



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS G 1256 : 1997

**Iron and steel — Method for X-ray
fluorescence spectrometric analysis**

ICS 77.080.01

Descriptors : iron, transition metals, steels, X-ray fluorescence spectrometry,
fluorimetry, X-ray analysis, spectroscopy

Reference number : JIS G 1256 : 1997 (E)

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently, JIS G 1256:1982 (former edition) is replaced with this Standard.

This revision intends not only to extend the determination range but also to improve the analytical precision with the specification of criteria on apparatus performance and of tolerance on verification and/or calibration of working curve.

Further, reference materials and calibration samples are definitely specified in this revision.

Date of Establishment: 1973-03-01

Date of Revision: 1997-04-20

Date of Public Notice in Official Gazette: 1997-04-21

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Iron and Steel

JIS G 1256 : 1997, First English edition published in 1997-12

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

Iron and steel— Method for X-ray fluorescence spectrometric analysis

1 Scope This Japanese Industrial Standard specifies the method for X-ray fluorescence spectrometric analysis of iron and steel and super alloy samples of lump form or plate form, which is applied to determination of each component as given in Table 1.

Table 1 Applicable components and determination range

Constituent	Determination range % (m/m)
Silicon	0.002 or over up to and incl. 10
Manganese	0.001 or over up to and incl. 30
Phosphorus	0.001 or over up to and incl. 1
Sulfur	0.001 or over up to and incl. 0.6
Nickel	0.002 or over up to and incl. 99.5
Chromium	0.001 or over up to and incl. 50
Molybdenum	0.001 or over up to and incl. 30
Copper	0.001 or over up to and incl. 10
Tungsten	0.002 or over up to and incl. 25
Vanadium	0.001 or over up to and incl. 6
Cobalt	0.002 or over up to and incl. 60
Titanium	0.001 or over up to and incl. 10
Aluminium	0.002 or over up to and incl. 12
Arsenic	0.002 or over up to and incl. 0.3
Tin	0.002 or over up to and incl. 0.6
Lead	0.002 or over up to and incl. 0.4
Zinc	0.001 or over up to and incl. 0.1
Zirconium	0.001 or over up to and incl. 2
Niobium	0.001 or over up to and incl. 10
Magnesium	0.003 or over up to and incl. 0.2
Calcium	0.001 or over up to and incl. 0.1
Tantalum	0.002 or over up to and incl. 15
Antimony	0.002 or over up to and incl. 0.7
Selenium	0.001 or over up to and incl. 0.5
Tellurium	0.002 or over up to and incl. 0.2
Bismuth	0.001 or over up to and incl. 0.2
Iron	0.003 or over up to and incl. 50
Lanthanum	0.002 or over up to and incl. 0.2
Cerium	0.003 or over up to and incl. 0.6
Praseodymium	0.002 or over up to and incl. 0.1
Neodymium	0.003 or over up to and incl. 0.3

Remarks 1 The normative references to this Standard are as follows.

JIS G 0203 *Glossary of terms used in iron and steel (products and quality)*

JIS G 0303 *General rules for inspection of steel*

- JIS G 0306 *Steel forgings—General technical requirements*
- JIS G 0307 *Steel castings—General technical requirements*
- JIS G 0321 *Product analysis and its tolerance for wrought steel*
- JIS G 1201 *General rules for chemical analysis of iron and steel*
- JIS K 0119 *General rules for X-ray fluorescence spectrometric analysis*
- JIS K 0211 *Technical terms for analytical chemistry (general part)*
- JIS K 0212 *Technical terms for analytical chemistry (optical part)*
- JIS K 0215 *Technical terms for analytical chemistry (analytical instrument part)*
- JIS R 6001 *Abrasive grain sizes*
- JIS Z 8402 *General rules for permissible tolerance of chemical analyses and physical tests*
- JIS Z 9901 *Quality systems—Model for quality assurance in design, development, production, installation and servicing*
- JIS Z 9902 *Quality systems—Model for quality assurance in production, installation and servicing*
- JIS Z 9903 *Quality systems—Model for quality assurance in final inspection and test*

- 2 The iron and steel in this Standard mean pig iron, cast iron, carbon steel, low alloy steel, high alloy steel, etc.

2 Definitions For the purposes of this Standard, the definitions given in JIS K 0119, JIS K 0211, JIS K 0212 and JIS K 0215 apply.

3 General matters General matters necessary for analysis shall be in accordance with JIS K 0119 and JIS Z 8402.

4 Summary Cut or machine a sample, finish the analytical surface into a plane by grinding or polishing, insert the sample so prepared into the sample chamber of an apparatus for X-ray fluorescence spectrometric analysis, irradiate the analytical surface with excited X-rays, and measure the intensity of fluorescent X-ray generated from the sample.

5 Apparatus and measuring conditions

5.1 Apparatus for X-ray fluorescence spectrometric analysis As described in 4 (Equipment) of JIS K 0119.

5.2 Adjustment of apparatus As described in 7 (Measuring operation) and 11 (Inspection on equipment) of JIS K 0119.

5.3 Criteria on apparatus performance The criteria on apparatus performance shall be as follows.

- (1) For the apparatus for X-ray fluorescence spectrometric analysis adjusted in 5.2, the measuring conditions shall be set up so that repeatable analytical precision and sensitivity become suitable.
- (2) The repeatable analytical precision shall be not more than the permissible value of repeatable analytical standard deviation as given in Table 2. The repeatable analytical precision means the standard deviation calculated from the such determination values that the intensity of fluorescent X-ray of a homogeneous sample, for example, iron and steel certified reference material for instrumental analysis or iron and steel reference material for instrumental analysis prepared by 7 is measured continuously 10 times according to the procedure of 8 and the determination value for each measurement is obtained.
- (3) The criteria on apparatus performance shall be examined periodically at a determined period, and, when the conditions of the apparatus are changed, such as change in measuring conditions, execution of overhaul, etc., the criteria on apparatus performance shall be necessarily examined.

