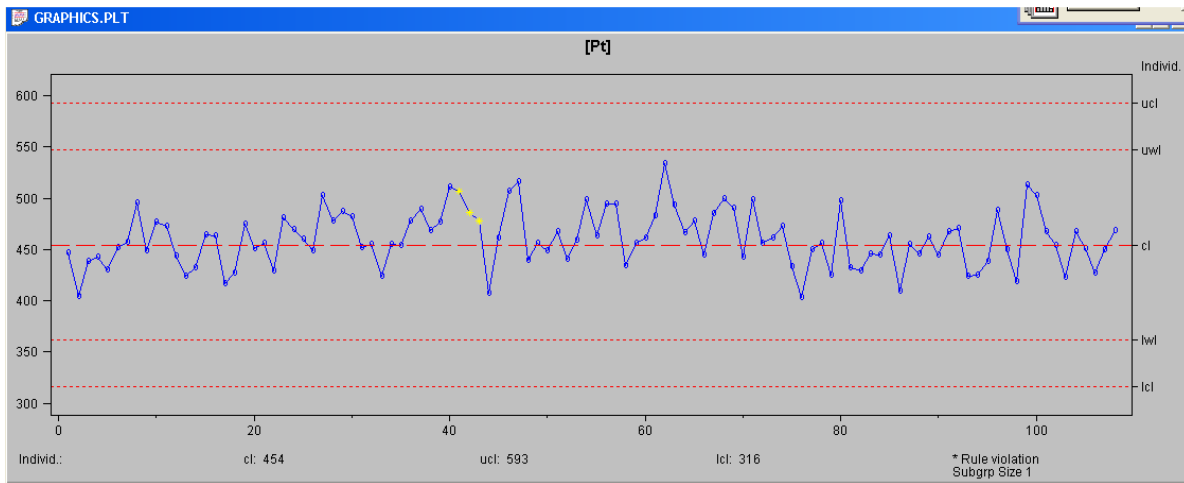


Fig.1 c: Variation in Pt for CDN-PGMS 23 reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

Table 2 below shows the certified values for Au, Pd and Pt for CDN-PGMS 23 along with mean and +/- 2 and +/- 3 standard deviations data for all standards analysed with samples from the Geological Survey of Ireland

**Table 3: Certified and mean values including standard deviations for CDN-PGMS 23 used in control charts**

<b>Mean</b>	1995.1	452.3	464.6
<b>+3x standard deviation</b>	2542.3	577.2	637.0
<b>-3x standard deviation</b>	1447.8	327.3	292.2
<b>+2x standard deviation</b>	2359.9	535.6	579.6
<b>-2x standard deviation</b>	1630.2	369.0	349.7
<b>Analyte symbol</b>	<b>Pd (ppb)</b>	<b>Pt (ppb)</b>	<b>Au (ppb)</b>
<b>CDN PGMS 23 Cert</b>	<b>2032 ± 166</b>	<b>456 ± 40</b>	<b>496 ± 58</b>

