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Gold Homogeneity in Certified Reference Materials; A Comparison of Five Manufacturers

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INTRODUCTION

Certified Reference Materials (CRMs) inserted into analytical batches are a requirement by the international codes governing the mineral industry (JORC 2012; NI43-101; SAMREC) and reporting to the standards laid out in these codes is a mandatory compliance for publicly listed companies on the Australian, New Zealand and Canadian Stock Exchanges. A CRM is a sample where the concentrations of one or more analytes have been quantified by valid methodologies and certified with valid documentation. These certified values are the consensus inter-laboratory mean for an analyte where typically each laboratory analyses multiple subsamples of the CRM.

For gold (Au) CRMs, one of the key properties is the homogeneity of the CRM sample (i.e. testing for nugget effects). Quantifying this homogeneity by the Relative Standard Deviation (RSD) is critical for assessment of laboratory results and the follow up of quality control (QC) failures.

There is vast literature discussing the nugget effect in gold mineralising systems and protocols in reducing the sampling error (e.g., Stanley & Smee 2007), but very little information has been published on the homogeneity of gold CRMs. By their very nature CRMs are assumed to be homogeneous and any variation is attributed to laboratory error (i.e. the variance attributed to the sampling error is less than the analytical error); so how homogenous are commercial gold CRMs?

The homogeneity of a gold CRM sample reflects the capability and competency of a manufacturer to eliminate any nugget effects and provide a homogeneous product that, when analysed, will provide a repeatable result within the statistical limits provided on the CRM certificate. Although all manufactures refer to the homogeneity of their CRMs, only one manufacturer (OREAS), measures the homogeneity of the CRMs and provides this information as routine with their gold CRM Certificate of Analysis. Rocklabs undertake a homogeneity and segregation test, but do not provide the actual homogeneity results.

This independent study evaluates the homogeneity of gold CRMs from commercial CRM manufacturers at four chosen gold grades (0.5 ppm Au, 1 ppm Au, 3 ppm Au and 9 ppm Au). These grades are typical in mining and exploration scenarios and reliable QC data at these grades is critical. This study provides a benchmark for further evaluations of potentially “nuggetty” CRM products including, but not restricted to, platinum group elements (PGEs), rare earth elements (REE), and Au.

CRM MANUFACTURERS AND THEIR PREPARATION

Four auriferous CRMS from each of five manufacturers were assessed (i.e. 20 CRMs in total). The CRMs were sourced from five manufacturers, as listed below alphabetically. A summary description of the preparation process is also provided and taken from their CRM certificates:

- African Mineral Standards (AMIS): http://www.amis.co.za/. The material was crushed, dry-milled and air-classified to <54 microns. Wet sieve particle size analysis of random samples confirmed the material was 98.5% <54 microns. It was then blended in a bi-conical mixer, systematically divided and then sealed into1 kg Laboratory Packs.
- CDN Resource Laboratories Ltd (CDN): http://www.cdnlabs.com/. Material was dried, crushed, pulverized and then passed through a 270 mesh screen. The +270 mesh material was discarded. The -270 mesh (53 microns) material was mixed for 5 days in a double-cone blender.
- Geostats Pty Ltd (GST): http://www.geostats.com.au/. All CRMs are dried in an oven for a minimum of 12 hours at 110 °C. The dry material is then pulverised to finer than 75 microns (nominal mean of 45 microns) using an air classifier. The material is then homogenised and stored in a sealed, stable container ready for final packaging.
- Ore Research and Exploration Pty Ltd (ORE): http://www.ore.com.au/. Materials are jaw crushed to minus 3 mm, dried to constant mass at 105 °C, barren materials are milled to >98% minus 75 microns and gold bearing material milled to 100% minus 20-30 microns, blending in appropriate proportions to achieve the desired grade, packaging into 60 g and 100 g units in laminated foil pouches and 1 kg units in plastic jars.
- Rocklabs (RLB): http://rocklabs.com/. Pulverized feldspar minerals, basalt rock and barren ironpyrites were blended with finely divided gold containing minerals that have been screened to ensure there is no nuggetty gold. (NOTE no sizing information provided).

See also Table 1 for a summary.

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Table 1: Summary of attributes of the various manufacturer's CRM as provided on their certificates.

Manufacturer	Material grain size	Homogeneity tested	Lab list provided	Statistical metrics	ISO accreditation	Major and trace element data
AMIS	< 54 um	Stated but details not provided	Yes	SD, Between-lab SD, Within-Lab SD, Combined Standard Uncertainty	Yes	Majors - certified; Traces - indicative
CDN	< 53 um	No	Yes	Between-lab SD	No	Majors only - indicative
Geostats	< 75 um	No	No	SD, 95% Confidence Interval	Yes	Majors & Traces - indicative
OREAS	< 30 µm	Yes (INAA subsample method)	Yes	SD, 95% Confidence limits, Tolerance limits	Yes	Majors & Traces - indicative
Rocklabs	Not specified	Yes (specific sampling/testing regime incl segregation test)	Yes	Between-lab SD, 95% Confidence limits	No	Majors only - indicative

Note: All manufacturers use multiple laboratory round robin for the certification process of gold by fire assay.

CRMS SELECTED

To allow a comparison between CRM manufactures four (4) auriferous grade ranges were chosen centred around 0.5 ppm; 1 ppm; 3 ppm and 9 ppm (see Table 2). The selection of CRMs from these grade ranges were determined by the availability of materials from the manufacturers, and preference given to CRMs that are siliceous in nature and manufactured within the last six years (to minimise any variation due to change in the manufacturing process and/or possible oxidation of sulphides). To this end, low sulphur samples were chosen where available.

Table 2: Summary of CRMs chosen for this study.