Table 2 Permissible value of repeatable analytical standard deviation

Unit : % (m/m)

Component	Range of component content	Permissible value of repeatable analytical standard deviation (1)
All elements	Determination range of Table 1	σ , obtained from $\log \sigma_r = 0.389 2 \cdot \log (X \%) - 2.110 7$

Note (1) The (X %) means the average value of the determination values [% (m/m)] obtained when repeatable analytical precision is obtained by 5.3 (2) on the component.

5.4 Measuring conditions Measuring conditions which satisfy the criteria on apparatus performance shall be selected according to the kind of analytical samples, the type of apparatus, the determination component and its content range, the content range of a coexisting component, etc. Principal points shall be as follows.

- (1) For the spectral line to be used for analysis, spectral lines suitable for the aim of the analysis shall be selected by considering the intensity of fluorescent X-ray of determination component, the interfering spectral lines of coexisting components, the background, etc. Spectral lines generally used are given in Table 3.
- (2) The target of an X-ray tube suitable for the determination component and its content shall be used, and the tube voltage and tube current shall be selected by considering the minimum excitation voltage of measuring spectral line, the miscalculation of a measuring circuit, and others.
- (3) For spectroscopic parts such as the width of slit, the dispersive crystal, and the detector, conditions suitable for the resolving power to the spectral line, and the content range of the component to be determined shall be selected.
- (4) When plural sample containers or masks are used, it shall be confirmed that the difference in the measured values obtained by use of each sample container or mask falls within the practically tolerable range.

- (5) It shall be confirmed that the analytical precision obtained by repeating insertion of the sample into the X-ray irradiation chamber has no significant difference from the repeatable analytical precision when the sample is being inserted therein.
- (6) For a scanning spectroscope, it shall be confirmed that the analytical precision obtained by repeating the resetting of the measuring wavelength has no significant difference from the repeatable analytical precision by means of a fixed spectroscope.
- (7) For the counting and recording part, setting conditions shall be selected according to the determination component, content, etc.

Further, measuring time shall be set up by considering the statistical fluctuation and the criteria on apparatus performance.

An example of measuring conditions is given in Table 4.

Table 3 Example of spectral lines

Component	Spectral line	Wavelength nm	Component	Spectral line	Wavelength nm
Silicon	Si $K\alpha_{1,2}$	0.712 6	Zirconium	Zr $K\alpha_{1,2}$	0.078 73
Manganese	Mn $K\alpha_{1,2}$	0.210 3	Niobium	Nb $K\alpha_{1,2}$	0.074 76
Phosphorus	P $K\alpha_{1,2}$	0.615 8	Magnesium	Mg $K\alpha_{1,2}$	0.989 0
Sulfur	S $K\alpha_{1,2}$	0.537 3	Calcium	Ca $K\alpha_{1,2}$	0.335 9
Nickel	Ni $K\alpha_{1,2}$	0.165 9	Tantalum	Ta $L\alpha_1$	0.152 2
Chromium	Cr $K\alpha_{1,2}$	0.229 1		Ta $L\beta_1$	0.132 7
Molybdenum	Mo $K\alpha_{1,2}$	0.071 07	Antimony	Sb $K\alpha_{1,2}$	0.047 18
Copper	Cu $K\alpha_{1,2}$	0.154 2		Sb $L\alpha_1$	0.343 9
Tungsten	W $L\alpha_1$	0.147 6	Selenium	Se $K\alpha_{1,2}$	0.110 6
Vanadium	V $K\alpha_{1,2}$	0.250 5	Tellurium	Te $K\alpha_{1,2}$	0.045 28
Cobalt	Co $K\alpha_{1,2}$	0.179 0		Te $L\alpha_1$	0.328 9
	Co $K\beta_{1,3}$	0.162 1	Bismuth	Bi $L\alpha_1$	0.114 4
Titanium	Ti $K\alpha_{1,2}$	0.275 0	Iron	Fe $K\alpha_{1,2}$	0.193 7
				Fe $K\beta_{1,3}$	0.175 7
Aluminium	Al $K\alpha_{1,2}$	0.834 0	Lanthanum	La $L\alpha_1$	0.266 6
Arsenic	As $K\alpha_{1,2}$	0.117 7		La $L\beta_1$	0.245 9
	As $K\beta_{1,3}$	0.105 7	Cerium	Ce $L\alpha_1$	0.256 1
Tin	Sn $K\alpha_{1,2}$	0.049 21		Ce $L\beta_1$	0.235 6
	Sn $L\alpha_1$	0.360 0	Praseodymium	Pr $L\alpha_1$	0.246 3
	Sn $L\beta_1$	0.338 5		Pr $L\beta_1$	0.225 9
Lead	Pb $L\alpha_1$	0.117 5	Neodymium	Nd $L\alpha_1$	0.237 0
	Pb $L\beta_1$	0.098 29		Nd $L\beta_1$	0.216 6
Zinc	Zn $K\alpha_{1,2}$	0.143 6			