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Methods for determination
of manganese 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 1274 : 1988** is replaced with **JIS H 1274 : 1998**.

In this revision, in order to make comparison with ISO, prepare JIS conforming with ISO, and make it easy to propose of the draft of ISO based on the JIS, **ISO 6351 : 1985**, *Nickel—Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents—Flame atomic absorption spectrometric method*, **ISO 7530-1 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution*, and **ISO 7530-6 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 6 : Determination of manganese content* are made a part of this Standard. Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Methods for determination of manganese in nickel and nickel alloys

Introduction This Standard is a Japanese Industrial Standard prepared so that its technical contents conform to the corresponding parts (a part relating to determination of manganese for **ISO 6351**, and the whole for **ISO 7530**) of the corresponding **ISO 6351 : 1985**, *Nickel—Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents—Flame atomic absorption spectrometric method*, **ISO 7530-1 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution*, and **ISO 7530-6 : 1990**, *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 6 : Determination of manganese content*.

Further, a determination method having no corresponding International Standard is specified as JIS.

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

Remarks : The corresponding International Standards to this Standard are shown 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 7530-1 : 1990 *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 1 : General requirements and sample dissolution*

ISO 7530-6 : 1990 *Nickel alloys—Flame atomic absorption spectrometric analysis—Part 6 : Determination of manganese content*

2 Normative reference The following standard contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent edition of the standard indicated below shall be applied.

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

3 General matters General matters common to the methods of chemical analysis shall be in accordance with **JIS H 1270**.

4 Classification of determination methods The method for determination of manganese shall be in accordance with any one of the following methods. The sample to which each determination applies shall be as given in Table 1.

- a) **Permanganic acid absorptiometry** This method applies to the sample of 0.05 % (m/m) or over up to and including 2.0 % (m/m) in manganese content.
- b) **Flame atomic absorption spectrometric method (method A)** This method applies to the sample of 0.01 % (m/m) or over up to and including 2.0 % (m/m) in manganese content.

- c) **Flame atomic absorption spectrometric method (method B)** This method applies to the sample of 0.01 % (m/m) or over up to and including 4.0 % (m/m) in manganese content.

Table 1 Determination method and sample to be applied

Sample		Determination method		
Alloy number	Alloy symbol	Permanganic acid absorptiometry	Flame atomic absorption spectrometric method (method A)	Flame atomic absorption spectrometric method (method B)
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 Permanganic acid absorptiometry

5.1 Summary After decomposing a sample with mixed acid of phosphoric acid, sulfuric acid and nitric acid, add sodium periodate, oxidize manganese to permanganic acid by boiling, and measure the absorbance with a photometer.

5.2 Reagents The reagents shall be as follows.

- Hydrochloric acid
- Hydrochloric acid (1+1)

- c) Nitric acid
- d) Mixed acid A Add 120 ml of phosphoric acid and 100 ml of sulfuric acid to 600 ml of water. After cooling to room temperature, add 170 ml of nitric acid, and dilute the solution amount to 1 000 ml with water.
- e) Mixed acid B Add 120 ml of phosphoric acid and 100 ml of sulfuric acid to 600 ml of water. After cooling to room temperature, dilute the solution amount to 1 000 ml with water.
- f) Nickel Nickel is not less than 99.9 % (m/m) in purity, and not more than 0.001 % (m/m) in manganese content.
- g) Iron Iron is not less than 99.9 % (m/m) in purity and not more than 0.001 % (m/m) in manganese content.
- h) Copper Copper is not less than 99.9 % (m/m) in purity and not more than 0.001 % (m/m) in manganese content.
- i) Chromium Chromium is not less than 99.9 % (m/m) in purity and not more than 0.001 % (m/m) in manganese content.
- j) Hydrogen peroxide
- k) Sodium nitrite solution (100 g/l)
- l) Sodium periodate solution (50 g/l)
- m) Ammonium molybdate solution (10 g/l) Weigh out 18.4 g of hexaammonium heptamolybdate tetrahydrate, and dissolve it in about 600 ml of warm water. After cooling to ordinary temperature, transfer the solution into a 1 000 ml one-mark volumetric flask with water, and dilute to the mark with water. Prepare this solution at each service.
- n) Urea solution (100 g/l)
- o) Standard manganese solution (100 µgMn/ml) Weigh out 1.000 g of manganese [99.9 % (m/m) or higher], transfer into a beaker (300 ml), cover with a watch glass, add 30 ml of hydrochloric acid (1+1), decompose by heating, and add 0.5 ml of hydrogen peroxide. After cooling to ordinary temperature, wash the bottom face of the watch glass with water, remove the watch glass, transfer the solution into a 1 000 ml one-mark volumetric flask with water, add 35 ml of hydrochloric acid, and dilute to the mark with water to prepare stock solution (1 000 µgMn/ml). Take 100.0 ml of the stock solution into a 1 000 ml one-mark volumetric flask, add 45 ml of hydrochloric acid, and dilute to the mark with water to prepare standard manganese solution.

5.3 Weighing out amount of sample The weighing out amount of a sample shall be as given in Table 2.