BS ISO 10378:2016

BSI Standards Publication

Copper, lead and zinc sulfide concentrates — Determination of gold and silver — Fire assay gravimetric and flame atomic absorption spectrometric method

bsi.

...making excellence a habit.™

Document Sharing Hub

Full document details: BS ISO 10378:2016

National foreword

This British Standard is the UK implementation of ISO 10378:2016. It supersedes BS ISO 10378:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NFE/36, Copper lead and zinc ores and concentrates.

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.

© The British Standards Institution 2016. Published by BSI Standards Limited 2016

ISBN 978 0 580 89275 2

ICS 73.060.99

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 29 February 2016.

Amendments issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS ISO 10378:2016

INTERNATIONAL STANDARD

ISO 10378

Third edition
2016-02-15

Copper, lead and zinc sulfide concentrates — Determination of gold and silver — Fire assay gravimetric and flame atomic absorption spectrometric method

Concentrés de sulfure de cuivre, de plomb et de zinc — Dosage de l'or et de l'argent — Méthode gravimétrique par essai au feu et spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 10378:2016(E)

Documents
Document Sharing Hub

© ISO 2016

Full document details: BS ISO 10378:2016

COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 10378:2016

Contents

Page

Foreword ... **v**

Introduction ... **vi**

1 Scope ... **1**

2 Normative references ... **1**

3 Principle ... **2**
3.1 General ... 2
3.2 Fusion ... 2
3.3 Cupellation ... 2
3.4 Parting ... 2
3.5 Retreatment ... 2
3.6 Correction for blank contamination ... 2

4 Reagents ... **2**

5 Apparatus ... **4**

6 Sample ... **5**
6.1 Test sample ... 5
6.2 Test portion ... 5

7 Procedure ... **6**
7.1 Number of determinations ... 6
7.2 Trial fusion ... 6
7.3 Blank tests ... 6
7.4 Charge preparation ... 6
7.5 Primary fusion ... 7
7.6 Cupellation ... 8
7.7 Retreatment of residues ... 8
7.8 Determination of gold in the primary bead ... 9
7.9 Determination of gold and silver in secondary beads and blanks, and of silver in prills ... 10
7.10 Determination of silver in the parting solution ... 11

8 Expression of results ... **12**
8.1 Mass fraction of gold ... 12
8.2 Mass fraction of silver ... 13

9 Precision ... **14**
9.1 Expression of precision ... 14
9.2 Method for obtaining the final result (see Annex H) ... 15
9.3 Precision between laboratories ... 15
9.4 Check of trueness ... 17
9.4.1 General ... 17
9.4.2 Type of certified reference material (CRM) or reference material (RM) ... 18

10 Test report ... **18**

Annex A (normative) **Procedure for the preparation and determination of the mass of a predried test portion** ... **19**

Annex B (normative) **Trial fusion** ... **21**

Annex C (normative) **Blank determination** ... **22**

Annex D (normative) **Inquartation** ... **23**

Annex E (normative) **Determination of vaporization loss of silver during the cupellation process** ... **24**

Annex F (normative) **Sulfuric acid — Parting** ... **25**

Annex G (normative) **Determination of impurities in parting solutions and washings** ... **27**

© ISO 2016 – All rights reserved

AVA Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

Annex H (normative) **Flowsheet of the procedure for the acceptance of analytical values for test samples (see 9.2)** **31**

Annex I (informative) **Flowsheet of the method** **32**

Annex J (informative) **Roasting method** **33**

Annex K (informative) **Guide to the preparation of dilutions for the determination of silver in parting solutions and residues** **34**

Annex L (informative) **Derivation of precision equations** **35**

Bibliography **50**

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 10378:2016

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

This third edition cancels and replaces the second edition (ISO 10378:2005), in which 6.2 has been technically revised and the warning notice in A.3.1 has been updated.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

Introduction

This International Standard describes a method for the determination of the mass fraction of gold and silver in copper, lead, and zinc sulfide concentrates. This International Standard was prepared to enable laboratories to determine the mass fraction of gold and silver in suitable samples using instrumental methods.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

INTERNATIONAL STANDARD ISO 10378:2016(E)

Copper, lead and zinc sulfide concentrates — Determination of gold and silver — Fire assay gravimetric and flame atomic absorption spectrometric method

WARNING — This International Standard may involve hazardous materials, operations, and equipment. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a fire assay gravimetric and flame atomic absorption spectrometric method for the determination of the mass fraction of gold and silver in copper, lead, and zinc sulfide concentrates as follows.

