

Chemical analysis of ferrous materials — Determination of phosphorus in steels and irons — Spectrophotometric method

The European Standard EN 10184:1989 has the status of a
British Standard

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This British Standard, having been prepared under the direction of the Iron and Steel Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 February 1992

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The following BSI references relate to the work on this standard:
Committee reference ISM/18
Draft for comment 84/38264 DC

ISBN 0 580 20578 9

Amendments issued since publication

Amd. No.	Date	Comments

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National foreword

This British Standard has been prepared under the direction of the Iron and Steel Standards Policy Committee and is the English language version of EN 10184:1989 “*Chemical analysis of ferrous materials — Determination of phosphorus in steels and irons — Spectrophotometric method*”, published by the European Committee for Standardization (CEN). It incorporates the Corrigendum EN 10184AC:1991. It supersedes BS 6200-3.24.1:1985, which is withdrawn.

A further method for the determination of phosphorus in ferrous materials will shortly be published as BS 6200-3.24.2. Full details of other British Standards relating to the chemical analysis of ferrous materials are given in BS 6200-3.0:1991.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Descriptors: Iron- and steel products, steels, cast iron, chemical analysis, determination of content, phosphorus, spectrophotometric analysis, phospho-vanado-molybdate, molecular absorption spectrophotometry.

English version

Chemical analysis of ferrous materials — Determination of phosphorus in steels and irons — Spectrophotometric method

Analyse chimique des matériaux
sidérurgiques — Dosage du phosphore dans les
aciers et fontes — Méthode
spectrophotométrique

Chemische Analyse von Eisenwerkstoffen —
Bestimmung von Phosphor in Stählen und
Eisen — Spektralphotometrisches Verfahren

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Brief history

This European Standard takes over the contents of EURONORM 184-87 "Chemical analysis of ferrous materials — Determination of phosphorus in steels and irons — Spectrophotometric method" prepared by ECISS/TC 20 "Methods of chemical analysis", the secretariat of which is allocated to the Dansk Standardiseringsraad (DS).

It was submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

According to the Common CEN/CENELEC Rules, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and United Kingdom.

Foreword

This European Standard specifies two methods for the spectrophotometric determination of phosphorus.

The first method is applicable to steels and irons and corresponds to International Standard ISO 2732:1984.

The second method is applicable to non-alloyed steels and irons.

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Method 1

Steel and cast iron — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method¹⁾

1 Scope and field of application

This European Standard specifies a spectrophotometric method for the determination of phosphorus in steel and cast iron.

The method is applicable to phosphorus contents between 0.005 and 1.5 % (m/m), provided that tungsten, niobium, tantalum and zirconium contents are not higher than 1 % (m/m) for each of these four elements and titanium content is not higher than 2 % (m/m).

2 Reference

Euronorm 18 — Selection and preparation of samples and test pieces for steel and iron and steel products.

3 Principle

Dissolution of a test portion in an oxidizing acid mixture.

Conversion of phosphorus to phosphovanadomolybdate in perchloric-nitric acid solution.

Extraction of phosphovanadomolybdate into 4-methyl-2-pentanone with citric acid present to complex arsenic.

Spectrophotometric measurement at a wavelength of about 425 nm.

4 Reagents

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

Verify by blank tests that the relevant reagents are free from phosphorus. Whenever necessary, the results shall be corrected accordingly. Grades giving high blank values are unsuitable and should be discarded.

4.1 Pure iron, containing 0.001 % (m/m) or less of phosphorus.

4.2 Hydrochloric acid, ρ about 1.19 g/ml.

4.3 Nitric acid, ρ about 1.40 g/ml.

4.4 Nitric acid, diluted 1 + 4.

4.5 Perchloric acid, ρ about 1.54 g/ml, with known low phosphorus content.

NOTE Perchloric acid (ρ about 1.67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1.54 g/ml) is equivalent to 79 ml of perchloric acid (ρ about 1.67 g/ml).

4.6 Citric acid, solution

Dissolve 500 g of citric acid monohydrate ($\text{H}_8\text{C}_6\text{O}_7 \cdot \text{H}_2\text{O}$) in water, dilute to 1 000 ml and mix.

4.7 4-Methyl-2-pentanone (isobutyl methyl ketone).

4.8 Hexaammonium heptamolybdate, solution.

Dissolve 15 g of hexaammonium heptamolybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in water, dilute to 100 ml and mix.

4.9 Ammonium metavanadate, solution.

Dissolve 2.5 g of ammonium metavanadate (NH_4VO_3) in water, dilute to 1 000 ml and mix.

