

#### **CERTIFICATE OF ANALYSIS FOR**

# Zn-Pb-Ag REFERENCE MATERIAL OREAS 134a

#### Summary Statistics for Key Analytes (see Table 1 for additional certified values).

Constituent (nam)	Certified	1SD	95% Confid	ence Limits	95% Tolerance Limits			
Constituent (ppm)	Value	130	Low	High	Low	High		
4-Acid Digestion								
Ag, Silver (ppm)	201	7	196	205	197	205		
Pb, Lead (wt.%)	12.79	0.766	12.34	13.23	12.49	13.08		
Zn, Zinc (wt.%)	17.27	0.553	16.95	17.59	16.97	17.57		

Please note: intervals may appear asymmetric due to rounding.



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Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 134a.

Constituent	Certified	460	95% Confid	lence Limits	95% Tolerance Limits		
Constituent	Value	1SD	Low	High	Low	High	
Fusion ICP*							
Ag, Silver (ppm)	197	10	186	209	190	205	
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	2.60	0.131	2.49	2.71	2.54	2.67	
As, Arsenic (ppm)	218	22	196	239	208	227	
Ba, Barium (ppm)	1500	156	1334	1667	1480	1521	
CaO, Calcium oxide (wt.%)	6.38	0.197	6.21	6.56	6.30	6.47	
Cd, Cadmium (ppm)	546	36	510	581	529	563	
Co, Cobalt (ppm)	100	13	90	110	98	103	
Fe, Iron (wt.%)	12.32	0.795	11.71	12.94	12.01	12.63	
MgO, Magnesium oxide (wt.%)	3.55	0.119	3.45	3.66	3.50	3.61	
Pb, Lead (wt.%)	12.95	0.623	12.49	13.40	12.67	13.22	
SiO <sub>2</sub> , Silicon dioxide (wt.%)	14.81	0.845	14.10	15.53	14.51	15.11	
Zn, Zinc (wt.%)	17.49	0.505	17.10	17.88	17.20	17.78	
4-Acid Digestion			1	1		•	
Ag, Silver (ppm)	201	7	196	205	197	205	
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	2.53	0.117	2.46	2.61	2.49	2.58	
As, Arsenic (ppm)	228	23	217	239	220	235	
CaO, Calcium oxide (wt.%)	6.04	0.385	5.79	6.29	5.94	6.13	
Cd, Cadmium (ppm)	536	36	514	557	523	548	
Co, Cobalt (ppm)	105	8	100	110	101	109	
Cu, Copper (ppm)	1291	45	1270	1311	1267	1314	
Fe, Iron (wt.%)	12.04	0.410	11.83	12.25	11.87	12.21	
MgO, Magnesium oxide (wt.%)	3.49	0.184	3.39	3.60	3.43	3.56	
Pb, Lead (wt.%)	12.79	0.766	12.34	13.23	12.49	13.08	
S, Sulphur (wt.%)	19.27	0.503	18.85	19.70	18.72	19.83	
Sb, Antimony (ppm)	115	15	107	124	112	119	
Zn, Zinc (wt.%)	17.27	0.553	16.95	17.59	16.97	17.57	
Aqua Regia Digestion				•			
Ag, Silver (ppm)	194	13	186	202	188	199	
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	0.774	0.085	0.714	0.833	0.755	0.792	
As, Arsenic (ppm)	226	24	212	239	219	232	
Cd, Cadmium (ppm)	547	44	520	573	537	556	
Co, Cobalt (ppm)	101	8	96	106	99	104	
Cu, Copper (ppm)	1279	78	1231	1328	1250	1309	
Fe, Iron (wt.%)	11.89	0.973	11.30	12.48	11.65	12.14	
MgO, Magnesium oxide (wt.%)	3.43	0.137	3.33	3.52	3.37	3.48	
Pb, Lead (wt.%)	12.64	0.759	12.11	13.17	12.34	12.93	
S, Sulphur (wt.%)	18.49	1.329	17.35	19.63	18.08	18.90	
Zn, Zinc (wt.%)	17.00	0.863	16.44	17.56	16.57	17.43	
Infrared Combustion						1	
S, Sulphur (wt.%)	19.11	0.413	18.81	19.42	18.81	19.41	

<sup>\*</sup>except for Ba where two laboratories used pressed powder pellet with XRF. Please note: intervals may appear asymmetric due to rounding.