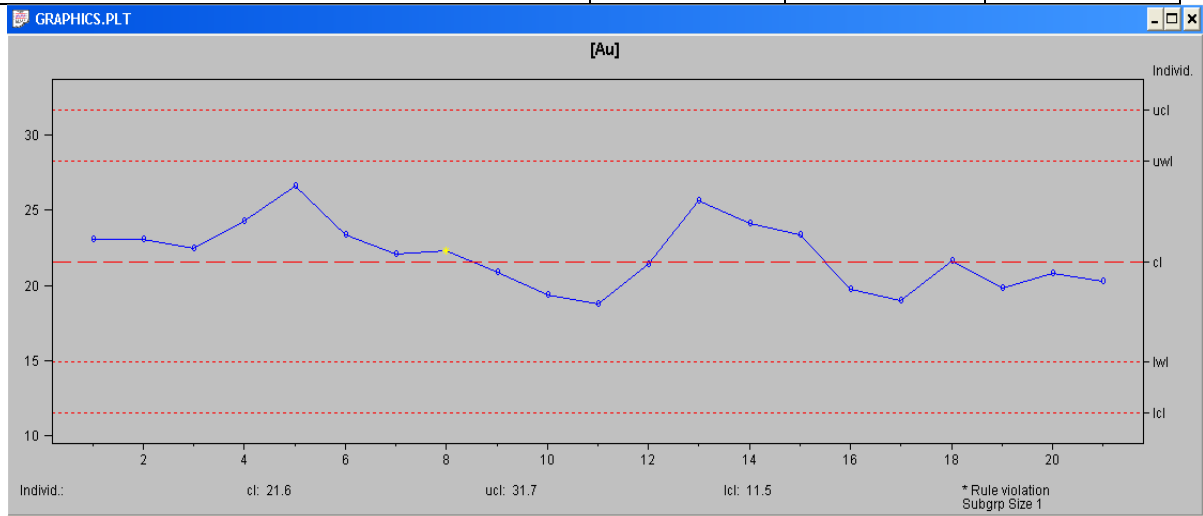


Fig.2 a: Variation in Au for Oreas 45d reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

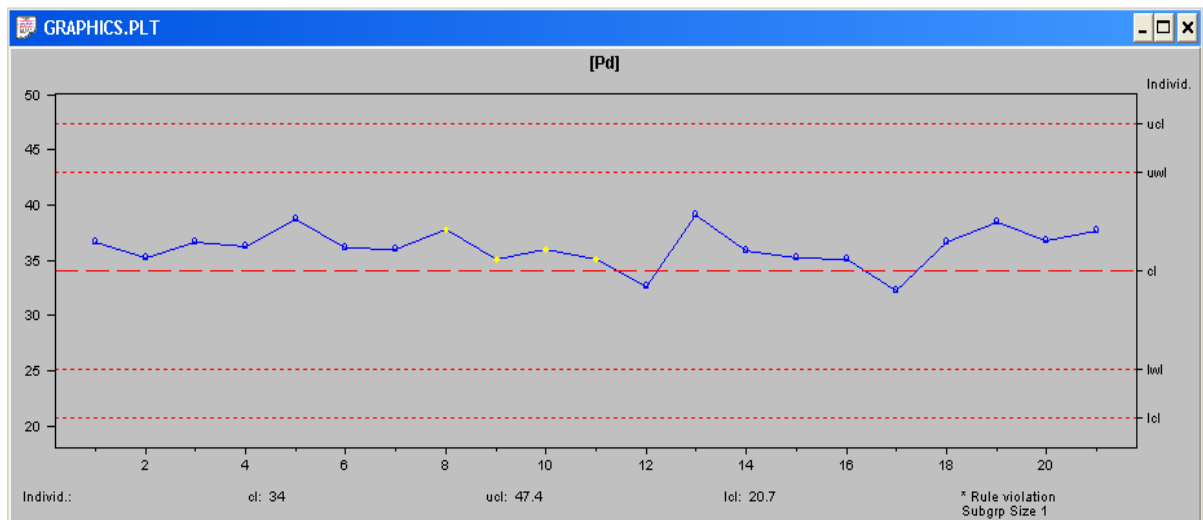


Fig.2 b: Variation in Pd for Oreas 45d reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

