

APPENDIX 2

2.1 Summary data obtained for the optimisation process of the SPECTRO 2000 ICP-MS.

Table 2A ICP-MS generator and ion optics parameters employed for PGM analysis of the RCCs.

Generator parameters	Specifications		Ion Optics parameters	Specifications
Plasma power (W)	1350		LO	-450
Pump step	2		LA	-110
Coolant step	3		LB	0.00
Auxiliary step	2		LC	-140
Nebulizer step	2		LD	-60
Neb. Flow (ml/min)	950		Field Axis	
Special valve	0		Pot.	5
Additional gas	0		Deflection (SEM)	
Mass flow (ml/min)	950		Inner	-500
Total flow			Outer	-200
Mass flow (ml/min)	2000			

2.2 Scanning graphs displayed to identify possible interferences on the isotopes of Pt, Pd, Au, Rh, Ru and Ir.

Figure 2A De-ionised blank and 100 ug L⁻¹ of Pt Spectrascan reference standard solution

Figure 2B Procedural blank, 100 ug L⁻¹ of Pt and 100 ug L⁻¹ of Cr, Fe, Se and Te Spectrascan reference standard solutions.

- Figure 2C** De-ionised blank and 100 ug L⁻¹ of Pd Spectrascan reference standard solution
- Figure 2D** Procedural blank, 100 ug L⁻¹ of Pd and 100 ug L⁻¹ of Cr, Fe, Se and Te Spectrascan reference standard solution.
- Figure 2E** De-ionised blank and 100 ug L⁻¹ of Rh Spectrascan reference standard solution
- Figure 2F** Procedural blank and 100 ug L⁻¹ of Rh and 100 ug L⁻¹ of Cr, Fe, Se and Te Specpure reference standard solution,
- Figure 2G** De-ionised blank and 100 ug L⁻¹ of Au Spectrascan reference standard solution
- Figure 2H** Firstly, scanning of 100 ug L⁻¹ of Au Spectrascan reference standard solution followed by De-ionised blank with a 1 minute washing period to demonstrate the memory effect of Au.
- Figure 2I** Procedural blank and 100 ug L⁻¹ of Au 100 ug L⁻¹ of Cr, Fe, Se and Te Spectrascan reference standard solution.
- Figure 2J** De-ionised blank and 100 ug L⁻¹ of Ru Spectrascan reference standard solution
- Figure 2K** Procedural blank and 100 ug L⁻¹ of Ru and 100 ug L⁻¹ of Cr, Fe, Se and Te Specpure reference standard solution.
- Figure 2L** De-ionised blank and 100 ug L⁻¹ of Ir Spectrascan reference standard solution
- Figure 2M** Procedural blank and 100 ug L⁻¹ of Ir and 100 ug L⁻¹ of Cr, Fe, Se and Te Spectrascan reference standard solution.

2.3 Microwave digestion parameters optimised for the digestion of the RCCs

Table 2B Multiwave 3000 digestion system operation parameters

Sample	Rotor	Sensor	Reagent	Temp/ Power	Ramp	Hold	Fan
Cleaning	HF 100-8	IR	6ml HNO ₃	500	5	15	1
				400		10	1
				0		15	3
Chromite (RCCs) *	HF 100-8	IR	8mls 1:1	500	10	30	1
		T	H ₂ SO ₄ :H ₃ PO ₄	0		15	3
		p					

Chromite – 0.2500g

IR – Infra red sensor, T – Temperature sensor, p – Pressure sensor

*Reduce the pressure increase rate to 0.3 bar/sec. Do not perform the digestion without the IR-sensor.

2.4 Hotplate digestion versus microwave digestion data for base metals.

Table 2C Significant testing data between HP and MW digestion for the elements Fe, Cr, Al and Mg.

	Fe259.941 MW - Fe	Fe259.941 HP - Fe	Cr266.871 MW - Cr2O3	Cr266.871 HP - Cr2O3	Al394.401 MW - Al2O3	Al394.401 HP - Al2O3	Mg280.270 MW - MgO	Mg280.270 HP - MgO
CHROM1	20.56	20.77	41.24	41.50	16.30	16.92	8.426	8.809
CHROM2	20.53	20.38	40.87	41.76	16.06	17.49	8.37	8.863
CHROM3	20.65	20.93	40.75	42.22	16.02	17.60	8.329	8.897
CHROM4	20.65	20.98	41.03	41.61	16.03	17.55	8.379	8.773
CHROM5	20.57	20.88	41.27	41.26	16.09	16.56	8.434	8.316
CHROM6	20.83	20.70	41.13	41.49	16.02	16.98	8.481	8.821
CHROM7	20.57	20.52	41.14	41.41	16.11	16.94	8.407	8.753
CHROM8	20.73	20.62	40.64	41.49	15.82	17.02	8.327	8.822

Mean	20.63	20.72	41.01	41.59	16.06	17.13	8.39	8.76
Stdev	0.10	0.21	0.23	0.29	0.13	0.37	0.05	0.18
%rsd	0.49	1.01	0.56	0.70	0.82	2.16	0.63	2.10
Tcalc		-1.07		-4.43		-7.73		-5.36
Tcrit(95%CI)		2.23		2.14		2.14		2.31

Table 2C Significant testing data between HP and MW digestion for the elements Mn, Ti and V.

	Mn257.611	Mn257.611	Ti336.121	Ti336.121	V292.464	V292.464
	MW - MnO	HP - MnO	MW - TiO2	HP - TiO2	MW - V2O5	HP - V2O5
CHROM1	0.454	0.482	1.185	1.108	0.479	0.448
CHROM2	0.441	0.437	1.151	1.135	0.469	0.453
CHROM3	0.442	0.433	1.156	1.135	0.471	0.453
CHROM4	0.442	0.445	1.146	1.166	0.467	0.461
CHROM5	0.445	0.469	1.151	1.226	0.469	0.476
CHROM6	0.439	0.424	1.142	1.099	0.467	0.441
CHROM7	0.448	0.424	1.172	1.102	0.473	0.44
CHROM8	0.447	0.426	1.163	1.11	0.475	0.443
Mean	0.287	0.286	1.158	1.135	0.471	0.452
Stdev	0.005	0.022	0.014	0.043	0.004	0.012
%rsd	1.68	7.65	1.24	3.79	0.89	2.67
Tcalc		0.29		1.44		4.29
Tcrit(95%CI)		2.31		2.26		2.26

APPENDIX 3

3.1 GFAAS parameters used during optimisation of the method for the elements Pt and Pd

Palladium

Measurement mode: Graphite furnace

Wavelength (nm): 244.8

Slit (nm): 0.2

High Voltage: 383.50

Lamp Current (mA) 6.0

Lamp Element: Pd

Background Correction: No

Table 3A GFAAS parameters developed for the element Pd

No.	Temp.	Elevation Time	Hold Time	Atomization	Internal Gas Flow
1	85	5	5		Mid
2	95	20	10		Mid
3	120	10	10		Mid
4	900	10	10		Mid
5	2500	0	2	*	Off
6	2500	0	1		Mid

Platinum

Measurement mode: Graphite furnace

Wavelength (nm): 265.9

Slit (nm): 0.2

High Voltage: 451

Lamp Current (mA) 7.0

Lamp Element: Pt

Background Correction: No

Table 3A GFAAS parameters developed for the element Pt

No.	Temp.	Elevation Time	Hold Time	Atomization	Internal Gas Flow
1	85	5	3		Mid
2	95	20	10		Mid
3	120	10	10		Mid
4	900	10	10		Mid
5	2500	0	2	*	Off
6	2500	0	1		Mid