Table 2. Indicative Values for OREAS 134a.

Constituent         Unit         Value         Constituent         Unit         Value         Constituent         Unit         Value           Fusion ICP           Cr         ppm         1.00         Na         ppm         653         Ti         ppm         613           Cr         ppm         80         P         ppm         297         V         ppm         16.4           Cu         ppm         1278         S         wt.%         19.39         Y         ppm         5.40           K         wt.%         0.929         Sb         ppm         118         Zr         ppm         30.2           LOI****         vt.%         15.12         Sc         ppm         2.00         Image: color sector se	Table 2. Indicative Values for OREAS 134a.									
Be         ppm         1.00         Na         ppm         653         Ti         ppm         613           Cr         ppm         80         P         ppm         297         V         ppm         16.4           Cu         ppm         1278         S         wt.%         19.39         Y         ppm         5.40           K         wt.%         15.12         Sc         ppm         118         Zr         ppm         30.2           LOI <sup>1000</sup> wt.%         15.12         Sc         ppm         2.00         ppm         30.2           Hon         ppm         3371         Sr         ppm         2.00         Sm         ppm         1.38           Ba         ppm         21.0         K         wt.%         0.795         Sm         ppm         1.38           Ba         ppm         21.0         L         ppm         6.10         Sn         ppm         1.13           Ba         ppm         20.0         Li         ppm         17.8         Sr         ppm         1.13           Ba         ppm         20.0         Li         ppm         100         Ta         ppb         25	Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value	
Cr         ppm         80         P         ppm         297         V         ppm         16.4           Cu         ppm         1278         S         wt.%         19.39         Y         ppm         5.40           K         wt.%         0.929         Sb         ppm         118         Zr         ppm         5.40           Mn         ppm         3371         Sr         ppm         2.00         L         ppm         22.02           Mn         ppm         21.0         K         wt.%         0.795         Sm         ppm         1.38           Ba         ppm         21.00         L         ppm         6.10         Sn         ppm         1.38           Ba         ppm         2.00         Li         ppm         17.8         Sr         ppm         1.33           Ce         ppm         13.2         Lu         ppb         100         Ta         ppb         45.00           Cr         ppm         13.2         Lu         ppb         100         Ta         ppb         45.00           Cr         ppm         1.66         Mn         ppm         3424         Tb         ppb	Fusion ICF	•								
Cu         ppm         1278         S         wt.%         19.39         Y         ppm         5.40           K         wt.%         0.929         Sb         ppm         118         Zr         ppm         30.2           LOI <sup>1000</sup> wt.%         15.12         Sc         ppm         2.00         V         ppm         30.2           4-Acid Digestion           Be         ppm         21.0         K         wt.%         0.795         Sm         ppm         1.38           Ba         ppm         2.400         Li         ppm         6.10         Sn         ppm         1.38           Be         ppm         2.00         Li         ppm         17.8         Sr         ppm         18.3           Ce         ppm         13.2         Lu         ppb         100         Ta         ppb         <500	Be	ppm	1.00	Na	ppm	653	Ti	ppm	613	
K         vt.%         0.929         Sb         ppm         118         Zr         ppm         30.2           LOI <sup>1000</sup> Mn         wt.%         15.12         Sc         ppm         2.00         r         ppm         30.2           4-Acid Digestion         B         ppm         21.0         K         wt.%         0.795         Sm         ppm         1.38           Ba         ppm         < 1400	Cr	ppm	80	Р	ppm	297	V	ppm	16.4	