Manufacturer	CRM	Certified Au (ppm)	Assay Method	Brief Material description	S (%)	SiO_2 (%)	Year of release	# labs
AMIS	AMIS0352	0.45	Fire assay	Andesitic-dacite tuffaceous agglomerate	0.56	62.13	2012	23
	AMIS0310	1.03		Basalt, volcanics & granite	1.58	69.38	2012	17
	AMIS0360	2.94		BIF, mafic volcanics and sediments	6.46	48.05	2014	24
	AMIS0267	9.05		Qtz-carbonate-adularia	0.75	83.27	2012	19
CDN	CND-GS-P5C	0.571	30 g Fire assay	Granitic	0.2	60.7	2014	15
	CND-GS-1M	1.07		Granitic	0.1	65.6	2013	15
	CND-GS-3L	3.18		Granitic	0.1	66.8	2013	15
	CND-GS-8C	8.59		Sourced from Cortez Hills Mine	0.6	56.6	2013	13
Geostats	G909-6	0.57	50 g Fire assay	Composite Gold Ores low sulphide	nr	nr	2009	132
	G313-1	1		Composite Mine Ore	0.035	64.56	2014	157
	G914-6	3.21		High Grade low sulphide ore	0.06	63.69	2015	179
	G914-7	9.81		High Grade low sulphide ore	0.04	60.87	2015	178
OREAS	OREAS 201	0.514	30-50 g Fire assay	Basaltic	0.39	53.69	2012	20
	OREAS 204	1.043		Basaltic	0.794	52.64	2012	20
	OREAS 17c	3.04		Basaltic	1.59	49.1	2009	18
	OREAS 62c	8.79		Andesitic volcanics	0.53	60.9	2009	16
Rocklabs	SE68	0.599	30 g Fire assay	Feldspar, basalt & iron pyrites with minor fine gold minerals	2.3	54.76	2012	53
	SG66	1.086			2.6	54.52	2012	53
	SJ80	2.656			3	56.26	2013	54
	SN75	8.671			3.3	56.17	2013	54

nr = not reported

MATERIAL RECEIVED

Amounts corresponding to the minimum manufacturers order were purchased. To this end:

- Two x 100 g sachets of each of the four CRM were purchased from AMIS andreceived in vacuum sealed foil pouches;
- Four x 100 g sachets of each of the four CRMs from CDN and received in paper bags and sealed in plastic;
- Four x 100 g sachets of each of the four CRMs from Geostats and received in plastic bags;
- Four x 60 g sachets of each of the four CRMs from OREAS and received in foil pouches;
- One 2.5 kg plastic jar for each CRM were ordered and received from Rocklabs.

The materials as received are shown in Photo 1.

Photo 1. CRM's "as received" from the five manufacturers.

SAMPLE PREPARATION AND ANALYSIS

Prior to dispatching the materials for analysis, each CRM was subsampled twenty times in a clean room. A 10 g aliquot of each CRM was placed into a Ziploc® plastic bag using a disposable plastic spatula to avoid any cross contamination. This procedure was repeated for each CRM so that the samples were sequenced in lots of 20, with each lot corresponding to one CRM.

For the homogeneity test work, Instrumental Neutron Activation Analysis (INAA) was used. INAA is a highly precise and unique assay method that focuses on the elements nucleus irrespective of the sample matrix or chemical form of the element. Each sample is subjected to a flux of neutrons to produce radioactive nuclides. These nuclides decay emitting gamma rays that are characteristic for each nuclide. When compared with a known standard, the intensity of the emitted gamma rays can be quantified into an element concentration (Lieser 2001).

The 400 x 10 g subsamples were dispatched to Activation Laboratories Limited (Actlabs) in Ancaster, Canada (www.actlabs.com) for INAA; Actlabs were informed of the purpose of the analysis and requested to weigh out 1 g

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of material from each sample. To minimise any effects of between batch bias, Actlabs were requested not to split any of the 20 sample CRM lots and all possible sources of measurement error (e.g., weighing, counting, detector geometry, flux monitor errors, etc.) be kept to a minimum. Minimising the sources of error and ensuring no sample lots were split provides confidence that the results are a true reflection of CRM sampling errors and hence CRM homogeneity, and that the analytical precision errors are minor in comparison to sampling errors. By subjecting all the samples to the identical non-destructive analytical technique provided by one laboratory that required no sample preparation, reagents or digestion, any laboratory error is constant for all samples and considered minimal. Thus variance in the spread of analytical results from each of the CRMs will represent the degree of homogeneity.

RESULTS

The results of this exercise are presented in Appendix 1, which is available for download from the EXPLORE page of the AAG website (www.appliedgeochemists.org). The certificates of the CRMs used in this study are downloadable from the AAG website (www.appliedgeochemists.org) and the Geochemical Services website (http://www.gspty.com.au/).

Precision/homogeneity

The Certified Value (CV), Relative Standard Deviation (RSD across the 20 x 1 g INAA values of each CRM), Sampling Constant (which is the minimum required sample mass to achieve a 1% RSD), mean INAA sample mass analysed (g), two Relative Standard Deviations at typical fire assay charge weights of 30 g (30 g 2RSD) and 50 g (50 g 2RSD) for each CRM by manufacturer is presented in Table 3. A mean RSD for each manufacturer is also presented as an indication of overall performance.

Table 3: Relative Standard Deviation comparison for the various CRM Manufacturers (based on 20 x 1 g INAA determinations per CRM).

Manufacturer	CRM	Certified Value (ppm)	Mean INAA subsample (g)	1 g RSD INAA	Mean RSD	Sampling Constant (g)	Based on 1 gm INAA data and the Sampling Constant	
							30g 2RSD	50g 2RSD
AMIS	AMIS0352	0.450	1.05	4.48%	12.08%	21.0	1.67%	1.30%
	AMIS0310	1.03	1.06	12.80%		172.7	4.80%	3.72%
	AMIS0360	2.94	1.06	3.60%		13.7	1.35%	1.05%
	AMIS0267	9.05	1.05	27.46%		794.8	10.29%	7.97%
CDN	CDN-GS-P5C	0.571	1.06	10.27%	9.70%	111.8	3.86%	2.99%
	CDN-GS-1M	1.07	1.05	15.85%		263.4	5.93%	4.59%
	CDN-GS-3L	3.18	1.05	11.53%		140.0	4.32%	3.35%
	CDN-GS-8C	8.59	1.05	1.16%		1.4	0.43%	0.34%
Geostats	G909-6	0.570	1.06	2.93%	3.09%	9.1	1.10%	0.85%
	G313-1	1.00	1.04	6.14%		39.4	2.29%	1.78%
	G914-6	3.21	1.06	2.15%		4.9	0.81%	0.63%
	G914-7	9.81	1.05	1.15%		1.4	0.43%	0.33%
OREAS	OREAS 201	0.514	1.06	1.87%	1.61%	3.7	0.70%	0.54%
	OREAS 204	1.04	1.05	1.77%		3.3	0.66%	0.51%
	OREAS 17c	3.04	1.05	1.61%		2.7	0.60%	0.47%
	OREAS 62c	8.79	1.04	1.18%		1.4	0.44%	0.34%
Rocklabs	SE68	0.599	1.06	1.90%	2.35%	3.8	0.71%	0.55%
	SG66	1.09	1.06	5.05%		27.1	1.90%	1.47%
	SJ80	2.66	1.05	1.20%		1.5	0.45%	0.35%
	SN75	8.67	1.04	1.26%		1.7	0.47%	0.36%

