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**Iron and steel—
Methods for determination
of manganese content**

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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 in accordance with the Industrial Standardization Law. Consequently **JIS G 1213 : 1981** is replaced with **JIS G 1213 : 2001**.

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Iron and steel— Methods for determination of manganese content

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

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 1201 *General rules for chemical analysis of iron and steel*

JIS K 8001 *General rule for test methods of reagents*

JIS K 8005 *Reference materials for volumetric analysis*

JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results—Part 6 : Use in practice of accuracy values*

3 General matters General matters common to the determination methods shall be given in JIS G 1201.

4 Classification of determination methods The method for determination of manganese shall conform to either of the following:

- a) **Ammonium peroxodisulfate oxidative sodium oxalate/potassium permanganate back titration method** This method is applied to the sample of $\geq 0.1 \%$ (*m/m*) and $\leq 30 \%$ (*m/m*) in manganese content, and the determination method shall be as given in Annex 1 (normative).
- b) **Permanganic acid absorptiometry** This method is applied to the sample of $\geq 0.01 \%$ (*m/m*) and $\leq 20 \%$ (*m/m*) in manganese content, and the determination method shall be as given in Annex 2 (normative).

Annex 1 (normative)

Ammonium peroxodisulfate oxidative sodium oxalate/potassium permanganate back titration method

1 Summary A sample is decomposed by a suitable acid, silver nitrate is used as a catalyst, and manganese is oxidized to manganese (VII) by ammonium peroxodisulfate. A specific amount of sodium oxalate standard solution is added and manganese (VII) is reduced to manganese (II). Thereafter, excess sodium oxalate is titrated by potassium permanganate standard solution.

2 Reagents The reagents shall be as follows:

- a) **Nitric acid (1+1)**
- b) **Perchloric acid**
- c) **Hydrofluoric acid**
- d) **Sulfuric acid (1+1, 1+10, 1+100)**
- e) **Aqua regia** (3 parts of hydrochloric acid and 1 part of nitric acid)
- f) **Mixed acid** (3 parts of sulfuric acid, 10 parts of phosphoric acid, 17 parts of water)
- g) **Sodium chloride**
- h) **Potassium disulfate**
- i) **Cobalt solution (0.02 gCo/ml)** 10 g of cobalt (II) nitrate hexahydrate is dissolved in about 70 ml of water, and is diluted to 100 ml by water.
- j) **Nickel solution (0.02 gNi/ml)** 9 g of nickel (II) sulfate hexahydrate is dissolved in about 70 ml of water, and is diluted to 100 ml by water.
- k) **Silver nitrate solution (10 g/l)**
- l) **Ammonium peroxodisulfate solution (200 g/l)** This solution is prepared per each use.
- m) **Manganese (II) sulfate solution** 10 g of manganese (II) sulfate pentahydrate is dissolved in 200 ml of water, and 50 ml of sulfuric acid (1+1) is added. After cooling, 0.02 mol/l potassium permanganate standard solution is added drop by drop while it is mixed by shaking until the red purple of the solution does not disappear for about 1 min, and is diluted to 1 000 ml by water.
- n) **0.05 mol/l sodium oxalate standard solution** Sodium oxalate specified in JIS K 8005 is dried following the drying condition given in Table 1 of JIS K 8005, its 6.700 g is transferred into a beaker (300 ml) by weighing out, it is dissolved by adding 500 ml of water, 50 ml of sulfuric acid (1+1) is added little by little. After cooling to ordinary temperature, the solution is transferred into a 1 000 ml volumetric flask by using water, and diluted to the mark by water. 1 ml of this standard solution corresponds to 0.001 009 g of manganese.

- o) **0.02 mol/l potassium permanganate standard solution** The preparation and the standardization methods shall be as given in 4.5 of JIS K 8001.

3 Weighing out amount of sample The weighing out amount of a sample shall be as given in Annex 1 Table 1.

Annex 1 Table 1 Weighing out amount of sample

Manganese content % (m/m)	Weighing out amount of sample g
0.1 or over to and excl. 0.3	2.0
0.3 or over to and excl. 1.5	1.0
1.5 or over to and excl. 2	0.50
2 or over to and excl. 5	0.25
5 or over up to and incl. 30	0.10

4 Operation

Informative reference : Warning The vapor of perchloric acid is in danger of exploding if ammonia, nitrous acid vapor or organic matter exists. The vaporization treatment of perchloric acid shall be carried out at a place equipped with a local exhaust facility which is safe though perchloric acid is used.

