INTERNATIONAL STANDARD

ISO 4298

Third edition
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Manganese ores and concentrates — Determination of manganese content — Potentiometric method

Minerais et concentrés de manganèse — Dosage du manganèse — Méthode potentiométrique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 132, *Ferroalloys*.

This third edition cancels and replaces the second edition (ISO 4298:1984), which has been technically revised.

The main changes are as follows:

- in Clause 4, the description of reaction has been reworded to better explain the method;
- in 5.12 (previously in 5.10), the detailed specification of “electrolytic manganese” has been added and described as “electrolytic manganese metal flakes”;
- in 5.13 (previously in 5.11), the commercially available standard solution has been added as an alternative, and variations in concentration shall be taken into account for the calculation of the results in 9.1;
- in 5.14 (previously 5.11.1), the amount of aliquot portion of the manganese standard reference solution and the saturated sodium pyrophosphate solution has been reduced, and the specification of the beaker has been changed;
- in 5.15 (previously 5.11.2), the amount of aliquot portion of the potassium permanganate solution and the saturated sodium pyrophosphate solution has been reduced, and the specification of the beaker has been changed;
- in Clause 6, the description of pH meter has been replaced with a list of potentiometric titration apparatus in line with the current status of the development of the instrument, including the apparatus for potentiometric titration in 6.1, pH determination in 6.2 and titration assembly in 6.3;
- in Clause 8, a new paragraph of the definition of test portion has been added in 8.1 and the amount of test portion in 8.1 (previously 8.2) has been reduced from 1,0 g to 0,50 g;
- in Clause 8, a new paragraph of the definition and requirement of determination of hygroscopic moisture content has been added in 8.3;

- in 8.4 (previously 8.2), the amount of acids has been reduced, the times of washing have been changed, and the detailed usage of watch-glass and the provision of filter volume have been added;
- in 8.6 (previously 8.4), the amount of aliquot portion of the solution and the saturated sodium pyrophosphate solution has been reduced, and the specification of one-mark volumetric flask and the beaker have been changed;
- in 9.1, the formula of the manganese content has been replaced with three new calculating formulae which take into account the hygroscopic moisture content of the test portion;
- in 9.2, the paragraph of the "permissible tolerances on results" has been replaced with the "general treatment of results";
- Clause 10, the test report has been added;
- Annex A, "Additional information on the international interlaboratory test" has been added;
- Annex B, "Flow sheet of the procedure for the acceptance of teat results" has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Manganese ores and concentrates — Determination of manganese content — Potentiometric method

1 Scope

This document specifies a potentiometric method for the determination of the manganese content of manganese ores and concentrates with manganese content equal to or greater than a mass fraction of 15 %.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 310, *Manganese ores and concentrates — Determination of hygroscopic moisture content in analytical samples — Gravimetric method*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4296-1, *Manganese ores — Sampling — Part 1: Increment sampling*

ISO 4296-2, *Manganese ores — Sampling — Part 2: Preparation of samples*

ISO 80000-1:2009, *Quantities and units — Part 1: General*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The method is based on the titration of manganous ion with permanganate ion in neutral pyrophosphate solution, the manganese(II) being oxidized, the permanganate ion reduced, to a pyrophosphate complex of the +3 state (the stoichiometry ratio of Mn^{2+} to MnO_4^- is 4:1).

Decomposition of a test portion by treatment with hydrochloric acid, nitric, perchloric and hydrofluoric acids. Separation of insoluble residue, and reservation of the filtrate as the main solution. Ignition of the residue, fusion with sodium carbonate, leaching of the melt with hydrochloric acid and combination with the main solution. Addition of an aliquot portion of the resulting solution to sodium pyrophosphate solution, adjustment of the pH to 7,0, and potentiometric titration with potassium permanganate standard volumetric solution.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 3 water as specified in ISO 3696.

5.1 Sodium carbonate (Na_2CO_3), anhydrous.

5.2 Hydrochloric acid, ρ = 1,19 g/ml.

5.3 Hydrofluoric acid, ρ = 1,14 g/ml.

5.4 Perchloric acid, ρ = 1,51 g/ml.

5.5 Nitric acid, ρ = 1,40 g/ml.

5.6 Potassium permanganate, degree of purity not less than 99,5 %.

5.7 Sodium carbonate, 50 g/l solution.

5.8 Hydrochloric acid, diluted 1 + 4.

5.9 Saturated sodium pyrophosphate solution. Dissolve 120 g of sodium pyrophosphate decahydrate ($Na_4P_2O_7{\cdot}10H_2O$) with 1 000 ml of water. After 24 h, the supernatant is obtained for use.