NOTE: Sampling Constant is the required grams to achieve a 1% RSD.

The RSD is used as a standardised measure of dispersion that indicates the precision or repeatability of an assay. The lower the RSD, the more repeatable, precise or homogeneous the CRM; conversely the higher the RSD the less homogeneous the CRM. Given the critical importance of CRMs and their mandatory use in the mining and exploration industry, the lower the RSD determined from replicate analysis via the INAA method on reduced analytical subsamples (e.g. 1 g) the more homogenous the reference material and the greater confidence and control the QC officer has in vetting data quality from a laboratory. The overall mean RSD for each manufacturer has been derived from the mean of the individual RSD's of the 1 g INAA data. In order of increasing mean RSD (corresponding to decreasing homogeneity) they range from OREAS: 1.61%, to Rocklabs: 2.35%, to Geostats: 3.09%, to CDN: 9.70%, to AMIS: 12.08%.

The Sampling Constant (Ingamells & Switzer 1973) has been calculated to show the minimum sample mass required to measure gold in each CRM by the 1 g INAA method to achieve a relative standard deviation of 1%. For high grade gold samples (~9 ppm Au), the sampling constant ranges from a low of 1.4 g from CDN, Geostats and OREAS to 794.8 g for AMIS. This suggests AMIS0267 is influenced by a nugget effect.

The 30 and 50 g 2RSDs have been calculated from the Sampling Constants for each CRM and in some instances

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reveal significant sampling errors. Typical measurement error for 30 or 50 g fire assay charge weights are usually around 5% at commercial laboratories for ore grade methods. The 2RSD values encapsulate the sampling error contribution from the CRM and these can be added to the nominal 5% measurement error laboratories tend to achieve to get a feel for what the overall errors would be in reported analytical results. Results from individual manufacturers are discussed below:

AMIS: homogeneity for individual AMIS CRMs varies from 3.60% RSD (AMIS0360; 2.94 ppm Au) to 27.46% RSD (AMIS0267; 9.05 ppm Au), a spread of 23.86% RSD showing no systematic change in homogeneity with changing grade. The sampling constant for AMIS0267 indicates that 794.8 g of sample would be required to ensure a RSD of 1% during analysis, and for a 30 g fire assay a sampling error of 10.29% applies. At this charge mass only AMIS0352 (30 g 2RSD sampling error of 1.67%) and AMIS0360 (30 g 2RSD of 1.35%) would be fit for purpose CRMs based on these calculations. The gold homogeneity of CRMs produced by AMIS is considered very poor to good.

CDN: homogeneity for individual CDN CRMs varies from 1.16% RSD (CDN-GS-8C; 8.59 ppm Au) to 15.85% RSD (CDN-GS-1M; 1.07 ppm Au), a spread of 14.69% RSD showing no systematic change in homogeneity with changing grade. Of the four CRMs evaluated only CDN-GS-8C (30 g 2RSD of 0.44%), the highest grade CDN gold CRM tested, would be suitable for a 30 g fire assay. For CDN-GS-P5C (0.571 ppm Au) a sample mass of 111.8 g would be required to obtain an RSD of 1% during analysis; equivalent to a sampling error (30 g 2RSD) of 3.86%; 263.4 g for CDN-GS-1M; equivalent to a 2RSD sampling error of 5.93% at 30 g; and 140.0 g for CDN-GS-3L (3.18 ppm Au), equivalent to a 2RSD sampling error of 4.32% at 30 g. While the homogeneity of CDN-GS-8C is very good, the remaining CDN CRMs tested are considered to be poor to very poor.

Geostats: homogeneity for individual Geostats CRM's vary from 1.15% RSD (G914-7; 9.81 ppm Au) to 6.14% RSD (G313-1; 1.00 ppm Au), a spread of 4.99% RSD showing no systematic change in homogeneity with changing grade. Three of the four CRM's (G909-6 at 0.570 ppm Au, G914-6 at 3.21 ppm Au & G914-7 at 9.81 ppm Au) are fit for purpose as grade control CRM's for 30 g fire assay whilst G313-1 at 1.00 ppm Au would be suitable for a 50 g fire assay based on the sampling constant. The 2RSD sampling errors at a 30 g charge weight vary from 0.43% to 2.29%. The homogeneity of CRM's produced by Geostats range from mediocre to very good.

OREAS: All OREAS CRM's show consistently low RSDs ranging from 1.18% RSD (OREAS 62c at 8.79 ppm Au) to 1.87% RSD (OREAS 201 at 0.514 ppm Au) for a spread of 0.69% RSD. An inverse correlation is apparent between grade and RSD suggesting a possible influence of analytical precision. All four OREAS CRM's tested are fit for purpose for 30 g fire assay with sampling constants indicating that a 4 g fire assay charge weight would provide a sampling error of 1% or less for all OREAS CRM's. The sampling error at a 30 g charge weight varies from 0.44% to 0.70%. The homogeneity of CRM's produced by OREAS is considered very good.

