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THE USE OF FLAME PROCEDURES FOR THE ANALYSIS OF MINERALS, ORES AND ELECTRIC FURNACE SLAGS

PART III: DETERMINATION OF SILICON IN ORES AND THEIR MIXTURES, AND IN ELECTRIC FURNACE SLAGS

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EXTRACTION METALLURGY DIVISION

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The Use of Flame Procedures for the Analysis of Minerals, Ores, and Electric Furnace Slags
Part III: Determination of Silicon in Ores and Their Mixtures, and in Electric Furnace Slags

by

R. J. Guest* and D. R. MacPherson**

SUMMARY

Rapid procedures, suitable for control and many other purposes, are described for the determination of silicon in electric furnace slags and associated materials and in ores and their mixtures. After sample dissolution by hydrofluoric acid in a Teflon bomb or after a sodium peroxide fusion and subsequent acidification, silicon is determined by an atomic absorption procedure. Other elements may be determined on the same solution as the silicon because no chemical separations have been made. Atomic absorption and chemical results are compared, and the precision found for the atomic absorption procedures is shown. The effectiveness of two atomizer-burner systems is compared and it is shown to be necessary, in one of these systems, to add a major contaminant to the comparison standard in order to obtain suitable accuracy.

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L'Utilisation du procédé à flamme pour l'analyse des minéraux, des minerais et des scories provenant du four électrique

3^e Partie: La Détermination du silicium dans les minerais et leurs mélanges et dans les scories provenant du four électrique

par

R. J. Guest* et D. R. MacPherson**

Résumé

Les auteurs décrivent les procédés rapides, qui sont convenables pour le contrôle et pour plusieurs autres buts, pour la détermination du silicium dans les scories provenant du four électrique et les minéraux associés et dans les minerais et leurs mélanges. Après la dissolution de l'échantillon par l'acide fluorhydrique dans une bombe Téflon ou après la fusion du peroxyde de sodium et l'acidification subséquente, le silicium est déterminé par un procédé d'absorption atomique. D'autres éléments peuvent être déterminés dans la même solution comme le silicium parce qu'il n'y a pas eu de séparations chimiques. Les auteurs ont comparé l'absorption atomique et les résultats chimiques, et la précision trouvé pour les procédés d'absorption atomique est démontrée. Ils ont comparé l'efficacité de deux systèmes de brûleur - atomiseur et il paraît nécessaire d'ajouter à un de ces systèmes une impureté majeure à la norme de comparaison afin d'obtenir une précision convenable.

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INTRODUCTION

In research at Mines Branch on the electric-furnace production of ferroalloys, analytical results must be rapid in order to achieve satisfactory process control. Recently to provide this analytical service, we have developed rapid, accurate procedures(1).

Of the elements required, among the most difficult to determine rapidly are silicon and aluminum. This report describes the application of an atomic absorption procedure to the determination of silicon in ores, slags, and associated materials. It is an extension of the work, described in Part II of this series(2), on the atomic absorption determination of silicon in minerals and their mixtures and it serves to widen the effective range of application of the procedure.

In Part I of this series(3), work of other investigators on silicon determination by atomic absorption was reviewed. This literature search revealed that little work had been reported on silicon determination in slags and associated materials from high-temperature furnace work and, in particular, on sample materials from the smelting of ilmenite and manganese ores.

Of the reported work, Langmyhr and Paus(4) described the atomic absorption analysis of silicon and several other elements in an iron ore and basic slag after a Teflon bomb dissolution. Galloway and Reid(5), Van de Vrande(6), and Reid et al(7) described the atomic absorption analysis of several elements, including silicon, in blast furnace slags and sinters, and/or in iron ores and slags, subsequent to dissolution in acid or to fusion techniques, not detailed.

The procedures, reported here, were applied primarily to materials high in iron and titanium or in iron-manganese materials. As suitable standard samples were not available for testing the procedure, mixtures of Certified Standard samples were prepared and analysed.

APPARATUS AND REAGENTS

Apparatus

Teflon bomb, model 4745, Parr Instrument Company, Moline, Illinois, U.S.A.

Zirconium crucibles, 45-ml.

