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**JIS G 1253** : 2002  
(JISF)

**Iron and steel—Method for  
spark discharge atomic emission  
spectrometric analysis**

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## Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Iron and Steel Federation (JISF) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS G 1253 : 1995** is replaced with this Standard.

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## Iron and steel—Method for spark discharge atomic emission spectrometric analysis

**1 Scope** This Japanese Industrial Standard specifies the method for atomic emission spectrometric analysis by spark discharge of iron and steel and shall be applicable to the quantitative determination of respective components shown in Table 1. However, the quantitative determination of nitrogen shall be applicable to only steel.

Remarks : The iron in this Standard means pig iron, cast iron and the like, and the steel means carbon steel, low alloy steel, high alloy steel and the like.

**Table 1 Components to be applied and determination range**

| Component  | Determination range<br>% (m/m) |                              |
|------------|--------------------------------|------------------------------|
| Carbon     | 0.001                          | or over up to and incl. 5.5  |
| Silicon    | 0.002                          | or over up to and incl. 6    |
| Manganese  | 0.003                          | or over up to and incl. 30   |
| Phosphorus | 0.000 5                        | or over up to and incl. 1.0  |
| Sulfur     | 0.000 2                        | or over up to and incl. 0.5  |
| Nickel     | 0.002                          | or over up to and incl. 40   |
| Chromium   | 0.002                          | or over up to and incl. 40   |
| Molybdenum | 0.001                          | or over up to and incl. 10   |
| Copper     | 0.001                          | or over up to and incl. 6    |
| Tungsten   | 0.01                           | or over up to and incl. 25   |
| Vanadium   | 0.001                          | or over up to and incl. 6    |
| Cobalt     | 0.001                          | or over up to and incl. 20   |
| Titanium   | 0.000 5                        | or over up to and incl. 3    |
| Aluminium  | 0.001                          | or over up to and incl. 5    |
| Arsenic    | 0.001                          | or over up to and incl. 0.3  |
| Tin        | 0.000 6                        | or over up to and incl. 0.3  |
| Boron      | 0.000 05                       | or over up to and incl. 0.5  |
| Nitrogen   | 0.001                          | or over up to and incl. 0.15 |
| Lead       | 0.001                          | or over up to and incl. 0.5  |
| Zirconium  | 0.001                          | or over up to and incl. 1    |
| Niobium    | 0.001                          | or over up to and incl. 2    |
| Magnesium  | 0.001                          | or over up to and incl. 0.2  |
| Calcium    | 0.000 1                        | or over up to and incl. 0.01 |
| Tantalum   | 0.02                           | or over up to and incl. 0.2  |
| Antimony   | 0.008                          | or over up to and incl. 0.5  |
| Selenium   | 0.003                          | or over up to and incl. 0.1  |
| Tellurium  | 0.003                          | or over up to and incl. 0.1  |
| Lanthanum  | 0.002                          | or over up to and incl. 0.05 |
| Cerium     | 0.005                          | or over up to and incl. 0.05 |

**2 Normative references** The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

- JIS G 0417 *Steel and iron—Sampling and preparation of samples for the determination of chemical composition*
- JIS G 1201 *Iron and steel—General rules for analytical methods*
- JIS K 0116 *General rules for atomic emission spectrometry*
- 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 *Bonded abrasive grain sizes*

**3 Definitions** For the purposes of this Standard, the definitions given in **JIS K 0116**, **JIS K 0211**, **JIS K 0212**, **JIS K 0215** and the following definitions apply.

- a) **measured value of emission strength** In the case of the emission strength method, the emission strength of quantitative component of the sample obtained by atomic emission spectrometry. In the case of the strength ratio method, the ratio of the emission strength of quantitative component of the sample to that of the internal standard element (iron, in general).
- b) **specific time integration method** The method in which the determination is carried out by integrating the strength of emitted spectral line for a specific time at the measurement of emission strength and then converting it to a digital signal value.
- c) **pulse distribution analysis (PDA method)** The method in which the emission strength is converted to a digital signal value for every single pulse at the measurement of emission strength and determined by using the numerical value (mean value, median value, etc.) treated statistically of the whole signal values of the pulse measured.
- d) **metallurgical hysteresis** The hysteresis of the sample such as the solidifying speed of melt sample, the heating temperature of heat treatment, rolling, forging, etc., influences the measured value of emission strength depending on the form of the metallic structure, precipitate, or intervening material, even if the chemical compositions of test portion are the same.