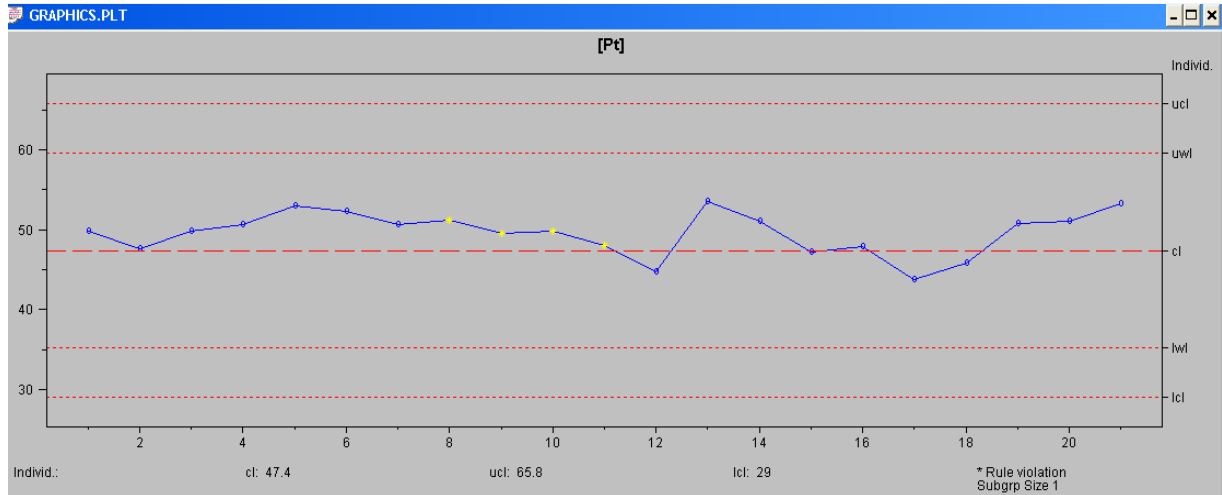


Fig.2 c: Variation in Pt for Oreas 45d reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

Table 3 below shows the certified values for Au, Pd and Pt for Oreas 45d along with mean and +/- 2 and +/- 3 standard deviations data for all standards analysed with samples from the Geological Survey of Ireland

**Table 4: Certified and mean values including standard deviations for Oreas 45d used in control charts**

<b>Mean</b>	34.7	48.4	22.0
<b>+3x standard deviation</b>	40.5	55.7	29.0
<b>-3x standard deviation</b>	29.0	41.1	15.1
<b>+2x standard deviation</b>	38.6	53.2	26.6
<b>-2x standard deviation</b>	30.9	43.5	17.4
<b>Analyte symbol</b>	<b>Pd (ppb)</b>	<b>Pt (ppb)</b>	<b>Au (ppb)</b>
<b>OREAS 45d Cert</b>	<b>35 ± 4</b>	<b>48 ± 6</b>	<b>23 ± 4</b>

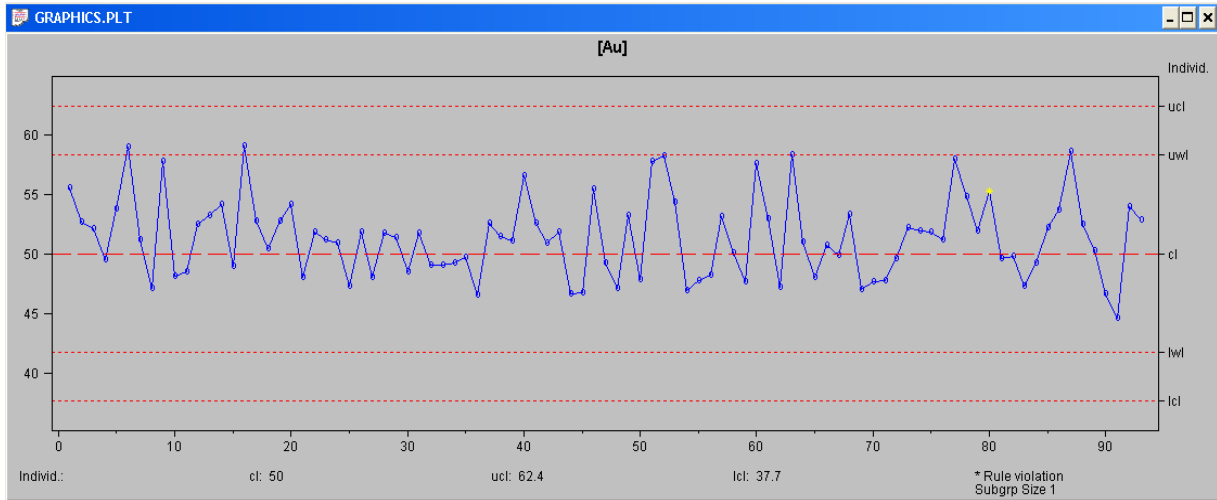


Fig.3 a: Variation in Au for Oreas 45e reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

