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Methods for determination of
iron in nickel and nickel alloys

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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 H 1273 : 1988** is replaced with **JIS H 1273 : 1998**.

In this revision, in order to make comparison with ISO, preparation of JIS agreeing with ISO, and proposal to the draft of ISO based on JIS easy, **ISO 6351 : 1985**, *Nickel—Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents—Flame atomic absorption spectrometric method*, **ISO 7528 : 1989**, *Nickel alloys—Determination of iron content—Titrimetric method with potassium dichromate*, **ISO 7530-1 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution*, and **ISO 7530-5 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 5 : Determination of iron content* are made a part of this Standard.

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Methods for determination of iron in nickel and nickel alloys

Introduction This Japanese Industrial Standard is prepared so as to conform in technical contents with corresponding parts of **ISO 6351** : 1985, *Nickel—Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents—Flame atomic absorption spectrometric method*, **ISO 7528** : 1989, *Nickel alloys—Determination of iron content—Titrimetric method with potassium dichromate*, **ISO 7530-1** : 1990, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution* and **ISO 7530-5** : 1990, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 5 : Determination of iron content* (in part of determination of iron in **ISO 6351**, and the whole in **ISO 7528** and **ISO 7530**).

Further, one determination method without including any corresponding ISO is specified as Japanese Industrial Standard.

1 Scope This Japanese Industrial Standard specifies the methods for determination of iron content in nickel and nickel alloys.

Remarks : The International Standards corresponding to this Standard are as follows.

ISO 6351 : 1985 *Nickel—Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents—Flame atomic absorption spectrometric method*

ISO 7528 : 1989 *Nickel alloys—Determination of iron content—Titrimetric method with potassium dichromate*

ISO 7530-1 : 1990 *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution*

ISO 7530-5 : 1990 *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 5 : Determination of iron content*

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 indicated below shall be applied.

JIS H 1270 *General rules for chemical analysis of nickel and nickel alloys*

JIS K 8005 *Reference materials for volumetric analysis*

JIS K 8580 *Tin*

3 General matters General matters common to the methods for chemical analysis shall be as specified in **JIS H 1270** and **JIS K 8005**.

4 Classification of determination methods The methods for determination of iron shall be in accordance with any one of the following methods, and applied reagents to each determination method shall be as given in Table 1.

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- a) **Iron hydroxide precipitate separation potassium dichromate titrimetric method (method A)** This method is applied to the sample of 0.3 % (m/m) or over up to and including 10 % (m/m) iron content.
- b) **Iron hydroxide precipitate separation potassium dichromate titrimetric method (method B)** This method is applied to the sample of 0.6 % (m/m) or over up to and including 50 % (m/m) iron content.
- c) **Flame atomic absorption spectrometric analysis (method A)** This method is applied to the sample of 0.01 % (m/m) or over up to and including 2.0 % (m/m) iron content.
- d) **Flame atomic absorption spectrometric analysis (method B)** This method is applied to the sample of 0.01 % (m/m) or over up to and including 4.0 % (m/m) iron content.

Table 1 Determination method and sample to be applied

Sample		Iron hydroxide precipitate separation potassium dichromate titrimetric method (method A)	Iron hydroxide precipitate separation potassium dichromate titrimetric method (method B)	Flame atomic absorption spectrometric analysis (method A)	Flame atomic absorption spectrometric analysis (method B)
Alloy number	Alloy symbol				
NW2200	Ni99.0	○	—	○	○
NW2201	Ni99.0-LC	○	—	○	○
	NATB	—	○	○	○
NW4400	NiCu30	○	—	—	○
NW4402	NiCu30-LC	○	—	—	○
NW5500	NiCu30Al3Ti	—	○	—	○
NW0001	NiMo30Fe5	—	○	—	—
NW0665	NiMo28	—	○	—	—
NW0276	NiMo16Cr15Fe6W4	—	○	—	—
NW6455	NiCr16Mo16Ti	—	○	—	○
NW6022	NiCr21Mo13Fe4W3	—	○	—	—
NW6007	NiCr22Fe20Mo6Cu2Nb	—	○	—	—
NW6985	NiCr22Fe20Mo7Cu	—	○	—	—
NW6002	NiCr21Fe18Mo9	—	○	—	—
Nickel casting		○	—	○	○
Nickel copper alloy casting		○	—	○	○
Nickel molybdenum alloy casting		—	○	—	—
Nickel molybdenum chromium alloy casting		—	○	—	—
Nickel chromium iron alloy casting		—	○	—	—

5 Iron hydroxide precipitate separation potassium dichromate titrimetric method (method A)

5.1 Summary Decompose a sample with nitric acid, precipitate iron (III) hydroxide by adding ammonium chloride and aqueous ammonia, and filter. Dissolve the precipitate in hydrochloric acid, reduce iron (III) to iron (II) by adding tin (II) chloride, and titrate with potassium dichromate standard solution.

5.2 Reagents The reagents shall be as follows.

- a) Hydrochloric acid (1+3, 1+10)
- b) Nitric acid (1+1)
- c) Mixed acid (3 parts of sulfuric acid, 3 parts of phosphoric acid, and 14 parts of water by vol.)
- d) Aqueous ammonia
- e) Aqueous ammonia (1+20)
- f) Ammonium chloride solution (250 g/l)
- g) Tin (II) chloride solution Dissolve 10 g of tin (II) chloride dihydrate in 100 ml of hydrochloric acid (1+1) by heating. After cooling to room temperature, dilute the solution amount to 200 ml with water, add a small amount of tin (**JIS K 8580**), and reserve in a brown bottle.
- h) Mercury (II) chloride solution (saturated, about 70 g/l)
- i) $\frac{1}{120}$ mol/l potassium dichromate standard solution Take a necessary amount of potassium dichromate (**JIS K 8580**). After drying at 150 °C for about 60 min, put into a desiccator, let it stand to cool, weigh out 2.4 g to 2.5 g thereof nearest to 0.1 mg, put it into a 1 000 ml one-mark volumetric flask, and dissolve by adding water. Add water to the mark, after mixing, put it into an airtight container, and reserve. Calculate the factor of the solution according to the following formula.

$$f = \frac{m_0}{2.452} \times \frac{P}{100}$$

where, f : factor of $\frac{1}{120}$ mol/l potassium dichromate standard solution

m_0 : weighed out mass of potassium dichromate (g)

P : potassium dichromate content in potassium dichromate standard material [% (m/m)]

- j) Sodium diphenylaminesulfonate solution Dissolve 0.2 g of sodium diphenylamine-sulfonate in a small amount of water, and dilute the liquid amount to 100 ml with water. Reserve the solution in a brown bottle.

5.3 Weighing out amount of sample The weighing out amount of a sample shall be 1.0 g.