



BSI Standards Publication

**Copper concentrates — Determination of  
mercury content — Cold vapour atomic  
absorption spectrometric method**

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## National foreword

This British Standard is the UK implementation of [ISO 13546:2021](#).

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 committee manager.

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# INTERNATIONAL STANDARD

**ISO**  
**13546**

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## **Copper concentrates — Determination of mercury content — Cold vapour atomic absorption spectrometric method**

*Concentrés de cuivre — Dosage du mercure — Méthode par  
spectrométrie d'absorption atomique de vapeur froide*



Reference number  
ISO 13546:2021(E)

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This document was prepared by Technical Committee ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

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# Copper concentrates — Determination of mercury content — Cold vapour atomic absorption spectrometric method

**WARNING** — The use of this document can involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

## 1 Scope

This document specifies an acid digestion and vapour generation atomic absorption spectrometric method for the determination of the mercury content in copper sulfide concentrates.

This document is applicable to mass fraction of mercury between 5 µg/g and 65 µg/g in copper sulfide concentrates.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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, zinc and nickel sulfide concentrates — Determination of hygroscopic moisture content of the analysis sample — Gravimetric method*

[ISO 12743:2018](#), *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

[ISO Guide 35](#), *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

## 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>



## 4 Principle

The test portion is decomposed by treatment with hydrochloric and nitric acid at a temperature between 60 °C and 80 °C followed by the addition of potassium permanganate as oxidizing agent. Subsequently, the potassium permanganate is reduced by hydroxylamine hydrochloride. The mercury vapour is generated by vapour generation using tin (II) chloride as reduction agent. The equipment is set to measure the absorbance at 253,7 nm. The absorbances of the test and calibration solutions, including those of certified or other reference materials, are compared to determine the mercury content.

## 5 Reagents

During the analysis, use only reagents of recognized analytical grade and grade 2 water in accordance with [ISO 3696](#).

Reagents shall be selected or purified for the lowest possible blank value.

**5.1 Tin (II) chloride dehydrate**, ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ), containing < 5 mg/g mercury.

**5.2 Potassium permanganate** ( $\text{KMnO}_4$ ).

**5.3 Hydroxylamine hydrochloride** ( $\text{HONH}_2 \cdot \text{HCl}$ ).

**5.4 Mercury (II) chloride** ( $\text{HgCl}_2$ ).

**5.5 Nitric acid**,  $\rho = 1,42 \text{ g/ml}$ .

**5.6 Hydrochloric acid**,  $\rho = 1,16 \text{ g/ml}$  to  $1,19 \text{ g/ml}$ .

**5.7 Sulfuric acid**,  $\rho = 1,84 \text{ g/ml}$ .

**5.8 Sulfuric acid solution**, diluted 1 + 5.

**5.9 Sulfuric acid solution**, diluted 1 + 9.

**5.10 Aqua regia**. Mix 300 ml of hydrochloric acid ([5.6](#)) and 100 ml nitric acid ([5.5](#)). Prepare freshly for each batch of mercury determination.

**5.11 Tin (II) chloride solution**, 100 g/l. Add 10 g of tin (II) chloride ([5.1](#)) to 80 ml sulfuric acid solution ([5.9](#)). Heat and swirl to dissolve. Cool the solution and dilute with deionized water to 100 ml and mix thoroughly.

Continuously stir the solution with a magnetic stirrer for at least 2 h before use and maintain stirring during analysis. Prepare weekly.

Hydrochloric acid may be used instead of sulfuric acid.

Alternative procedure:

Tin (II) chloride solution, 100 g/l: add 10 g of tin (II) chloride ([5.1](#)) to 20 ml of deionized water. Add continuously 60 ml of sulfuric acid solution ([5.8](#)). Heat and swirl to dissolve. Cool, dilute to 100 ml with deionized water and mix thoroughly.

**5.12 Potassium permanganate ( $\text{KMnO}_4$ ) solution**, 2 g/l. Add 0,2 g of potassium permanganate ([5.2](#)) to 100 ml of deionized water. Store in a glass bottle.



**5.13 Hydroxylamine hydrochloride solution**, 20 g/l. Add 2 g of hydroxylamine hydrochloride ([5.3](#)) to 100 ml of deionized water.