No.   No.	Cu	ppm	1278	S	wt.%	19.39	Y	ppm	5.40	
Mn		wt.%	0.929	Sb	ppm	118	Zr	ppm	30.2	
Mn	LOI <sup>1000</sup>	wt.%	15.12	Sc	ppm	2.00				
B         ppm         21.0         K         wt.%         0.795         Sm         ppm         1.38           Ba         ppm         < 1400		ppm	3371	Sr	ppm	28.8				
Ba         ppm         < 1400         La         ppm         6.10         Sn         ppm         1.13           Be         ppm         2.00         Li         ppm         17.8         Sr         ppm         18.3           Ce         ppm         13.2         Lu         ppb         100         Ta         ppb         < 500	4-Acid Dig	estion								
Be         ppm         2.00         Li         ppm         17.8         Sr         ppm         18.3           Ce         ppm         13.2         Lu         ppb         100         Ta         ppb         <500	В	ppm	21.0	K	wt.%	0.795	Sm	ppm	1.38	
Ce         ppm         13.2         Lu         ppb         100         Ta         ppb         <500           Cr         ppm         16.6         Mn         ppm         3424         Tb         ppb         180           Cs         ppm         1.14         Mo         ppm         6.25         Te         ppb         90           Dy         ppm         0.80         Na         ppm         303         Th         ppm         1.44           Er         ppm         0.56         Nb         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         7.42         Ti         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Rb         ppm         27.4         W         ppm	Ва	ppm	< 1400	La	ppm	6.10	Sn	ppm	1.13	
Cr         ppm         16.6         Mn         ppm         3424         Tb         ppb         180           Cs         ppm         1.14         Mo         ppm         6.25         Te         ppb         90           Dy         ppm         0.80         Na         ppm         303         Th         ppb         90           Eu         ppb         300         Nd         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         7.42         Tl         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         1.47           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         200         Sc         ppm         1.40         Yb         ppb         520	Be	ppm	2.00	Li	ppm	17.8	Sr	ppm	18.3	
Cs         ppm         1.14         Mo         ppm         6.25         Te         ppb         90           Dy         ppm         0.80         Na         ppm         303         Th         ppm         1.44           Er         ppm         0.56         Nb         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         7.42         Ti         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         1.22           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         200         Sc         ppm         1.40         Yb         ppb         520           In         ppm         1.16         Se         ppm         7.44         Zr         ppm	Ce	ppm	13.2	Lu	ppb	100	Та	ppb	< 500	
Cs         ppm         1.14         Mo         ppm         6.25         Te         ppb         90           Dy         ppm         0.80         Na         ppm         303         Th         ppm         1.44           Er         ppm         0.56         Nb         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         7.42         Tl         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hf         ppb         880         Rb         ppm         27.4         W         ppm         12.2           Hg         ppb         200         Sc         ppm         1.40         Yb         ppb         520           In         ppb         200         Sc         ppm         1.44         Zr         ppm         5	Cr		16.6	Mn		3424	Tb		180	