Jarrell-Ash atomic absorption spectrophotometer, model 82-300, dual double-beam fully compensated unit

Techtron atomic absorption spectrophotometer, model AA-3, with model AA-5 burner-atomizer

Drying oven

Plastic bottles, various sizes

Volumetric flasks, various sizes

Muffle furnace

Reagents

Hydrofluoric acid, concentrated

Hydrochloric acid, concentrated

Nitric acid, concentrated

Boric acid, reagent grade

Sodium Peroxide, reagent grade

Sodium solution (peroxide, chloride or nitrate), 5% (w/v); store in plastic

Nitrous oxide cylinder

Acetylene cylinder

Standard Silicon Solution

Weigh out fused silica powder and take it into solution with either a Teflon bomb or a sodium peroxide fusion procedure. Treat the standard in the same manner as the samples and as described later under Dissolution Procedures. This solution should contain between 200 and 500 ppm of silicon.

Take an aliquot of the main silicon solution, add enough hydrochloric acid to ensure that the final solution will be acid, add anough sodium solution to bring the sodium content to about 6000 ppm, and dilute the solution to volume.

If solutions of silicon plus contaminants are required, add the contaminant to the diluted silicon standard just before making up the standard to the mark in the volumetric flask.

The final solution should contain between 10 and 200 ppm silicon.

PROCEDURE

Dissolution Procedures

1. Teflon Bomb Procedure

Weigh between 0.2 and 0.5 g of sample into the sample container of the Teflon bomb. Add 2 to 3 ml of aqua regia as a wetting agent, then 3 ml of concentrated hydrofluoric acid. Cover the Teflon cup and enclose it in the metal shell, close the bomb hand-tight. Place the bomb in a drying oven set at 140°C for about 45 minutes, then cool it to room temperature in a cold-water bath before loosening its screw-top. Transfer the sample to a plastic beaker; rinse the Teflon cup with 20 ml of warm 14% boracic acid solution, and transfer the sample solution to a 100-ml volumetric flask and dilute to the mark with water. Mix, and transfer the solution to a plastic bottle for further dilution, for direct atomization, or for storing.

2. Sodium Peroxide Fusion

Weigh between 0.5 and 2 g of sample into a zirconium crucible, add sodium peroxide from a 5-g weighed portion and mix intimately, finally covering with the remaining sodium peroxide. Cover the crucible with a zirconium cover and place it in a muffle furnace at 640°C for 30 minutes. Cool.

Place the crucible and contents in a plastic beaker and add water gradually to keep the reaction moderate, until the melt is leached from the crucible. Add concentrated hydrochloric acid to the beaker containing the crucible until the solution becomes acid and clears up. After starting to add acid, add the entire amount at once, otherwise silicon may come out of solution. Add enough acid to bring the solution to between 3 and 5% hydrochloric acid, remove the crucible and rinse it with distilled water. Transfer the solution to an appropriate volumetric flask and dilute to the mark.

Atomic Absorption Procedure

Take an aliquot of the main sample solution and place it in an appropriate volumetric flask. Add enough of a sodium solution (see reagents) so that the final dilution for atomization will be about 6000 ppm in sodium, making sure that the sample solution remains well on the acid side by adding hydrochloric acid. Dilute to the mark with water. This solution should contain between 10 and 200 ppm of silicon.

Atomize the sample directly, and, on the Jarrell-Ash atomic absorption spectrophotometer, compare readings with pure silicon standards which are close to the amount of silicon expected in the sample and which, preferably, span the sample content.

Atomic Absorption Parameters with the Jarrell-Ash Dual Double-Beam (8) Spectrophotometer, Model 82-300

Wave length - 251.6 nm

Lamp current - 10 mA

Burner - Hetco total consumption, with Tri-Flame laminar-flow head

Burner height - 19 cm from the burner top to the bottom of the burner holder

Range - variable

Slit - 100 and 150 microns

Fuel mixture - Nitrous oxide and acetylene

Flame type - just luminous

Atomic Absorption Parameters with the Techtron Spectrophotometer, Model AA-3 (9)

Wave length - 251.5 nm

Lamp current - 12 mA

Burner - burner and atomizer assembly for the Model AA-5, with plain-slot and grooved burner heads

Slit - 50 microns

Fuel mixture - nitrous oxide and acetylene

Flame type - just luminous

EXPERIMENTAL

A. Application of the Procedures to Certified Standard Samples and their Mixtures

A number of Certified Standard samples were chosen, and mixtures of them prepared, to approximate in content the type of sample material encountered in high-temperature furnace work. The composition of these sample mixtures, as shown in Table 1, leaned heavily toward high-titanium sample material, as methods to obtain gravimetric silicon results on slags from smelting of ilmenite ore were especially time-consuming and not always reliable. All samples analysed were found to be readily dissolved by the Teflon bomb and sodium peroxide fusion procedures.

