THE ANALYSIS OF SLAGS AND RELATED OXIDE-TYPE MATERIALS

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 $^{\odot}$ by American Society for Testing and Materials 1973

W. Ramsden

The Application of Multi-Channel Spectrometers to the Elemental Analysis of Oxide Materials





Fig. 2 Effect Of Grinding Time on XRF Analysis Of Sinters.





















TABLE 1

cc	OMPOSITION (OF INDUS	TRIALLY	MPORTANT	OXIDES	
.	Iron Ore	Fe Rei	fractories (Cement	Class	Cryolite
Constituent	Sinter			Imestone	Glass	40-60
Nan O	0.1-1	0.1-10	0.05-0.2	0.05-1	10-15	20-30
A1203	0.4-25	0.3-25	0.1-98	0.5-20	1-3	10-65
Mg 0	0.1-6	0.1-25	0.01-65	0.1-6	0.1-5	
Si 02	0.5-40	0.2-60	0.5-99	1-65	65-75	1-40
P2 05	0.01-10	0.1-20				
S 03	0.005-0.5	0.01-10		0.02	0.05-0.5	
K2 0	0.2-1.5		0.01-11	0.1-3	0.5-1.5	
Co 0	0.1-25	0.4-60	0.06-3	1-55	10-15	3-6
Ti 02	0.04-1	0.02-2.5	0.01-2	0.1-0.2		0.5-3
Cr2 03		0.01-6.5	0.01-40		0.15-0.25	i
Mn 0	0.05-2.5	0.1-20	0.01-0.5	0.05-0.2		0.5-50
Fe2 03			0.03-16	0.1-10	0.04-0.5	
Fe	18-70	0.2-70				

TABLE 2

DAY-TO-DAY REPRODUCIBILITY OF IRON ORE SINTER ANALYSIS (QUANTOVAC - BRIQUETTE)

Day	Fe	CoO	SiO ₂	P2O5	MnO	MgO	A12O3	S
1	54.8	7.2	8.05	0.33	1,55	1.10	3.00	0.010
2	54.6	7.3	8.12	0.35	1.57	1,10	3.03	0.009
3	54.6	7.3	8.20	0.33	1.53	1.07	2.96	0,009
4	54.7	7.4	8.07	0,35	1.56	1,13	2,79	0.009
5	55.0	7.2	7,83	0.34	1.58	1,10	2.79	0,009
6	54.7	7,2	8.09	0.34	1,49	1.10	3.06	0.007
7	54.4	7.6	8.22	0.35	1.56	1.09	2.92	0.007
8	54.4	7.5	8.22	0.34	1.56	1.12	2.98	0.008
9	54.5	7.4	8.18	0.35	1,56	1.08	3.00	0.008
10	54.5	7.4	8.24	0.34	1.57	1.09	3.02	0.009
11	54.7	7.2	8.18	0.35	1,60	1,10	3.08	0.007
Mean analysis	54.6	7.3	8.13	0.34	1.56	1.10	2.97	0.008
Range:min.	54.4	7.2	7.83	0.33	1,49	1.07	2,79	0.007
mox.	55.0	7.6	8.24	0.35	1.60	1,13	3.08	0.010
Standard deviation	0.18	0.14	0.118	0.008	0.028	0.017	0.089	0.0010