Dy         ppm         0.80         Na         ppm         303         Th         ppm         1.44           Er         ppm         0.56         Nb         ppm         1.86         Ti         ppm         479           Eu         ppb         300         Nd         ppm         7.42         Tl         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hif         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         <1	Cs		1.14	Мо		6.25	Te		90	
Eu         ppb         300         Nd         ppm         7.42         TI         ppm         93           Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         <1	Dy	ppm	0.80	Na	ppm	303	Th	ppm	1.44	
Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         < 1	Er	ppm	0.56	Nb	ppm	1.86	Ti	ppm	479	
Ga         ppm         4.88         Ni         ppm         21.3         Tm         ppb         100           Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         < 1	Eu		300	Nd		7.42	TI	' '	93	
Gd         ppm         1.24         P         ppm         294         U         ppm         1.47           Ge         ppb         2080         Pr         ppm         1.68         V         ppm         12.2           Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         < 1	Ga		4.88	Ni		21.3	Tm	' '	100	
Hf         ppb         880         Rb         ppm         27.4         W         ppm         0.52           Hg         ppb         2000         Re         ppb         <1	Gd		1.24	Р	ppm	294	U		1.47	
Hg         ppb         2000         Re         ppb         < 1         Y         ppm         5.18           Ho         ppb         200         Sc         ppm         1.40         Yb         ppb         520           In         ppm         1.16         Se         ppm         7.44         Zr         ppm         27.8           Aqua Regia Digestion           Au         ppb         < 0.5	Ge	ppb	2080	Pr	ppm	1.68	V	ppm	12.2	
Ho	Hf	ppb	880	Rb	ppm	27.4	W	ppm	0.52	
In	Hg	ppb	2000	Re	ppb	< 1	Y	ppm	5.18	
Aqua Regia Digestion           Au         ppb         < 0.5	Но	ppb	200	Sc	ppm	1.40	Yb	ppb	520	
Au         ppb         < 0.5         In         ppm         1.20         Se         ppm         6.92           B         ppm         < 10	In	ppm	1.16	Se	ppm	7.44	Zr	ppm	27.8	
B         ppm         < 10         K         wt.%         0.150         Sm         ppm         1.44           Ba         ppm         < 600	Aqua Regi	a Digesti	ion							
Ba         ppm         < 600         La         ppm         6.54         Sn         ppm         0.93           Be         ppm         0.70         Li         ppm         15.3         Sr         ppm         23.8           CaO         wt.%         6.09         Lu         ppb         < 100	Au	ppb	< 0.5	In	ppm	1.20	Se	ppm	6.92	
Be         ppm         0.70         Li         ppm         15.3         Sr         ppm         23.8           CaO         wt.%         6.09         Lu         ppb         < 100         Ta         ppb         < 50           Ce         ppm         13.0         Mn         ppm         3854         Tb         ppb         200           Cr         ppm         12.9         Mo         ppm         6.80         Te         ppb         100           Cs         ppm         0.68         Na         ppm         6.80         Te         ppb         100           Cs         ppm         0.68         Na         ppm         161         Th         ppm         2.18           Dy         ppm         1.11         Nb         ppm         < 0.1         Ti         ppm         60           Er         ppm         0.56         Nd         ppm         6.37         Tl         ppm         67           Eu         ppb         360         Ni         ppm         21.6         Tm         ppb         < 100           Ga         ppm         1.34         Pr         ppm         1.54         V         ppm	В	ppm	< 10	K	wt.%	0.150	Sm	ppm	1.44	
CaO         wt.%         6.09         Lu         ppb         < 100         Ta         ppb         < 50           Ce         ppm         13.0         Mn         ppm         3854         Tb         ppb         200           Cr         ppm         12.9         Mo         ppm         6.80         Te         ppb         100           Cs         ppm         0.68         Na         ppm         161         Th         ppm         2.18           Dy         ppm         1.11         Nb         ppm         < 0.1	Ва	ppm	< 600	La	ppm	6.54	Sn	ppm	0.93	