— Copper concentrates

The method is applicable to the determination of mass fractions of gold from 0,5 g/t to 300 g/t and of mass fractions of silver from 25 g/t to 1 500 g/t in copper sulfide concentrates containing mass fractions of copper from 15 % to 60 %.

— Lead concentrates

The method is applicable to the determination of mass fractions of gold from 0,1 g/t to 25 g/t and of mass fractions of silver from 200 g/t to 3 500 g/t in lead sulfide concentrates containing mass fractions of lead from 10 % to 80 %.

— Zinc concentrates

The method is applicable to the determination of mass fractions of gold from 0,1 g/t to 12 g/t and of mass fractions of silver from 10 g/t to 800 g/t in zinc sulfide concentrates containing mass fractions of zinc up to 60 %.

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 385, *Laboratory glassware — Burettes*

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 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

ISO 9599, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 10378:2016

3 Principle

3.1 General

Fire assaying for the determination of gold and silver comprises a series of steps to separate firstly the precious metals from most of the associated metals, followed by separation of the gold from silver and other metals pre-concentrated into a precious-metal alloy.

The stages that comprise the determinations are described in 3.2 to 3.6 inclusive.

3.2 Fusion

The samples are fused in a crucible after mixing with a litharge-based flux which, under reducing conditions, collects the precious metals in a metallic lead button.

3.3 Cupellation

The base metals present in the lead button are substantially separated from the precious metals by oxidizing fusion. Cupellation produces a bead largely comprising a silver-gold alloy with small quantities of other metals.

3.4 Parting

Gold is separated from the primary bead by treatment with nitric acid. The gold prill is weighed. Gold prills having a mass less than 50 µg are dissolved in aqua regia and the gold is determined by atomic absorption spectrometry (AAS). Silver is determined in the parting solution by AAS.

3.5 Retreatment

All residues are retreated to maximize the recovery of gold and silver. The addition of collectors for either gold or silver is not required, as both metals are present in sufficient amounts to be readily visible after the cupellation stage. The second bead is dissolved in acids followed by analysis of both metals by AAS.

3.6 Correction for blank contamination

Contamination by gold and silver impurities in the reagents is corrected for by fusing the reagents without the test portion.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water that complies with grade 2 of ISO 3696.

4.1 Sodium carbonate, anhydrous.

4.2 Litharge (PbO), assay grade having a mass fraction of gold of less than 0,01 g/t and a mass fraction of silver of less than 0,2 g/t.

4.3 Silica, precipitated.

4.4 Potassium nitrate or sodium nitrate

NOTE If sodium nitrate is used, the masses specified for potassium nitrate will have to be modified:

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

$$\text{g of } KNO_3 \times \frac{85,0}{101,1} = \text{g of } NaNO_3$$

4.5 Flour

4.6 Borax, fused anhydrous sodium tetraborate (borax glass powder).

4.7 Nitric acid, concentrated (ρ_{20} 1,42 g/ml), chloride concentration <0,5 µg/ml.

4.8 Nitric acid, diluted 1+1.

Slowly add 500 ml of concentrated nitric acid (4.7) to 500 ml of water, while stirring.

4.9 Lead, foil, having a mass fraction of gold of less than 0,01 g/t and a mass fraction of silver of less than 0,2 g/t.

4.10 Silver, of minimum purity 99,99 %.

4.11 Hydrochloric acid (ρ_{20} 1,16 g/ml to 1,19 g/ml).

4.12 Thiourea, 10 g/l solution.

Add 1 g of thiourea to 100 ml of water.

4.13 Aqua regia

Mix three parts of hydrochloric acid (4.11) with one part of nitric acid (4.7). Prepare freshly as required.

4.14 Standard solutions

Standard solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.14.1 Silver, standard stock solution A (500 µg of Ag/ml).

