BRITISH STANDARD

BS EN 13086:2000

Lead and lead alloys — Lead oxides

The European Standard EN 13086:2000 has the status of a British Standard

ICS 77.150.60

National foreword

This British Standard is the official English language version of EN 13086:2000.

The UK participation in its preparation was entrusted to Technical Committee NFE/22, Lead and lead alloys, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 18, an inside back cover and a back cover.

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Lead and lead alloys - Lead oxides

Plomb et alliages de plomb - Oxydes de plomb

Blei und Bleilegierungen - Bleioxide

This European Standard was approved by CEN on 19 May 2000.

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 306 "Lead and lead alloys", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by December 2000, and conflicting national standards shall be withdrawn at the latest by December 2000.

The annexes A, B, C and D are normative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies the requirements, acceptance tolerances and chemical and physical test procedures for lead oxides, including litharges (powder or granulated), battery oxides (Barton or Mill) and crystal red lead.

WARNING Lead and lead compounds are toxic by inhalation and/or ingestion.

2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 23923-2, *Metallic powders - Determination of apparent density - Part 2 : Scott volumeter method (ISO 3923-2 : 1981)*.

EN ISO 787-11, *General methods of test for pigments and extenders - Part 11 : Determination of tamped volume and apparent density after tamping (ISO 787-11:1981)*.

3 Terms and definitions

For the purpose of this European Standard, the following terms and definitions apply :

3.1
lead oxide
product obtained from oxidation of lead metal with air

3.2
battery oxide
product obtained from partial oxidation of lead metal with air and composed of lead monoxide (PbO) and lead metal (Pb)

NOTE There are two varieties used in batteries : Barton (see 3.2.1) and Mill (see 3.2.2).

3.2.1
Barton
type of battery oxide obtained from oxidation of molten lead metal with air

3.2.2
Mill
type of battery oxide obtained from oxidation of lead metal with air together with a grinding process

3.3
litharge
product composed of lead monoxide (PbO) and obtained from oxidation of lead metal, supplied either as powder or granulated

3.4
red lead
product essentially composed of lead(II)-lead(IV) oxides ($2PbO.PbO_2$) (also known as lead orthoplumbate, trilead tetroxide, minium, etc.) obtained from oxidation of lead metal

4 Production process

The process for the production of lead oxides shall be left to the discretion of the producer.

5 Requirements

The requirements for lead oxides differ, depending on the type and the application of the oxide. The lead oxides shall be in accordance with the requirements given in the relevant table (see Tables 1 to 7). The test method shall be in accordance with the relevant method (see clause 7).

NOTE 1 Additional requirements or a range of values within, but narrower than, those stated in this European Standard can be specified in the order by agreement between the supplier and the purchaser.

NOTE 2 In all cases, the level of impurities is a consequence of impurities present in the lead used.

5.1 Battery oxides

Table 1 - Battery oxide - Mill type

Requirements	Units	Values
PbO content (mass fraction)	%	65 to 82
Free Pb content (mass fraction)	%	18 to 35
Apparent density (Scott)	g/cm^3	1,1 to 1,5
Tamped density	g/cm^3	2,9 to 3,5
Residue in sieve over 63 μm	%	0,5 max.
H_2SO_4 absorption index	mg/g	240 to 270

Table 2 - Battery oxide - Barton type

Requirements	Units	Values
PbO content (mass fraction)	%	65 to 82
Free Pb content (mass fraction)	%	18 to 35
Apparent density (Scott)	g/cm^3	1,0 to 2,0
Tamped density	g/cm^3	3 to 4
Residue in sieve over 63 μm	%	0,5 max.
H_2SO_4 absorption index	mg/g	150 to 210

5.2 Litharges

Table 3 - Litharge - Powder

Description		
Denomination		Litharge
Colour		Yellow/orange
Formulae		PbO
Requirements	**Units**	**Values**
PbO content (mass fraction)	%	99,5 min.
PbO_2 content (mass fraction)	%	0,05 max.
Free Pb content (mass fraction)	%	0,05 max.
Apparent density (Scott)	g/cm^3	1,8 to 2,6
Tamped density	g/cm^3	3,7 to 5,1
Residue in sieve over 63 µm	%	0,3 max.
Characteristics [a]	**Units**	**Values**
Melting point	°C	886
Molecular mass	g/mol	223,2
[a] The following characteristics are given for information only.		