Ce         ppm         13.0         Mn         ppm         3854         Tb         ppb         200           Cr         ppm         12.9         Mo         ppm         6.80         Te         ppb         100           Cs         ppm         0.68         Na         ppm         161         Th         ppm         2.18           Dy         ppm         1.11         Nb         ppm         < 0.1	Be	ppm	0.70	Li	ppm	15.3	Sr	ppm	23.8	
Cr         ppm         12.9         Mo         ppm         6.80         Te         ppb         100           Cs         ppm         0.68         Na         ppm         161         Th         ppm         2.18           Dy         ppm         1.11         Nb         ppm         < 0.1	CaO	wt.%	6.09	Lu	ppb	< 100	Та	ppb	< 50	
Cs         ppm         0.68         Na         ppm         161         Th         ppm         2.18           Dy         ppm         1.11         Nb         ppm         < 0.1	Ce	ppm	13.0	Mn	ppm	3854	Tb	ppb	200	
Dy         ppm         1.11         Nb         ppm         < 0.1         Ti         ppm         60           Er         ppm         0.56         Nd         ppm         6.37         Tl         ppm         67           Eu         ppb         360         Ni         ppm         21.6         Tm         ppb         < 100	Cr	ppm	12.9	Мо	ppm	6.80	Te	ppb	100	
Er         ppm         0.56         Nd         ppm         6.37         TI         ppm         67           Eu         ppb         360         Ni         ppm         21.6         Tm         ppb         < 100	Cs	ppm	0.68	Na	ppm	161	Th	ppm	2.18	
Eu         ppb         360         Ni         ppm         21.6         Tm         ppb         < 100           Ga         ppm         3.13         P         ppm         281         U         ppm         0.96           Gd         ppm         1.34         Pr         ppm         1.54         V         ppm         7.40           Ge         ppb         475         Rb         ppm         11.7         W         ppm         0.24           Hf         ppb         280         Re         ppb         3         Y         ppm         3.00           Hg         ppb         2260         Sb         ppm         101         Yb         ppb         480	Dy	ppm	1.11	Nb	ppm	< 0.1	Ti	ppm	60	
Ga         ppm         3.13         P         ppm         281         U         ppm         0.96           Gd         ppm         1.34         Pr         ppm         1.54         V         ppm         7.40           Ge         ppb         475         Rb         ppm         11.7         W         ppm         0.24           Hf         ppb         280         Re         ppb         3         Y         ppm         3.00           Hg         ppb         2260         Sb         ppm         101         Yb         ppb         480	Er	ppm	0.56	Nd	ppm	6.37	TI	ppm	67	
Gd         ppm         1.34         Pr         ppm         1.54         V         ppm         7.40           Ge         ppb         475         Rb         ppm         11.7         W         ppm         0.24           Hf         ppb         280         Re         ppb         3         Y         ppm         3.00           Hg         ppb         2260         Sb         ppm         101         Yb         ppb         480	Eu	ppb	360	Ni	ppm	21.6	Tm	ppb	< 100	
Ge         ppb         475         Rb         ppm         11.7         W         ppm         0.24           Hf         ppb         280         Re         ppb         3         Y         ppm         3.00           Hg         ppb         2260         Sb         ppm         101         Yb         ppb         480	Ga	ppm	3.13	Р	ppm	281	U	ppm	0.96	
Hf         ppb         280         Re         ppb         3         Y         ppm         3.00           Hg         ppb         2260         Sb         ppm         101         Yb         ppb         480	Gd	ppm	1.34	Pr	ppm	1.54	V	ppm	7.40	
Hg ppb 2260 Sb ppm 101 Yb ppb 480	Ge	ppb	475	Rb	ppm	11.7	W	ppm	0.24	
	Hf	ppb	280	Re	ppb	3	Y	ppm	3.00	
Ho   ppb   200   Sc   ppm   < 0.5   Zr   ppm   10.4	Hg	ppb	2260	Sb	ppm	101	Yb	ppb	480	
Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are	1									