Weigh 0,500 0 g of silver metal to the nearest 0,1 mg. Transfer to a 100 ml beaker, add 20 ml of diluted nitric acid (4.8), and warm to dissolve. Cool and add 20 ml of concentrated nitric acid (4.7). Transfer to a 1 000 ml volumetric flask, fill up with water nearly to the mark, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.14.2 Silver, standard solution B (50 µg of Ag/ml).

Pipette 10,00 ml of silver standard stock solution A (4.14.1) into a 100 ml volumetric flask, fill up with water nearly to the mark, mix and cool to room temperature; then fill up exactly to the mark and mix again.

Prepare a fresh solution per batch.

4.14.3 Gold, standard solution (1 000 µg of Au/ml).

Weigh 1,000 g of gold metal to the nearest 0,1 mg. Transfer to a 200 ml beaker, add 25 ml of aqua regia solution (4.13), and warm to dissolve. Cool and transfer to a 1 000 ml volumetric flask. Add 75 ml of hydrochloric acid (4.11), fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

4.14.4 Gold and silver, standard solution (100 µg of Au/ml + 50 µg of Ag/ml).

Pipette 10,00 ml of silver standard stock solution A (4.14.1) into a 100 ml volumetric flask. Add 40 ml of hydrochloric acid (4.11). Pipette 10,00 ml of gold standard solution (4.14.3) into the volumetric flask. Fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.15 Calibration solutions

Calibration solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.15.1 Gold/silver calibration solutions

Pipette 0,0 ml, 1,00 ml, 2,00 ml, 5,00 ml, and 10,00 ml of gold and silver standard solution (4.14.4) into a series of 100 ml one-mark volumetric flasks.

Add 40 ml of hydrochloric acid (4.11) to each flask, fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

These solutions contain 0,0 µg of Au/ml, 1,00 µg of Au/ml, 2,00 µg of Au/ml, 5,00 µg of Au/ml, and 10,00 µg of Au/ml; and 0,0 µg of Ag/ml, 0,50 µg of Ag/ml, 1,00 µg of Ag/ml, 2,50 µg of Ag/ml, and 5,00 µg of Ag/ml, and shall be freshly prepared.

4.15.2 Silver calibration solutions

Pipette 0,0 ml, 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml, 8,00 m,l and 10,00 ml of silver standard solution B (4.14.2) into a series of 100 ml volumetric flasks. Add 10 ml of nitric acid (4.7), fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

These solutions contain 0,0 µg of Ag/ml, 0,50 µg of Ag/ml, 1,00 µg of Ag/ml, 2,00 µg of Ag/ml, 3,00 µg of Ag/ml, 4,00 µg of Ag/ml, and 5,00 µg of Ag/ml, and shall be freshly prepared.

Contamination by gold and silver impurities in the reagents is corrected for by fusing the reagents without the test portion.

5 Apparatus

5.1 Assay crucible furnace, with a maximum required operating temperature of 1 200 °C.

5.2 Muffle furnace, with a maximum required operating temperature of 1 100 °C. Temperature indication, automatic temperature control, and controlled air flow are preferable.

5.3 Assay crucibles, made of fire clay, of nominal capacity 200 ml to 600 ml, capable of withstanding corrosion by the samples and fluxes at 1 100 °C.

The crucible shall be of such a size that the charge does not fill the crucible to a depth greater than 3/4 the depth of the crucible.

5.4 Cupels, made of magnesium oxide, or bone-ash cupels having a nominal capacity of 50 g of molten lead.

The inside bottom of the cupel shall be concave, as recommended in the fire assay texts referred to in the Bibliography.

5.5 Conical mould, made of cast iron, of sufficient capacity to contain all of the molten lead plus slag from the crucible fusion.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

5.6 Analytical balance, sensitive to 1 mg.

5.7 Microbalance, sensitive to 1 µg or less.

5.8 Ordinary laboratory glassware, washed free of chlorides.

5.9 Volumetric glassware, of class A complying with ISO 385, ISO 648, and ISO 1042, and used in accordance with ISO 4787.

5.10 Atomic absorption spectrometer (AAS), equipped with background correction and a glass bead in the spray chamber.

5.11 Inductively coupled plasma (ICP) atomic emission spectrometer.

5.12 Pulverizer.

5.13 Hotplate.

6 Sample

6.1 Test sample

Prepare an air-equilibrated test sample in accordance with ISO 9599.