A.C.Knott, E.D.Aldaya. J.Iron & Steel Institute November 1964

	PERFORM	ANCE OF	MULTI	CHAN	NEL XRF	SYSTEMS	
		ON OXI	DE MA	TERIAL	s (1960-6	5)	
	Material	Constituent	Preci s.d. d	ision at %	Accura Av. Dev.	<u>cy</u> <u>% Range</u>	
	Slag Slag Slag Sinter Sinter Cement	Fe Fe 0 Fe 0 Fe Fe Fe ₂ 0 ₃	0.055 (0.02 (0.02 (0.055 (0.01 (0.009(25% 25% 27% 61% 57% 3.4%	0.25 0.45 0.6 0.25 0.1 0.08	52-64% 14-30% 20-30% 52-64% 55-65% 2-4.5%	
	Slag Slag Sinter Sinter Cement	Ca 0 Ca 0 Ca 0 Ca 0 Ca 0	0.07@ 0.038@ 0.004@ 0.01@ 0.02@	35% 38% 7.5% 5.3% 41%	0.15 0.37 0.25 0.09 0.10	14-54% 35-45% 5-10% 2-10% 38-48%	
	Slag Slag Sinter Sinter Cement	Si 02 Si 02 Si 02 Si 02 Si 02 Si 02	0.15@ 0.08@ 0.06@ 0.17@ 0.11@	24% 33% 5.4% 6% 17%	0.28 0.6 0.1 1.5 0.07	5-56% 30-36% 4-13% 4-7% 8-18%	
	EFFECT	OF GRI INSITIES	TABLE	<u>4</u> 3 TIA 0N	AE ON ORE SIN	XRF NTER	
Grinding	lime (m	in). 0	0.5	1	2	4	8
Low Gro Apparent Apparent Apparent	ide Ore Fe % Ca 0 % Si 02 %	38.9 17.6 13.1	39.5 17.9 13.9	39.6 18.0 13.8	39.9 18.1 13.3	40.2 17.7 12.7	40.5 17.5 11.7
High Gr Apparent Apparent Apparent	ade Ore Fe % Ca 0 % Si 02 %	58.2 5.6 5.6	60.i5 5.6 5.3 TABLE	61.1 5.8 4.9 5	61.1 5.7 4.5	61.4 5.7 3.8	61.1 5.5 2.9
EFFECT	OF G	RINDING	TIME IRON	ON ORE	XRF I	NTENSIT	IES
Grindi	ng time	(min), 0.1	5	4		·	
Appare Appare Appare	ent Fe % ent Ca 0 ent Si 0 ₂	59.0 % 6.0 % 5.5)) 5	59.0 5.8 5.5	-) ;		

TABLE 3

TABLE 6

TYPICAL	<u>72000 X-RA</u>	Y QUANT	OMETER ARRAY FOR
	OXIDE M	ATERIALS	
Element	Wave -length	Crystal	Detector
No Kat Mg Kat Al Kat Si Kat S Kat Co Kat Ti Kat Cr Kat Mn Kat Fe Kat	11.909 Å 9.889 Å 8.339 Å 7.126 Å 6.155 Å 5.373 Å 3.744 Å 3.360 Å 2.750 Å 2.291 Å 2.103 Å 1.937 Å	4" RAP 4" ADP 4" E DDT 4" E DDT 4" Ge 4" Ge 4" Ge 4" LiF 4" LiF 4" LiF 4" LiF 4" LiF	Flow Prop. P-10. P.H.D. Ne Prop. Al Window P.H.D. Ne Prop. Al Window P.H.D. Ne Prop. Be Window P.H.D. Ne Prop. Be Window P.H.D. Ne Prop. Be Window P.H.D. Ne Multitron Be Window Ne Multitron Be Window Ne Multitron Be Window Ne Multitron Be Window

X-ray Tube: Rh; 50 Kv 40 ma

TABLE 7

<u>co</u>	OMPARISON	OF SHORT-TERM	PRECISION	<u>O</u> F
72	000 - FUSIO	N TECHNIQUE (1972) AND	
BR	IQUETTING	TECHNIQUE (1960 -	- 1965)	
Constituent	Material	Briquette (1962)	72000 - Fu	usion (1972)
		S.D.	Dil. Factor	<u>S.D.</u>
Fe	Sinter Ores Slag	0.01-0.05@50-60% 0.008@1%	11,28 5.6	0.02-0.04@45-67% 0.001-0.002@0.4-2%

Fe	Sinter Ores	0.01-0.05@50-60%	11,28	0.02-0.04@ 45-67%
	Slag	0.008 @ 1%	5.6	0.001-0.002@0.4-2%
Mn	Any	0.001-0.1@0.2-2%	5.6, 11,28	0.001-0.004@0.05-2%
Si 02	Any	0.06-0.4@5-35%	5.6, 11,28	0.02-0.07@ 1-40%
Ca O	Any	0.01-0.07@ 30-40%	5.6, 11,28	0.01-0.04 @ 1-45%
Mg 0	Any	0.05-0.06@2-10%	5.6, 11,28	0.02-0.04 @0.02-6.5%
Al2 03	Any	0.03-0.1@2-10%	5.6, 11,28	0.01-0.02@0.2-15%

R. Jenkins

Current Status of X-ray Emission Analysis for the Analysis of Slags and Related Oxide-Type Materials



Note: All errors quoted are given in terms of 1 σ

Figure #1



Phase	Fluorescing element	Absorption for measured wavelength	Effect
Phase 1 Phase 2	Present Absent	Same	Grain size
Phase 1 Phase 2	Present Absent	Different	Inter-mineral
Phase 1 Phase 2	Present Present	Different	Mineralogical

The particle effect in X-ray fluorescence spectrome try (see text).