1. Comparison of Silicon Results Using Both Pure- and Contaminant-Added Standards

a) Hydrofluoric-Boric Acid Medium

Two mixtures of Certified iron ore and titanium dioxide were analysed for silicon using both Teflon bomb and peroxide fusion procedures. In all cases, hydrofluoric and boric acids were present or added, and atomic absorption measurements were compared with both pure and contaminant-added silicon standards. The samples were analysed in two sets, designated as sets A and B, which were done at different times but are disparate primarily because of the varying atomizer efficiency encountered during their analyses. For these tests, a Techtron Atomic Absorption Spectrophotometer, Model AA-3, was used.

TABLE 1

Composition of Certified Standard Reference Samples Analysed for Silicon Using Atomic Absorption Procedures

Sample Type		Silicon Present % SiO_2	Aluminum Present % Al	Calcium Present % CaO	Magnesium Present % MgO	Titanium Present % TiO_2	Iron Present % Fe
BCS 27C - Mesabi Ore		Certified 2.08	-	-	-	-	65
NBS 116a - Ferrotitanium		6.68	3	-	-	25	65
BCS 301 - Lincolnshire Iron Ore		7.20	2	22	2	0.1 (Ti)	25
BCS 302 - Iron Ore		20.0	4	3	1	0.4	36
BCS 303 - Iron Ore Sinter		16.5	4	20	2	0.2 (Ti)	36
Mixture of 208/1 (Ferromanganese) and BCS305 (Ferrosilicon)	Ratio 6:1	25.0	0.2	0.1	-	0.1	16
Mixture of NBS 27C (Mesabi Ore) and NBS 154a (Titanium Dioxide)	1:2	0.69	-	-	-	66	22
	2:1	1.39	-	-	-	33	43
Mixture of BCS 301 (Iron Ore) and NBS 154a (Titanium Dioxide)	1:2	2.40	1	7	0.6	66	8
	1:1	3.60	1	11	1	50	12
Mixture of BCS 303 (Iron Ore) and NBS 154a (Titanium Dioxide)	1:2	5.50	1	7	1	66	12
	1:1	8.25	2	10	1	50	18

As can be seen from Table 2, Sets A and B gave results which agreed well with each other and with results on Certified silicon samples when compared with contaminant-added silicon standards. Agreement between Sets A and B was poor, however, if compared with pure silicon standards, and agreement with Certified results was poor and inconsistent. Also, the precision found was superior for atomic absorption measurements made against contaminant-added standards, because the coefficient of variation found was 2%, compared with 4% using pure-silicon standards.

It was found that there was a marked variance in performance shown by the Techtron atomizer system over a period of time because of a gradual deterioration in atomizer efficiency. A second atomizer acted in much the same way. The Jarrell-Ash atomizer system on the contrary did not seem to be affected in a similar manner, or, at least, to such a noticeable extent.

b) Hydrochloric Acid Medium

The same two mixtures of Certified iron ore and titanium dioxide as in A.1.a)(above), were fused with sodium peroxide and the slag was first treated with water and then with hydrochloric acid. Atomic absorption measurements were then made against pure silicon standards and against silicon standards that contained invididual contaminants in different ratios of contaminant:silicon. The sodium contents were between 6000 and 12,000 ppm. All measurements were made with the Techtron instrument used in the previous test, A.1.a).

TABLE 2

Atomic Absorption Results Obtained for Silicon on Mixtures of Certified Standard Samples Using Pure-and Contaminant-Added Standards for Comparison; HF-H_3BO_3 Medium

