Applied Mechanics and Materials *Online: 2013-01-11*
ISSN: 1662-7482, Vol. 273, pp 473-477
doi:10.4028/www.scientific.net/AMM.273.473

ICP-AES Determination of the Four Trace Elements (Al, Ca, P, Ti)In The Ferrosilicon

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Keywords: ICP-AES; Ferrosilicon Sample; Trace Elements (Al, Ca, P, Ti); Recoveries

Abstract. Ferrosilicon by swinging open vessel acid decomposition method after acid decomposition processing, based on national standards GB/T24194-2009, using inductively coupled plasma atomic emission spectrometry (ICP-AES), determination of the four trace elements (Al, Ca, P, Ti) content in ferrosilicon sample, The use of modern instruments with background correction technology to eliminate polyatomic ion interference on the part of the measured elements. The results of the various elements of the recoveries were between 98.994% and 103.01%, all the precision (RSD) of the detected value is less than 3%, so the high technical accuracy, able to meet the requirements of the simultaneous determination of a variety of elements in ferrosilicon samples.

Introduction

Ferrosilicon is iron alloy that composed of iron and silicon. Ferrosilicon is silicon ferroalloys made of coke, steel scrap, quartz as raw material. Silicon and oxygen is very easy synthesis of carbon dioxide, ferrosilicon, commonly steelmaking deoxidizer. Ferrosilicon as an alloying element can also join agents, widely used in low-alloy structural steel, spring steel, bearing steel, heat-resistant steel, and electrical silicon steel.these steel have special uses so parts of the technical indicators of the steel very demanding , the more stringent requirements for samples of ferrosilicon (steel-making raw materials) for export. Therefore controlling the amount of good ferrosilicon of Al, Ca, P, Ti and other trace elements is critical.Not only the quality of the steel plays a vital role, but also to meet the export requirements.

In the early years, the inductively coupled plasma is a new light source of emission spectrometric analysis. It is seen as the fastest progress in the field of atomic spectroscopy, and the forefront of an analytical technique. Qinghai Entry-Exit Inspection and Quarantine uses the IRIS model ICP-AES, it uses Echelle optical system and a unique (CID) solid detector to provide a complete and full wavelength coverage of the analysis of spectra, ICP-AES as the conventional means for the quantitative determination of trace elements of a variety of samples in many of the world laboratories. Widely used,it has proven to that ICP-AES multi-element determination has many advantages such as high analytical accuracy , precision and the matrix interference was small.

In this paper, ferrosilicon by swinging open vessel acid decomposition method after acid decomposition processing, based on national standards GB/T24194-2009, using inductively coupled plasma atomic emission spectrometry (ICP-AES) to meet the simultaneous determination of a variety of elements in ferrosilicon samples.

Experimental Materials and Methods

Materials

Black powder ferrosilicon sample (the Qinghai objects Group Industry Co., Ltd.)

Into a particle size of less than 125um samples for the analysis of the experimental sample was prepared in accordance with the requirements of GB/T4010.

Fig.1 Lump ferrosilicon

Fig. 2 Powder-like ferrosilicon

Experimental Apparatus and Reagents

IRIS the full spectrum of direct reading inductively coupled plasma atomic emission spectrometer (ICP-AES), (thermoelectric) device number: 200363AC009; A240 electronic balance (Switzerland Mette music Torres); ultrapure water treatment systems (Millpore). Singlet pipette indexing pipette and single-scale volumetric flask, in line with GB/T12806 GB/T12807, GB/T12808.

Nitric acid(density 1.42g/mL), hydrochloric acid(density 1.19g/mL), hydrofluoric acid(density 1.15g/mL), perchloric acid(density 1.67g/mL), hydrochloric acid (1+3),these are AR, laboratory preparation of aluminum, calcium, phosphorus, titanium standard solution, respectively, for the 100ug / L.

Fig. 3 ICP-AES

Instrument Working Conditions

Table 1 ICP-AES detection working conditions

Incident power (W)	1150	Spray chamber pressure (PSI)	25
Auxiliary gas(L / min.)	0.5	Frequency (MHz)	27.12
Peristaltic pump speed (r / min.)	130	Cooling gas (L / min.)	14
The shortwave integral time (s)	10	The longwave integral time (s)	5

Table 2 Determination of the wavelength and the detection limit and measuring range

Element	Wavelength (nm)	Range (PPM)	Element	Wavelength(nm)	Range (PPM)
Al	308.215	0.0070~500	P	178.287	0.008-100
Ca	393.366	0.00006~10	Ti	334.941	0.0002-10

Table 3 GB01432 Concentration

Elment	GB01432 content (%)	Determination of value range (%)	Allow poor (%)	Instrument measuring range (%)
Al	0.24	0.25~0.50	0.020	0.01~3.00
Ca	0.064	0.05~0.10	0.008	0.01~2.50
P	0.0093	0.005~0.01	0.0015	0.005~0.10
Ti	0.032	0.03~0.05	0.0030	0.005~0.10