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

#### INTRODUCTION

OREAS reference materials are intended to provide a low cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

#### **SOURCE MATERIALS**

OREAS 134a is one of eight pigeon paired CRM's prepared from zinc-lead mineralised material from Xstrata's Black Star and George Fisher orebodies located in Mt Isa in NW Queensland, Australia. OREAS 134a contains a 4.3% and 4.2% lower relative offset in Pb and Zn grades respectively, to OREAS 134b. The orebodies are sediment hosted 'SEDEX' Zn-Pb-Ag deposits located within the Urquart Shale Formation of the Mount Isa Group, a weakly metamorphosed, 5 km thick sequence composed predominantly of Mesoproterozoic carbonate siltstones, mudstones and shales. The Urquart Shale consists of a sequence of alternating pyrite-rich dolomitic siltstone and shale beds up to 1000 metres thick and was deposited in a lacustrine setting within an intracratonic rift basin. The orebodies lie within the upper 650m and are bounded by the Mount Isa fault on the west and by volcanic greenstones to the east. Comprising galena and sphalerite with pyrite and pyrrhotite, the lead-zinc-silver orebodies are concordant with carbonaceous dolomitic sediments and interfinger with the silica-dolomitic mass hosting copper. OREAS 134a was prepared from a blend of Black Star waste rock, Black Star ore and George Fisher ore.

#### COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 134a was prepared in the following manner:

- drying to constant mass at 65°C;
- crushing and milling to 100% minus 30 microns;
- homogenisation and bagging into 20kg lots;
- packaging into 10g units sealed under nitrogen in laminated foil pouches.

#### ANALYTICAL PROGRAM

Fifteen commercial laboratories participated in the analytical program to certify Ag, Al<sub>2</sub>O<sub>3</sub>, As, Ba, CaO, Cd, Co, Cu, Fe, MgO, Pb, S, Sb, SiO<sub>2</sub> and Zn by a range of analytical methods. Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 134a DataPack.xlsx**).

The intent of the certification program was to characterise the analytes by:

- fusion methods sodium peroxide fusion or lithium borate fusion with ICP (except for Ba where two laboratories used pressed powder pellet with XRF);
- four acid (HF-HCl-HNO<sub>3</sub>-HClO<sub>4</sub>) digest with ICP or AAS;
- agua regia digest with ICP or AAS;

# • Leco for sulphur only.

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. Aqua regia is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions which can include the ratio of nitric to hydrochloric acids, acid strength, temperatures, leach times and secondary digestions. Recoveries for sulphide-hosted base metal sulphides approach total values, however, other analytes, in particular the lithophile elements, show greater sensitivity to method parameters. This can result in lack of consensus in an inter-laboratory certification program for these elements. The approach applied here is to report certified values in those instances where reasonable agreement exists amongst a majority of participating laboratories. The results of specific laboratories may differ significantly from the certified values, but will, nonetheless, be valid and reproducible in the context of the specifics of the aqua regia method in use. Users of this reference material should, therefore, be mindful of this limitation when applying the certified values in a quality control program.

For the round robin program a batch of five 25g vacuum-packed pulp samples was submitted to each of the participating laboratories for analysis. The five samples comprising each batch were scoop-split from a random selection of five of ten or more 400g master samples. The latter were taken at regular intervals during the bagging stage and immediately following homogenisation. Table 1 presents the 37 certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows 115 indicative values. Table 3 provides performance gate intervals for the certified values of each method group based on their pooled 1SD's.

#### STATISTICAL ANALYSIS

Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if >2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

**Certified Values** are the means of accepted laboratory means after outlier filtering. Indicative (uncertified) values (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) inter-laboratory consensus is poor; or iii) a significant proportion of results are outlying.

**95% Confidence Limits** are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. *95% Confidence Limits should not be used as control limits for laboratory performance.* 

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**Standard Deviation** values (1SDs) are reported in Table 1 and provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. The SD values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e. after removal of any individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-lab bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

Table 3 shows **Performance Gates** calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned. A second method utilises a 5% window calculated directly from the certified value. Standard deviation is also shown in relative per cent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow.

**Tolerance Limits** (ISO Guide 3207) were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for Zn by 4-acid digestion, where 99% of the time (1- $\alpha$ =0.99) at least 95% of subsamples ( $\rho$ =0.95) will have concentrations lying between 16.97 and 17.57 wt.%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

Table 3. Performance Gates for OREAS 134a.

