#### BS EN 15703-2:2014



## **BSI Standards Publication**

# Copper and copper alloys — Determination of manganese content

Part 2: Flame atomic absorption spectrometric method (FAAS)



BS EN 15703-2:2014 BRITISH STANDARD

#### National foreword

This British Standard is the UK implementation of EN 15703-2:2014.

The UK participation in its preparation was entrusted to Technical Committee NFE/34/1, Wrought and unwrought copper and copper alloys.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Copper and copper alloys - Determination of manganese content - Part 2: Flame atomic absorption spectrometric method (FAAS)

Cuivre et alliages de cuivre - Détermination de manganèse - Partie 2: Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des Mangangehaltes - Teil 2: Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

This European Standard was approved by CEN on 8 November 2014.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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#### **Foreword**

This document (EN 15703-2:2014) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to prepare the following standard:

EN 15703-2, Copper and copper alloys — Determination of manganese content — Part 2: Flame atomic absorption spectrometric method (FAAS)

This is one of two Parts of the standard/Technical Specification for the determination of manganese content in copper and copper alloys. The other Part is:

CEN/TS 15703-1, Copper and copper alloys — Determination of manganese content — Part 1: Spectrophotometric method

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

#### 1 Scope

This European Standard specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the manganese content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having manganese mass fractions between 0,001 0 % and 6,0 %.

#### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1811-1, Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products

ISO 1811-2, Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings

#### 3 Principle

Dissolution of a test portion in hydrochloric acid and nitric acid followed, after suitable dilution and the addition of lanthanum chloride to mask the effect of interfering ions, by aspiration into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 279,5 nm or 403,1 nm line emitted by a manganese hollow-cathode lamp.

#### 4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

- **4.1 Hydrochloric acid**, HCl ( $\rho$  = 1,19 g/ml)
- 4.2 Nitric acid, HNO<sub>3</sub> ( $\rho$  = 1,40 g/ml)
- **4.3 Hydrofluoric acid**, HF ( $\rho$  = 1,13 g/ml)

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

#### 4.4 Nitric acid solution, 1 + 1

Add 500 ml of nitric acid (4.2) to 500 ml of water.

#### 4.5 Lanthanum(III) chloride solution, 100 g/l

Dissolve 100 g of lanthanum(III) chloride ( $LaCl_3 \cdot 7H_2O$ ) in a 600 ml beaker with water and transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

#### 4.6 Sulphuric acid, $H_2SO_4$ ( $\rho = 1.84$ g/ml)

#### 4.7 Sulphuric acid solution, 1 + 9

Add 20 ml of sulphuric acid (4.6) in 180 ml water.

#### 4.8 Manganese stock solution, 1,0 g/l Mn

#### 4.8.1 Cleaning of manganese metal

Transfer several grams of electrolytic manganese (purity > 99,9 %) into a 250 ml beaker containing about 150 ml of sulphuric acid solution (4.7). Stir, then allow the manganese to settle for several minutes. Decant and discard the sulphuric acid solution. Rinse several times with water and finally with acetone. Dry the metal for about 2 min at 100°C and cool in a desiccator.

#### 4.8.2 Preparation of stock solution

Weigh,  $(1 \pm 0,001)$  g, of manganese cleaned as in 4.8.1 and transfer it into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.1) and 20 ml of the nitric acid solution (4.4). Cover with a watch glass and, if necessary, heat gently to assist dissolution. When dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1,0 mg of Mn.

#### 4.9 Manganese standard solution, 0,10 g/l

Transfer 20,0 ml of manganese stock solution (4.8) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,10 mg of Mn.

#### 4.10 Manganese standard solution, 0,010 g/l

Transfer 5,0 ml of manganese stock solution (4.8) to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,010 mg of Mn.

