Round robin standard composition: HCFeMn reference material

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INTRODUCTION

In order to test a novel electrical configuration for the production of high-carbon ferromanganese (HCFeMn) at Mintek's pilot facilities, internal reference standards (IRMs), for HCFeMn grade B metal and associated slag (ASTM International, 2009) were produced to use for quality control purposes.

METHODOLOGY

The as-received samples were crushed, screened and milled in preparation for analysis according to the ISO standards (ISO Guide 31, 2000; ISO Guide 32, 1997; ISO Guide 33, 2000; ISO Guide 34, 2000; ISO Guide 35, 1989). Three laboratories, namely Mintek Analytical Services Division (ASD), and two laboratories based at HCFeMn production plants in South Africa, participated in the collaborative analytical programme. Each laboratory was allowed to use its preferred analytical technique. The phase chemistry of the samples was analysed by Mintek's Minerology Division using the X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive spectrometry (SEM-EDS), and electron microprobe analysis (EPMA).

RESULTS AND DISCUSSION

The particle size distribution (PSD) of the milled samples was determined to confirm the target grind of D_{95} passing 75 µm (Rivera and Rodríguez, 2010). This was done to render the sample as homogeneous as possible to meet the laboratory requirements.

The three statistical parameters calculated for each data-set are the mean or average (µ), standard deviation (STD, σ), and the relative standard deviation (RSD). RSD (Equation [1]) is used to express the precision and repeatability of an assay and is mainly used for quality assurance studies

$$RSD = (\frac{\sigma}{\mu})\times 100 \qquad [1]$$

Table I and Table II show the compositions of the alloy and slag respectively as analysed by the three laboratories. The RSD values of 16.64, 65.77, and 14.28 (for Si analysis) from Table I and RSD values of 25.64, 51.12, and 18.92 (for FeO analysis) from Table II indicate that the composition mean was random and spread out. The robust RSD of the data-sets must be less than 33% (Hansen, Ring, and Steele, 1983) with a confidence level of 68%. A reasonable explanation for this is the low concentration of Si in the alloy and FeO in the slag. The RSD values for Si and FeO by Laboratory 1 are too high, making the analyses unreliable, and this data was therefore excluded when calculating the composition of the IRMs.

Table I. Bulk chemical composition of the alloy

Lab/detection limit		C	Mn	Si	Fe
ASD (0.05%)	Mean	6.8	78.0	0.27	16.1
	STD	0.2	1.5	0.03	0.4
	RSD	2.4	1.9	16.64	2.5
Laboratory 1	Mean	6.73	76.55	0.36	12.71
	STD	0.02	0.13	0.23	0.12
	RSD	0.36	0.17	65.77	0.91
Laboratory 2	Mean	-	76.32	0.042	16.33
	STD	-	0.38	0.006	0.17
	RSD	-	0.50	14.28	1.05

Table II. Bulk chemical composition of the slag

Lab/detection limit		MgO	MnO	FeO	Al_2O_3	CaO	SiO_2
ASD (MgO [0.08], MnO [0.06], FeO [0.06], Al_2O_3 [0.09], CaO [0.07], SiO_2 [0.07])	Mean	6.9	26.7	0.24	3.20	31.48	28.1
	STD	0.2	0.6	0.06	0.13	0.48	1.1
	RSD	2.7	2.2	25.64	4.05	1.54	3.8
Laboratory 1	Mean	7.61	25.60	0.113	2.62	29.84	29.72
	STD	0.04	0.16	0.058	0.05	0.12	0.10
	RSD	0.52	0.61	51.327	1.88	0.41	0.35
Laboratory 2	Mean	7.546	25.89	0.1825	3.2143	30.593	30.515
	STD	0.286	0.46	0.0345	0.2067	0.337	0.216
	RSD	3.790	1.77	18.9041	6.4306	1.102	0.707

CONCLUSIONS

IRMs for high-carbon ferromanganese (HCFeMn) grade B alloy and slag were prepared. The IRM compositions of both alloy and slag are summarized in Table III and Table IV respectively. The HCFeMn alloy and slag IRMs were used for quality control purposes to validate the analytical data generated during the pilot plant campaign.

Table III. Alloy composition

	C	Mn	Si	Fe
Reference	6.79	78.03	0.272	16.09
Campaign	6.75	76.01	0.260	15.93
Campaign RSD	6.71	1.44	8.785	0.87

Table IV. Slag composition

	MgO	MnO	FeO	Al_2O_3	CaO	SiO_2
Reference	7.36	26.06	0.22	3.01	30.64	29.452
Campaign	7.27	26.20	0.29	3.01	30.31	29.830
Campaign RSD	0.74	2.09	7.03	4.07	2.20	2.965

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