5.10 Potassium-permanganate ($KMnO_4$), recrystallized. Dissolve 250 g of potassium permanganate (5.6) with 800 ml of hot water (90 °C) in a 1 000 ml beaker. Filter the solution under vacuum through a filter crucible with a sintered glass plate No.3 (6.5). Cool the filtered solution in an ice bath to 10 °C, while stirring vigorously. Allow the fine-grained precipitate to settle. Then pour out the solution, transfer the crystalline mass to the crucible with sintered glass plate No.3 (6.5) and place under suction. Repeat the recrystallization.

After thorough suction, transfer the crystalline mass thus obtained to a glass or porcelain dish and dry in air in the dark, protecting from dust. When the crystalline mass no longer sticks together when crushed with a glass rod, dry it at 80 °C to 100 °C for 2 h to 3 h in a drying oven. Then transfer it to a stoppered brown glass bottle.

Potassium permanganate thus obtained contains 34,76 % (mass fraction) of manganese and is non-hygroscopic.

5.11 Bromothymol blue indicator, 0,4 g/l solution.

5.12 Manganese, standard reference solution, corresponding to 1 g of Mn per litre. Place 10,00 g of electrolytic manganese metal flakes (purity not less than 99,95 %) in a 400 ml or 500 ml beaker. Add a mixture of 50 ml of water and 5 ml of the nitric acid (5.5) and leave for a few minutes until the surface becomes bright. Wash the treated manganese six times with water, then with acetone, and dry at 100 °C for 10 min.

Place 1,00 g of the treated electrolytic manganese in a 400 ml or 500 ml beaker, and add 20 ml of sulfuric acid (ρ = 1,84 g/ ml, diluted 1 + 1) and about 100 ml of water. Boil the solution for a few minutes, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of Mn.

5.13 Potassium permanganate, standard volumetric solution, c (1/5 $KMnO_4$) ≈ 0,1 mol/l. Dissolve 3,20 g of potassium permanganate (5.6) in 1 000 ml of water, allow to stand for six days, then transfer to a brown glass bottle and mix.

Alternatively, commercially available standard solutions of known concentration can be used instead of standard solutions produced in the laboratory. Variations in concentration shall be taken into account for the calculation of the results.

5.14 Standardization with the manganese standard reference solution (5.12).

Transfer 50,00 ml of the manganese standard reference solution (5.12) into a 400 ml beaker and add, while stirring, 150 ml of the saturated sodium pyrophosphate solution (5.9). Adjust the pH of the solution to 7,0 with hydrochloric acid (5.8) [check the pH using either the pH electrode (6.2) or the bromothymol blue indicator (5.11)] and titrate with the potassium permanganate solution (5.13) using the potentiometric titration apparatus (6.1, 6.2, 6.3) until the maximum peak deflection on the voltmeter is observed.

Carry out a blank test, omitting the standard manganese solution.

The mass concentration of the potassium permanganate solution, ρ_1, is given by Formula (1):

$$\rho_1 = \frac{m_1}{V_1 - V_2} \tag{1}$$

where

m_1 is the mass, in grams, of manganese present in the aliquot portion of the manganese standard reference solution (5.12);

V_1 is the volume, in millilitres, of the potassium permanganate solution used for titration of the manganese standard reference solution;

V_2 is the volume, in millilitres, of the potassium permanganate solution used for titration of the blank test solution.

5.15 Standardization with the potassium permanganate (5.10).

Place 1,50 g of potassium permanganate (5.10) in a 250 ml or 300 ml fluoroplastic or polytetrafluoroethylene beaker, add 30 ml to 40 ml of water and mix thoroughly. Add 20 ml of hydrochloric acid (5.2), cover the beaker with a watch-glass and heat. When the reaction is complete, rinse the watch-glass, transferring the washings into the beaker. Add 10 ml of perchloric acid (5.4) and 20 ml of hydrofluoric acid (5.3) to the cooled solution and evaporate until dense white fumes of perchloric acid appear. Cool the solution, add 20 ml of hydrochloric acid (5.8) and heat until the pink colour of the solution is discharged.