Rocklabs: Individual Rocklabs CRM homogeneity varies from 1.20% RSD (SJ80 at 2.66 ppm Au) to 5.05 % RSD (SG66 at 1.09 ppm Au), a spread of 3.83% RSD with the CRM's showing no systematic change in homogeneity with changing grade. Three of the four CRM's (SE68 at 0.599 ppm Au, SJ80 at 2.66 ppm Au and SN75 at 8.67 ppm Au) are fit for purpose for 30 g fire assay while SG66 would probably be more fit for purpose for 50 g fire assay based on the sampling constant. The sampling errors of a 30 g charge weight range from 0.45% to 1.90%. The homogeneity of CRM's produced by Rocklabs is considered good to very good.

Table 4: Summary Statistics for each CRM.

Manufacturer	CRM	Certified Value Au (ppb)	NAA Mean Au (ppb)	NAA Min Au (ppb)	NAA Max Au (ppb)	NAA SDev Au (ppb)
AMIS	AMIS0352	450	445.3	393	467	20
	AMIS0310	1030	1080	946	1450	138
	AMIS0360	2940	3014	2810	3280	109
	AMIS0267	9050	8849	7450	17900	2425
CDN	CDN-GS-P5C	571	492.7	423	642	51
	CDN-GS-1M	1070	975.4	774	1480	155
	CDN-GS-3L	3180	3136	2340	3800	365
	CDN-GS-8C	8590	8238	8100	8440	96
Geostats	G909-6	570	545.8	510	576	16
	G313-1	1000	971.8	885	1130	60
	G914-6	3210	3269	3110	3390	70
	G914-7	9810	9657	9440	10000	112
OREAS	OREAS 201	514	546.2	531	567	10
	OREAS 204	1043	1020	990	1050	18
	OREAS 17c	3040	3009	2940	3110	48
	OREAS 62c	8790	8411	8100	8570	99
Rocklabs	SE68	599	615.5	591	638	12
	SG66	1086	1087	1030	1300	55
	SJ80	2656	2520	2450	2570	30
	SN75	8671	8000	7720	8150	100

INTERNAL CRM VARIATION

Control charts presented below show the variation in results of each CRM for each manufacturer. Figures 1 to 5 (A-D plots) show graphical representation of the 20 x 1 g INAA data where the X axis represents the order of analysis (analytical subsamples 1 to 20) from left to right; and the Y axis shows the measured INAA concentration in Au ppb. A solid red line shows the mean INAA value (see Table 4) for each CRM and for each grade range (i.e. 0.5 ppm Au, 1 ppm Au, 3 ppm Au and 9 ppm Au). The same Y axis concentration range and scale has been used where possible to facilitate visual comparison between the manufacturers. The Y axis in Figures 1-5 (E-H plots) shows the percentage difference from the calculated INAA mean. These diagrams are not intended as verification charts for each CRM by INAA, but as a standardised graphical communication tool.

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AMIS: Figure 1 graphically depicts results for the AMIS CRMs. Individual values for each CRM show a wide spread of almost random data with no systematic variation. AMIS0267 (cert 9.05 ppm) ranges from 7.45 ppm Au to 17.90 ppm Au (Fig. 1D) which represents -15.8 % to +102% difference from the INAA mean of this CRM (Fig. 1H). Two samples show a significant departure from the INAA mean, reporting +43.5% (12.70 ppm Au) and +102% (17.90 ppm Au) of the mean value (Fig. 1H) and reflects inhomogeneity (presumably a nugget effect) of the CRM. AMIS0310 (cert 1.03 ppm) also shows (Figs. 1B and F) significant departure from the INAA mean for two samples (+29.7% and

Figure 1. Control charts for AMIS CRMs showing INAA concentration (Au ppb) in relation to the certified value [A-D] and the percentage difference from the INAA mean value [E-H].

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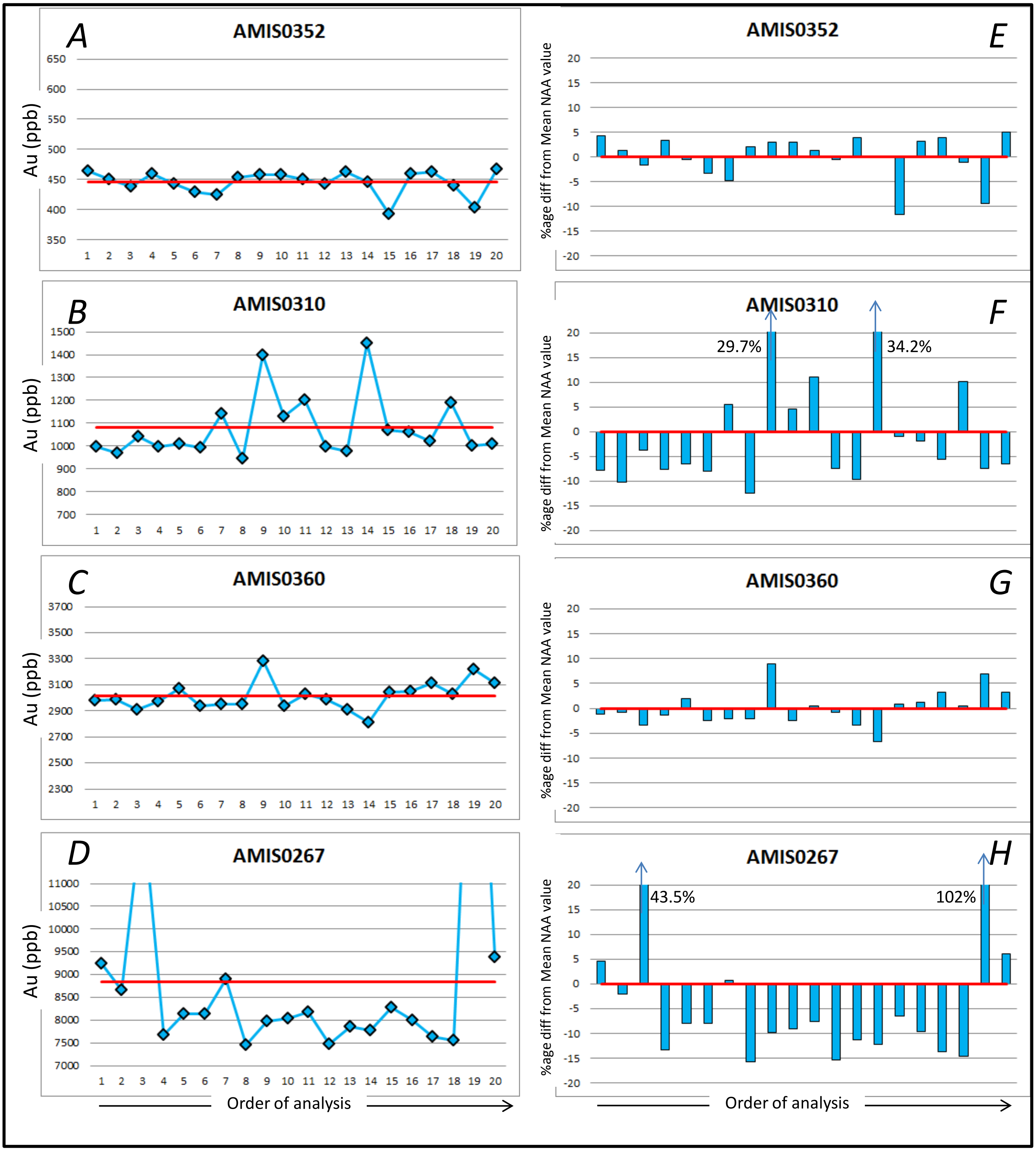