**4 General items** The general items required in analysis shall be in accordance with **JIS G 1201** and **JIS K 0116**.

**5 Summary** After cutting-off or cutting of the sample, it is finished to a plane surface by grinding or polishing, fixed to the sample support of emission spectrophotometer to make an electrode, and a spark is generated by using silver or tungsten as the counter electrode. The spectral line is then resolved by a spectroscope and the strength of the spectral line of the quantitative component of the sample is measured.

## 6 Equipment and analytical condition

**6.1 Emission spectrophotometer** The emission spectrophotometer shall be in accordance with 1.2 of Annex (normative), and shall be as follows:

- a) **Counter electrode** Mould the top end of silver rod or tungsten rod of 1 mm to 7 mm diameter to a cone of 20° to 120° in angle or a truncated cone having a plane surface of 1 mm diameter by a moulding machine.
- b) **Argon** It is at least 99.99 % (*m/m*) in purity with a small amount of impurities such as oxygen, hydrocarbon, nitrogen. Since the purity of argon will influence greatly the quantitative value, in the case of supplying it to the equipment by using a cylinder, when the lot of the cylinders is changed or the residual quantity has reduced, confirm the determination result according to clause 12.

**6.2 Adjustment of equipment** The adjustment of equipment shall be in accordance with 1.3 of Annex (normative).

**6.3 Equipment performance reference** The equipment performance reference shall be as follows:

- a) For the emission spectrophotometer adjusted according to 6.2, the analytical condition (analytical line, excitation condition, photometry condition, etc.) shall be set so that the repeatability and sensitivity become adequate.
- b) The repeatability shall be not more than the tolerance on repeatability standard deviation shown in Table 2 and Table 3.

The repeatability means the standard deviation of quantitative independent values obtained from the measured values of at least 6 measurements of the emission strength of a homogeneous sample, for example, the reference material of iron and steel for instrument analysis carried out continuously according to the procedure of clause 9.

- c) The checking of the equipment performance reference, in addition to those periodical, shall be carried out every time the analytical condition is changed or the condition of equipment is changed by repairs, adjustments, etc.

**Table 2 Repeatability standard deviation tolerance (steel)**

Unit: % (m/m)