Table 4 - Litharges – Granulated

Description		
Denomination		Litharge - granulated
Colour		Yellow/orange
Formulae		PbO
Requirements	**Units**	**Values**
PbO content (mass fraction)	%	99,5 min.
Free Pb content (mass fraction)	%	0,05 max.
Characteristics [a]	**Units**	**Values**
Melting point	°C	886
Molecular mass	g/mol	223,2
[a] The following characteristics are given for information only.		

5.3 Red lead oxides

Table 5 - Red leads for battery

Description		
Denomination		Red lead (battery)
Colour		Orange/red
Formulae		Pb_3O_4 ($2PbO\ PbO_2$)
Requirements	**Units**	**Values**
PbO content (mass fraction)	%	28,3 max.
PbO_2 content (mass fraction)	%	25,0 min.
Pb_3O_4 content (mass fraction)	%	71,7 min.
Apparent density (Scott)	g/cm^3	1,1 to 1,9
Tamped density	g/cm^3	2,7 to 4,0
Residue in sieve over 63 µm	%	2 max.
Characteristics [a]	**Units**	**Values**
Melting point	°C	500
Molecular mass	g/mol	685,6
[a] The following characteristics are given for information only.		

Table 6 - Crystal red lead for glass

Description		
Denomination		Red lead (glass)
Colour		Orange/red
Formulae		Pb_3O_4 ($2PbO\ PbO_2$)
Requirements	**Units**	**Values**
PbO content (mass fraction)	%	22,6 max.
PbO_2 content (mass fraction)	%	27,0 min.
Pb_3O_4 content (mass fraction)	%	77,4 min.
Residue in sieve over 63 µm	%	2 max.
Characteristics [a]	**Units**	**Values**
Melting point	°C	500
Molecular mass	g/mol	685,6
[a] The following characteristics are given for information only.		

Table 7 - Red lead for ceramic

Description		
Denomination		Red lead (ceramic)
Colour		Orange/red
Formulae		Pb_3O_4 ($2PbO\ PbO_2$)
Requirements	**Units**	**Values**
PbO content (mass fraction)	%	22,6 max.
PbO_2 content (mass fraction)	%	27,0 min.
Pb_3O_4 content (mass fraction)	%	77,4 min.
Residue in sieve over 63 µm	%	2 max.
Characteristics [a]	**Units**	**Values**
Melting point	°C	500
Molecular mass	g/mol	685,6

[a] The following characteristics are given for information only.

6 Quality control

The supplier shall be responsible for any inspections and tests required by the relevant specification given in clause 5, prior to shipment of the product. If the purchaser wishes to inspect the product prior to shipment, he shall notify the supplier at time of placing the order.

7 Test methods

7.1 Apparent density

The apparent density shall be determined in accordance with EN 23923-2.

7.2 Tamped density

The tamped density shall be determined in accordance with EN ISO 787-11.

7.3 Free lead in lead oxide

The free lead in lead oxide shall be determined in accordance with annex A.

7.4 Lead dioxide in red lead and litharge

The lead dioxide in red lead and litharge shall be determined in accordance with annex B.

7.5 Oversize residue

The oversize residue shall be determined in accordance with annex C.

7.6 Sulfuric acid absorption index

The sulfuric acid absorption index shall be determined in accordance with annex D.

8 Ordering information

The purchase order shall define the product required and shall contain the following details :

a) denomination of the product (see Tables 1 to 7) ;

b) number of this European Standard (EN 13086) ;

c) quantity (mass) ;

d) packaging and shipping conditions ;

e) any other requirement agreed between the purchaser and the supplier.

9 Labelling

Each product shall be clearly labelled by the manufacturer, in accordance with the relevant regulations, either directly on the package or by an adhesive label on the shipping unit with the following information :

a) manufacturer's name or trade mark ;

b) denomination of the product (see Tables 1 to 7) ;

c) year and month of manufacture, and/or production reference.

Annex A
(normative)

Determination of free lead in lead oxides

WARNING Due to the hazards inherent in a laboratory, both general and specific, this procedure shall only be carried out by fully trained personnel, or for those in the course of training, under the full supervision of trained personnel.

A.1 Scope

This method specifies the determination of the percentage of free lead (metallic lead) in litharge and battery lead oxide (Barton and Mill). The lead monoxide content is calculated as specified in A.6.2.

A.2 Principle

A weighed test portion is treated with acetic acid in order to dissolve the lead monoxide. Any undissolved lead dioxide (PbO_2) is reduced in ascorbic acid.

A.3 Apparatus

A.3.1 500 ml conical flask.

A.3.2 2 000 ml flat-bottomed flask.

A.3.3 Sintered glass filters, Nº 4 porosity.

