



The Determination of Selenium in Steels

Application Note

Atomic Absorption

Authors

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Introduction

The determination of selenium by atomic absorption spectroscopy [1,2] and also by atomic fluorescence spectroscopy [3] has been reported by several workers. Improvements in detection limits have been achieved both with the use of an argon-hydrogen-entrained air flame [4], as well as with a nitrogen separated air-acetylene flame [5].

The relatively poor sensitivity and detection limit for selenium together with the large chemical interference from iron and acids make the accurate determination of the very low concentrations of selenium, found in steels, very difficult.

In the procedure described below interferences are largely overcome by carrying out a separation of the selenium from steel, as well as maintaining a closer control over the final acidity of the resultant sample solutions.

Experimental

Instrumental

Standard selenium solutions are prepared, each containing 15% vol./vol. HClO_4 (S.G. 1.54).

The atomic absorption measurements are made at the selenium 1960.3 Å resonance line using a spectral band pass of 6.6 Å and the hollow cathode lamp operating at 8 mA. An argon-hydrogen entrained-air flame together with scale expansion are employed. Other flames may also be employed, for example, nitrogen-hydrogen entrained-air, air-hydrogen, and air-acetylene flames.



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Samples

Approximately 2 g of the steel sample are weighed out accurately, transferred to a 250-mL conical flask and dissolved in 30 ml of a 2 vol. HClO_4 (S. G. 1.54) - 2 vol. HNO_3 (S. G. 1.42) - 1 vol. H_3PO_4 (S. G. 1.75) acid mixture.

This mixture is gently heated until all of the sample has dissolved and is then evaporated to fumes.

The solution is allowed to fume gently for 2 minutes, cooled and 20 mL of HCl (S. G. 1.16) are added and then boiled for 5 minutes.

The solution is cooled once more to 20 °C and 10 mL of a 35% SnCl_2 solution are added and the whole mixture is allowed to stand for 15 minutes.

Note: The stannous chloride solution is prepared by dissolving 35 g of SnCl_2 in 50 mL HCl (S.G. 1.16) with beating, followed by cooling and dilution to 100 mL with distilled water. The addition of SnCl_2 reduces the selenium to the metallic state thus enabling its separation from iron to take place.

The solution is filtered on a tight pulp pad and the precipitate is washed with 50% HCl until it is free from iron.

The precipitate and the pad are transferred to a beaker, 20 mL of HNO_3 and 15 mL of HClO_4 are added and the whole evaporated to fumes in order to destroy the organic matter. The solution is then cooled and diluted to exactly 100 mL with distilled water.

2 g of high purity iron are treated in exactly the same manner in order to obtain a selenium free blank solution.

Typical Results

Component	% Selenium	
	By atomic absorption	By colorimetric method
MAC 89 Steel	(a) 0.018	0.013
	(b) 0.0175	0.013
MAC 103 Steel	(a) 0.0385	0.039
	(b) 0.0390	0.039

Conclusion

For the MAC 103 steel the atomic absorption results are in excellent agreement with the colorimetric method results.

The high results obtained for MAC 89 steel may be due to chemical interference from tellurium (0.018% Te in MAC 89). Chakrabarti 2 has shown that 160 ppm Te cause the absorbance of a 1 ppm Se solution to be enhanced by about 15%. Thus, in order to compensate for this enhancement both the sample and standard solution should contain approximately equal amounts of tellurium.

This method for the determination of selenium in steels is readily adaptable to the determination of tellurium in steels, using the 2142.8 Å tellurium resonance line.

References

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