After cooling, transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix. Take a 50,00 ml aliquot portion and transfer it with continuous stirring into a 400 ml beaker containing 150 ml of the saturated sodium pyrophosphate solution (5.9). Adjust the pH of the solution to 7,0 with hydrochloric acid (5.8) or the sodium carbonate solution (5.7) [check the pH using either the pH electrode (6.2) or the bromothymol blue indicator (5.11)] and titrate with the potassium permanganate solution (5.13) using the potentiometric titration apparatus (6.1, 6.2, 6.3) until the maximum peak deflection on the voltmeter is observed.

Carry out a blank test, omitting the solid potassium permanganate.

The mass concentration of the potassium permanganate solution, ρ_2, is given by Formula (2):

$$\rho_2 = \frac{m_2 \times 0,347\,6}{V_3 - V_4} \tag{2}$$

where

m_2 is the mass, in grams, of the potassium permanganate (5.10) present in the aliquot portion of solution taken for titration;

V_3 is the volume, in millilitres, of the potassium permanganate solution used for titration of manganese;

V_4 is the volume, in millilitres, of the potassium permanganate solution used for titration of the blank test solution;

0,347 6 is the conversion factor from potassium permanganate to manganese.

6 Apparatus

All volumetric glassware shall be Class A, in accordance with ISO 648 and ISO 1042. Ordinary laboratory apparatus and the following shall be used.

6.1 Apparatus for potentiometric titration: Indicator electrode (of platinum) with reference electrode (of tungsten, platinum, calomel or silver/silver chloride) or combined electrode of equivalent performance.

6.2 Apparatus for pH determination: Glass electrode and reference electrode, or combined electrode of equivalent performance.

6.3 Titration assembly, consisting of a 400 ml beaker, two 20 ml or 25 ml dosing units and a magnetic stirrer.

6.4 Platinum crucible

6.5 Filter crucible with a sintered glass plate No.3.

7 Sampling

The sampling of manganese ores shall be in accordance with ISO 4296-1. The preparation of samples shall be in accordance with ISO 4296-2.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,000 1 g, approximately 0,50 g of the test sample.

8.2 Blank test

Carry out a blank test in parallel with the determination.

8.3 Determination of hygroscopic moisture content

Determine the hygroscopic moisture content in accordance with ISO 310, simultaneously with the taking of the test portion (8.1) for the determination of manganese content.

8.4 Decomposition of test portion

Place the test portion (8.1) in a 250 ml or 300 ml fluoroplastic or polytetrafluoroethylene beaker, moisten with a few drops of water, add 10 ml of hydrochloric acid (5.2) and 2 ml of nitric acid (5.5), cover the beaker with a watch-glass and heat until nitrous fumes cease to be evolved, and cool. Add 5 ml of perchloric acid (5.4) and heat, gently at first, and then strongly to decompose the ore. Cool the solution, rinse the watch-glass, transferring the washings into the beaker. Add 5 ml of hydrofluoric acid (5.3) and evaporate until dense white fumes of perchloric acid appear. Cool the solution, add 10 ml of hydrochloric acid (5.8) and heat until the salts dissolve. Filter the insoluble residue on a medium texture filter containing a small amount of paper pulp, and wash with warm water 8 to 10 times. The filter volume should be no more than 120 ml. Reserve the filtrate (A).

8.5 Treatment of residue

Place the filter paper containing the residue in a platinum crucible (6.4), dry, char the paper at 600 °C to 700 °C and finally fuse with 2 g of sodium carbonate (5.1) at 900 °C to 1 000 °C. Cool the crucible containing the melt, place in a 250 ml beaker, add 10 ml of hydrochloric acid (5.2) and 30 ml to 40 ml of water, and heat to dissolve the melt. Remove the crucible and rinse it into the beaker with water. Cool the solution and add it to the filtrate (A).

8.6 Titration

Transfer the combined solution into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

Take a 50,00 ml aliquot portion of the solution and transfer it with continuous stirring into a 400 ml beaker containing 150 ml of the saturated sodium pyrophosphate solution (5.9).

The solution obtained should be clear. If a residue is formed, it is necessary to take either a small aliquot portion or a greater amount of the saturated sodium pyrophosphate solution (5.9).