Sample	Set ***	Dissolution Procedure	Acid Medium Present	Sodium Present ppm Na	Type of Standard Used for Measurement	Silica Found % SiO_2	Deviation %
(3.60% SiO_2) Mixture of Certified Iron Ore and Titanium Dioxide -1:1 BCS 301 and NBS 154a <u>Ratio</u> 1 SiO_2 : 3 CaO 14 TiO_2 4 Fe 0.3 Al_2O_3 0.3 MgO	A	Bomb	1.5% HF-H_3BO_3	12,000	SiO_2	2.75	- 23.6
		Bomb	1.5% HF-H_3BO_3	12,000	Multi-Contaminant*	3.59	- 0.3
		Fusion	1.5% HF-H_3BO_3	12,000	SiO_2	2.79	- 22.5
		Fusion	1.5 to 3% HF-H_3BO_3	12,000	Multi-Contaminant*	3.37	- 6.4
	B	Bomb	1.5 to 3% HF-H_3BO_3	6,000 and 12,000	SiO_2	3.92	+ 8.9
					Multi-Contaminant**	3.70	+ 2.8
		Fusion	1.5 to 3% HF-H_3BO_3	6,000 and 12,000	SiO_2	4.00	+ 11.1
					Multi-Contaminant**	3.55	- 1.4

TABLE 2 (cont'd)

Sample	Set ***	Dissolution Procedure	Acid Medium Present	Sodium Present ppm Na	Type of Standard Used for Measurement	Silica Found % SiO_2	Deviation %
(5.50% SiO_2 Mixture of Certified Iron Ore and Titanium Dioxide - 1:2 BCS 303 and NBS 154a Ratio 1 SiO_2 : 1 CaO 11 TiO_2 2 Fe 0.2 Al O_3 0.2 MgO	B	Bomb	1.5 to 3% $HF-H_3BO_3$	6,000 and 12,000	SiO_2	5.87	+ 6.7
					Multi-Contaminant*	5.54	+ 1.1
		Fusion	1.5 to 3% $HF-H_3BO_3$	6,000 and 12,000	SiO_2	6.02	+ 9.4
					Multi-Contaminant**	5.41	- 1.6
	A	Bomb	1.5% $HF-H_3BO_3$	12,000	SiO_2	4.43	-19.5
		Bomb	1.5% $HF-H_3BO_3$	12,000	Multi-Contaminant*	5.54	+ 0.7
		Fusion	1.5% $HF-H_3BO_3$	6,000	SiO_2	4.57	-16.9
		Fusion	1.5% $HF-H_3BO_3$	12,000	SiO_2	4.53	-17.6
		Fusion	1.5% $HF-H_3BO_3$	12,000	Multi-Contaminant*	5.46	- 0.7
		Fusion	1.5% $HF-H_3BO_3$	12,000	$SiO_2:Al_2O_3$ -1:12	5.43	- 1.3

* ratio of 1 SiO_2 to 15 Fe, 5 TiO_2, 3 MgO, and 5 Al_2O_3.

** ratio of 1 SiO_2 to 3 Fe, 23 TiO_2, 5 CaO.

*** set A was done at a different time than set B.

It was found that results obtained from comparison with silicon standards containing a major component found in the sample, were better than those obtained from comparison with pure silicon standards. Each of the elements, iron, titanium, aluminum, calcium, and magnesium, when added to the silicon standards, gave a comparison solution which provided results in good agreement with the given Certified silicon result (Table 3).

It was concluded from the results of these two series of tests A.1.a) and A.1.b) on the mixtures of Certified Standard samples, that

a) it was necessary to use standards containing a major contaminant for atomic absorption comparison, if analysing this type of sample material and if using the Techtron atomizer, because the contaminated standard provided a superior indication of the atomizer's efficiency:

b) the addition of one of the major contaminants to the silicon standard appeared to work as well as preparing a multi-contaminant standard:

c) a hydrochloric acid solution following a peroxide fusion gave as efficient a medium for silicon determination as a hydrofluoric-boric acid medium and was simpler to use;

d) the Techtron atomizer system was difficult to keep operating efficiently in this highly salted media and it had to be cleaned frequently.

TABLE 3

Atomic Absorption Results for Silicon on Mixtures of Certified Standard Samples Using Pure and Contaminant-Added Standards for Comparison - HCl Medium