A.3.4 Hotplate.

A.3.5 Oven-capable of maintaining 105 °C ± 2 °C.

A.3.6 Balance minimum accuracy ± 0,001 g.

A.3.7 Graduated flask 1 000 ml.

A.4 Reagents

A.4.1 General

All reagents shall be of analytical grade.

A.4.2 Deionized water, or water of equivalent quality.

A.4.3 Acetic acid, glacial (ρ_{20} = 1,05 g/ml).

A.4.4 Acetic acid, 20 % (volume fraction).

To 1 000 ml of deionized water in a 2 000 ml flask, add 400 ml of acetic acid, make up to the mark with water and mix.

A.4.5 Ascorbic acid (solution 2 %). Dissolve 20 g ± 0,1 g of ascorbic acid in water in a 1 000 ml graduated flask, make up to the mark with water and mix.

A.5 Procedure

A.5.1 General

The procedures for the litharge and for the battery oxides differ slightly as regards the mass of the test portion and the quantities of reagents used. The procedure for each is therefore detailed separately.

A.5.2 Litharge

A.5.2.1 Weigh between 50 g ± 0,1 g and 100 g ± 0,1 g of the litharge sample and transfer quantitatively to a 500 ml conical flask (A.3.1).

A.5.2.2 Add 250 ml of acetic acid 20 % (volume fraction) (A.4.4) and warm gently with shaking, until dissolution of the oxide in the sample is complete: any free lead will remain as a sediment. If the solution has a red-brown colour caused by lead dioxide (PbO_2), add ascorbic acid solution (A.4.5) dropwise until the red-brown colour is discharged.

A.5.2.3 After any red-brown colour has disappeared, allow 5 min for all particles to settle, then decant and discard the bulk of the supernatant liquid, ensuring that none of the sediment is lost.

A.5.2.4 Filter the remaining contents of the flask through a pre-weighed sintered glass filter (A.3.3), ensuring the transfer of all the sediment.

A.5.2.5 Wash the sediment retained on the filter four times with cold water and finally with acetone.

A.5.2.6 Transfer the filter to an oven at 105 °C ± 2 °C and dry the filter until the mass is constant.

A.5.2.7 Calculate the results in accordance with A.6.

A.5.3 Battery oxides

A.5.3.1 Weigh 5 g ± 0,001 g of the oxide sample and transfer quantitatively to a 500 ml conical flask (A.3.1).

A.5.3.2 Add 50 ml of acetic acid (A.4.4) and warm gently, with shaking, until the dissolution of the lead monoxide (PbO) is complete. If the solution has a red-brown colour due to the lead dioxide (PbO_2), add ascorbic acid solution (A.4.5) dropwise until the red-brown colour is discharged.

A.5.3.3 After any red-brown colour has disappeared, filter the solution through a pre-weighed sintered glass filter (A.3.3), ensuring the transfer of all the sediment.

A.5.3.4 Wash the sediment retained on the filter, four times with cold water and finally with acetone.

A.5.3.5 Dry the filter in an oven at 105 °C ± 2 °C until the mass is constant.

A.5.3.6 Calculate the results in accordance with A.6.

A.6 Calculation of results

A.6.1 The free lead, mass fraction in percent, shall be calculated as follows :

$$\text{Free lead (mass fraction)} = \frac{(\text{mass of filter plus sediment}) - (\text{mass of filter})}{(\text{mass of sample})} \times 100$$

A.6.2 The lead monoxide, mass fraction in percent, shall be calculated as follows :

Lead monoxide (PbO) = (100 – free lead) %.

Annex B
(normative)

Determination of lead dioxide in red lead and litharge

WARNING Due to the hazards inherent in a laboratory, both general and specific, this procedure shall only be carried out by fully trained personnel, or for those in the course of training, under the full supervision of trained personnel.

B.1 Scope

This method specifies the determination of the percentage of lead dioxide (PbO_2) present in litharge and in the red leads used in battery, glass and in ceramic glazes applications.

B.2 Principle

A weighed quantity of the sample is reacted with an excess of potassium iodide. The iodine liberated is titrated with sodium thiosulfate and lead dioxide calculated as specified in B.7.

B.3 Apparatus

B.3.1 Conical flasks, 250 ml, 500 ml.

B.3.2 Burette, 50 ml × 0,1 ml sub-divisions.

B.3.3 Micro-burette, 5 ml × 0,02 ml sub-divisions.

B.3.4 Graduated flasks, 500 ml, 2 000 ml.

B.3.5 Graduated cylinder, 100 ml.