#### 4.11 Copper matrix solution, 20 g/l Cu

Transfer  $(10 \pm 0,01)$  g of manganese-free copper  $(Cu \ge 99,95 \%)$  into a 600 ml beaker. Add 100 ml of hydrochloric acid (4.1) and, cautiously, 100 ml of the nitric acid solution (4.4). Cover with a watch glass and heat gently until the copper has been completely dissolved, then heat up to the boiling point until the nitrous fumes have been expelled. Allow to cool and transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

#### 4.12 Copper matrix solution, 2 g/l Cu

Transfer 50,0 ml of the copper matrix solution (4.11) into a 500 ml one-mark volumetric flask. Add 90 ml of hydrochloric acid (4.1) and 90 ml of the nitric acid solution (4.4). Dilute to the mark with water and mix well.

#### 5 Apparatus

#### **5.1 Atomic absorption spectrometer**, fitted with an air/acetylene burner

#### 5.2 Manganese hollow-cathode lamp

#### 6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

Test samples shall be in the form of fine drillings, chips or millings with a maximum thickness of 0,5 mm.

#### 7 Procedure

#### 7.1 Preparation of the test portion solution

#### 7.1.1 Test portion

Weigh  $(1 \pm 0,001)$  g of the test sample.

#### 7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.1) and 10 ml of the nitric acid solution (4.4). Cover with a watch glass and heat gently until the test portion is completely dissolved. Allow to cool. If undissolved matter remains, indicating the presence of silicon, filter the solution. Place the filter and retained residues in a platinum crucible and ash, taking care that the filter does not flame. Calcine at about 550 °C. Allow to cool and add 5 ml of hydrofluoric acid (4.3) and five drops of nitric acid (4.2). Evaporate to dryness and calcine again for several minutes at about 700 °C to completely volatilize the silicon. Cool, and then dissolve the residue with the least possible volume of nitric acid solution (4.4). Filter, if necessary, and add this filtrate quantitatively to the original filtrate.

#### 7.1.3 Manganese mass fractions between 0,001 % and 0,020 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Add 10 ml of the lanthanum(III) chloride solution (4.5), dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions between 0,015 % and 0,020 % results are better when the determination is carried out following 7.1.3.

#### 7.1.4 Manganese mass fractions between 0.020 % and 0.50 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 5,0 ml of this solution into a 100 ml one-mark volumetric flask. Add 4,5 ml of hydrochloric acid (4.1), 4,5 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

#### 7.1.5 Manganese mass fractions between 0,50 % and 2,0 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 10,0 ml of this solution into a 100 ml one-mark volumetric flask. Add 9 ml of hydrochloric acid (4.1), 9 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions near 0,5 % results are better when the determination is carried out following 7.1.5.

#### 7.1.6 Manganese mass fractions between 2,0 % and 6,0 %

Transfer the dissolved test portion or the combined filtrates quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Transfer 2,5 ml of this solution into a 100 ml one-mark

volumetric flask. Add 2,3 ml of hydrochloric acid (4.1), 2,3 ml of the nitric acid solution (4.4) and 10 ml of the lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

NOTE The validation exercise of this method showed that for manganese mass fractions near 2,0 % results are better when the determination is carried out following 7.1.6.

#### 7.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents as used for the determination, but substituting pure copper for the test portion.

#### 7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of a reference material or a synthetic sample containing a known amount of manganese and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

#### 7.4 Establishment of the calibration curve

#### 7.4.1 Preparation of the calibration solutions

#### 7.4.1.1 **General**

In all cases, copper, chloride and nitrate concentrations, and acidity in the calibration solutions shall be similar to those of the test portion solutions.

The presence of copper in the calibration solutions compensates for chemical interaction effects of copper in the test portion solution. Normally no similar additions are required to compensate for the effect of alloying elements. If an alloying element is present in the material to be analysed in mass fraction > 10 %, an appropriate mass of this element shall be added to the calibration solutions. The volumes of copper matrix solution added (4.11 or 4.12) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Overcompensation may occur if the same volumes are added when the test samples are copper-based alloys where the percentage of copper is lower. In these cases the volumes of copper matrix solution shall be decreased to match the copper content of the test sample in solution.