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+34.2%). This inhomogeneity of AMIS CRMs has potentially significant implications for laboratory reporting whereby a user would question the laboratory results based on the assumption that the CRM is homogeneous.

CDN: Figure 2 graphically displays the results for the CDN CRMs. With the exception of CDN-GS-8C (cert 8.59 ppm) which has a very tight spread of data around the INAA mean ranging from -1.7% to +2.5 % (Fig. 2H). The lower three of the four CDN CRMs (CDN-GS-P5C, CDN-GS-1M and CDN-GS-3L), show a wide scatter of data that commonly exceeds ±20% of the INAA mean value; CDN-GS-

Figure 2. Control charts for CDN CRMs showing INAA concentration (Au ppb) in relation to the certified value [A-D] and the percentage difference from the INAA mean value [E-H].

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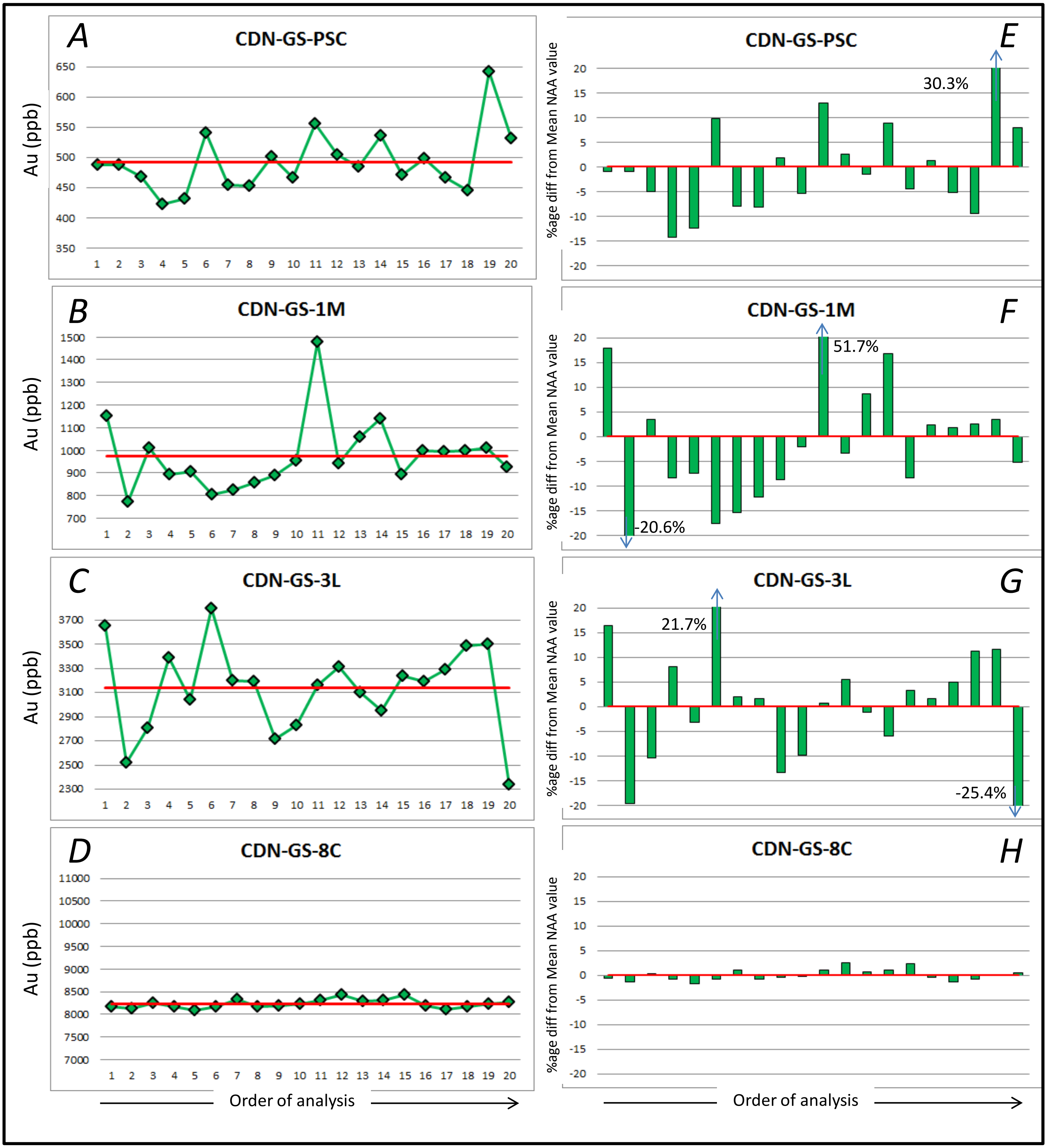

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P5C (cert .0.571 ppm) shows a range of -14.2 to +30.3% from the INAA mean (Figs. 2A and 2E); CDN-GS-1M (cert 1.07 ppm) ranges from -20.6 to +51.7 (Figs. 2B and 2F) and CDN-GS-3L (cert 3.18 ppm) ranges from -25.4 to +21.2% (Figs. 2C & 2G). This indicates inhomogeneity in three of these CRMs produced by CDN. The exception is CDN-GS-8C (cert 8.59 ppm) which has a very tight spread of data around the INAA mean ranging from -1.7% to +2.5 % (Figs. 2D and 2H).