Sample	Sodium[a] Present ppm Na	Ratio Silica:Contaminant in Comparison Standard	Silica Found % SiO_2
Mixture of Certified Iron Ore and Titanium Dioxide -1:1 BCS 301 and NBS 154 a Ratio - 1 SiO_2: 3 CaO 14 TiO_2 4 Fe 0.3 Al_2O_3 0.3 MgO (3.60% SiO_2)	12,000	SiO_2:no contaminant	3.67[b], 2.96[b]
	6,000	SiO_2:no contaminant	2.55[b], 3.89[b]
	6,000 and 12,000	1 SiO_2:10 Fe	3.60
	6,000	1 SiO_2: 4 Fe	3.66
	6,000 and 12,000	1 SiO_2:10 TiO_2	3.67
	6,000	1 SiO_2: 4 TiO_2	3.61
	6,000 and 12,000	1 SiO_2:10 Al_2O_3	3.60
	6,000	1 SiO_2: 4 Al_2O_3	3.59
	6,000 and 12,000	1 SiO_2:10 MgO	3.56
	6,000	1 SiO_2: 4 MgO	3.59
	6,000 and 12,000	1 SiO_2:10 CaO	3.57
	6,000	1 SiO_2: 4 CaO	3.59
	12,000	1 SiO_2:multi-contaminant[c]	3.70
Mixture of Certified Iron Ore and Titanium Dioxide -1:2 BCS 303 and NBS 154 a Ratio - 1 SiO_2: 1 CaO 11 TiO_2 2 Fe 0.2 Al_2O_3 0.2 MgO (5.50% SiO_2)	6,000	SiO_2:no contaminant	4.05
	6,000 and 12,000	1 SiO_2:10 Fe	5.69
	6,000	1 SiO_2: 4 Fe	5.42
	6,000 and 12,000	1 SiO_2:10 TiO_2	5.63
	6,000	1 SiO_2: 4 TiO_2	5.42
	6,000 and 12,000	1 SiO_2:10 Al_2O_3	5.55
	6,000	1 SiO_2: 4 Al_2O_3	5.48
	6,000 and 12,000	1 SiO_2:10 MgO	5.55
	6,000	1 SiO_2: 4 MgO	5.47
	6,000 and 12,000	1 SiO_2:10 CaO	5.55
	6,000	1 SiO_2: 4 CaO	5.44

a - 100 ppm SiO_2 present per 12,000 ppm Na; 50 ppm SiO_2 present per 6,000 ppm Na.
b - results obtained on different sets using different atomizer-burner combinations.
c - ratio 1 SiO_2:15 Fe, 5 TiO_2, 3 MgO, and 5 Al_2O_3.

2. Comparison of Atomic Absorption Results with the Certified Chemical Results

Five Certified Standard samples, and seven mixtures made from Certified Standard samples, were put into solution by the Teflon bomb procedure. Also, six of these samples were fused with sodium peroxide and dissolved in hydrochloric acid medium. Atomic absorption results found using the Jarrell-Ash spectrophotometer with pure silicon standards, and the Techtron spectrophotometer with contaminant-added standards, were then compared with Certified results.

As can be seen from Table 4, results were in good agreement with Certified values, and the average deviation found between Certified values and atomic absorption results was within 2%. This indicated that either dissolution procedure was satisfactory for application to these types of sample material, with ease of sample dissolution and handling being the determinant.

B. Application of the Atomic Absorption Procedures to Slags and Associated Materials

1. Comparison of Atomic Absorption and Gravimetric Results on Typical Sample Material

A number of typical samples of slag and associated materials from high-temperature furnace work were analysed for silicon using the Teflon bomb procedure. The composition of these samples is shown in Table 5. In all cases the Jarrell-Ash spectrophotometer was used and comparison of the sample with pure-silicon standards was done.

TABLE 4

Comparison of Atomic Absorption with Chemical Results for Silicon on Certified Standard Samples and Their Mixtures

Sample[a] Type	Silica Value Given % SiO_2	Silica Found by A.A.[b] Teflon Bomb Dissolution % SiO_2	Silica Found by A.A.[b] Fusion Dissolution % SiO_2	Deviation from Chemical Results - % Bomb	Deviation from Chemical Results - % Fusion
BCS 27C - Iron Ore	2.08	2.04	-	-1.9	-
BCS 301 - Iron Ore	7.20	7.21	-	+0.14	-
BCS 302 - Iron Ore	20.0	19.85	19.3	-0.75	-3.5
BCS 303 - Iron Ore Sinter	16.5	16.5	-	0.0	-
NBS 116a - Ferrotitanium	6.68	6.50	6.79	-2.7	+1.65
6:1 Mixture of BCS 208/1 and BCS 305	25.0	24.25	24.2	-3.0	-3.2
1:2 Mixture of BCS 27C and NBS 154a	0.69	0.71	-	+2.9	-
2:1 Mixture of BCS 27C and NBS 154a	1.39	1.46	-	+5.0	-
1:2 Mixture of BCS 301 and NBS 154a	2.40	2.37	-	-1.25	-
1:1 Mixture of BCS 301 and NBS 154a	3.60	3.60 3.65[c]	3.46[c] 3.61[d]	0.0 +1.4	-3.9 +0.28
1:2 Mixture of BCS 303 and NBS 154a	5.50	5.48 5.54[c]	5.43[c] 5.52[d]	-0.36 +0.73	-1.3 +0.36
1:1 Mixture of BCS 303 and NBS 154a	8.20	8.22	8.30	+0.24	+1.2