NOTE A test sample is not required if predried test portions are to be used (see Annex A).

6.2 Test portion

Taking multiple increments, extract a test portion from the test sample in such a manner that it is representative of the whole contents of the dish or tray. Weigh to the nearest 1 mg approximately 10 g to 20 g of the test sample. At the same time as test portions are being weighed for analysis, weigh test portions for the determination of hygroscopic moisture in accordance with ISO 9599.

NOTE 1 The precision of determination of the mass of contained gold in a lot is the sum of sampling and analytical precision. The presence of particulate gold in a concentrate contributes to imprecision of the analytical component of total precision. When it has been established that the presence of particulate gold in a concentrate causes unacceptable imprecision, the following mixing and sampling methods are recommended.

a) Mix the sample by either of the following preferred mixing methods as described in ISO 12743:2006:

— 15.3.2 c), strip mixing;

— 15.3.2 e), riffle, or rotary divider.

Mixing methods in items a), b), and d) in ISO 12743:2006, 15.3.2 *are not recommended.*

b) Extract a test portion from the test sample using either of the following methods described in ISO 12743:2006:

— 15.4.1 a), rotary sample division;

— 15.4.1 f), ribbon division.

Methods in items b) to e) and g) in ISO 12743:2006, 15.4.1 *are not recommended*.

Alternatively, the method specified in Annex A may be used to prepare predried test portions directly from the laboratory sample.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

If a mass fraction of arsenic above 2 % is present in the sample, this element should be removed by following the procedure in Annex J; otherwise, interference with the cupellation stage may occur.

NOTE 2 If the mass fraction of copper is greater than 30 %, a 10 g or 15 g test portion is preferable (see the fourth paragraph of 7.4).

For lead concentrates, the test portion should be 10 g to ensure an adequate supply of lead.

7 Procedure

7.1 Number of determinations

Carry out the determinations at least in duplicate, as far as possible under repeatability conditions, on each test sample.

NOTE 1 Repeatability conditions exist where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment, within short intervals of time.

NOTE 2 In the case where the ratio of silver to gold does not exceed 2,5 to 1 and the procedure specified in 7.10 is carried out for the silver determination, separate determinations for gold and silver will be necessary (see Annex D). Four test portions, therefore, are required, i.e. two for determinations of gold and two for silver.

NOTE 3 Annex I contains a flowsheet of the method.

7.2 Trial fusion

Carry out a trial fusion as described in Annex B, to ensure that the mass of the lead button is between 30 g and 45 g.

7.3 Blank tests

Carry out a reagent blank test as described in Annex C in parallel with the analysis, using the same quantities of all reagents, with the addition of sufficient flour (4.5) to the flux to give a lead button of between 30 g and 45 g. Omit the test portion and the potassium nitrate. The total blank should not exceed 5 µg of gold or 100 µg of silver.

7.4 Charge preparation

Determine the mass of potassium nitrate (4.4) and flour (4.5) required in the charge, as indicated by the trial fusion (see Annex B), and include this reagent in the flux mixture. Typical masses of the flux components for copper, lead, and zinc concentrates are shown in Tables 1 to 3 respectively.

Thoroughly mix the test portion with a flux.

Intimate mixing of flux components and the test portion is very important. All flux components should be in a finely divided state with a preferred particle size of less than 0,5 mm.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

Table 1 — Typical masses of flux components for copper concentrates

Flux components	Mass g
Sodium carbonate (4.1)	30
Litharge (4.2)	210
Silica (4.3)	25
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	20

If the mass fraction of copper is greater than 30 %, the mass of litharge should be 30 times that of copper plus 35 g for the lead button. Alternatively, a 10 g or 15 g test portion can be used while retaining the flux composition given in Table 1. If there are difficulties experienced in achieving a fluid melt, the amount of silica recommended in Table 1 can be reduced to 19 g, while including 6 g borax.

Table 2 — Typical masses of flux components for lead concentrates

Flux components	Mass, g
Sodium carbonate (4.1)	30
Litharge (4.2)	100
Silica (4.3)	10
Borax (4.6)	10
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	10

Table 3 — Typical masses of flux components for zinc concentrates

Flux components	Mass, g
Sodium carbonate (4.1)	30
Litharge (4.2)	120
Silica (4.3)	10
Borax (4.6)	10
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	20

Place the mixture in an assay crucible (5.3).