GEOSTATS: Figure 3 shows the individual results for the Geostats CRMs. G914-6 (cert 3.21 ppm) and G914-7 (cert

Figure 3. Control charts for Geostats CRMs showing INAA concentration (Au ppb) in relation to the certified value [A-D] and the percentage difference from the INAA mean value [E-H].

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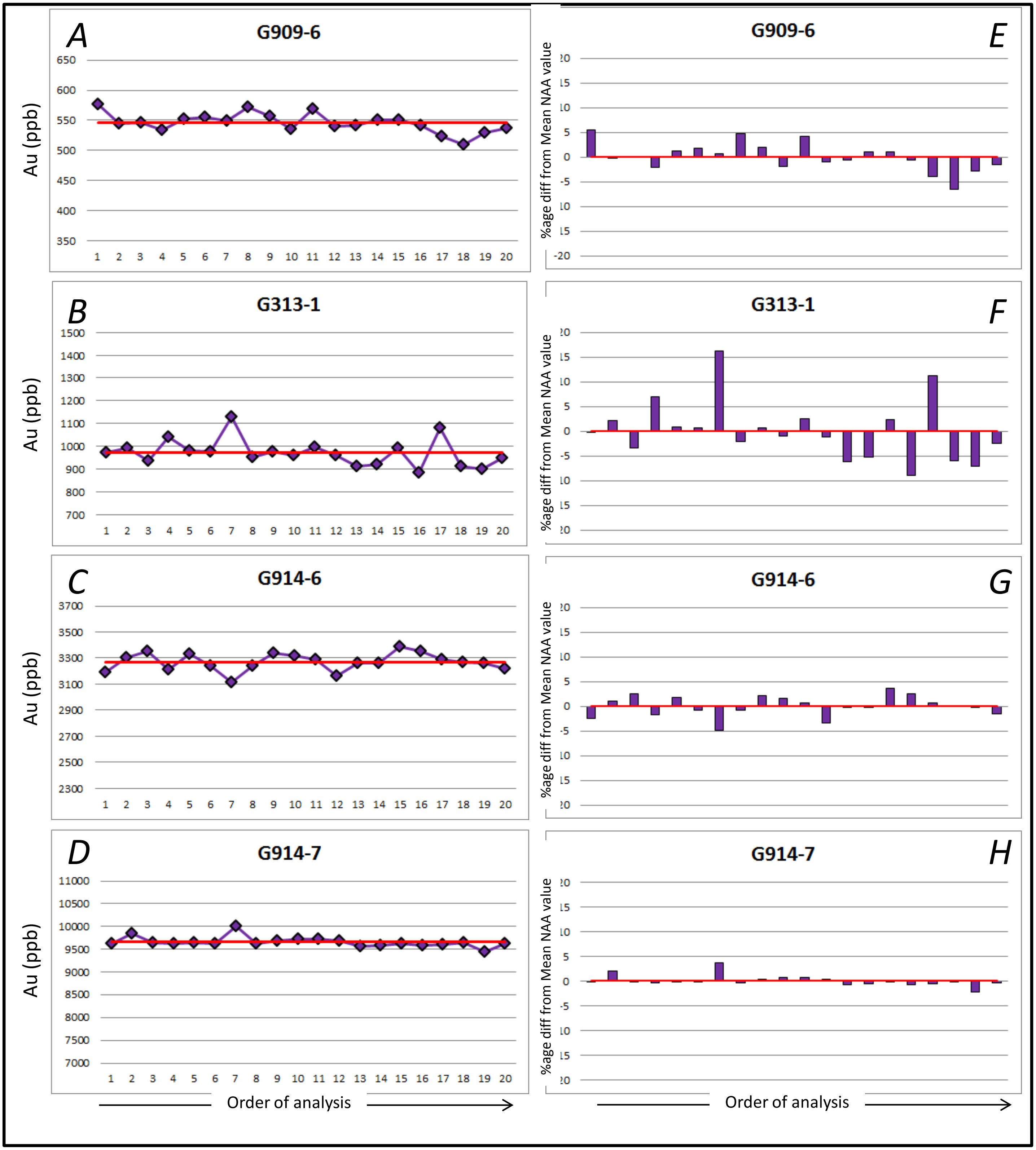

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9.81 ppm) show a relatively tight cluster around the INAA mean with G914-6 ranging from -4.9% to +3.7% (Figs. 3E and 3G) and G914-7-ranging from -2.2 to +3.6% (Figs.3 D and H). G909-6 (cert 0.57 ppm) shows a wider scatter whilst G313-1 (cert1.0 ppm) shows the widest scatter of all Geostats CRMs ranging from -8.9% to +16.3% of the INAA mean (Figs. 3B and 3F).

OREAS: Figure 4 shows the individual results for the OREAS CRMs. All OREAS CRMs show a consistent

Figure 4. Control charts for OREAS CRMs showing INAA concentration (Au ppb) in relation to the certified value [A-D] and the percentage difference from the INAA mean value [E-H].