a - for chemical composition, see Table 1.
b - sodium content, 5000 to 6000 ppm Na.
c - Techtron vs contaminated standards in HF-H_3BO_3 medium.
d - Techtron vs contaminated standards in HCl medium.

TABLE 5

Composition of Typical Slag Samples Analysed for Silicon Using Atomic Absorption Procedures

Sample Type	Silicon Present % SiO_2	Aluminum Present % Al_2O_3	Calcium Present % CaO	Magnesium Present % MgO	Titanium Present % TiO_2	Iron Present % Fe	Manganese Present % MnO
EMP 1552 - Slag from Iron Ore Smelting	27	15	38	8	-	6	-
EMP 2958 - Slag from Ilmenite Smelting	5	6	0.5	4	67	13	-
EMP 2715 - Ilmenite Head Sample	1.5	2	0.1	2	38	42	-
Slag - B Manganese Ore Smelting	24	21	6	3	-	0.7	43
Slag - H Manganese Ore Smelting	24	19	3	1	-	0.6	49

Several of the samples were put into solution with a sodium peroxide fusion-hydrochloric acid treatment, and comparisons were made similar to those above. All samples contained about 6000 ppm sodium.

The comparison of results obtained on these samples by atomic absorption and gravimetric procedures is shown in Table 6. Most of the samples analysed were ferromanganese slags, on which gravimetric results were assumed to have reasonably good credibility because results were from two different laboratories. With high-titanium material, however, gravimetric results on the high-titanium slags and ilmenite head samples were considered to be less reliable due to the difficulty in analysing for silicon on this type of sample material. It has been found in our laboratory, for example, that results will be erratic and often low unless special precautions are taken when analysing this type of sample gravimetrically.

Agreement between gravimetric and atomic absorption procedures was found to be generally satisfactory, although, on one sample a titanium-bearing slag (EMP 2958), apparently high results were obtained from the fusion procedure. The reason for this was not clear because the ratio, silicon:contaminants, in this sample is less than would be expected to cause trouble (see Part II) (2). Also, the amount of contaminant present was less than in the ilmenite head sample, EMP 2715, for which good agreement was found. Moreover, agreement was good between chemical and atomic absorption procedures on Certified sample material similar to EMP 2958 (see Tables 1, 4 and 5). As a

TABLE 6

Comparison of Atomic Absorption Results
With Chemical Results for Silicon on Typical Slag Samples

Sample Type	Silica Value (Gravimetric) % SiO_2	Silica Found by A.A. Teflon Bomb Dissolution % SiO_2	Silica Found by A.A. Fusion Dissolution % SiO_2	Deviation of A.A. Results from Chemical Results - % Bomb	Deviation of A.A. Results from Chemical Results - % Fusion
EMP-1552, Slag from Iron Ore Smelting	27.3[b]	26.9	27.3	-1.5	0.0
EMP-2846, Slag from Ilmenite Smelting	4.27[b]	-	4.14[c]	-	-3.0
EMP-2958, Slag from Ilmenite Smelting	4.81[b]	5.03	5.51	+4.6	+14.6
EMP-2715, Ilmenite Head Sample	1.53[b]	1.535	1.54	+0.33	+0.65
Ferromanganese Slag - A	23.9[a,b]	23.6	-	-1.25	-
Ferromanganese Slag - B	24.45[a,b]	24.3	-	-0.6	-
Ferromanganese Slag - C	23.45[a,b]	24.8	-	+5.8	-
Ferromanganese Slag - D	23.9[a,b]	25.1	-	+4.8	-
Ferromanganese Slag - E	24.7[a,b]	24.4	-	-1.2	-
Ferromanganese Slag - F	22.0[a]	22.7	-	+3.2	-
Ferromanganese Slag - G	23.0[a]	24.2	-	+5.2	-
Ferromanganese Slag - H	23.7[a]	24.5	-	+3.4	-
Ferromanganese Slag - I	22.9[a]	22.4	-	-2.2	-
Ferromanganese Slag - J	25.4[a]	24.8	-	-2.4	-
EMP-2711, Ferromanganese Slag	24.4[b]	25.0	-	+2.4	-

a - results provided by George Ascroft, Chief Chemist,
Union Carbide Canada Ltd., Welland, Ontario.

b - gravimetrically by Extraction Metallurgy Division

c - Techtron vs contaminated standards

precaution, atomic absorption was applied to standards made up to simulate this slag sample, but there was no difference in results.