B.3.6 Magnetic stirrer.

B.4 Reagents

B.4.1 General

All reagents shall be of analytical grade.

B.4.2 Deionized water, or equivalent quality.

B.4.3 Acetic acid, glacial (ρ_{20} = 1,05 g/ml).

B.4.4 Sodium acetate trihydrate.

B.4.5 Potassium iodide.

B.4.6 Starch, soluble.

B.4.7 Sodium thiosulfate.

B.5 Preparation of reagent solutions

B.5.1 Dissolution mixture

Add in the following order to a 2 000 ml graduated flask (B.3.4) :

- approximately 1 000 ml of water (B.4.2) ;
- 65 ml acetic acid, glacial (B.4.3) ;
- 120 g sodium acetate trihydrate (B.4.4) ;
- 5 g potassium iodide (B.4.5).

Make up to the mark with water and mix thoroughly.

NOTE This dissolution mixture should be freshly prepared.

B.5.2 Starch solution

Prepare a solution of 5 g soluble starch in about 400 ml of hot water and add to a 500 ml graduated flask. Then make up to the mark with water and mix.

B.5.3 Sodium thiosulfate solutions

B.5.3.1 0,1 N solution.

NOTE 1 ml 0,1 N sodium thiosulfate is equivalent to 0,0011959 g PbO_2.

B.5.3.2 0,02 N solution.

NOTE 1 ml 0,02 N sodium thiosulfate is equivalent to 0,00023918 g PbO_2.

The 0,02 N and 0,1 N solutions of sodium thiosulfate are available commercially. For solutions prepared on-site, the titre shall be checked.

B.6 Procedure

B.6.1 General

Due to the different levels of lead dioxides contents in litharge and red lead, the sample mass and the quantities of reagents differ. The procedure for each is therefore detailed separately.

B.6.2 Red lead

Weigh 1 g ± 0,01 g of the red lead sample, transfer to a 250 ml conical flask and add 60 ml of the dissolution mixture (B.5.1). Add 20 ml of sodium thiosulfate solution 0,1 N (B.5.3.1). Agitate to ensure that all the sample has reacted, if necessary using a glass rod to break down any agglomerates. Add 60 ml of the dissolution mixture (B.5.1) and 2 ml of the starch solution (B.5.2). Immediately titrate the liberated iodine with sodium thiosulfate solution, 0,1 N (see B.5.3.1) until the blue colour is just discharged. Note the volume in millilitres of sodium thiosulfate solution used.

Calculate the results in accordance with B.7.

B.6.3 Litharge

Weigh 10 g ± 0,1 g of the litharge sample, transfer to a 500 ml conical flask and add 100 ml of the dissolution mixture (B.5.1).

Agitate to ensure that all the sample has reacted, using, if necessary, a glass rod to break up any agglomerates. Immediately titrate the liberated iodine with sodium thiosulfate solution, 0,02 N (B.5.3.2) until the blue colour is just discharged. Note the volume in millilitres of sodium thiosulfate solution used.

Calculate the results in accordance with B.7.

B.7 Calculation of results

The lead dioxide content, mass fraction in percent, shall be calculated as follows :

$$\mathrm{PbO_2}\ \text{(mass fraction)} = \frac{V \times N \times 11{,}959}{m} \times 100$$

where

N is the normality of the sodium thiosulfate solution ;

V is the volume used of the sodium thiosulfate solution, expressed in millilitres ;

m is the mass of the sample, expressed in grams.

Annex C
(normative)

Determination of oversize residue by water method using a manual sieving procedure

C.1 Scope

This method specifies the determination of the proportion of material that will not pass through a 63 μm sieve and is suitable for lead oxides used for all purposes.

NOTE Other test methods for sieving exist but the results given may not be comparable.

C.2 Principle

A weighed portion of the oxide is made into a slurry with water and washed through a sieve with the aid of a water jet. The oversize residue is dried, weighed and the percentage calculated.

C.3 Apparatus

C.3.1 Sieve, aperture 63 μm.

C.3.2 Oven, capable of maintaining 105 °C ± 2 °C.

C.3.3 Balance, accurate to at least ±0,001 g and with pan dimensions to accommodate the sieve.

C.3.4 Wash bottle, plastic containing deionized water.

C.3.5 Brush, hog bristle, approximately 5 mm thick, 20 mm wide and 35 mm long.

C.3.6 Desiccator, with desiccant.

C.3.7 Beakers, various.

C.4 Procedure

C.4.1 General

If the approximate percentage of oversize residue is unknown, carry out a preliminary test in order to assess the suitable sample mass.