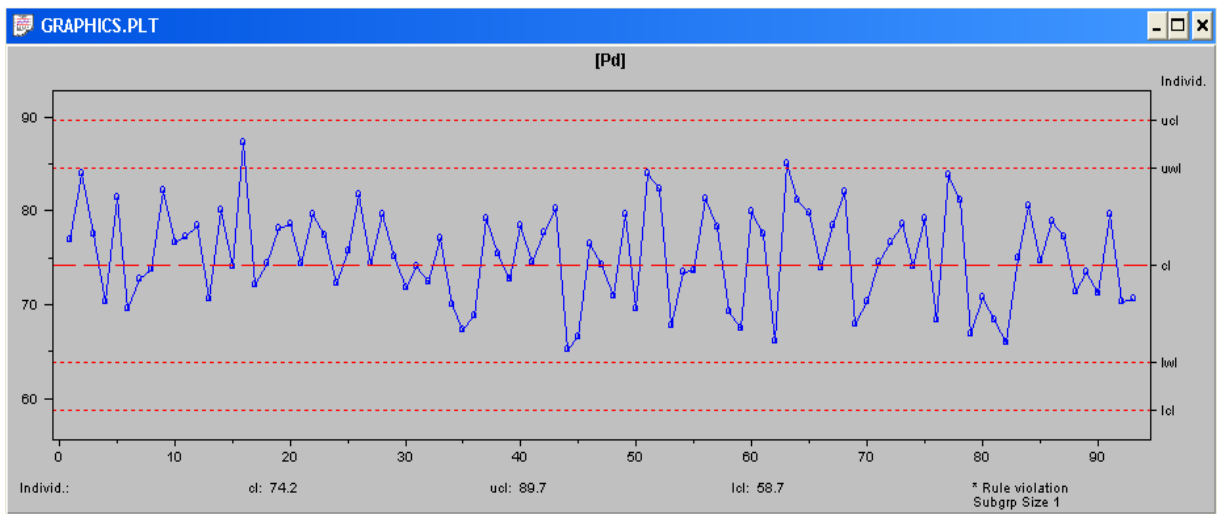


Fig.3 b: Variation in Pd for Oreas 45e reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.