| Component  | Range of component content      |         | Repeatability standard deviation tolerance <sup>(1)</sup> |
|------------|---------------------------------|---------|-----------------------------------------------------------|
| Carbon     | 0.013 or over to and excl.      | 0.14    | $3.0 \times (0.013\ 08 \times C \% + 0.000\ 45)$          |
|            | 0.14 or over up to and incl.    | 0.49    | $3.0 \times (0.001\ 98 \times C \% + 0.002\ 01)$          |
| Silicon    | 0.10 or over up to and incl.    | 1.42    | $3.0 \times (0.009\ 40 \times Si \% - 0.000\ 38)$         |
| Manganese  | 0.12 or over up to and incl.    | 1.79    | $3.0 \times (0.005\ 08 \times Mn \% + 0.000\ 11)$         |
| Phosphorus | 0.004 or over up to and incl.   | 0.038   | $3.0 \times (0.012\ 57 \times P \% + 0.000\ 09)$          |
| Sulfur     | 0.002 or over up to and incl.   | 0.041   | $3.0 \times (0.026\ 30 \times S \% + 0.000\ 06)$          |
| Nickel     | 0.015 or over to and excl.      | 4.00    | $3.0 \times (0.005\ 98 \times Ni \% + 0.000\ 34)$         |
|            | 4.00 or over up to and incl.    | 20.6    | $3.0 \times (0.007\ 40 \times Ni \% - 0.005\ 33)$         |
| Chromium   | 0.011 or over to and excl.      | 3.3     | $3.0 \times (0.004\ 13 \times Cr \% + 0.000\ 43)$         |
|            | 3.3 or over to and excl.        | 15.0    | $3.0 \times (0.001\ 48 \times Cr \% + 0.009\ 20)$         |
|            | 15.0 or over up to and incl.    | 25.6    | $3.0 \times (0.008\ 20 \times Cr \% - 0.091\ 60)$         |
| Molybdenum | 0.010 or over up to and incl.   | 2.43    | $3.0 \times (0.006\ 03 \times Mo \% + 0.000\ 16)$         |
| Copper     | 0.024 or over up to and incl.   | 0.49    | $3.0 \times (0.007\ 19 \times Cu \% + 0.000\ 25)$         |
| Vanadium   | 0.010 or over up to and incl.   | 0.40    | $3.0 \times (0.004\ 13 \times V \% + 0.000\ 09)$          |
| Cobalt     | 0.013 or over up to and incl.   | 0.27    | $3.0 \times (0.002\ 44 \times Co \% + 0.000\ 29)$         |
| Titanium   | 0.012 or over up to and incl.   | 0.10    | $3.0 \times (0.009\ 27 \times Ti \% + 0.000\ 01)$         |
| Arsenic    | 0.005 or over up to and incl.   | 0.046   | $3.0 \times (0.004\ 74 \times As \% + 0.000\ 24)$         |
| Tin        | 0.006 or over up to and incl.   | 0.066   | $3.0 \times (0.008\ 75 \times Sn \% + 0.000\ 08)$         |
| Boron      | 0.001 4 or over up to and incl. | 0.007   | $3.0 \times (0.004\ 73 \times B \% + 0.000\ 03)$          |
| Nitrogen   | 0.001 or over up to and incl.   | 0.15    | $3.0 \times (0.009\ 99 \times N \% + 0.000\ 39)$          |
| Zirconium  | 0.008 or over up to and incl.   | 0.039   | $3.0 \times (0.020\ 45 \times Zr \% + 0.000\ 06)$         |
| Niobium    | 0.010 or over up to and incl.   | 0.050   | $3.0 \times (0.003\ 08 \times Nb \% + 0.000\ 26)$         |
| Calcium    | 0.000 6 or over up to and incl. | 0.002 9 | $3.0 \times (0.022\ 23 \times Ca \% + 0.000\ 02)$         |

Note (1) For the elementary symbol for each component, followed by %, the mean value of the quantitative values [% (m/m)] obtained during the procedure of determining the repeatability of each component in 6.3 b) is inserted.

**Table 3 Repeatability standard deviation tolerance (iron)**

Unit: % (m/m)

| Component  | Range of component content         | Repeatability standard deviation tolerance <sup>(1)</sup> |
|------------|------------------------------------|-----------------------------------------------------------|
| Carbon     | 2.2 or over up to and incl. 4.32   | $3.0 \times (0.005\ 01 \times C \% + 0.002\ 9)$           |
| Silicon    | 0.32 or over to and excl. 1.60     | $3.0 \times (0.006\ 09 \times Si \% + 0.001\ 7)$          |
|            | 1.60 or over up to and incl. 3.44  | $3.0 \times (0.014\ 59 \times Si \% - 0.011\ 8)$          |
| Manganese  | 0.18 or over up to and incl. 2.42  | $3.0 \times (0.003\ 92 \times Mn \% + 0.001\ 3)$          |
| Phosphorus | 0.010 or over up to and incl. 0.57 | $3.0 \times (0.010\ 40 \times P \% + 0.000\ 3)$           |
| Sulfur     | 0.003 or over up to and incl. 0.23 | $3.0 \times (0.032\ 82 \times S \% + 0.000\ 2)$           |
| Nickel     | 0.03 or over up to and incl. 5.27  | $3.0 \times (0.008\ 26 \times Ni \% + 0.000\ 1)$          |
| Chromium   | 0.02 or over up to and incl. 2.11  | $3.0 \times (0.006\ 42 \times Cr \% + 0.000\ 2)$          |
| Copper     | 0.02 or over up to and incl. 2.11  | $3.0 \times (0.046\ 70 \times Cu \% + 0.000\ 3)$          |
| Titanium   | 0.009 or over up to and incl. 0.17 | $3.0 \times (0.018\ 66 \times Ti \% - 0.000\ 1)$          |

**6.4 Analytical condition** The analytical condition shall be set so as to satisfy the equipment performance reference depending on the classification of the test portion, the coexisting components, the classification of simultaneous quantitative component of the sample and the content of quantitative component of the sample. The examples of the analytical conditions and the related matters are shown in Table 4 to Table 7. The emission strength becomes in some cases unstable immediately after exchanging to a newly moulded counter electrode and under the condition of discharge repeated multiple times. Therefore, the range of number of times for analysis, in which an emission strength is stably obtained shall be previously investigated.