Adjust the pH of the solution to 7,0 with hydrochloric acid (5.8) or the sodium carbonate solution (5.7) [check the pH using either the pH electrode (6.2) or the bromothymol blue indicator (5.11)] and titrate with the potassium permanganate solution (5.13) using the potentiometric titration apparatus (6.1, 6.2, 6.3) until the maximum peak deflection on the voltmeter is observed.

9 Expression of results

9.1 Calculation of manganese content

The manganese (Mn) content, expressed as a percentage mass fraction, ω_{Mn} (%), is given by Formula (3), (4) or (5):

$$\omega_{\mathrm{Mn}} = \frac{\rho_1 \times (V_5 - V_6) \times 100}{m_3 \times \gamma} \times \frac{100}{100 - K} \tag{3}$$

$$\omega_{\mathrm{Mn}} = \frac{\rho_2 \times (V_5 - V_6) \times 100}{m_3 \times \gamma} \times \frac{100}{100 - K} \tag{4}$$

$$\omega_{\mathrm{Mn}} = \frac{\rho_3 \times (V_5 - V_6) \times 4 \times 54{,}938 \times 100}{m_3 \times \gamma \times 1000} \times \frac{100}{100 - K} \tag{5}$$

where

ρ_1 is the concentration obtained by Formula (1), expressed in grams of manganese per millilitre, of the potassium permanganate solution (5.13);

ρ_2 is the concentration obtained by Formula (2), expressed in grams of manganese per millilitre, of the potassium permanganate solution (5.13);

ρ_3 is the certified value of amount-of-substance concentration of the commercially available potassium permanganate solution (5.13) in the certificate accompanying the certified reference material, $c\,(\mathrm{KMnO_4}) = 1/5\ c\,(1/5\mathrm{KMnO_4})$, in moles per litre;

4 is the stoichiometry ratio of Mn^{2+} to $\mathrm{MnO_4}^{-}$;

54,938 is the molecular weight, in grams per mole, of manganese;

V_5 is the volume, in millilitres, of the potassium permanganate solution (5.13) used for the titration of the aliquot portion of the test solution;

V_6 is the volume, in millilitres, of the potassium permanganate solution (5.13) used for the titration of the blank test solution;

m_3 is the mass, in grams, of the test portion of ore;

γ is the volume ratio of moving solution;

K is the hygroscopic moisture content of the test portion, as a mass fraction, determined in accordance with ISO 310.

9.2 General treatment of results

9.2.1 Expression of precision

A planned trial of this method was carried out by 13 laboratories at 10 levels of manganese content, each laboratory making three determinations for each level.

The results obtained were treated statistically in accordance with ISO 5725-2[1].

The relationship between manganese content and repeatability limit and reproducibility limit of the test results are summarized in Table 1. The detailed results for manganese content obtained from the international interlaboratory test and a graphical representation of the precision data are shown in Annex A. The data show a linear relationship between manganese content and repeatability, but the linear relationship between manganese content and reproducibility is not significant. Therefore, the precision measures can be expressed by the average for reproducibility.

Table 1 — Repeatability limit and reproducibility limit

Manganese content % (mass fraction)	Repeatability limit r	Reproducibility limit R
14,45	0,12	0,48
15,74	0,18	0,43
22,93	0,21	0,42
25,00	0,23	0,50
30,99	0,17	0,28
34,67	0,21	0,54
38,81	0,26	0,39
45,39	0,22	0,38
49,17	0,26	0,38
58,88	0,29	0,56

The difference between two single and independent results found on identical test material by one analyst using the same apparatus within a short time interval in the normal and correct operation of the test method will exceed the repeatability, *r*, on average, in only one case in 20.

The difference between two single and independent results found by two operators working in different laboratories on identical test material in the normal and correct operation of the test method will exceed the reproducibility, *R*, on average, in only one case in 20.

9.2.2 Determination of analytical result

Having computed the independent duplicate results according to Formula (3), (4) or (5), compare them with the repeatability limit, *r*, using the procedure given in Annex B, and obtain the final laboratory result.

9.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the procedure described in 9.2.2. Compute the following quantity using Formula (6):

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \tag{6}$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of the final results.

If $|\mu_1 - \mu_2| \le \mathrm{R}$, the final results are in agreement.

