Daehan Hwahak Hwoejee
Vol. 10, Number 3, 1966
Printed in Republic of Korea

Atomic Absorption Spectrophotometric Analysis of Chromium, Nickel and Manganese in Stainless Steel

by

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(Received Oct. 14, 1966)

原子吸光分析에 의한 鋼鐵속에 있는 Cr, Mn 및 Ni의 定量

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(1966. 10. 14 受理)

要 約

Stainless steel 속에 들어 있는 chromium, nickel, 및 manganese 를 原子吸光分光分析法(atomic absorption spectrophotomer)으로써 보다 빨리 定量할 수 있는 方法을 記述하였다.

現行되고 있는 濕式分析法과 比較한 結果 原子吸光分光分析法도 stainless steel 의 分析에 믿을만한 한가지 分析法임을 알게 되었다.

Abstract

Rapid methods for determining chromium, nickel, and manganese in stainless steel by atomic absorption spectrophotometer are described. Use of suitable standards eliminates the need to separate each element prior to analysis.

Comparison with the conventional wet chemical analysis shows that atomic absorption spectrophotometer is a reliable analytical tool in these applications.

Introduction

The atomic absorption spectrophotometer, which is economic, highly sensitive, simple in operation, rapid in analysis, and nearly completely free from spectral interferences, has in recent years found great applications in the field of metals and alloy analysis.

Several workers(1-5) have determined manganese, copper, chromium, nickel, and magnesium in steel. Beyer(6) also determined chromium, copper, magnesium, manganese, and nickel in cast iron and steel. Very recently Barnes (7) used the same technique to analyze chromium in low alloy steels.

But, very little work has been done on stainless steel by atomic absorption spectrophotometer. The apparent reasons are those interferences reported in chromium analysis (3,8) and errors which may be introduced by successive dilutions of the original sample solution prepared.

In an attempt to solve these problems, various experimental conditions have been tried in this laboratory. The following efforts have given thus far the most satisfactory results:

The mutual spectral interferences of chromium by nickel and iron and of nickel by chromium and iron were minimized by using lean flame and suitable standard sample which were obtained from National Bureau of Standards (Appendix I).

The unknown sample for a particular element of which concentration is specified is bracketed with two standards so that the element is approximately halfway between the limits of the concentration of the standards to be used.

Successive dilution of the sample solution was avoided by utilizing suppression effect due to high concentration of iron present.

Experimental Analytical Procedure

Transfer 0.1000-gram of sample to 150ml beaker, add 10ml 1:4 (V./V.) HNO_3-HCl, and cover with a watch glass. Place on a hot plate at moderate heat until all soluble material is dissolved. Add 10 ml of $HClO_4$ and boil it vigorously to expel brown NO_2 gas. Allow the solution to cool and filter the residue, if any, through S & S No. 589 black ribbon filter paper. Preliminary spectrographic analysis of the residue showed that the residue, if any, at this stage of sample preparation is made entirely of silica.

Wash both the beaker and filter paper with hot water; cool and dilute to the mark of 100ml volumetric flask. Solutions thus made are ready for analysis of chromium, nickel and manganese.

All reagents used throughout the work were Baker's analyzed reagent grade.

Apparatus

The Perkin-Elmer model 290 atomic absorption spectrophotometer without recorder was used. As a light source, a multi-element (chromium, nickel, cobalt, copper, and manganese) tube, Perkin-Elmer Serial No. 783C, was also used for chromium, nickel, and manganese determinations.

Since sensitivities of the elements were not of primary interest and concern in the present work, the operating conditions were fixed for each element as follows:

Operating Conditions:

Source current	5ma
Slit disc	7A
Meter damping	3
Air flow scale	14.40
Fuel (acetylene) flow scale	14.50
Atomic absorption burner -atomizer	Perkin-Elmer 303-0110

Element setting

Chromium	340.0
Nickel	115.5
Manganese	199.4

Results and Discussion

Three stainless steel samples from General Electric Co. and one N.B.S. No. 442 sample as a monitor were analyzed both by the present technique and conventional wet chemical method[8].

Results are shown in Table I.

Table I Comparison of Results* of Analyses Methods

Samples	ASTM Method			Present Technique		
	Cr	Ni	Mn	Cr	Ni	Mn
No. 1	15.03±0.03	4.47±0.03	0.89±0.02	14.95±0.12	4.28±0.08	0.89±0.01
No. 2	15.35±0.05	4.15±0.05	0.77±0.01	15.45±0.15	4.20±0.08	0.78±0.01
No. 3	15.49±0.05	4.15±0.05	0.81±0	15.43±0.20	4.30±0.09	0.81±0.01
N.B.S. 442	Reported			Found by Present Technique		
	Cr	Ni	Mn	Cr	Ni	Mn
	16.15	9.83	2.80	16.05±0.30	9.98±0.21	2.75±0.02

* Each sample was run in duplicate.

Chromium, nickel and manganese in the samples were analyzed by Persulfate method (9), Gravimetric dimethylglyoxime method and Persulfate method (9) respectively. Average accuracy of the analysis of chromium, nickel and manganese by the present technique compared to ASTM method is found to be ±0.6% for chromium, ±3% for nickel, and ±0.3% for manganese respectively.

Considering the average precision of the results, ±1% for chromium, ±2% for nickel, and ±1% for manganese respectively by the present technique, however, there is still much to refine on the accuracy of the results reported here and valid concentration ranges of the elements considered.

The improvement both in accuracy and precision can be achieved by careful control of amounts of interfering elements, use of a recorder, narrower slit diso, and nitrous oxide-acetylene flame which may completely eliminate chemical interferences.

In summary one will find straightforward and rapid the present technique which requires minimum sample preparation to reduce error and contamination, if any, during otherwise extreme further dilution of the sample solution.

Furthermore the fixed operating conditions as well as use of the multielement lamp source are found a useful time saver.

Treatment of sample solution with perchloric acid prior to final evaporation of the sample facilitates filtering the residue because of its dehydrating characteristic.

It is safe to say that atomic absorption spectrophotometry is a reliable and satisfactory analytical technique in the analysis of chromium, nickel and manganese in stainless steels.

Futher work on copper and cobalt in stainless steels is under investigation by the same method in this laboratory.

Acknowledgment

Authors express their gratitude to Mr. Arnold Greene and Mr. Robert J. Halliday for their continuous support which made this work possible. Thanks are also due to Mrs. Arlene E. Pope who kindly read and typed the whole manuscript for this publication.

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Appendix I Chemical Compositions of the Standards Used

(In% Units)

Sample No.	Mn	Si	Cu	Ni	Cr	Mo
NBS 845	0.77	0.52	0.065	0.28	13.31	0.92
NBS 846	0.53	1.19	0.19	9.11	18.35	0.43
NBS 847	0.23	0.37	0.19	13.26	23.72	0.059
NBS 848	2.13	1.25	0.16	0.52	9.09	0.33
NBS 850	—	0.12	0.36	24.80	2.99	—
NBS 808A	0.76	0.28	0.10	1.20	0.655	0.065