7.5 Primary fusion

Place the crucibles in the furnace (5.1) preheated to approximately 900 °C.

If oil-fired or gas-fired furnaces are used, the fuel should be turned off immediately before opening the furnace.

Slowly raise the furnace temperature to 1 000 °C to 1 050 °C. Maintain this temperature until the fusion has been tranquil for at least 10 min.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

The optimum furnace setting temperature depends on the furnace structure and the position of the thermometer. If unfused material remains in the bottom of assay crucibles, the setting temperature of the furnace should be raised and the assay repeated.

To minimize crucible corrosion and build-up of impurities in the lead button, the overall fusion time should not exceed 40 min. However, should the fusion take an extended time to settle, the fusion time may need to exceed 40 min.

It is recommended that a fire-clay lid or a layer of salt or borax approximately 12 mm thick be used with this fusion to prevent loss of material by dusting or ebullition. If borax is used as a cover, the amount used in the flux may be reduced accordingly. If some "shotting" of the lead is noted in the slag (this is common for zinc concentrates), it is recommended that the extra borax be retained.

Pour the mixture into a dry conical mould (5.5), taking care that no loss of lead or slag occurs. Reserve the crucible for retreatment fusion.

Allow the mixture to cool and carefully separate the lead button from the slag. Hammer the lead button as necessary to remove any small particles of adhering slag. Reserve the slag for retreatment.

Weigh the lead button. If the button weighs less than 30 g or more than 45 g, discard the button and slag and repeat the assay after appropriate adjustment of the oxidizing agent (see Annex B).

NOTE Buttons weighing less than 30 g may show poor collection efficiencies, whereas those exceeding 45 g may contain higher amounts of copper and other base metals.

7.6 Cupellation

Place the lead button obtained in 7.5 into a preheated cupel (5.4) in a muffle furnace (5.2) at 900 °C. Allow the cupellation to proceed at the lower muffle temperature of approximately 860 °C with a steady air flow.

Variations depend on the cupel type and furnace conditions. In the case of cupels made of bone ash, a cupellation temperature of 820 °C is recommended.

NOTE 1 In the case where only gold is being determined, it could be effective to raise the furnace temperature to 900 °C to finish the cupellation after the visible lead melt on the cupel is approximately 10 mm in diameter [approximately 80 % (mass fraction) of lead absorbed].

High cupellation temperatures will cause higher silver losses and low temperatures can cause "freezing" of the bead and incomplete cupellation. It is recommended that loss of silver during the cupellation process be determined, to decide upon the furnace conditions (see Annex E).

Remove the cupel from the furnace and cool.

Carefully extract the primary bead and remove any adhering cupel material with a brush. Flatten the bead slightly and place in a 30 ml porcelain crucible.

NOTE 2 A test tube can be used instead of a porcelain crucible.

NOTE 3 If the ratio of silver to gold in the primary bead is greater than 2,5 to 1, the silver can be determined by the gravimetric method instead of the procedure specified in 7.10. For the gravimetric method, weigh the primary bead, in micrograms, to the nearest 1 µg (m_0), carry out the parting by the procedure specified in 7.8, and determine the impurities in the parting solutions and washings by the procedure specified in Annex G.

NOTE 4 If it is difficult to recover the bead because of its small size, 1 mg of palladium can be added before fusion. In this case, the palladium bead is dissolved and determined by the procedure specified in 7.9.

Reserve the cupel for retreatment of residues.

7.7 Retreatment of residues

Place both the cupel and the slag in a pulverizer (5.12) and pulverize for about 20 s to reduce the material to minus 150 µm.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

If magnesium cupels are used, it is recommended that the slag and the cupel be retreated separately.

NOTE 1 Longer grinding can cause caking of the material and heating of the grinding barrel.

NOTE 2 The pulverizer may be cleaned between samples by grinding small portions of broken glass or quartz.

Thoroughly mix the ground residues with a flux. Typical composition of the flux is shown in Table 4.