2. Precision Found for the Atomic Absorption Procedures

The precision found was calculated for a number of slag samples and one mixture of Certified Standard samples, using the method of Dean and Dixon (10), and Bauer (11). The results of these tests, given in Table 7, showed satisfactory precision for either material and the average coefficient of variation was 1.3%.

DISCUSSION

It was found that the Jarrell-Ash and Techtron atomizer-burner systems behaved quite differently with the type of highly salted sample solutions used in this work. The Techtron atomizer would block-up during operation and changes in air pressure could be observed when partial blockage of the atomizer began; this seriously affected absorption readings. This concurs with the finding of Reid et al (7) who reported trouble with nebulizer blockage when using a 2% boric acid solution. With the Techtron instrument, the use of standard solutions containing one or more of the major contaminants found in the samples was necessary for compensation of the atomization changes taking place, and good results could be obtained in this way. However, eventually the atomizer's efficiency, in spite of repeated cleaning, lessened to the point where results became too erratic for use. A second atomizer was tried with similar results. The Jarrell-Ash atomizer did not undergo the blockage shown by the Techtron atomizer and comparison of atomic absorption readings could be made against pure silicon standards on each material tried.

TABLE 7

Precision Obtained for Silicon Analyses Following Sample Dissolution by Teflon Bomb and Sodium Peroxide Fusion

Sample Type	Sample Treatment	Individual Determinations Using Atomic Absorption % SiO_2	Average Result % SiO_2	Precision Found		
				Standard Deviation S.D.	Coefficient of Variation %	95% Confidence Limits for Average Results
Slag from Iron Ore Smelting	Teflon Bomb	26.10, 26.75 26.80, 26.90 26.00	26.51	0.39	1.46	0.47
	Fusion - HCl	27.30, 27.90 27.20, 27.15 27.00	27.31	0.39	1.42	0.47
Slag from Ilmenite Smelting	Teflon Bomb	4.97, 4.95 5.06, 5.06 4.99	5.01	0.047	0.94	0.059
Slag from Manganese Ore Smelting	Teflon Bomb	24.10, 24.55 24.30, 25.00 24.80	24.55	0.39	1.58	0.47
Mixture of Certified Ferromanganese and Ferrosilicon	Fusion - HCl	24.15, 24.05 24.35, 23.80 24.15	24.10	0.24	0.98	0.29

The two atomizer types are quite different, therefore, they would not necessarily react in the same manner to the highly salted solutions. In the Techtron laminar-flow system, the sample flow mixes with the support gas inside the atomizer and makes a right-angle turn before going through the burner slot. The Jarrell-Ash atomizer-burner system uses a Hetco total-consumption burner, converted to a laminar-flow burner by means of a Tri-Flame head, with the Hetco burner serving as an aspirator-atomizer for the burner head. The flow of gases passes directly up the Hetco burner and through the screen below the burner head, where it is mixed with the sample flow.

Two types of burner head were used during this work: a flat-top burner head (with both spectrophotometers) and a grooved burner head (with the Techtron). It was found that the Techtron flat-top burner head was more prone to carbonization than the Jarrell-Ash burner head, whereas the grooved Techtron burner head showed only minor carbonization. However, solids formed on the edge of the grooved burner slot; this presented problems like those reported by Goguel (12) who found that high background noise and extensive baseline drift was caused by crust formation on the grooved burner slot. Much less formation of solids on the burner slot was found when using the flat-top burner head.

CONCLUSIONS

Atomic absorption procedures for determining silicon in ores, in slags, and in associated materials are especially well-suited to operational control. The procedures described are fast, accurate, and precise; also, the sample solutions are suitable for other analyses.

No contaminant need normally be added to the comparison standard if using the Jarrell-Ash atomizer-burner on highly salted samples. A major contaminant must be added to the comparison standard if using the Techtron atomizer-burner.

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