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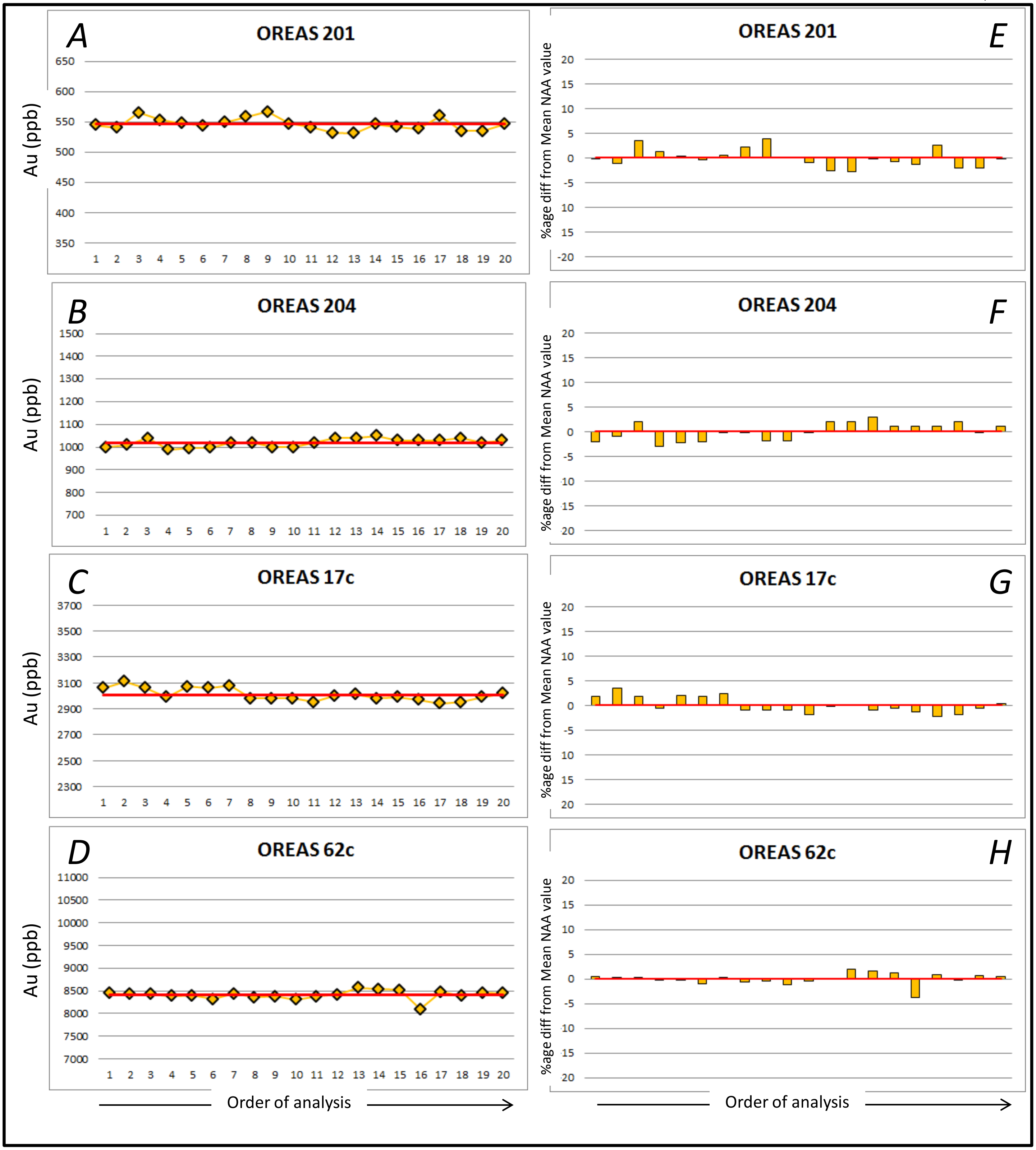

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tight range within ±5% of the INAA mean indicating a consistent homogenous CRM product over the range of CRMs tested.

Rocklabs: Figure 5 shows the individual results for the Rocklabs CRMs. Three of the four CRMs evaluated show a tight range within ±5% of the INAA mean indicating a consistent homogenous product. SG66 (cert 1.086 ppm) is slightly less homogenous with values ranging from -5.2% to +19.6% including one outlier (Figs. 5B and 5F).

Figure 5. Control charts for Rocklabs CRMs showing INAA concentration (Au ppb) in relation to the certified value [A-D] and the percentage difference from the INAA mean value [E-H].

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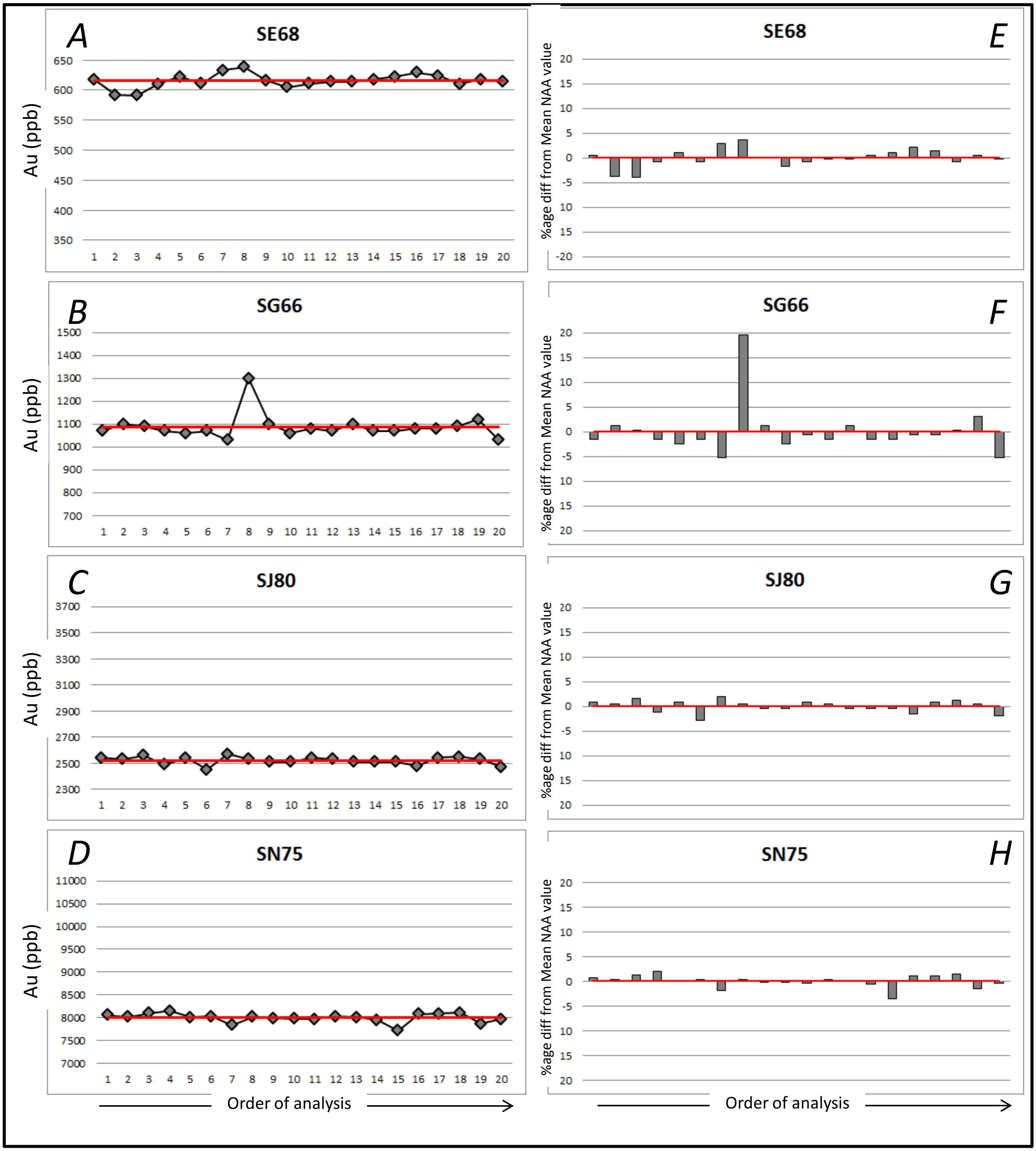