Figure #2



Figure #3



ALGORITHMS FOR MATRIX CORRECTION $I_{i} = \frac{W_{i} \quad K_{i}}{\sum_{j} \quad W_{j} \quad \alpha_{j}}$ $I_{i} = \frac{W_{i} \quad K_{i}}{1 \quad + \quad \sum_{j} \quad \alpha_{ij} \quad W_{j}}$ $I_{i} = \frac{W_{i} \quad K_{i}}{1 \quad + \quad \sum_{j} \quad k_{ij} \quad I_{j}}$

Figure # 5



Comparative resolutions of the flow counter, the scintillation counter, the semiconductor counter, and the crystal spectrometer.

Figure #6

M. D. Amos

Atomic Absorption Spectrophotometry in the Analysis of Slags and Oxides

Concentration Ranges (%) (5 g / 100 ml)								
Detection Limit Abs = 0.88 Burner at 90°	Zn 0·000002 0·004 0·08	Fe 0:00001 0:025 0:5	AI 0-00004 0-3 6	Si 0·0005 0·6 12	Ca 0·000004 0·012 0·24			
	F	7 IG . 1						
Concentration of Analyte = 0.100 % Solution of 1g / 1000 ml Dilution is 1000:1 Concentration in Solution is 1.00 mg/l If Analytical Measurement is in Error by 1 % (+ve) Concentration Found will be 1.01 mg/l and Result Obtained will be 0.101 % i.e. 1 % Error FIG. 2								
100 mg Ceme 500 mg Li ₂ B ₄ Fusi 75 ml HCl with La +Cs J Dilu 100 ml	ort O ₂ ion ition Difution	→ AI+: (0-5- → Ca,1	Sidet [≞] -5%)(41g+Fe 35%)(1	l. 8-25 %) det ⁿ . ⋅3-3⋅3 %) (0-8-4 %)			

FIG. 3



FIG. 4

If Dilution Error of 1% (-ve) is Made, Result will be 0.102% i.e. 2% Error

If Dilution Error of 0.1% (-ve) is Made,Result will be 0.1011 % i.e. 1.1 % Error

FIG. 5

DISADVANTAGES OF SOLUTION METHODS

- 1. Requirement to Devise Solution Method
- 2. Additional Handling
- 3. Possibility of Dilution or Calculation Errors
 - FIG. 6

ADVANTAGES OF SOLUTION METHODS

- 1. Minimization of Inhomogeneity Errors
- 2. Ideal "Iso Formation"
- 3. Ease of Standardization
- 4. Ease of Dilution

FIG. 7

	Si O ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO
1	14.93	2.23	2.62	42.74
2	15.09	2.28	2.62	42.60
3	15.04	2.20	2.54	42.74
4	15.09	2.20	2.62	42.74
5	14.85	2.23	2.62	42.46
6	15.09	2.18	2.62	42.87
7	14.89	2.20	2.62	42.87
8	15.09	2.29	2.69	42.60
9	14.82	2.23	2.62	42.60
10	14.99	2.14	2.62	42.46
11	15.09	2.15	2.62	43.01
12	14.99	2.20	2.62	42.72
Deviation	0.336	0.029	0.037	0.166
Average	14.98	2.21	2.62	42.70
C.O.V.	0.80%	2.0%	1.18%	0.39%

FIG. 8

CaO → Ca[°] + (O)

CaOSi O_2 \rightarrow Ca O. Si O_2

 $\begin{array}{c}
\text{Ca O} \\
\text{Si O}_2 \\
\text{Sr O}
\end{array} \xrightarrow{} \begin{array}{c}
\text{Ca}^\circ + (O) \\
\text{Sr O}_2 \\
\text{Sr O}_2 \\
\text{Sr O}_2
\end{array}$

FIG. 9

$$K^{\circ} \Longrightarrow K^{+} + e^{-}$$

FIG. 10

Concentration Ranges (%) (0.1 g / 100 ml)

	Zn	Fe	AI	Si	Ca
Detection Limit	0.0001	0.0002	0.002	0.025	0.0002
Abs = 0.88	0·2	1.25	15	30	0.6
Burner at 90°	4	25	100	100	12
	F	IG. 11			

Concentra	tion Ran	ges (%))(1g/	100 ml	<u>)</u>
	Zn	Fe	AI	Si	Ca
Detection Limit	0.00001	0.00005	0.0002	0.0025	0.0002
Abs = 0.88	0.02	0.125	1.5	3	0.06
Burner at 90°	0.4	2.5	30	60	1.2
	F	IG. 12			

J. R. Ryan and R. K. Scott

Utility of the Optical Emission Spectrometer in the Analysis of Refractories, Slags, and Other Oxide Materials

No Figures or Tables.