Table 4 — Typical masses of flux components for the retreatment of residues

Flux components	Mass, g	
	Magnesium oxide cupel	Bone ash cupel
Sodium carbonate (4.1)	50 to 60	40
Litharge (4.2)	50 to 60	45
Silica (4.3)	50 to 60	20
Flour (4.4)	4	2 to 3
Borax (4.6)	30 to 50	15

The mass of flour shown in Table 4 is typical. The mass should be sufficient to produce a 30 g to 45 g lead button.

The combined mass of slag and cupel of the primary fusion, in addition to the flux components given in Table 4, may exceed the capacity of the assay crucibles, or the re-fusion may be so reactive that the fusions may froth over. In these cases, it is permissible to split the residues into equal halves and fuse separately in two crucibles. The lead buttons obtained should be cupelled separately, or be scorified together and the resultant lead button cupelled.

Place the mixture in the original assay crucible.

Carry out the fusion as detailed in 7.5 and discard the crucible and slag.

Cupel the lead button as detailed in 7.6 to obtain a second bead and discard the cupel.

7.8 Determination of gold in the primary bead

Add 10 ml of dilute nitric acid (4.8) to the primary bead in the porcelain crucible prepared in 7.6 and heat gently on a hotplate (5.13) for 20 min or until the reaction ceases.

NOTE 1 It is essential that chloride be absent during parting; otherwise, some of the gold may dissolve.

When the bead is treated with hot dilute nitric acid, silver will start to dissolve provided that the ratio of silver to gold in the bead exceeds 2,5 to 1. The rate of dissolution increases with increasing mass fraction of silver of the bead. Rapid attack of the bead should be avoided by further dilution and slow heating to prevent disintegration of the gold. Should the ratio of silver to gold be less than 2,5 to 1, as shown by failure to part in hot dilute nitric acid, the bead should be inquarted (see Annex D).

If there is danger of the gold sponge crumbling during the parting operation, it is recommended that the operation be carried out with sulfuric acid (see Annex F).

Carefully pour the solution into a 200 ml beaker by decantation to avoid losses.

Add 15 ml of warm dilute nitric acid (4.8) to the porcelain crucible and continue heating gently until parting is complete. This should take approximately 25 min.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

Carefully pour the solution into the 200 ml beaker by decantation to avoid losses. Wash the crucible and gold with four 15 ml volumes of hot water. Collect all the washings in the same 200 ml beaker. Reserve the solution for the determination of silver as specified in 7.10.

NOTE 2 The possibility of gold particles occurring in the collected parting and washing solutions can be determined by evaporating the solutions slowly down to 2 ml to 3 ml, then continuing with the determination as specified in 7.9.

Dry the gold sponge in the porcelain crucible on the hotplate.

Place the crucible in the muffle furnace (5.2) to anneal the gold at dull red heat for approximately 5 min.

Cool and weigh the resultant gold prill, in micrograms, to the nearest 1 µg (m_1).

If the mass of the gold is less than 50 µg, it is recommended that the gold be dissolved and determined by the procedure specified in 7.9.

If the mass of the gold is less than 50 µg, repeat the fusion and cupellation, then dissolve the prepared bead and determine the gold and silver concentration as specified in 7.9 without the parting operation. This alternative procedure is recommended where there is a danger of the gold sponge crumbling during the parting operation. The procedure, however, cannot be applied if the product of mass of test portion by mass fraction of silver, i.e. mass in test portion, is larger than 7 500 µg.

NOTE 3 If the sensitivity of the microbalance is 0,1 µg, the applicable range of the gravimetric method can be extended to 5 µg of gold. In such a case, weigh the gold prill, in micrograms, to the nearest 0,1 µg (m_1).

Reserve the gold prill to determine silver in the prill. The prill is dissolved and the silver concentration is determined as specified in 7.9. Several of the weighed prills of the same laboratory sample can be combined for the determination.

Platinum and palladium are removed from the prill during parting with nitric acid. If the determination of these elements remaining in the prill is considered necessary, determine these by the procedure specified in 7.9, followed by the addition of platinum and palladium to the standard solutions in relevant proportions. If a sufficient detection limit for AAS or ICP cannot be obtained on a single prill basis, a large number of prills of the same laboratory sample should be combined.

7.9 Determination of gold and silver in secondary beads and blanks, and of silver in prills

For blanks and samples determined by the procedure specified in the fourth last paragraph of 7.8 without the parting operation, the primary and secondary beads should be combined and treated together.