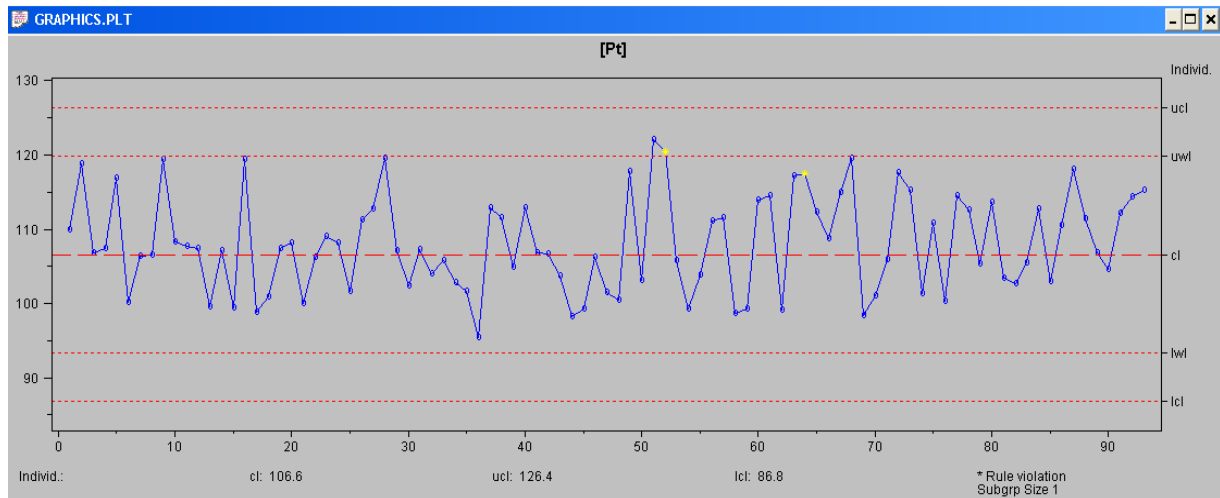


Fig.3 c: Variation in Pt for Oreas 45e reported for all reports for samples from the Geological Survey of Ireland showing data for standards as well as the mean, upper/lower warning limits and upper/lower control limits; where ucl is the upper control limit, uwl is the upper warning limit, lcl is the lower control limit and lwl is the lower warning limit.

Table 4 below shows the certified values for Au, Pd and Pt for Oreas 45e along with mean and +/- 2 and +/- 3 standard deviations data for all standards analysed with samples from the Geological Survey of Ireland

**Table 5: Certified and mean values including standard deviations for CDN-PGMS 23 used in control charts**

<b>Mean</b>	74.2	106.6	50.0
<b>+3x standard deviation</b>	89.7	126.4	62.4
<b>-3x standard deviation</b>	58.7	86.8	37.7
<b>+2x standard deviation</b>	84.5	119.8	58.3
<b>-2x standard deviation</b>	63.8	93.4	41.8
<b>Analyte symbol</b>	<b>Pd (ppb)</b>	<b>Pt (ppb)</b>	<b>Au (ppb)</b>
<b>OREAS 45e Cert</b>	<b>75 ± 10</b>	<b>110 ± 12</b>	<b>53 ± 6</b>

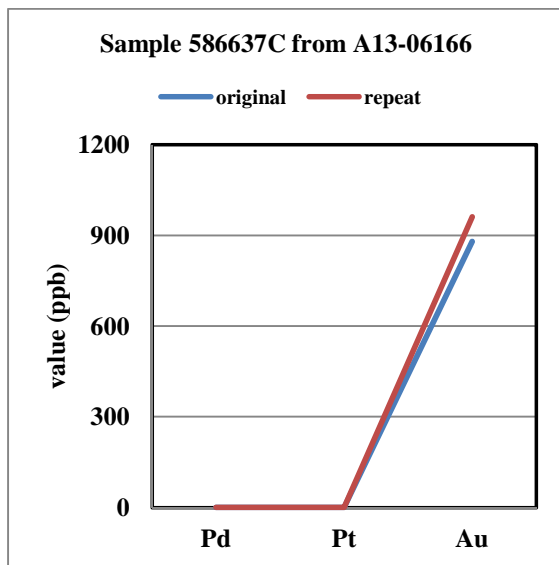
For the 222 standards (CDN PGMS 23 – 108, Oreas 45d – 21 and Oreas 45e – 93) reported in the quality control sections of reports for samples from the Geological survey of Ireland, none had any value for the three analytes that was outside 3 standard deviations of the mean values (upper/lower control limits). In fact over 99% of values were within 2 standard deviations of the mean (below the upper/lower warning limits).

The error associated with the reported data can be summarized as:

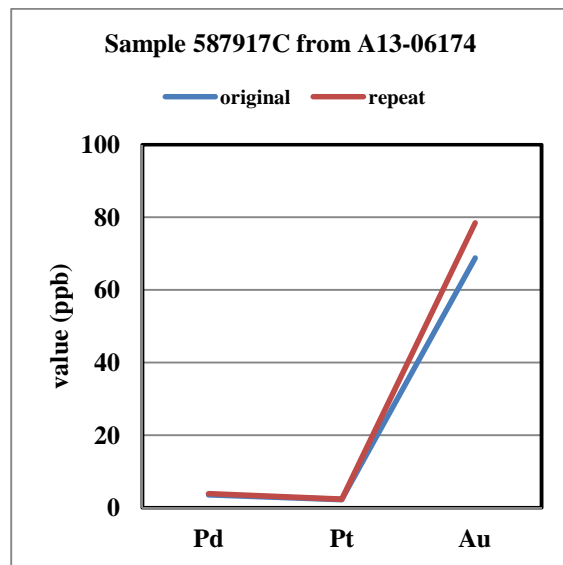
± 100 % at the reporting limit (~ 5 ppb), ± 10 % at 10x the reporting limit (~50 ppb) and ± 5 % at 100x the reporting limit (~500 ppb).