4.1 Preparation of sample solution The preparation of sample solution shall conform to any one of the following.

- a) **Sample easy to be decomposed by mixed acid and nitric acid** A sample is weighed out, transferred into an Erlenmeyer flask (500 ml), 30 ml of mixed acid and 10 ml of nitric acid (1+1) are added, it is decomposed by heating, and successively heated to remove nitrogen oxide or the like⁽¹⁾ ⁽²⁾.

Notes ⁽¹⁾ When residues of free carbon and the like are found, after adding about 50 ml of water, the solution is filtered by using filter paper (class 5 B) and filter paper pulp, the filter paper is washed 4 or 5 times by sulfuric acid (1+100), and the filtrate and washings are collected in the Erlenmeyer flask (500 ml).

- ⁽²⁾ For a sample containing tungsten, the sample solution is successively heated to generate white fumes. After being allowed to cool down, about 50 ml of water is added, and soluble salts are dissolved.

- b) **Sample containing not less than 15 mg of chromium in the weighed out sample** Weigh out a sample, transfer into an Erlenmeyer flask (500 ml), add 30 ml of aqua regia and decompose by heating. Add 20 ml of perchloric acid, successively heat, add several drops of hydrofluoric acid just before generation of white fumes of perchloric acid, and oxidize chromium to dichromic acid by successively heating. Add 1 g to 2 g of sodium chloride by dividing several times while heating, and repeat the operation of adding sodium chloride until generation of brown fumes disappears. Successively heat to decrease the amount of the

solution to 5 ml or under. After allowing to cool down, dissolve soluble salts by adding about 50 ml of water⁽³⁾, and add 30 ml of mixed acid.

Note (3) When precipitates of tungstic acid, silicic acid and the like are recognized, filter the solution by using filter paper (class 5 B) and filter paper pulp, wash the filter paper 4 or 5 times with sulfuric acid (1+100), and collect the filtrate and the washings into the Erlenmeyer flask (500 ml).

- c) **Sample difficult to be decomposed by acid** Weigh out a sample, transfer into a beaker (300 ml), cover with a watch glass, add 30 ml of mixed acid and nitric acid (1+1), decompose by heating, and remove nitrogen oxide or the like by successively heating. Wash the bottom surface of the watch glass with a small amount of water, remove the watch glass, add about 50 ml of water, filter the solution with filter paper (class 5 B), wash the filter paper 4 or 5 times with sulfuric acid (1+100), then 2 or 3 times with warm water, and collect the filtrate and the washings in an Erlenmeyer flask (500 ml), and reserve as the main solution. Transfer the insoluble residues together with the filter paper into a platinum crucible (No. 30), dry the filter at a low temperature, further incinerate the filter paper by heating at about 800 °C. Thereafter, allow to cool down. Fuse the residue by adding 1 g to 2 g of potassium disulfate. After allowing to cool down, dissolve the melt by adding a small amount of warm water and 1 ml of sulfuric acid (1+10). Filter the solution with filter paper (class 5 B), wash the filter paper with warm water, and add the filtrate and washings to the previously preserved main solution.

4.2 Oxidation of manganese Add 10 ml of silver nitrate solution to the solution obtained in a), b) or c) of 4.1, and dilute to about 200 ml with hot water. Add 10 ml of ammonium peroxodisulfate solution [2 l)], and boil by heating. After small bubbles by decomposition of ammonium peroxodisulfate have changed to large bubbles and the surface of solution has become still, further heat for 30 s to 60 s.

4.3 Titration Immediately add about 50 ml of water and 1.0 ml to 1.5 ml of manganese sulfate solution [2 m)] to the solution obtained in 4.2⁽⁴⁾ ⁽⁵⁾, vanish the red purple of the solution with 0.05 mol/l sodium oxalate standard solution [2 n)] by using a burette⁽⁶⁾, and further add 2 ml excessively, thereafter, read accurately the used amount of 0.05 mol/l sodium oxalate standard solution [2 n)]. Immediately titrate excess sodium oxalate with 0.02 mol/l potassium permanganate standard solution [2 o)], take the point wherein the solution shows dimly red purple as the end point⁽⁷⁾, and obtain the used amount of 0.02 mol/l potassium permanganate standard solution [2 o)].

Notes (4) When a great amount of nickel is contained in a sample, add cobalt solution [2 i)] so that the ratio of the nickel amount in sample solution to the added cobalt amount becomes 4 : 1.

(5) When a great amount of cobalt is contained in a sample, add nickel solution [2 j)] so that the ratio of the cobalt amount in sample solution to the added nickel amount becomes 1 : 4.

(6) When vanadium is contained in a sample, slowly add sodium oxalate standard solution [2 n)].

(7) When vanadium is contained in a sample, take the point wherein red purple color lasts for about 1 min as the end point.