9.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM). Calculate the analytical result, μ_c, for the CRM/ RM, and compare it with the reference or certified value A_c. There are two possibilities:

a) $|\mu_c - A_c| \le C$, in which case the difference between the reported result and the certified/reference value is statistically insignificant;

b) $|\mu_c - A_c| > C$, in which case the difference between the reported result and the certified/reference value is statistically significant;

where

μ_c is the final result for the certified reference material;

A_c is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used.

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.[2] C shall be calculated using Formula (7):

$$C = \frac{1}{\sqrt{2}}\sqrt{R^2 - \frac{n-1}{n}r^2 + 8u^2} \tag{7}$$

where

R is the between-laboratories reproducibility;

r is the within-laboratory repeatability;

n is the number of replicate determinations carried out on the CRM/RM;

u is the uncertainty of certified values of CRM/RM.

9.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operation specified in Annex B, calculated to four decimal places and rounded off to the second decimal place as follows:

a) where the figure in the third decimal place is less than 5, it shall be discarded and the figure in the second decimal place is kept unchanged, in accordance with ISO 80000-1:2009, B.3, Rule A;

b) where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;

c) where the figure in the third decimal place is 5 and there is a Figure 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

10 Test report

The test report shall include the following information:

a) the name and address of the testing laboratory;

b) the date of issue of the test report;

c) a reference to this document, i.e. ISO 4298;

d) the details necessary for the identification of the sample;

e) the results and unit in which they are expressed;

f) any characteristics noticed during the determination and any operations not specified in this document which may have had an influence on the result, for either the test sample or the certified reference material(s).

Annex A
(informative)

Additional information on the international interlaboratory test

Detailed results for manganese content obtained from the international interlaboratory test are shown in Table A.1.

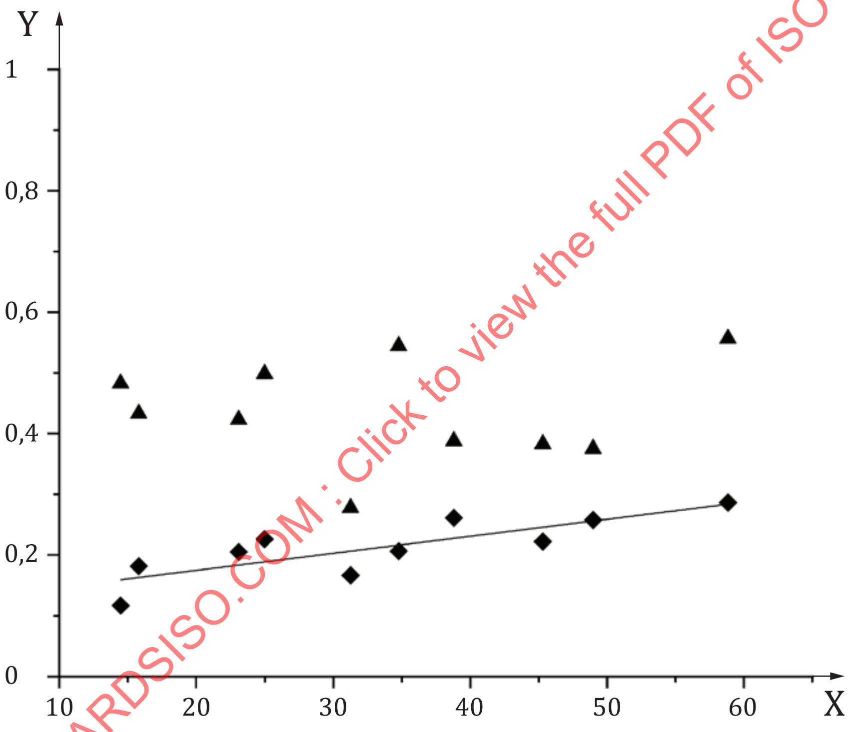
The precision data are presented in graphical form in Figure A.1.