## Repeat analyses

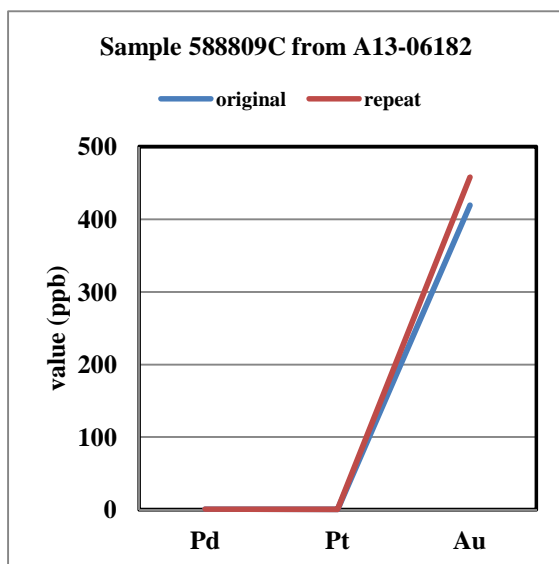
On August 9<sup>th</sup>, an email was sent listing select samples from A13-06036, A13-06079, A13-06166, A13-06168 and A13-06182 for which data reported was indicated to be higher or lower than expected. These samples were re-analysed from original solutions since there was no material available for re-assay. The relation between the original and repeat analyses data for these samples is shown in various charts below. As summarized in an email sent on August 15<sup>th</sup>, no significant difference was noted between the two sets of data. The repeat data confirmed the reliability of data originally reported.