**Table 4 An example of analytical condition**

| Item                                           | Contents                                                         |
|------------------------------------------------|------------------------------------------------------------------|
| Pressure in spectroscope                       | 2.7 Pa max.                                                      |
| Reciprocal linear dispersion of spectroscope   | 1 nm/mm max.                                                     |
| Inlet slit width                               | 20 $\mu$ m to 50 $\mu$ m                                         |
| Photometry method                              | Specific time integration method<br>Time resolved PDA photometry |
| Gap between test portion and counter electrode | 4.0 mm to 6.0 mm                                                 |
| Flow rate of argon gas at the time of emission | 4 L/min to 18 L/min                                              |
| Wavelength of analytical line                  | See Table 5                                                      |
| Excitation condition and discharge condition   | See Table 6 and Table 7                                          |

**Table 5 An example of analytical line and internal standard line**

| Component  | Wavelength of analytical line nm | Component | Wavelength of analytical line nm |
|------------|----------------------------------|-----------|----------------------------------|
| Carbon     | C I 156.14                       | Aluminium | Al I 308.22                      |
|            | C I 165.81                       |           | Al I 394.40                      |
|            | C I 193.09                       |           | Al I 396.15                      |
| Silicon    | Si I 212.42                      | Arsenic   | As I 197.26                      |
|            | Si I 251.61                      |           | As I 228.81                      |
|            | Si I 288.16                      |           | As I 234.98                      |
| Manganese  | Mn II 290.02                     | Tin       | Sn I 189.99                      |
|            | Mn II 293.31                     |           | Sn I 317.50                      |
| Phosphorus | P I 177.50                       |           | Sn I 326.23                      |
|            | P I 178.29                       | Boron     | B I 182.58                       |
|            | P I 214.91                       |           | B I 182.64                       |
| Sulfur     | S I 180.73                       |           | B II 206.72                      |
|            |                                  |           | B I 208.96                       |
| Nickel     | Ni II 225.39                     |           | B I 249.68                       |
|            | Ni II 227.02                     | Nitrogen  | N I 149.26                       |
|            | Ni II 227.73                     |           |                                  |
|            | Ni II 231.60                     | Lead      | Pb II 220.35                     |
|            | Ni I 341.48                      |           | Pb I 283.31                      |
|            | Ni II 243.79                     |           | Pb I 405.78                      |
| Chromium   | Cr II 265.85                     | Zirconium | Zr II 339.20                     |
|            | Cr II 267.72                     |           | Zr II 343.82                     |
|            | Cr II 276.65                     | Niobium   | Nb II 319.50                     |
|            | Cr II 286.09                     |           | Nb II 320.63                     |
|            | Cr II 286.26                     | Magnesium | Mg II 279.55                     |
|            | Cr II 298.92                     |           | Mg II 280.27                     |
|            | Cr I 428.97                      | Calcium   | Ca II 393.37                     |
| Molybdenum | Mo II 202.03                     |           | Ca II 396.85                     |
|            | Mo II 277.54                     | Tantalum  | Ta II 240.06                     |
|            | Mo II 281.62                     |           |                                  |
|            | Mo I 313.26                      | Antimony  | Sb I 187.12                      |
|            | Mo I 317.03                      |           |                                  |
|            | Mo I 386.41                      | Selenium  | Se I 196.09                      |
|            |                                  |           |                                  |
|            |                                  | Tellurium | Te I 214.28                      |
|            |                                  |           |                                  |
|            |                                  | Lanthanum | La II 408.67                     |
|            |                                  |           |                                  |