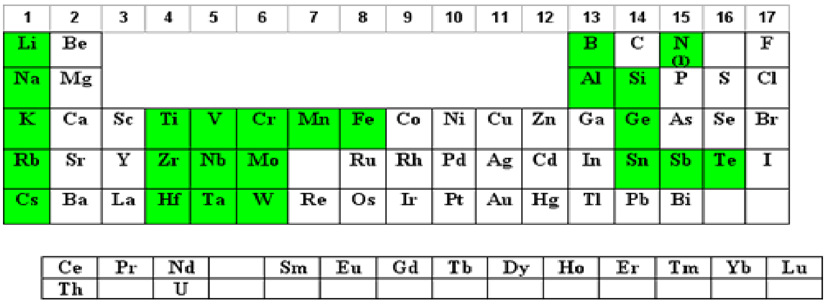


Fig. 4 ICP-AES determination of the four trace elements

Experimental Methods

The Method

Accurately weigh 0.5000g sample specimen,it shall be placed in a 200mL Teflon beaker, wetting with a little water, then add 10mL of nitric acid(density 1.42g/mL) with a plastic tube carefully dropping the about 5mL hydrofluoric acid(density 1.15g/mL), when a fierce reaction stopped ,then add 5mL hydrochloric acid(density 1.19g/mL), and heated slowly to the test material dissolved completely, add about 8mL perchloric acid(density 1.67g/mL) on low heat to produce perchloric acid smoke, rinse with water to beaker wall, to continue the low-temperature heating to smoke when the left about 2mL to 3mL solution,then add 20mL of hydrochloric acid (1 +3) to the coolish, heated to dissolve the salts, cooled to room temperature, the test solution moved into a 100mL volumetric flask, dilute with water to scale mixing, in parallel with a blank test solution. Preparation of standard night.

The Measurement

Calibration Solution

First inhalation of the standard solution, using the zero-calibration solution according to the order inhaled deionized water between each inhalation solution, repeat the test twice, take two readings average.

Test Solution

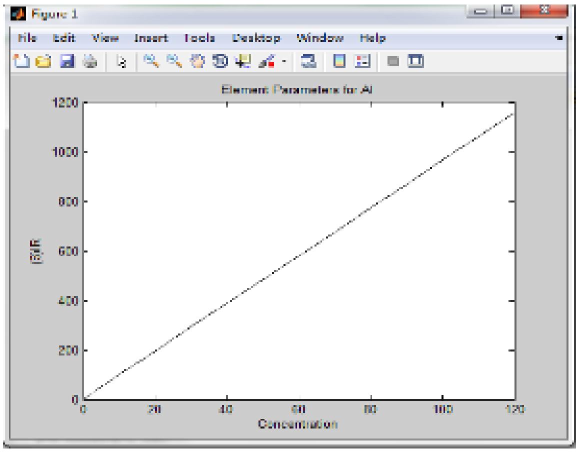
Calibration solution, immediately measure the experimental solution, inhalation of deionized water between each measurement; the test should be repeated twice.

The Experimental Results

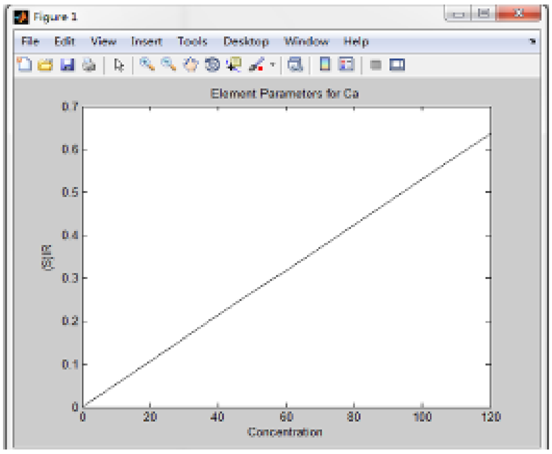
The Standard Curve

Element	MDL	MQL	Linear regression equation	Correlation coefficient (r)	curvature
Al	0.128927	0.429756	Y=9.635095X+2.258177	0.999988	0.000000
Ca	0.110597	0.368655	Y=0.005298X+0.001189	0.999992	0.000000
P	0.085330	0.284434	Y=0.008021X+0.000034	0.999899	0.000000
Ti	0.041416	0.138054	Y=0.035979X+0.018728	0.999995	0.000000