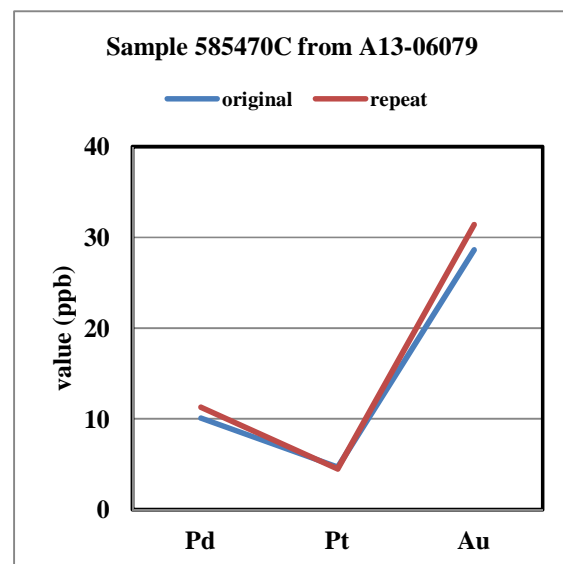
a



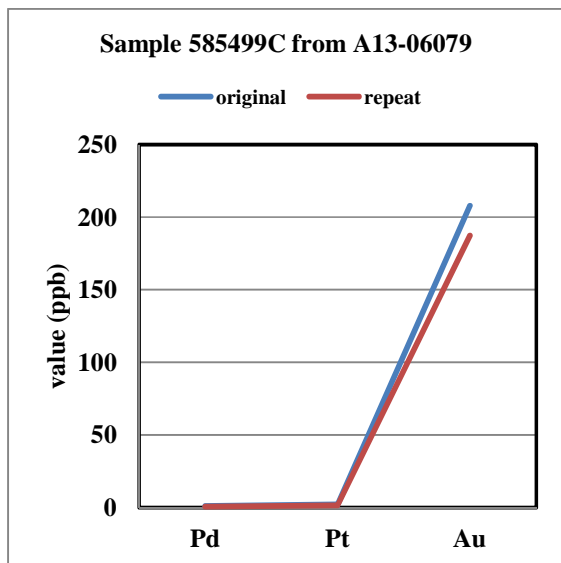
b



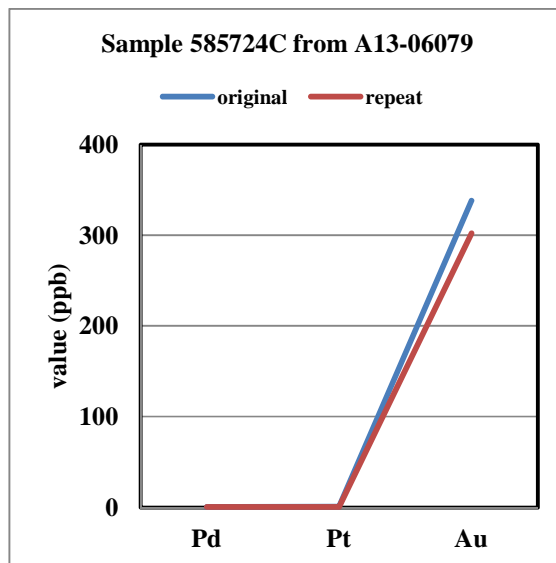
c



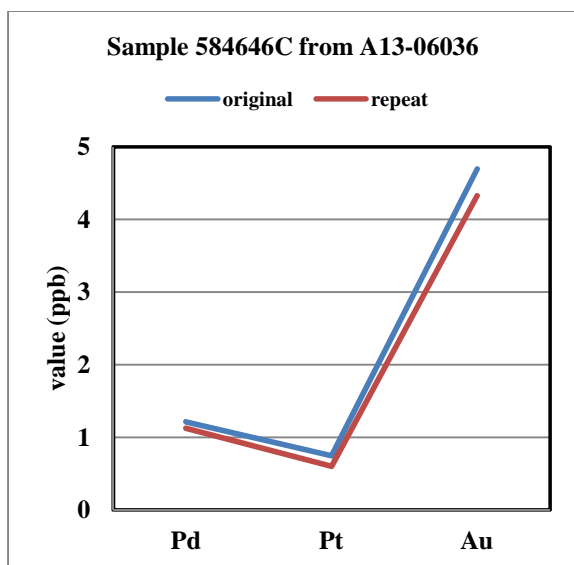
d



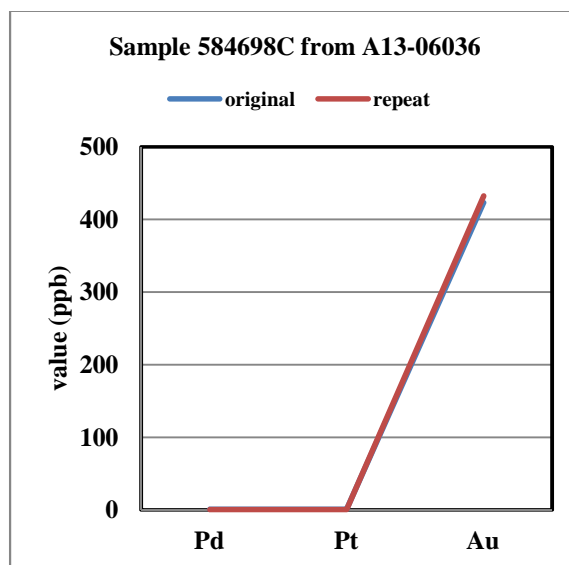
e



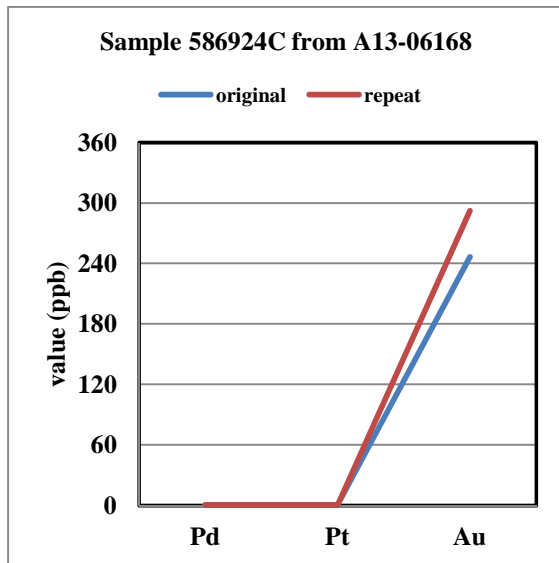
f



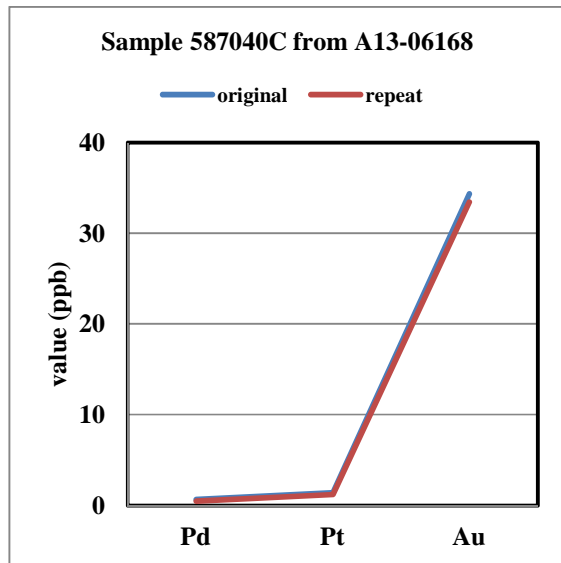
g



h



i



j

Figures 4a-j: Line graphs showing relation between original and repeat analysis data for 10 samples selected from A13-06036, A13-06079, A13-06166, A13-06168, A13-06174 and A13-06182.

\*A summary of the method used in analysing samples is provided for your reference.

## 1C - Exp 2 - Fire Assay -Au, Pd, Pt- ICP/MS

---

A sample size of 5 to 50 grams can be used but the routine 30 g size is applied for rock pulps, soils or sediments (exploration samples). The sample is mixed with fire assay fluxes (borax, soda ash, silica, litharge) and with Ag added as a collector and the mixture is placed in a fire clay crucible, the mixture is preheated at 850°C, intermediate 950 °C and finish 1060 °C, the entire fusion process should last 60 minutes. After cooling, the lead button is separated from the slag and cupelled at 950°C to recover the Ag (doré bead) + Au, Pt, Pd.