Table 3. Performance Gates for OREAS 134a.											
Constituent	Certified	Absolute Standard Deviations					Relative Standard Deviations			5% window	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Fusion ICP*											
Ag, ppm	197	10	177	217	167	227	5.06%	10.11%	15.17%	188	207
Al <sub>2</sub> O <sub>3</sub> , wt.%	2.60	0.131	2.34	2.86	2.21	3.00	5.04%	10.08%	15.12%	2.47	2.73
As, ppm	218	22	174	262	152	284	10.09%	20.19%	30.28%	207	229
Ba, ppm	1500	156	1189	1811	1033	1967	10.37%	20.74%	31.11%	1425	1575
CaO, wt.%	6.38	0.197	5.99	6.78	5.80	6.97	3.08%	6.16%	9.23%	6.07	6.70
Cd, ppm	546	36	474	617	438	653	6.56%	13.12%	19.68%	519	573
Co, ppm	100	13	74	127	61	140	13.15%	26.30%	39.45%	95	105
Fe, wt.%	12.32	0.795	10.73	13.91	9.94	14.71	6.45%	12.91%	19.36%	11.71	12.94
MgO, wt.%	3.55	0.119	3.32	3.79	3.20	3.91	3.35%	6.69%	10.04%	3.38	3.73
Pb, wt.%	12.95	0.623	11.70	14.19	11.08	14.81	4.81%	9.62%	14.43%	12.30	13.59
SiO <sub>2</sub> , wt.%	14.81	0.845	13.12	16.50	12.28	17.35	5.71%	11.41%	17.12%	14.07	15.55
Zn, wt.%	17.49	0.505	16.48	18.50	15.98	19.01	2.89%	5.78%	8.66%	16.62	18.37
4-Acid Digest	ion										
Ag, ppm	201	7	187	214	181	221	3.36%	6.72%	10.09%	191	211
Al <sub>2</sub> O <sub>3</sub> , wt.%	2.53	0.117	2.30	2.77	2.18	2.88	4.61%	9.23%	13.84%	2.41	2.66
As, ppm	228	23	182	274	159	297	10.06%	20.12%	30.18%	216	239
CaO, wt.%	6.04	0.385	5.27	6.81	4.88	7.19	6.37%	12.75%	19.12%	5.73	6.34
Cd, ppm	536	36	463	608	427	644	6.73%	13.47%	20.20%	509	562
Co, ppm	105	8	88	121	80	130	7.85%	15.70%	23.54%	100	110
Cu, ppm	1291	45	1201	1381	1156	1426	3.49%	6.98%	10.47%	1226	1355
Fe, wt.%	12.04	0.410	11.22	12.86	10.81	13.27	3.41%	6.82%	10.23%	11.44	12.64
MgO, wt.%	3.49	0.184	3.13	3.86	2.94	4.05	5.27%	10.54%	15.81%	3.32	3.67
Pb, wt.%	12.79	0.766	11.25	14.32	10.49	15.09	5.99%	11.99%	17.98%	12.15	13.43
S, wt.%	19.27	0.503	18.27	20.28	17.77	20.78	2.61%	5.22%	7.82%	18.31	20.24
Sb, ppm	115	15	86	145	71	160	12.86%	25.71%	38.57%	110	121
Zn, wt.%	17.27	0.553	16.16	18.37	15.61	18.92	3.20%	6.40%	9.60%	16.40	18.13
Aqua Regia D	igestion										
Ag, ppm	194	13	167	220	154	233	6.80%	13.60%	20.41%	184	203
Al <sub>2</sub> O <sub>3</sub> , wt.%	0.774	0.085	0.603	0.944	0.517	1.030	11.05%	22.10%	33.15%	0.735	0.812
As, ppm	226	24	177	274	153	298	10.68%	21.35%	32.03%	214	237
Cd, ppm	547	44	458	636	413	680	8.13%	16.25%	24.38%	519	574
Co, ppm	101	8	85	118	77	126	8.09%	16.19%	24.28%	96	106
Cu, ppm	1279	78	1123	1436	1044	1515	6.13%	12.25%	18.38%	1216	1343
Fe, wt.%	11.89	0.973	9.95	13.84	8.97	14.81	8.18%	16.36%	24.55%	11.30	12.49
MgO, wt.%	3.43	0.137	3.15	3.70	3.01	3.84	4.01%	8.02%	12.04%	3.25	3.60
Pb, wt.%	12.64	0.759	11.12	14.16	10.36	14.91	6.01%	12.01%	18.02%	12.01	13.27
S, wt.%	18.49	1.329	15.84	21.15	14.51	22.48	7.18%	14.37%	21.55%	17.57	19.42
Zn, wt.%	17.00	0.863	15.27	18.73	14.41	19.59	5.08%	10.16%	15.24%	16.15	17.85
Infrared Com	bustion										
S, wt.%	19.11	0.413	18.29	19.94	17.87	20.35	2.16%	4.32%	6.48%	18.16	20.07
	i .					with VDE					·