The manganese concentration of the calibration solutions shall be adjusted to suit the sensitivity of the spectrometer used, so that the curve of absorbance as a function of concentration is a straight line.

#### 7.4.1.2 Manganese mass fractions between 0,001 0 % and 0,020 %

Into each of a series of seven 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.10) and copper matrix solution (4.11) shown in Table 1. Introduce also 10 ml of lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

Table 1 — Calibration for manganese mass fractions between 0,001 0 % and 0,020 %

Manganese standard solution volume (4.10)	Corresponding manganese mass	Corresponding manganese concentration after final dilution	Copper matrix solution volume (4.11)	Corresponding copper mass	Corresponding manganese mass fraction of sample
ml	mg	μg/ml	ml	g	%
<sub>0</sub> a	0	0	50	1,000	0
1	0,010	0,10	50	1,000	0,001 0
3	0,030	0,30	50	1,000	0,003 0
6	0,060	0,60	50	1,000	0,006 0
10	0,10	1,0	50	1,000	0,010
15	0,150	1,5	50	1,000	0,015 0
20	0,20	2,0	50	1,000	0,020
a Blank test on reagents for calibration.					

<sup>7.4.1.3</sup> Manganese mass fractions between 0,020 % and 0,5 %

Into each of a series of eight 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.10) and copper matrix solution (4.12) shown in Table 2. Introduce also 10 ml lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

Table 2 — Calibration for manganese mass fractions between 0,020 % and 0,5 %

Manganese standard solution volume (4.10)	Corresponding manganese mass	Corresponding manganese concentration after final dilution	Copper matrix solution volume (4.12)	Corresponding copper mass	Corresponding manganese mass fraction of sample
ml	mg	μg/ml	ml	g	%
<sub>0</sub> a	0	0	25	0,05	0
1	0,01	0,1	25	0,05	0,02
3	0,03	0,3	25	0,05	0,06
6	0,06	0,6	25	0,05	0,12
10	0,10	1,0	25	0,05	0,20
15	0,15	1,5	25	0,05	0,30
20	0,20	2,0	25	0,05	0,40
25	0,25	2,5	25	0,05	0,50
a Blank test on reagents for calibration.					

#### 7.4.1.4 Manganese mass fractions between 0,5 % and 2,0 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.9) and copper matrix solution (4.12) as shown in Table 3. Introduce also 10 ml lanthanum(III) chloride solution (4.5). Dilute to the mark with water and mix well.

Table 3 — Calibration for manganese mass fractions between 0,5 °	5 % and 2,0 %
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Manganese standard solution volume (4.9)	Corresponding manganese mass	Corresponding manganese concentration after final dilution	Copper matrix solution volume (4.12)	Corresponding copper mass	Corresponding manganese mass fraction of sample
ml	mg	μg/ml	ml	g	%
<sub>0</sub> a	0	0	50	0,1	0
5	0,5	5	50	0,1	0,5
7,5	0,75	7,5	50	0,1	0,75
10	1,0	10	50	0,1	1,0
15	1,5	15	50	0,1	1,5
20	2,0	20	50	0,1	2,0
a Blank test on reagents for calibration curve.					

#### 7.4.1.5 Manganese mass fractions between 2,0 % and 6,0 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.9) and copper matrix solution (4.12) as shown in Table 4. Introduce also 10 ml lanthanum(III) chloride solution (4.5) Dilute to the mark with water and mix well.

Table 4 — Calibration for manganese mass fractions between 2,0 % and 6,0 %

Manganese standard solution volume (4.9)	Corresponding manganese mass	Corresponding manganese concentration after final dilution	Copper matrix solution volume (4.12)	Corresponding copper mass	Corresponding manganese mass fraction of sample
ml	mg	μg/ml	ml	g	%
<sub>0</sub> a	0	0	12,5	0,025	0
5	0,5	5	12,5	0,025	2,0
7,5	0,75	7,5	12,5	0,025	3,0
10	1,0	10	12,5	0,025	4,0
12,5	1,25	12,5	12,5	0,025	5,0
15	1,5	15	12,5	0,025	6,0
a Blank test on reagents for calibration.					