J. C. Cline and R. A. Pontello

X-Ray Emission Spectrometer Analysis of Slags and Related Materials



FIG. 1



FIG. 2

	1	ABLE 1				TA	IBLE 2		
CR203			*SWE	DE SLAGS	<u>\$102</u>			*SWEDE	SLAGS
NO.	<u>CERTIFIED</u>	<u>DIRECT</u>	<u>EUSION</u>	HV. ABS. F <u>uston</u>	<u>NO.</u>	CERTIFIED	DIRECT	FUSION	EUSION
1	.20	.28	.22	-	1	19,40	19.64	18.94	19.26
2	.10	,15	.080	-	2	5.50	5,89	5.46	5.80
3	0	-	-	-	3	34.0	32,88	34,41	33,90
4	0	-	-	-	4	34.10	31,35	34.40	33.44
5	,15	.37	.25	-	5	57.00	57.20	56,91	56.39
6	.09	.082	.081	-	6	23.70	26.03	24.20	24.00
7	.46	.45	,43	-	7	20.50	19,68	20.01	20,00
8	3.70	3,45	3.82	-	8	39,90	40.90	40.82	40.79
۸DJ	COEF OF COREL	,9973	.9991	-	ADJ.	COEF OF COREL.	.9918	, 9992	,9990
STD.	ERROR OF EST.	.0741	.0442		STD.	ERROR OF EST.	1.4098	,4321	.4883

	TA	BLE <u>3</u>				TA	NBLE 4		
EE0			*SWE	DE SLAGS	MNO			*SWEDI	E SLAGS
NO.	<u>CERTIFIED</u>	<u>DIRECT</u>	FUSION	EUSTON	<u>NO.</u>	<u>CERTIFIED</u>	DIRECT	FUSION	HV ABS. FUSTON
1	16,60	17,83	17.11	16,45	1	18.60	15.92	18.56	18.40
2	25,00	20,45	21.21	23.66	2	4.80	4.16	4.60	4.65
3	1.10	1.01	1.16	1,32	3	2.40	2.50	2,45	2,33
4	,40	.35	.44	,60	4	1.60	1.94	1.76	1.58
5	26.80	26.40	26.77	28.75	5	9,80	13.26	13.17	10.59
6	4,20	4.07	3,92	4,27	6	2.20	2.17	2.09	2.13
7	18,70	18.05	17.65	17,74	7	5,30	4.67	5.13	5,10
8	,80	.70	, 94	1.06	8	4.70	4.67	4,97	4.73
		08/15	0007	0075	ADJ.	COEF OF COREL	. 9291	, 9624	,9971
	COEF OF COREL	C POE -	, 5 5 0 7		STD.	ERROR OF EST.	1,5039	1.0947	.3017
STD.	ERROR OF EST.	1,4050	1.1108	.9137					

		TABLE <u>5</u>				TA	BLE <u>6</u>		
AL203			*SWE	DE SLAGS	<u>CA0</u>			*SWE	DE SLAGS
NO.	<u>CERTIFIED</u>	<u>DIRECT</u>	FUSION	HV. ABS. FUSION	NO.	<u>CERTIFIED</u>	DIRECT	FUSION	HV ABS. Fusion
1	3.10	3.50	3.07	3,13	1	32,60	34.56	33.42	33,07
2	.70	.57	.67	.69	2	43,90	45.68	45.04	44,18
3	9,40	9.67	9,33	9,29	3	42.20	38.61	40.53	41.76
4	12.40	12.26	12.25	12.11	4	40.00	36.35	38.84	39,55
5	1,70	1.57	1,74	1.66	5	2.20	3.32	2,55	2.22
6	1,90	1.79	1.83	1.81	6	59,10	58.25	58.92	59.25
7	2.10	2.17	2,06	2.07	7	44.00	45.15	45.09	43,90
8	6,80	6.86	6.94	6,92	8	36,50	35.60	35,95	36,30
ADJ,	COEF OF COREL	9980	,9997	, 9995	ADJ.	COEF OF COREL.	,9842	.9964	,9996
STD.	ERROR OF EST.	,1913	.0758	,0922	STD.	ERROR OF EST.	2.0401	.9753	,3078