<sup>\*</sup>except for Ba where two laboratories used pressed powder pellet with XRF.

Note: intervals may appear asymmetric due to rounding.



Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 134a is fit-for-purpose as a certified reference material (see 'Intended Use' below).

#### PARTICIPATING LABORATORIES

- 1. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 2. Actlabs, Ancaster, Ontario, Canada
- 3. ALS, Brisbane, QLD, Australia
- 4. ALS, Johannesburg, South Africa
- 5. ALS, Perth, WA, Australia
- 6. ALS, Vancouver, BC, Canada
- 7. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 8. Bureau Veritas Amdel Laboratories, Perth, WA, Australia
- 9. Intertek Genalysis, Perth, WA, Australia
- 10. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 11. Intertek Testing Services, Cupang, Muntinlupa, Philippines
- 12. SGS Australia Mineral Services, Perth, WA, Australia
- 13. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 14. SGS Mineral Services, Townsville, QLD, Australia
- 15. Bureau Veritas Geoanalytical, Perth, WA, Australia

#### PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 134a has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd

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37A Hosie Street

Fax: +613-9729 8338

Bayswater North VIC 3153

Web: www.ore.com.au

AUSTRALIA

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It is available in 10g units sealed under nitrogen in laminated foil pouches.

#### **INTENDED USE**

OREAS 134a is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological samples;
- for the verification of analytical methods for analytes reported in Table 1;
- for the calibration of instruments used in the determination of the concentration of analytes reported in Table 1.

# STABILITY AND STORAGE INSTRUCTIONS

OREAS 134a has been prepared from a blend of sulphide-bearing Black Star waste, Black Star ore and George Fisher ore. To prolong its shelf life it has been packaged under nitrogen in robust foil laminate pouches. It is considered to have long-term stability under normal storage conditions. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

#### INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 134a refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

# **TRACEABILITY**

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis. The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs) for a particular analytical method, analyte, or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified and non-certified (indicative) values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

#### **QMS ACCREDITED**

ORE Pty Ltd is accredited to ISO 9001:2008 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.





# HANDLING INSTRUCTIONS

Fine powders pose a risk to eyes and lungs and therefore standard precautions such as the use of safety glasses and dust masks are advised.

# **LEGAL NOTICE**

Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the use of this material and information.

#### **CERTIFYING OFFICER**

Sem

Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager - ORE P/L

Date of certification: March 14, 2008

First revision: April 11, 2016

Reasons: i) The Standard Deviations (SD's) were revised to bring them into line with the method used for all other OREAS CRMs (pooled SD method). The original certification used a different method (involving standardising the laboratory means) that generated SD's that were overly constrained for practical use; ii) Indicative values have been added (see Table 2).

#### **REFERENCES**

ISO Guide 30 (1992), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2000), Reference materials – Contents of certificates and labels.

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.