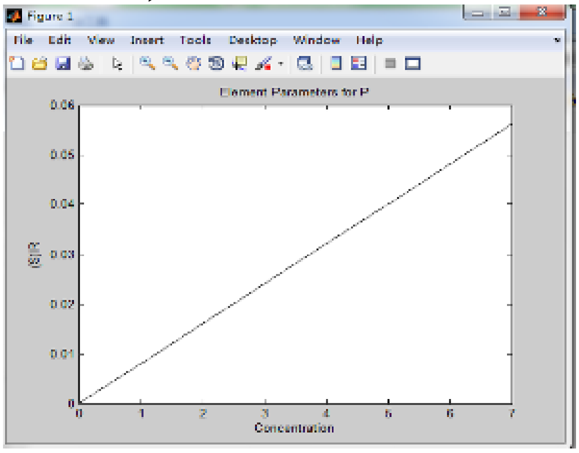
The standard blank solution and standard solution of the selected instrument conditions and measurement, the instrument automatically draw the curve, the linear correlation coefficient greater than 0.999899.



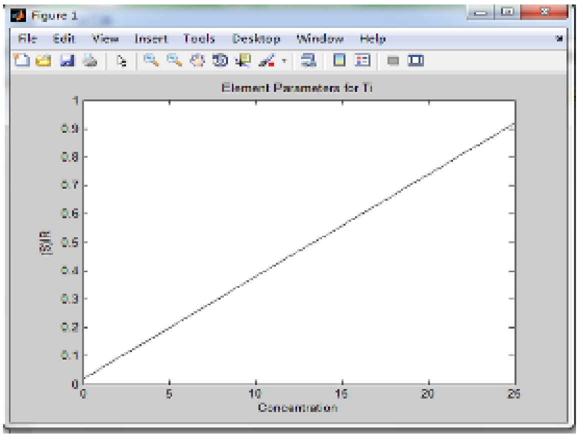
Al ;Y=9.635095X+2.258177



Ca; Y=0.005298X+0.001189



P; Y=0.008021X+0.000034



Ti; Y=0.035979X+0.018728

Sample Determination Results

The three sample (the ferrosilicon national standards sample (GBW 01432) , the three ferrosilicon samples , blank sample) which the determination of value of the four trace elements, content is measured by ICP-AES, using two-sample test, the table 5 below is the determination of the value, testing in accordance with GB/T24194-2009.

Inspection lot	Sample volume (g)	Al content (%)	Ca content (%)	P content (%)	Ti content (%)
GBW 01432	0.5012	0.2433	0.0970	0.0079	0.0312
63120926(1)	0.5004	0.2754	0.1057	0.0158	0.0703
63120926(2)	0.5006	0.2788	0.1042	0.0159	0.0700
63120927(1)	0.5006	0.3797	0.2115	0.0161	0.0673
63120927(2)	0.5004	0.3748	0.1994	0.0158	0.0679
63120928(1)	0.5005	0.4161	0.2886	0.0164	0.0775
63120928(2)	0.5008	0.4166	0.2823	0.0167	0.7440
Blank	0.0000	0.0080	0.0347	0.0002	0.0004

Result analysis: From the table,we can see that the determination of the instrument GBW 01432is stable.The four trace elements(Al ,Ca, P,Ti)content is less than the manufacturers requirements (Al 0.5 ;Ca 0.35 ;P 0.04; of Ti 0.02) data.the determination of ferrosilicon is qualified.

Precision and Recovery Test

Further verify the accuracy of the test methods in the ferrosilicon sample recovery experiment.The spiked recoveries of the elements shown in Table 4, the various elements of the recoveries were between 98.994% and 103.01%; the same sample 3 Determination, its value and the precision (RSD) in Table 6,all RSD less than 3%.

Element	Add scalar (ug/L)	Measured values (ug/L)	The relative standard deviation (%)	Recoveries (%)
Al	10.000	10.1771	1.77	101.77
	50.000	50.049	0.097	100.09
	100.00	99.774	0.226	99.774
Ca	10.000	9.9795	0.205	99.795
	50.000	49.708	0.583	99.416
	100.00	100.31	0.312	100.31
P	1.0000	1.030	3.000	103.00
	2.0000	2.0202	1.010	101.01
	5.0000	4.9497	1.010	98.994
Ti	5.0000	4.9858	0.285	99.716
	10.000	9.9576	0.424	99.576
	20.000	20.057	0.283	100.28

The Conclusion

After this experiment that ferrosilicon by swinging open vessel acid decomposition method after acid decomposition processing, based on national standards GB/T24194-2009, using inductively coupled plasma atomic emission spectrometry (ICP-AES), determination of the four trace elements (Al, Ca, P, Ti) content in ferrosilicon sample, The use of modern instruments with background correction technology to eliminate polyatomic ion interference on the part of the measured elements. The results of the various elements of the recoveries were between 98.994% and 103.01%, all the precision (RSD)of the detected value is less than 3%, so the precision and accuracy are high, it able to meet the analytical results needs. This method is of great significance for the detection of the export of raw materials.

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