The Ag doré bead is digested in hot (95°C) HNO<sub>3</sub> + HCl. After cooling for 2 hours the sample solution is analyzed for Au, Pt, Pd by Perkin Elmer Sciex ELAN 6000, 6100 or 9000 ICP/MS. A blank and a digested standard are run every 15 samples. Instrument is recalibrated every 45 samples. Duplicates are run when sample duplicates are received by the ICP/MS department.

Smaller sample splits are used for high chromite or sulphide samples to ensure proper fluxing and metal recoveries.

If values exceed upper limits, reanalysis by fire assay Au, Pt, Pd (Code 8) is recommended.

### Code 1C-Exploration Elements and Detection Limits (ppb)

Element	Detection Limit	Upper Limit
Au	1	30,000
Pt	0.5	30,000
Pd	0.5	30,000

### References:

Hoffman, Eric L. and Dunn, Bernie, 2002. Sample Preparation and Bulk Analytical Methods for PGE.CIM Special Volume 54 The Geology, Geochemistry and Mineral Beneficiation of Platinum Group Elements Edited by Louis J. Cabri, pp.1-11.

Hoffman, Eric L., Clark, John R. and Yeager, James R., 1998. Gold Analysis – Fire Assaying and Alternative Methods. *Explor. Mining Geology*, Volume 7, Nos. 1 and 2, pp. 155-160.