#### 7.4.2 Adjustment of the atomic absorption spectrometer

Fit the manganese hollow-cathode lamp (5.2) into the atomic absorption spectrometer (5.1), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 279,5 nm for manganese mass fractions up to 0,5 % (Tables 1 and 2) or 403,1 nm for manganese mass fractions over 0,5 % (Tables 3 and 4) to minimum absorbance.

Following manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of

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acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

#### 7.4.3 Spectrometric measurement of the calibration solutions

Aspirate the relevant series of calibration solutions (7.4.1.2, 7.4.1.3, 7.4.1.4, 7.4.1.5 depending on the expected manganese content) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray water through the burner after each measurement (see note).

NOTE For certain types of apparatus, instead of water it is preferable to use a solution containing the attack reagents, in the same concentrations as in the test portion solutions.

#### 7.4.4 Calibration curve

Establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or an off-line computer for regression calculations or prepare a graphical representation.

#### 7.5 Determination

#### 7.5.1 General

The analyses shall be carried out independently, in duplicate.

#### 7.5.2 Preliminary spectrometric measurement

Carry out a preliminary measurement on the test portion solution (7.1.3, 7.1.4, 7.1.5 or 7.1.6) following the procedure specified in 7.4.2 and 7.4.3 at the same time as the spectrometric measurements are carried out on the calibration solutions (see 7.4.1) and using the appropriate wavelength, either 279,5 nm or 403,1 nm. Estimate the preliminary analyte amount by using the calibration curve (7.4.4).

#### 7.5.3 Spectrometric measurements

#### 7.5.3.1 Use of the calibration curve

Repeat the measurements and determine the concentration directly using the calibration curve.

#### 7.5.3.2 Use of bracketing method

Carry out a second measurement on the test portion solution (7.1.3, 7.1.4, 7.1.5 or 7.1.6) following the procedure specified in 7.4.3, by bracketing between two new calibration solutions with composition similar to that of the calibration solutions (see 7.4.1), but having manganese contents slightly higher and slightly lower (± 10 %) than the estimated manganese concentration of the test portion solution.

To prepare these calibration solutions, follow the procedure specified in 7.4.1 using, however, suitable quantities of manganese standard solutions (4.9 or 4.10).

#### 8 Expression of results

#### 8.1 Use of the calibration curve

Calculate the manganese mass fraction, in per cent (%), according to Equation (1):

$$w_{\mathsf{Mn}} = \frac{c_1}{m} \times V_{\mathsf{f}} \times D_{\mathsf{f}} \times 100 \tag{1}$$

#### where

- $w_{Mn}$  is the manganese mass fraction in percent (%);
- $c_1$  is the manganese concentration from the calibration curve, in microgram per millilitre ( $\mu$ g/ml);
- m is the sample mass of the test portion, in micrograms ( $\mu$ g);
- $V_{\rm f}$  is the volume of the test portion solution (7.1.3, 7.1.4, 7.1.5 or 7.1.6), in millilitres (ml);
- $D_{r}$  is the dilution ratio.
- for 7.1.4,  $D_r = 20$ ;
- for 7.1.5,  $D_r = 10$ ;
- for 7.1.6,  $D_r = 40$ .

#### 8.2 Use of the bracketing method

Calculate the manganese mass fraction, in per cent (%), according to Equation (2):

$$w_{\mathsf{Mn}} = \frac{c_2}{m} \times V_{\mathsf{f}} \times D_{\mathsf{f}} \times 100 \tag{2}$$

#### where

- $c_2$  is the manganese concentration, calculated according to Equation (3), in micrograms per millilitre ( $\mu$ g/ml);
- m is the sample mass of the test portion, in micrograms ( $\mu$ g);
- $V_{\rm f}$  is the volume of the test portion solution (7.1.3, 7.1.4, 7.1,5 or 7.1.6), in millilitres (ml);
- $D_{\perp}$  is the dilution ratio.
- for 7.1.4,  $D_r = 20$ ;
- for 7.1.5,  $D_r = 10$ ;
- for 7.1.6,  $D_r = 40$ .