4.10 Boron fluoride, solution.

Dissolve 40 g of boric acid (H_3BO_3) in 300 ml of water in a plastic beaker, add 100 ml of hydrofluoric acid ρ about 1.13 g/ml, dilute to 1 000 ml and mix.

Keep the solution in a plastic bottle.

4.11 Potassium permanganate, 10 g/l solution.

4.12 Sodium nitrite, 50 g/l solution.

¹⁾ The text of this European Standard corresponds with the text of ISO 2732:1984.

4.13 Phosphorus, standard solution.

4.13.1 Phosphorus, 0.1 g/l stock solution.

Weigh, to the nearest 0.0001 g, 0.4393 g of potassium dihydrogen orthophosphate (KH_2PO_4), previously dried to constant mass at 105 °C and cooled in a desiccator.

Dissolve in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0.1 mg of P.

4.13.2 Phosphorus, 0.01 g/l standard solution.

Transfer 100.0 ml of the stock solution (4.13.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

This solution should be prepared immediately before use.

1 ml of this solution contains 0.01 mg of P.

5 Apparatus

Ordinary laboratory apparatus and spectrophotometer.

6 Sampling

Carry out sampling in accordance with Euronorm 18.

7 Procedure

WARNING: Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

7.1 Test portion

Weigh, to the nearest 0.001 g, approximately 0.5 g (m) of the test sample.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents.

7.3 Determination

7.3.1 Preparation of test solution

7.3.1.1 For phosphorus content equal to or less than 0.08 % (m/m)

Introduce the test portion (7.1) into a 125 ml conical beaker. Add 5 ml of the nitric acid (4.3) and 5 ml of the hydrochloric acid (4.2), cover the beaker with a watch-glass and heat until solvent action ceases (see Note below).

Add 10 ml of the perchloric acid (4.5) and evaporate to fuming. Continue fuming for 5 to 10 minutes at such a temperature that a steady reflux of white perchloric acid fumes on the walls of the beaker is maintained (see 9.1).

Cool, add 25 ml of the nitric acid (4.4) and a few glass beads and then boil for 1 to 2 minutes (see 9.2).

Add 5 ml of the potassium permanganate solution (4.11), boil for 2 minutes, then add 10 ml of the sodium nitrite solution (4.12) and boil until free of nitrous fumes.

NOTE For highly alloyed samples which do not dissolve readily in this acid mixture, further 5 ml additions of the hydrochloric acid (4.2) may be made up to a maximum of 20 ml.

7.3.1.2 For phosphorus content greater than 0.08 % (m/m)

Carry out the same procedure as specified in the first paragraph of 7.3.1.1.

Dilute to about 100 ml with water, and filter if necessary to remove graphite.

Cool, transfer quantitatively into a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Take a suitable volume (V) of the solution, containing not more than 0.4 mg of phosphorus.

Proceed as specified in 7.3.1.1 from the second paragraph beginning at "Add 10 ml of the perchloric acid (4.5) ..." to the end of 7.3.1.1.

7.3.2 Colour development

Cool to about 20 °C.

Add 10.0 ml of the ammonium metavanadate solution (4.9) and 15.0 ml of the hexaammonium heptamolybdate solution (4.8) and then allow to stand at a temperature between 18 and 25 °C for a minimum of 7 minutes.

Transfer the solution to a 250 ml separating funnel marked at 100 ml, dilute to the mark with water and mix. Add 10 ml of the citric acid solution (4.6), mix and immediately add 40.0 ml of the 4-methyl-2-pentanone (4.7), and shake the funnel for 30 seconds.

Allow the two layers to separate and discard the lower (aqueous) layer.

Dry the inside of the stem of the separating funnel with a small piece of filter paper. Filter the 4-methyl-2-pentanone layer through a dry rapid paper into a small dry beaker.

7.3.3 Spectrophotometric measurement

Carry out the spectrophotometric measurements at 20 ± 1 °C in cells of 2 cm optical path at a wavelength of about 425 nm after having adjusted the spectrophotometer (Clause 5) to zero against the 4-methyl-2-pentanone (4.7).

Correct the absorbance with the blank test solution.

7.4 Establishment of the calibration graph

7.4.1 Preparation of calibration solutions

Introduce into a series of nine 125 ml conical beakers 0.5 g of the pure iron (4.1) and then respectively the volumes of the phosphorus standard solution (4.13.2) indicated in the following table:

Volume of phosphorus standard solution (4.13.2)	Corresponding mass of phosphorus
ml	mg
0	0
5.0	0.05
10.0	0.10
15.0	0.15
20.0	0.20
25.0	0.25
30.0	0.30
35.0	0.35
40.0	0.40

Proceed as specified in 7.3.1.1 from the first paragraph beginning at “Add 5 ml of the nitric acid (4.3), 5 ml of the hydrochloric acid (4.2)...” to the end of 7.3.2, but omitting 7.3.1.2.

