

Methods for the analysis of ingot tin — Determination of impurity element contents in tin grades 99,90 % and 99,85 % by atomic spectrometry

The European Standard EN 13615:2001 has the status of a
British Standard

ICS 77.040.30; 77.150.60

National foreword

This British Standard is the official English language version of EN 13615:2001, including Corrigendum December 2002.

The UK participation in its preparation was entrusted to Technical Committee NFE/27, Tin and tin alloys, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/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.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

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

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Engineering Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 23 July 2002

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 15 and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments
13789 Corrigendum No. 1	24 October 2003	Deletion of the last paragraph in 7.2

© BSI 24 October 2003

ISBN 0 580 40129 4

English version

**Methods for the analysis of ingot tin - Determination of impurity
element contents in tin grades 99,90 % and 99,85 % by atomic
spectrometry**

Méthodes pour l'analyse des lingots d'étain - Détermination
des teneurs en impuretés dans l'étain de qualité 99,90 % et
99,85 % par spectrométrie atomique

Verfahren für die Analyse von Zinn in Masseln -
Bestimmung des Gehaltes an Verunreinigungselementen in
Zinn der Reinheitsgrade 99,90 % und 99,85 % durch
Atomspektrometrie

This European Standard was approved by CEN on 5 October 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

	page
Foreword	3
1 Scope.....	3
2 Normative references.....	3
3 Principle	3
4 Reagents	4
5 Apparatus.....	6
6 Sampling	6
7 Procedure.....	6
7.1 Preparation of the solution of the sample under test.....	6
7.2 Preparation of calibration solutions.....	6
7.3 Certified reference materials (CRM).....	7
7.4 Spectrometric measurements.....	7
8 Expression of results.....	7
8.1 Determining the metal content	7
8.2 Evaluation of precision.....	8
9 Test report.....	8
Annex A (normative) Method for checking atomic absorption spectrometer performance (based on ISO 7530-1:1990)	9
Annex B (normative) Method for checking inductively coupled plasma atomic emission spectrometer performance.....	12
Bibliography	14

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 220 "Tin and tin alloys" the secretariat of which is held by BSI.

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 2002, and conflicting national standards shall be withdrawn at the latest by June 2002.

The annexes A and B 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 atomic spectroscopic methods (Atomic Absorption Spectrometry (AAS) or inductively coupled plasma Atomic Emission Spectrometry (ICP-AES)) intended for the analysis of ingot tin. It is written for use by experienced analysts familiar with atomic spectrometric techniques.

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 610, *Tin and tin alloys — Ingot tin*.

3 Principle

The test sample is dissolved in hydrochloric acid plus nitric acid and tartaric or citric acid, and the concentration of the element sought is measured using atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry. Interference is minimised by matching sample and reference materials and by the choice of instrument parameters.

4 Reagents

4.1 General

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

4.2 *Hydrochloric acid*, concentrated ($\rho \approx 1,18$ g/ml).

4.3 *Dilute hydrochloric acid (1 + 1)*. Dilute 100 ml hydrochloric acid (4.2) with 100 ml water.

WARNING The acid should be added to the water for safety reasons.

4.4 *Dilute hydrochloric acid (1 + 19)*. Dilute 10 ml hydrochloric acid (4.2) with 190 ml water.

4.5 *Nitric acid*, ($\rho \approx 1,42$ g/ml).

4.6 *Sulphuric acid*, ($\rho \approx 1,84$ g/ml).

4.7 *Tartaric acid or citric acid*.

4.8 *Acid mixture*. Add 250 ml hydrochloric acid (4.2) to 250 ml water. Cool. Add 250 ml nitric acid (4.5) and 50 g tartaric (or citric) acid (4.7). Dilute to 1 l with water.

4.9 *Dilute nitric acid (1 + 1)*. Add 100 ml nitric acid (4.5) to 100 ml water.

4.10 *Dilute nitric acid (1 + 4)*. Add 50 ml nitric acid (4.5) to 200 ml water.

4.11 *Dilute nitric acid (1 + 9)*. Add 25 ml nitric acid (4.5) to 225 ml water.

4.12 *Dilute nitric acid (1 %)*. Dilute 5 ml nitric acid (4.5) up to 500 ml.

4.13 *Standard solutions of metals*. Freshly purchased standard metal solutions may be used or standard metal solutions should be made up as follows (4.13.1 to 4.13.25).

4.13.1 *Standard antimony solution (1 ml contains 1 mg Sb)*. Place 0,10 g Sb in 5 ml sulphuric acid (4.6) and heat to complete dissolution. Cool. Carefully add approximately 10 ml water and cool again. Transfer to a 100 ml volumetric flask with dilute hydrochloric acid (1 + 1) (4.3).

4.13.2 *Standard antimony solution (1 ml contains 0,2 mg Sb)*. Transfer 10,00 ml of Sb solution 4.13.1 to a 50 ml volumetric flask and make up to volume with dilute hydrochloric acid (1 + 1) (4.3).

4.13.3 *Standard copper solution (1 ml contains 1 mg Cu)*. Dissolve 0,10 g Cu in 10 ml dilute nitric acid (1 + 4) (4.9) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.4 *Standard copper solution (1 ml contains 0,2 mg Cu)*. Transfer 10,00 ml of Cu solution 4.13.3 to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.5 *Standard lead solution (1 ml contains 1 mg Pb)*. Dissolve 0,10 g Pb in 10 ml dilute nitric acid (1 + 4) (4.10) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.6 *Standard lead solution (1 ml contains 0,1 mg Pb)*. Transfer 5,00 ml of Pb solution 4.13.5 to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.7 *Standard bismuth solution (1 ml contains 1 mg Bi)*. Dissolve 0,10 g Bi in 10 ml dilute nitric acid (1 + 1) (4.9) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.