The Analysis of Iron Ores

Application Note

Atomic Absorption

Authors

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Introduction

The atomic absorption technique is ideally suited for the rapid analysis of a wide range of elements in ores. A description is given of the methods employed at Agilent Technologies in the analysis of iron ore samples for fifteen different elements.

Agilent Technologies

Experimental

Fe, Al and Si Sample Solutions

Fuse 0. 5 g of the finely ground sample of iron ore with 2 g of NaOH pellets in a zirconium crucible.

Cool the melt and dissolve it in a solution of 100 mL of distilled water plus 2 mL of 10 vol. H_2O_2 and 9.5 mL of conc. HNO_3. Boil until a clear solution is obtained and then cool.

Transfer the solution quantitatively into a 200 mL volumetric flask and make up to volume with distilled water.

Aluminium and silicon can be determined directly on this solution.

For the determination of iron the above solution must be diluted 50-fold.

Notes on the Individual Determinations

Aluminium

No serious interferences occur provided that the standard and the sample solutions are matched with respect to their Na and Fe content.

Standard solutions are prepared to cover the range 0–150 ppm Al, for example, for Al concentrations of less than 6% in the original ore sample.

The atomic absorption measurements are made at the 3092.7, 3092.8 Å doublet, using the $N_20\text{-}C_2H_2$ flame.

Silicon

Standard solutions are prepared to cover the range 0–100 ppm Si, containing similar concentrations of Na and Fe as are found in the sample solution.

A standard Si stock solution containing 1000 ppm Si may be prepared by fusing 1.069 g of ignited SiO_2 with 2 g of NaOH, dissolving and making up to 500 mL with distilled water.

It should be noted that silica does not precipitate readily from acid solutions, provided that the normality does not exceed 3N and provided that the acidic solution is not boiled.

The atomic absorption measurements are made at the 2516.1 Å resonance line, using a slightly fuel-rich (reducing) $N_2O\text{-}C_2H_2$ flame.

Iron

The use of the less sensitive (9 times) 3719.9 Å line is recommended because of the high concentration of iron in solution and because of its much higher intensity than the more commonly used 2483.3 Å line, resulting in a better signal to noise ratio and thus improved precision.

Care should be taken to ensure, when any free acids are present (particularly HCl and $HClO_4$), that a fuel-lean flame is used in order to overcome the suppressing effect of these acids. It is advisable to match the standard and sample solutions with respect to their acid content.

- **Trace Metals**
  The relatively low concentrations of other elements generally found in iron ores necessitate the use of sample solutions which contain approximately 2 g of ore per 25 mL of solution. At such high concentrations (~8%) severe blockage of both the nebulizer and the burner can occur. In order to overcome this problem the bulk of the iron is first removed by extraction into iso-butyl acetate.
- **Extraction Procedure**
  2 g of finely ground ore are digested with 25 mL of cone. HCL The mixture is evaporated to dryness and the residue is redissolved in a minimum amount of 50% v/v HCl

  This acidic solution is filtered and the residue (A) is washed with minimum amounts of hot 20% v/v HCl and distilled water.
- **Treatment of Filtrate (Plus Washings)**
  The filtrate is evaporated down to a paste and redissolved in 20 mL of conc. HCl

  This solution is oxidized by the dropwise addition of cone. HNO_3. After cooling it is washed into a 100 mL separating funnel with conc. HCl, giving a final total volume of 40 mL.

  50 mL of iso-butyl acetate are added and the mixture is shaken for 30 seconds. The lower aqueous layer is run into a second separating funnel.

  The residual organic layer is washed with 5 mL of cone. HCl and the washings added to the second funnel.

  The above extraction procedure, using 30 mL of iso-butyl acetate followed by a 5 mL conc. HCl wash, is repeated on the aqueous acid solution in the second separating funnel.

  The combined aqueous layer and washings are evaporated almost to dryness and the paste is redissolved in 5 mL of 50 % v/v HNO_3 (Solution B).

- **Treatment of Residue (A)**
  The residue (A) is ignited and then treated with a mixture of cone. HF (5 mL) and conc. H_2SO_4 (2–3 drops) in order to remove SiO_2.

  The residue is ignited once more before fusion with 4 times its weight of anhydrous sodium carbonate. The cooled melt is extracted with 10 % v/v $HN0_3$ and combined with solution (B).

  Finally, the combined solutions are transferred to a 25 mL graduated flask and made up to volume with distilled water.

  The atomic absorption determination of each element by comparison with prepared standard solutions is carried out, using the conditions mentioned below.

Magnesium

The atomic absorption measurements are made at the 2852.1 Å line, using the air-C_2H_2 flame.

It has been reported that Si causes a depression of the Mg absorption in the air-C_2H_2 flame. It is recommended that either the hotter N_2O-C_2H_2 be used (together with excess Na as an ionization suppressant) or that when the air-C_2H_2 flame is used that both the standard and sample solutions are matched with respect to the Si content.

Calcium

In the determination of Ca it is recommended that the N_2O-C_2H_2 flame, rather than the cooler air-C_2H_2 flame, be used. This flame gives an increase in sensitivity and virtually eliminates interferences.

Sufficient Na is available from the fusion step to suppress Ca ionization in this flame.

The atomic absorption measurements are made at the 4226.7 Å line.

Sodium

It is obvious that Na cannot be determined on this solution. Treatment of another sample must be carried out in which the Na fusion of residue (A) is omitted. The residue (A) is dissolved in 5 mL of 10% v/v HNO_3. A blank should be run with the sample to compensate for sodium pick-up from reagents and glass-ware.

The atomic absorption measurements are made at the 5890.0 Å line, using either an air-C_2H_2 or an air-propane flame.

Chromium

The atomic absorption measurements are made at the 3578.7 Å line, using either a fuel-rich air-C_2H_2 or a N_2O-C_2H_2 flame.

All the other elements are determined in an air-C_2H_2 flame.

Element	Resonance line
Pb	2170.0 Å
Ni	2320.0 Å
Zn	2138.6 Å
Cu	3247.5 Å
K	7664.9 Å
Bi	2230.6 Å
Mn	2794.8 Å
Co	2407.3 Å

Other metals which may also be determined in such sample solutions are: As, Mo, Sb, Sn, Ti, V.

Some typical results

Element	Concentration	Unit
Fe	60.0	%
Si	3.50	%
Al	2.31	%
Bi	3.0	ppm
Ca	41	ppm
Co	4.5	ppm
Cr	35	ppm
Cu	18	ppm
K	40	ppm
Mg	10	ppm
Mn	39	ppm
Na	110	ppm
Ni	3.5	ppm
Pb	20	ppm
Zn	28	ppm

Conclusion

It is estimated that a suitably equipped laboratory set up for routine analysis of iron, silicon and aluminium could carry out the determination, of these elements within 3 hours of receipt of the sample. For twelve elements the time required would be approximately 6 to 8 hours.

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