$$c_2 = C_1 + (C_2 - C_1) \times \frac{S_0 - S_1}{S_2 - S_1}$$
(3)

#### where

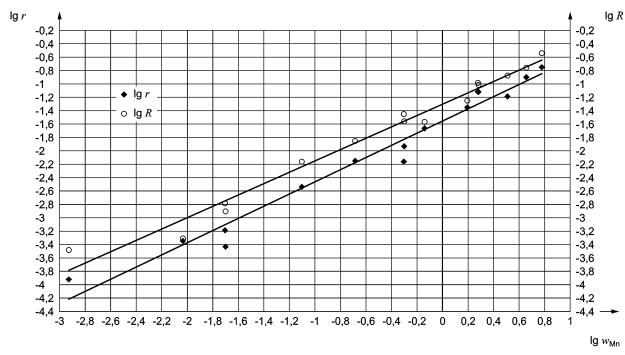
- $C_1$  is the lower manganese concentration of the calibration solution used, in micrograms per millilitre ( $\mu$ g/ml);
- $C_2$  is the higher manganese concentration of the calibration solution used, in micrograms per millilitre ( $\mu$ g/ml);
- $S_0$  is the absorbance value of the test portion solution;
- $S_1$  is the absorbance value of the calibration solution corresponding to the concentration  $C_1$ ;
- $S_2$  is the absorbance value of the calibration solution corresponding to the concentration  $C_2$ .

#### 9 Precision

Five laboratories co-operated in validating this method and obtained the results summarized in Table 5 and Figure 1 respectively.

Table 5 — Statistical information

Level	Reference value %	Found %	Repeatability r	Reproducibility R
1 (7.1.3)	0,001 44	0,001 18	0,000 12	0,000 33
2 (7.1.3)	0,009 21	0,009 25	0,000 45	0,000 49
3 (7.1.3)	0,020 28	0,019 97	0,000 37	0,001 25
4 (7.1.4)	0,020 28	0,019 77	0,000 65	0,001 64
5 (7.1.4)	0,079 6	0,079 0	0,002 9	0,006 9
6 (7.1.4)	0,211 8	0,206 8	0,007 1	0,014 0
7 (7.1.4)	0,497 3	0,496 6	0,006 9	0,035 5
8 (7.1.5)	0,497 3	0,500 7	0,011 7	0,027 6
9 (7.1.5)	0,723	0,722 9	0,021 7	0,027 1
10 (7.1.5)	1,56	1,564 3	0,044 6	0,056 3
11 (7.1.5)	1,9	1,891 7	0,076 5	0,103 9
12 (7.1.6)	1,9	1,913 1	0,076 2	0,096 7
13 (7.1.6)	3,27	3,226 5	0,065 1	0,133 0
14 (7.1.6)	4,54	4,526 9	0,126 1	0,174 8
15 (7.1.6)	5,9	5,994 6	0,177 7	0,288 1



#### Key

 $\lg r = 0.909 \ 3 \ \lg w_{Mn} - 1.553$   $\lg R = 0.848 \ 1 \ \lg w_{Mn} - 1.302 \ 7$ 

Figure 1 — Ig relationship between manganese mass fraction  $w_{\mbox{Mn}}, r$  and R

#### 10 Test report

The test report shall contain the following information:

- a) identification of the test sample;
- b) reference to this European Standard (EN 15703-2);
- c) test method used;
- d) results;
- e) any unusual characteristics noted during the determination;
- f) any operation not included in this European Standard or in the document to which reference is made or regarded as optional;
- g) date of the test and/or date of preparation or signature of the test report;
- h) signature of the responsible person.

### **Bibliography**

- ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions
- ISO 5725-2, Accuracy (trueness and precision) of measurement methods and results Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method
- ISO 5725-3, Accuracy (trueness and precision) of measurement methods and results Part 3: Intermediate measures of the precision of a standard measurement method



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