7.4.2 Spectrophotometric measurement

Carry out spectrophotometric measurements of each solution in cells of 2 cm optical pathlength at a wavelength of about 425 nm after having adjusted the spectrophotometer (Clause 5) to zero absorbance against the zero term of the calibration solutions (7.4.1).

7.4.3 Plotting of the calibration graph and calculation of the angular coefficient a

Plot the absorbance against the known mass of phosphorus, in milligrams in 40 ml of 4-methyl-2-pentanone.

Calculate the angular coefficient a from the slope of the calibration graph, if it is a straight line.

8 Expression of results

The phosphorus (P) content, as a percentage by mass, is obtained in accordance with the calibration graph, or calculated from the following formula:

for phosphorus content equal to or less than 0.08 % (m/m):

$$\frac{A}{a} \cdot \frac{1}{m} \cdot \frac{1}{10^3} \cdot 100 = \frac{A}{10 a m}$$

or

for phosphorus content greater than 0.08 % (m/m):

$$\frac{200}{V} \cdot \frac{A}{a} \cdot \frac{1}{m} \cdot \frac{1}{10^3} \cdot 100 = \frac{20 A}{V a m}$$

where

- a is the angular coefficient or absorbance of a solution containing 1 mg of phosphorus in 40 ml of 4-methyl-2-pentanone with an optical pathlength of 2 cm,
- A is the absorbance of the test solution corrected by the absorbance of its blank test,
- m is the mass, in grams, of the test portion,
- V is the volume, in millilitres, taken from the test solution for phosphorus content greater than 0.08 % (m/m).

9 Notes

9.1 For samples containing more than 25 % (m/m) of chromium, remove the chromium by volatilization as follows:

To the fuming solution, with the chromium fully oxidized, add 5 ml of the hydrochloric acid (4.2), then continue fuming until the remaining chromium is again fully oxidized.

Repeat the treatment with hydrochloric acid followed by further fuming to reoxidize the small amount of residual chromium.

9.2 For samples containing titanium, zirconium, niobium or tantalum contents as specified in Clause 1, after evaporation of perchloric acid, add 20 ml of the boron fluoride solution (4.10) in addition to the nitric acid (4.4).

10 Test report

The test report shall include the following information:

- a) the method used by reference to this European Standard;
- b) the results, and the form in which they are expressed;
- c) any unusual features noted during the determination;
- d) any operation not specified in this European Standard or in the European Standard to which reference is made, or any optional operation which may have influenced the results.

Method 2

Determination of phosphorus in non-alloyed steels and irons — Molybdenum blue spectrophotometric method

1 Scope and field of application

This European Standard specifies a spectrophotometric method for the determination of phosphorus in non-alloyed steels and irons.

The method is applicable to non-alloyed steels and irons with phosphorus contents from 0.005 to 0.25 % (m/m).

2 Reference

Euronorm 18 — Selection and preparation of samples and test pieces for steel and iron and steel products.

3 Principle

Dissolution of a test portion in nitric and hydrochloric acids and controlled addition of perchloric acid. Formation of the phosphomolybdate complex after removal of silicon and arsenic and reduction with hydrazine sulphate to molybdenum blue.

Spectrophotometric measurement of the blue complex at a wavelength of 680 nm or 825 nm.

4 Reagents

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

Verify by blank tests that the relevant reagents are free from phosphorus. Whenever necessary, the results shall be corrected accordingly. Grades giving high blank values are unsuitable and should be discarded.

4.1 Hydrochloric acid, ρ about 1.19 g/ml.

4.2 Nitric acid, ρ about 1.40 g/ml.

4.3 Perchloric acid, ρ about 1.67 g/ml.

4.4 Hydrofluoric acid, ρ about 1.13 g/ml, diluted 1 + 9.

4.5 Hydrobromic acid, ρ about 1.47 g/ml, diluted 1 + 1.

4.6 Perchloric acid, ρ about 1.67 g/ml, diluted 42 + 58.

Carefully pour 420 ml perchloric acid (4.3) into 400 ml of water. Swirl, cool, transfer to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

4.7 Sulphuric acid, ρ about 1.84 g/ml, diluted 3 + 37.

Carefully pour 37.5 ml sulphuric acid (ρ about 1.84 g/ml) into 300 ml water. Swirl, cool and transfer to a 500 ml volumetric flask. Dilute to the mark with water and mix.