Table A.1 — Detailed results obtained in the international cooperative test

Lab	Manganese content, S, % (mass fraction)									
	S-1	S-2	S-3	S-4	S-5	S-6	S-7	S-8	S-9	S-10
1	14,491	15,570	22,916	24,986	31,023	34,696	38,898	45,254	49,101	58,857
	14,426	15,638	22,921	24,836	31,221	34,600	38,859	45,104	49,201	58,870
	14,487	15,779	22,939	24,801	31,155	34,555	38,918	45,350	49,150	58,808
2	14,290	15,645	23,122	24,579	31,392	34,382	38,594	45,137	48,901	59,089
	14,344	15,739	23,309	24,765	31,371	34,389	38,814	45,321	48,920	58,729
	14,339	15,691	23,230	24,716	31,389	34,557	38,575	45,385	48,923	58,852
3	14,669	15,971	23,261	25,131	31,253	34,916	38,972	45,093	48,883	58,908
	14,708	16,036	23,115	25,215	31,339	34,791	38,942	44,981	49,077	58,757
	14,741	15,936	23,212	25,303	31,213	34,997	39,110	45,203	49,123	59,008
4	14,312	15,442	22,852	24,634	31,292	34,815	38,461	45,064	49,223	58,397
	14,238	15,678	22,778	24,889	31,339	34,923	38,691	45,229	48,954	58,544
	14,223	15,482	22,993	24,934	31,489	34,944	38,662	45,174	48,913	58,622
5	14,152	16,015	22,967	25,186	31,124	34,599	38,799	45,422	49,175	59,322
	14,139	15,944	23,005	25,199	31,217	34,704	38,717	45,334	48,953	59,191
	14,253	16,098	23,186	25,102	31,250	34,642	38,821	45,394	49,079	59,139
6	14,372	15,540	22,924	24,881	31,112	34,696	38,843	45,254	49,001	59,170
	14,349	15,543	22,881	24,686	31,101	34,603	39,095	45,104	49,109	58,899
	14,244	15,659	22,905	24,658	31,280	34,554	38,998	45,350	49,289	58,862
7	14,788	15,839	23,288	25,122	31,272	34,837	38,744	45,356	48,969	58,918
	14,751	15,918	23,319	25,144	31,247	34,945	38,774	45,313	48,921	58,858
	14,709	15,815	23,373	25,206	31,226	35,056	38,956	45,322	48,886	58,884
8	14,483	15,772	23,082	24,908	31,273	34,827	38,834	45,326	48,762	58,734
	14,452	15,802	23,130	24,943	31,309	34,793	38,866	45,355	48,803	58,701
	14,496	15,798	23,193	25,032	31,206	34,845	38,807	45,382	48,734	58,772
9	14,463	15,763	22,935	25,055	31,187	34,585	39,037	45,465	48,967	58,903
	14,501	15,814	23,110	25,091	31,129	34,688	38,731	45,511	48,954	58,998
	14,521	15,774	23,142	25,068	31,156	34,668	38,825	45,449	48,969	58,706
10	14,637	15,904	23,115	25,052	31,322	35,023	38,890	45,294	48,879	58,830
	14,610	15,900	23,184	25,092	31,294	35,059	38,757	45,353	48,948	58,621
	14,589	15,872	23,074	25,005	31,300	34,949	38,860	45,374	48,987	58,700

Table A.1 *(continued)*

Lab	**Manganese content, S**, % (mass fraction)									
	S-1	S-2	S-3	S-4	S-5	S-6	S-7	S-8	S-9	S-10
11	14,564	15,893	23,228	25,063	31,354	34,819	38,689	45,421	49,016	58,670
	14,535	15,830	23,196	25,054	31,334	35,005	38,793	45,430	48,860	58,699
	14,444	15,871	23,175	25,030	31,346	34,886	38,839	45,374	49,015	58,682
12	14,525	15,869	23,082	24,971	31,234	34,619	38,706	45,051	48,707	58,605
	14,568	15,745	22,974	24,951	31,272	34,532	38,706	45,213	48,936	58,708
	14,541	15,880	22,931	25,093	31,315	34,553	38,724	45,110	48,819	58,630
13	14,570	15,906	23,148	25,091	31,400	34,999	38,788	45,434	48,887	58,700
	14,593	15,836	23,231	25,096	31,351	34,927	38,711	45,461	48,911	58,692
	14,553	15,889	23,221	25,060	31,373	35,014	38,702	45,387	48,974	58,718



Key

◆	*r* (%)
▲	*R* (%)
——	*r*
X	manganese content, % (mass fraction)
Y	precision, % (mass fraction)
r	$= 0{,}002\,804\, m + 0{,}118\,8$
R	$= 0{,}44$

where *m* is the average mass fraction of manganese obtained from three determinations in each laboratory.

Figure A.1 — Relationship between manganese content and repeatability limit and reproducibility limit