C.4.2 Weigh between 10 g ± 0,01 g and 100 g ± 0,1 g, depending on the oversize residue present and transfer to a beaker of suitable size.

Add 300 ml to 600 ml of water depending on the sample weight taken plus a suitable dispersion agent (0,2 % to 0,5 % of the mass) if the sample is agglomerated. Agitation can also be necessary to break up any agglomerates.

C.4.3 Pour the suspension, in portions if necessary, through the pre-weighed sieve (C.3.1) using the wash bottle jet (C.3.4) to aid the passage of the undersize material in order to facilitate sieving. Wash all of the sample from the beaker to the sieve with the wash bottle jet using the brush (C.3.5) to transfer any particles adhering to the beaker. Wash the residue well using the wash bottle jet.

C.4.4 Dry the sieve in an oven (C.3.2) at 105 °C ± 2 °C for 1 h, allow to cool in a desiccator (C.3.6) and weigh. Dry again for 30 min and re-weigh. Repeat the drying and weighing until the weight is constant.

C.4.5 After the weight of the residue has been determined, examine the residue and report any type of foreign matter found.

C.5 Calculation of results

The mass fraction of material over 63 µm, in percent, shall be calculated as follows :

$$\text{Material over 63 µm (mass fraction)} = \frac{\text{mass of residue}}{\text{mass of sample taken}} \times 100$$

Annex D
(normative)

Determination of sulfuric acid absorption index in battery oxides

WARNING Due to the hazards inherent in a laboratory, both general and specific, this procedure shall only be carried out by fully trained personal, or for those in the course of training, under the full supervision of trained personnel.

D.1 Scope

This method specifies the determination of the sulfuric acid absorption index (mg H_2SO_4/g oxide) of the battery oxides.

D.2 Principle

A weighed quantity of the sample is reacted with an excess of sulfuric acid.

The non-reacted acid is then titrated with sodium hydroxide solution and the sulfuric acid absorption index is calculated as specified in D.6.

D.3 Apparatus

D.3.1 Conical flasks, 250 ml, 500 ml.

D.3.2 Burette, 50 ml × 0,1 ml sub-divisions.

D.3.3 Pipettes, 10 ml.

D.3.4 Magnetic stirrer.

D.3.5 Balance, accurate to at least ±0,01 g.

D.3.6 Sintered glass filter, N° 4 porosity.

D.3.7 250 ml graduated cylinder.

D.3.8 500 ml glass beaker.

D.4 Reagents

D.4.1 Sulfuric acid 96 % (ρ_{20} = 1,84 g/ml).

D.4.2 1,0 N sodium hydroxide solution.

D.4.3 1 % phenolphthalein solution.

D.5 Procedure

D.5.1 Prepare a sulfuric acid solution (ρ_{20} = 1,150 g/ml) by carefully adding 130 g of sulfuric acid 96 % (ρ_{20} = 1,84 g/ml) (D.4.1) to 870 ml of deionized water, while agitating the solution. Allow the solution to cool.

D.5.2 Weigh 50 g ± 0,01 g of battery lead oxide, transfer to a 500 ml conical flask (D.3.1) and, using a 250 ml graduated cylinder (D.3.7), add 125 ml of sulfuric acid solution prepared in D.5.1 (ρ_{20} = 1,150 g/ml).

D.5.3 Agitate the contents for 15 min using the magnetic stirrer (D.3.4).

D.5.4 Allow to settle for 5 min.

D.5.5 Filter the supernatant liquid to a 500 ml beaker (D.3.8) through a sintered glass filter, N° 4 porosity (D.3.6).

D.5.6 With a 10 ml pipette transfer from the beaker to a 250 ml conical flask, 10 ml of filtrate (D.5.5). Add 3 drops of phenolphthalein indicator (D.4.3) and titrate with sodium hydroxide solution 1,0 N (D.4.2) to a permanent peak end point. Note the volume in millilitres of sodium hydroxide used (V_1).

D.5.7 Repeat the titration (D.5.6) with 10 ml of the sulfuric acid original solution D.5.1. Note the volume in millilitres of sodium hydroxide used (V_2).

D.6 Calculation of results

The sulfuric acid absorption index shall be calculated as follows :

$$\text{mg } H_2SO_4/\text{g oxide} = (V_2 - V_1) \times 12{,}26$$

where

V_1 is the volume of the sodium hydroxide solution, used with the sample, expressed in millilitres ;

V_2 is the volume of the sodium hydroxide solution, used with the blank, expressed in millilitres.

blank

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