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DISCUSSION

The application of CRMs in the mining and exploration industry is to monitor laboratory quality and to comply with mandatory reporting requirements (e.g., TSX, ASX). They are also utilised during ore reserve calculations to understand the uncertainty in a resource that ultimately feeds into an economic model. It is thus essential that users have confidence in the quality and homogeneity of their CRMs. The CRM certified values are consensus values derived by inter-laboratory round robin programs. The methods used by some manufacturers to filter outliers can mask potential issues with the homogeneity of the materials. Only outliers confidently reasoned to be analytical should be removed and this confidence only exists where homogeneity has been independently validated.

Results from this study show that the degree of homogeneity of CRMs available from the five manufacturers is variable; users and analysts of these materials need confidence that the CRMs are homogenous to a level fit for purpose so that QC failures are genuine and not a function of sampling error (i.e. inhomogeneous materials). A ranking system based on the values for the 30 g 2RSD sampling error would provide a guide to homogeneity of a CRM and enable users to select appropriate CRMs for their projects such that <1% is considered Very Good; 1-2% considered Good; 2-3% considered Mediocre; 3-4% considered Poor and >4% considered Very Poor.

AMIS (McWha & Smee 2012a-c, 2014) certificates contain no evidence of homogeneity testing but state, “Samples were randomly selected for homogeneity testing and third party analysis. Statistical analysis of both homogeneity and the consensus test results were carried out by independent statisticians”. CDN certificates (Sanderson & Smee 2013a-c, 2014) make no mention of homogeneity; Geostats certificates (Geostats 2009, 2014, 2015a,b) provide an unsupported statement that “materials are tested regularly to ensure stability and homogeneity”. OREAS publishes homogeneity test results with their gold CRM certificates (Hamlyn, 2009a,b, 2012a,b). Rocklabs (Smith & Ball 2012a,b,2013a,b) certificates contain a ‘Homogeneity Assessment’ section with the RSD reported but do not provide the actual results. Segregation/Settling information is also provided.

To enable the exploration and mining industry to have confidence in the quality of gold CRMs used for QC and mandatory reporting, manufacturers need to provide data on the homogeneity of every gold CRM. This homogeneity test work could be through the Reduced Analytical Subsample Method as utilised routinely by OREAS and chosen for this study or through the Replicates of Large and Small Sample Mass as described by Bagley et al. (2015). Without CRM manufacturers providing transparency on the homogeneity through test work, users should not assume all CRMs represent quality products.

CONCLUSIONS

The homogeneity of twenty commercial gold ore CRMs produced by AMIS (South Africa), CDN (Canada), Geostats (Australia), OREAS (Australia) and Rocklabs (New Zealand) have been evaluated and compared. The CRMs range in gold content from 0.45 to 9.81 ppm, typical of the levels commonly encountered in mining exploration projects.

The CRMs of AMIS, CDN, Geostats and OREAS are produced from naturally occurring gold ores to which variable quantities of barren/waste rock material has been added to achieve target grades. Rocklabs CRMs are produced from a range of rock and mineral products to which fine gold dust particles have been added in concentrations to achieve desired grades.

The investigation has shown a remarkable contrast in homogeneity between the various producers. These contrasting levels of homogeneity have serious ramifications with regard to sampling errors, which in some instances are of a magnitude equal to or greater than typical 30 g fire assay measurement errors rendering them of questionable value as a QC tool. Sampling errors for a typical fire assay charge weight (25 to 50 g) should be very minor compared to laboratory measurement errors. Otherwise the CRM cannot be deemed fit for purpose as the user is unable ascertain the source of the error seen in analytical data.

The homogeneity of the 20 gold CRM’s were evaluated using the Sampling Constant, the minimum sample mass (charge weight) required to achieve a 1% relative standard deviation in repeat analyses. For the four AMIS CRM’s tested, the Sampling Constants returned a range from 14 to 791 g. CDN had one CRM with a Sampling Constant of 1.4 g while the other three ranged from 111.8 to 263.4 g. Geostats had one CRM with a Sampling Constant of 1.4 g with the other three ranging from 4.9 to 39.4 g.

The OREAS CRM’s all returned Sampling Constants of less than 4 g. For Rocklabs, three of the four CRM’s returned Sampling Constants of less than 4 g with the fourth coming in at 27.1 g. Currently, OREAS is the only CRM manufacturer routinely evaluating and publishing the results of homogeneity test work on all their gold CRM’s using the Reduced Analytical Subsample INAA method.

This study clearly demonstrates that there is a wide range in quality amongst commercially available gold ore CRM’s and that the homogeneity of gold CRM’s from most manufacturers varies from one product to the next. It is proposed that all manufacturers be encouraged to undertake and publish results of homogeneity test work on gold in CRM’s, thereby providing end users irrefutable data on the magnitude of CRM sampling errors and their impact on QC protocols.

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