TABLE 7

Ŧ 0		-			MGO				*SWEDE SLAGS
1102			*SWED	E SLAGS	NO	CEPTIEIED	DIRECT	FUSTON	HV.ABS.
NO.	<u>CERTIFIED</u>	DIRECT	FUSION	HV.ABS. <u>EUSION</u>	<u>1124</u>		<u>KTUCKT</u>		<u>103101</u>
					1	8.00	6.48	8.37	8.08
1	,53	.57	.60	.61	2	8,70	6,56	8.89	8,58
2	.17	.16	.18	.21	3	5,20	6.29	5.03	5,02
3	,89	.75	.85	.84	4	6,70	7,60	6,91	6,14
4	1.25	1.10	1.23	1.24	5	1,50	2.32	1.94	1.37
5	,42	.78	.65	,63	6	5,90	6.30	5,99	5.73
6	.18	.13	.16	.136	7	6.70	6.10	6.55	6,45
7	, 34	.32	.35	,35	8	5,70	6.67	5.45	5,61
8	,26	.25	.26	.30	ADJ,	COEF OF CORE	EL, .8542	,9879	, 9939
ADJ,	COEF OF COREL	.8726	, 956	.9604	STD,	ERROR OF EST	r8322	.2393	.1698
STD,	ERROR OF EST.	1360	.0799	.0759					

TABLE <u>8</u>

<u>FE0</u>		(R'Fe = R	; # RFE EXP & RCA) 1					
<u>10</u> .	<u>CERT IFIED</u>	FUS Observed	io:i <u>corrected</u>	II.A. F <u>Odserved</u>	usion <u>corrected</u>			
1	16.6	17.1	17.6	16.5	16.9			
2	25.0	21.2	24.2	23.7	25.5			
3	1.1	1.16	,96	1.32	1.07			
4	.4	,44	.21	.60	,28			
5	26.8	26.8	26.6	28,8	26.8			
6	4.2	3.92	4.10	4,27	4,41			
7	18.7	17.7	19.1	17,7	18,8			
8	.8	.94	,73	1.06	,78			
ADJ. STD.	COEF OF COREL.	,9903 1,1108	,9981 ,4884	.9335 .9137	,9998 ,168			

TABLE 3A (R'Fe = RFE exp g Rca

H. G. Zelinske and D. H. Arendt

Analysis of Slags by Atomic Absorption Spectrophotometry

PERCENT	CONCENTRATION	RANGES	OF	SLAG	CONSTITUENTS

CONSTITUENT	% CONC. RANGE
SILICON OXIDE	o — so.o
CALCIUM OXIDE	0 - 80.0
ALUMINUM OXIDE	o — 60.0
IRON OXIDE	0 — 50.0
CHROMIUM OXIDE	0 — 4 8.0
MAGNESIUM OXIDE	0 40.0
MANGANESE OXIDE	0 - 30.0
TITANIUM OXIDE	0- 1.25







FIG. 3



FIG. 4



FIG. 5



FIG. 6



FIG. 7



FIG. 8



FIG. 9



FIG. 10

8.0.F. SLA	<u>G</u>	
CONSTITUENT	LAB.A	AMSTED
CHROMIUM OXIDE	-	.13
MANGANESE OXIDE	4.8	4.56
IRON OXIDE	26.5	25.76
CALCIUM OXIDE	44.2	45.0
MAGNESIUM OXIDE	5.5	5.68
ALUMINUM OXIDE	1.52	2.0
SILICON OXIDE	15.7	16.7
SILICON OXIDE	15.7	16.7

FIG. 11

ELECTRIC FL	JRNACE SLAG					
STAINLES	S STEEL					
CONSTITUENT	LAB.A	AMSTED				
CHROMIUM OXIDE		.20				
MANGANESE OXIDE	. 96	.98				
IRON OXIDE	3.46	3.21				
CALCIUM OXIDE	24.48	24.8				
MAGNESIUM OXIDE	6.30	6.12				
ALUMINUM OXIDE	17.19	18.4				
SILICON OXIDE	45.60	45.5				
FIG . 12						

BASIC SLAG 8.C.S. NO. 174-1 CONSTITUENT B.C.S. AMSTED CHROMIUM OXIDE -_ MANGANESE OXIDE 5.11 5.04 IRON OXIDE 10.88 10.95 44.83 44.1 CALCIUM OXIDE MAGNESIUM OXIDE 7.18 7.2 ALUMINUM OXIDE 1.72 1.9 SILICON OXIDE 14.69 15.20

IRON			
CONSTITUENT	LAB.A	LAB.8	AMSTED
CHROMIUM OXIDE	_	-	.328
MANGANESE OXIDE	4.33	3.85	4.29
IRON OXIDE	6.94	7.72	8.55
CALCIUM OXIDE	15.50	15.4	16.0
MAGNESIUM OXIDE	10.00	9.77	10.15
ALUMINUM OXIDE	12.44	9.66	9.4
SILICON OXIDE	50.8	49.72	50.8

FIG. 13

FIG. 14