4.8 Sodium metabisulphite, 100 g/l solution.

Dissolve 100 g of dry sodium metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 500 ml of warm water, filter and collect the filtrate in a 1 000 ml volumetric flask. Cool, dilute to the mark with water and mix.

4.9 Molybdate reagent

4.9.1 Hydrazine sulphate, 1.5 g/l solution.

4.9.2 Ammonium molybdate, 20 g/l solution.

Carefully pour 300 ml of sulphuric acid (ρ about 1.84 g/ml) into 500 ml of water. Dissolve 20 g of ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in about 100 ml of water. Add this solution to the preceding solution. Swirl, cool and transfer to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

Prepare immediately before use the following mixture:

Add, in order, to a 1 000 ml volumetric flask, swirling between additions:

- 500 ml of water,
- 250 ml of molybdate solution (4.9.2),
- 100 ml of hydrazine sulphate solution (4.9.1).

Dilute to the mark with water and mix.

4.10 Phosphorus, 5 mg/l standard solution.

Weigh, to the nearest 0.0001 g, 0.4393 g of potassium dihydrogen phosphate (KH_2PO_4), previously dried to constant mass at 105 °C and cooled in a desiccator. Dissolve in water and add 40 ml perchloric acid, ρ about 1.67 g/ml, diluted 1 + 5. Transfer to 1 000 ml volumetric flask, dilute to the mark with water and mix.

Transfer 50.0 ml of this solution to a 1 000 ml volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 5 µg of phosphorus.

5 Apparatus

Ordinary laboratory equipment and spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 680 nm or 825 nm, together with 2 cm or 1 cm optical cells.

6 Sampling

Sampling shall be carried out in accordance with Euronorm 18.

Chips of thickness less than 2 mm.

7 Procedure**7.1 Test portion**

Weigh a mass (m) to the nearest 0.001 g:

- $m = 1 \text{ g} \pm 5 \%$ for $0.005 < P (\%) < 0.050$,
- $m = 0.5 \text{ g} \pm 5 \%$ for $0.050 < P (\%) < 0.1$,
- $m = 0.2 \text{ g} \pm 5 \%$ for $0.1 < P (\%) < 0.25$.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents.

7.3 Determination**7.3.1 Preparation of test solution**

Introduce the test portion (7.1) into a wide necked 250 ml conical flask. Add 10 ml of nitric acid (4.2), 20 ml of hydrochloric acid (4.1) and 5 ml of water. Cover with a watch-glass and warm gently until dissolution is complete. Remove the watch-glass and add 17 ml of perchloric acid (4.3). Heat until the appearance of perchloric acid fumes. Remove from the heat and allow to cool.

Add 10 ml of hydrofluoric acid (4.4). Heat again until the appearance of perchloric acid fumes. Remove from the heat. Allow to cool (see the note in Clause 9).

Add 10 ml of hydrobromic acid (4.5). Heat again to perchloric acid fumes. When the fumes collect at the bottom of the cylindrical part of the neck of the 250 ml conical flask, maintain for about 1 minute.

Add 30 ml of water and heat to dissolve the salts. Bring to the boil, then remove from the source of heat and allow to cool. Transfer to a 100 ml volumetric flask, dilute to the mark with water and mix.

7.3.2 Formation of the reduced phosphomolybdate complex

Transfer a 20 ml aliquot of the solution, obtained in 7.3.1, into each of two wide-necked, 100 ml conical flasks, the one to be used as the test solution and the other as a compensating solution. By means of burettes, make additions as follows:

Test solution

Add 15 ml of sodium metabisulphite solution (4.8). Swirl, boil for one minute to expel sulphur dioxide. Without removing from the source of heat add exactly 50 ml of molybdate reagent (4.9). Bring just to the boil and place in a water-bath at 85 to 90 °C for 20 minutes or bring to the boil and maintain for 4 minutes. Remove from the source of heat and cool quickly to ambient temperature under cold water.

Transfer to a 100 ml volumetric flask, dilute to the mark with water and mix.

Compensating solution

Add 15 ml sodium metabisulphite solution (4.8). Swirl, then boil for one minute to expel sulphur dioxide. Without removing from the source of heat add 50 ml of sulphuric acid (4.7).

Remove from the source of heat and allow to cool to ambient temperature.

Transfer to a 100 ml volumetric flask. Dilute to the mark with water and mix.