Transfer the bead(s) or prill(s) to a test tube or a porcelain crucible. Add 2 ml of nitric acid (4.7) and warm in a heating block or a sand bath set at approximately 98 °C. Add 6 ml of hydrochloric acid (4.11) and heat again to dissolve the gold. If necessary, add a further 2 ml of nitric acid (4.7). Take the above solution, or that prepared according to Annex C, and heat almost to dryness.

The solution should not be allowed to evaporate to dryness; otherwise, metallic gold will form.

Remove the test tube or the crucible from the heating block or the sand bath and allow cooling. Add 10 ml of hydrochloric acid (4.11) and mix or swirl to dissolve any salts. Transfer quantitatively to a 50 ml volumetric flask, add 10 ml of hydrochloric acid (4.11), make up to the mark with water, and mix well.

Depending upon the mass fraction of silver (see Annex K), it may be necessary to make dilutions so that the concentration of silver in the test solutions is in the range covered by the silver calibration solutions (4.15.1). Hydrochloric acid (4.11) should be added so that 40 ml of the acid is contained per 100 ml of the diluted test solutions.

Aspirate the test solutions and gold/silver calibration solutions (4.15.1) into the atomic absorption spectrometer (5.10) and measure the absorbance. As a guide, the atomic absorption settings shown in Table 5 are recommended; however, the instrument should be optimized to be free from any interference and to give maximum sensitivity and as near as practical to a linear relationship between absorbance and concentration.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016

Table 5 — Recommended atomic absorption settings

Parameter	Gold	Silver
Flame	air/acetylene (oxidizing)	
Wavelength	242,8 nm	328,1 nm
Lamp current	4 mA	5 mA
Background corrector	on	off
Aspiration rate	optimize for maximum signal	
Integration time	3 s	
Number of integrations	5	
Absorbance of 5 µg/ml calibration solution	0,17	0,55

Perform three measurements on each test solution and calibration solution. Calculate, to three significant figures, the mean absorbance for each solution, provided that the range of values does not exceed 0,003 absorbance units. If this range is exceeded, repeat the measurement.

In order to clean out the nebulizer system, it is recommended to aspirate a cleaning solution, which is, for instance, prepared by carefully adding 500 ml of hydrochloric acid (4.11) and 100 ml of concentrated nitric acid (4.7) to 400 ml of water, between measurements.

Plot a calibration graph of absorbance versus concentration and determine the gold and/or silver concentrations, in micrograms per millilitre, in the test solutions, followed by calculation of the mass (m_a), in micrograms, of the gold and/or silver using Formula (1):

$$m_a = \rho \times \mathrm{TDF} \tag{1}$$

where

ρ is the mass concentration of gold and/or silver;

TDF is the total dilution factor.

Alternatively, an ICP atomic emission spectrometer (5.11) can be used for the determination of gold and silver at the appropriate wavelength. Typical wavelengths are 242,8 nm for gold and 328,1 nm for silver; however, the instrument should be optimized to be free from any interference and to give maximum sensitivity and as near as practical to a linear relationship between absorbance and concentration. In order to improve the precision, it is recommended that a simultaneous internal standard correction be adopted. Yttrium is generally used as an internal standard.

During all AAS or ICP determinations, the test solutions and calibration solutions should have the same temperature, as well as the same acid concentration.

7.10 Determination of silver in the parting solution

NOTE 1 This determination is applicable where the ratio of silver to gold is greater than 2,5 to 1 and the silver has been parted from the gold as indicated in 7.8.

NOTE 2 The silver can be determined by the gravimetric method instead of the procedure specified in this subclause. For the gravimetric method, determine the impurities in the parting solutions and washings by the procedure specified in Annex G.

Take the parting solutions and washings, which were reserved in 7.8. Heat to evaporate to approximately 20 ml, cool, and then add 2 ml of nitric acid (4.8). Transfer quantitatively to a 100 ml volumetric flask, make up to the mark with water, and mix well.

If the solution turns cloudy, add 1 % of thiourea solution (4.12) drop by drop, while stirring until the solution is clear. Add 2 ml in excess, then dilute to volume.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 10378:2016