7.3.3 Spectrophotometric measurements

Set the spectrophotometer to zero absorbance in relation to water. Carry out spectrophotometric measurements on the blank, the test solution, and the compensating solution at the maximum of the absorption curve, either at a wavelength of about 680 nm in 2 cm optical cells or at a wavelength of about 825 nm for 1 cm optical cells.

Subtract the absorbance of the compensating solution from that of the test solution. Convert the net readings for the test portion solution and for the blank test solution to micrograms of phosphorus by reference to the calibration graph (7.4.4).

7.4 Establishment of the calibration graph

7.4.1 Preparation of calibration solutions

In a series of six wide-necked 100 ml conical flasks introduce phosphorus standard solution (4.10) as indicated in the following table:

Volume of phosphorus standard solution (4.10) [ml]	Corresponding mass of phosphorus [µg]	% P for a test portion of 1 g and a 20 ml aliquot	% P for a test portion of 0.5 g and a 20 ml aliquot	% P for a test portion of 0.2 g and a 20 ml aliquot
0	0	0	0	0
4	20	0.010	0.020	0.050
8	40	0.020	0.040	0.100
12	60	0.030	0.060	0.150
16	80	0.040	0.080	0.200
20	100	0.050	0.100	0.250

Add 6.5 ml of perchloric acid solution (4.6) and evaporate to perchloric acid fumes.

When the fumes collect at the bottom of the cylindrical part of the neck of the 100 ml conical flask, maintain for about one minute. Dilute with 20 ml of water, remove from the source of heat and allow to cool.

7.4.2 Formation of the reduced phosphomolybdate complex

Continue as in 7.3.2 from "Add 15 ml of sodium metabisulphite solution (4.8)...".

7.4.3 Spectrophotometric measurements

Set the spectrophotometer to zero absorbance in relation to water. Carry out spectrophotometric measurements on the calibration solutions as described in 7.3.3.

7.4.4 Plotting of the calibration graph

Subtract the value obtained for the solution with no added phosphorus from each of the absorbance readings.

Prepare the calibration graph by plotting the net absorbance readings against micrograms of phosphorus.

8 Expression of results

The phosphorus (P) content, as a percentage by mass, is obtained in accordance with the calibration graph, or calculated from the following formula:

$$\frac{(m_1 - m_o)}{m \cdot 1000} \cdot 100 = \frac{m_1 - m_o}{10 m}$$

where

- m is the mass, in milligrams, of the test portion (7.1),
- m_o is the mass, in micrograms, of phosphorus, found in the blank test solution,
- m_1 is the mass, in micrograms, of phosphorus, found in the test portion solution.

9 Note

For steels and irons with high silicon contents ($\text{Si} > 1 \%$), repeat the step given in the second paragraph of 7.3.1 as often as necessary for the complete removal of silicon.

10 Test report

The test report shall include the following information:

- a) the method used by reference to this European Standard;
- b) the results, and the form in which they are expressed;
- c) any unusual features noted during the determination;
- d) any operation not specified in this European Standard or in the European Standard to which reference is made, or any optional operation which may have influenced the results.

Annex Precision data

Planned trials of this method were carried out by analysts from 15 laboratories; each analyst carried out two determinations on each of five samples. From the results obtained 95 % (2 s) confidence limits have been calculated in accordance with ISO 5725 and are tabulated as follows:

Alloy type	Content of phosphorus %	Repeatability r	Reproducibility R
Non-alloyed iron ^a	0.02	0.0013	0.0023
Non-alloyed steel	0.02	0.0014	0.0019
Non-alloyed steel	0.024	0.0019	0.0028
Non-alloyed steel	0.043	0.0027	0.0041
Non-alloyed steel	0.012	0.0024	0.0030
^a In the case of this iron, only five laboratories participated in the tests.			

Repeatability, r

The difference between two single results found on identical material by one analyst using the same apparatus within a short time interval will exceed the repeatability, r , on average not more than once in 20 cases in the normal and correct operation of the method.

Reproducibility, R

The difference between two single and independent results found by two operators working in different laboratories on identical test material will exceed the reproducibility, R , on average not more than once in 20 cases in the normal and correct operation of the method.

National annex NA (informative)

Committees responsible

The United Kingdom participation in the preparation of this European Standard was entrusted by the Iron and Steel Standards Policy Committee (ISM/-) to Technical Committee ISM/18, upon which the following bodies were represented.

BCIRA

British Steel Industry

Department of Trade and Industry (Laboratory of the Government Chemist)

Ferro Alloys and Metals Producers' Association

Ministry of Defence

BSI — British Standards Institution

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