**5.14 Mercury trapping solution**. Add 10 g of potassium permanganate ([5.2](#)) to 100 ml of water and mix well. This reagent is used to oxidize mercury vapour to its  $\text{Hg}^{2+}$  state and trap it in solution.

**5.15 Nitric acid solution**, diluted (1+9).

**5.16 Mercury standard solution A**, 100  $\mu\text{g}/\text{ml}$ . Weigh 0,1354 g of mercury (II) chloride ([5.4](#)) into a 250 ml beaker and dissolve in 100 ml of nitric acid ([5.5](#)). Quantitatively transfer to a 1-l volumetric flask with water. Mix and store in a labelled glass container.

Alternatively, use a suitable high-quality commercial standard solution.

**5.17 Mercury standard solution B**, 10  $\mu\text{g}/\text{ml}$ . Pipette 10 ml of mercury standard solution A ([5.16](#)) into a 100-ml volumetric flask containing 10 ml nitric acid ([5.5](#)). Dilute to volume with water. Mix and store in a labelled volumetric flask.

Prepare the solution monthly.

**5.18 Mercury standard solution C**, 0,1  $\mu\text{g}/\text{ml}$ . Pipette 10 ml of mercury standard solution B ([5.17](#)) into a 1-l volumetric flask containing 100 ml of nitric acid ([5.5](#)). Dilute to volume with water. Mix and store in a labelled volumetric flask.

The solution should be freshly prepared.

## 6 Apparatus

All laboratory glassware and equipment shall be free of mercury contamination. Use ordinary laboratory apparatus, including pipettes and volumetric flasks conforming with the specifications of [ISO 648](#) and [ISO 1042](#), respectively, and the following:

**6.1 Analytical balance**, sensitive to 0,1 mg.

**6.2 Magnetic stirrers**.

**6.3 Laboratory glassware**, of class A conforming with [ISO 385](#), [ISO 648](#), [ISO 1042](#) and used in accordance with [ISO 4787](#).

**6.4 Atomic absorption spectrometer**, equipped with a mercury hollow cathode lamp or electrodeless discharge lamp or a continuum radiation source.

**WARNING — Follow the manufacturer's instructions to avoid possible explosion hazards when igniting and extinguishing the air-acetylene flame and possible burning from the hot electric furnace. Wear tinted safety glasses whenever the atomic absorption spectrometer is in operation. Good ventilation is necessary to prevent poisoning by mercury hydride.**

The atomic absorption spectrometer used in this method shall meet the following criteria:

- Minimum sensitivity: the absorbance of the highest concentration calibration solution (see [8.7](#)) is at least 0,5  $\mu\text{g}/\text{ml}$ .
- Graph linearity: the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) is not less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.



- c) Minimum stability: the standard deviation of the absorbance of the most concentrated calibration solution and that of the zero-calibration solution, each being calculated from a sufficient number of repetitive measurements, are less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated solution.

The use of a strip-chart recorder, digital readout device or both is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

Parameters will vary with each instrument.

#### 6.5 Stopwatch.

#### 6.6 Hotplate.

## 7 Sampling and sampling preparation

### 7.1 Test sample

Prepare an air-equilibrated test sample and a hygroscopic moisture test sample in accordance with [ISO 9599](#).

Mercury can evaporate from samples at elevated temperatures, so a separate chemical analysis sample shall be prepared as described in [ISO 12743:2021](#), 16.2.

### 7.2 Test portion

Taking multiple increments, extract the test portion from the test sample as specified in [Table 1](#) and weigh to the nearest 0,1 mg. At the same time as the test portions are being weighed for analysis, weigh test portions for the determination of hygroscopic moisture in accordance with [ISO 9599](#).

**Table 1 — Recommended test portion masses**

Mercury content of sample µg/g	Mass of test portion g
< 50	1,0000
50 to 65	0,5000

## 8 Procedure

### 8.1 Number of determinations

Carry out the determinations at least in duplicate in accordance with [Annex A](#), independently, on each test sample.

NOTE The expression “independently” means that the second and any subsequent result is not affected by previous results. For this particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

### 8.2 Blank test

Carry out a blank test in parallel with the analysis using the same quantities of all reagents but omitting the test portion. The purpose of the blank test in this method is to check the quality of reagents. If a significant value is obtained as a result of the blank test, check all reagents and repeat the analysis.



### 8.3 Dissolution of the test portion

Transfer the test portion (7.2) to a conical beaker. Wash down the walls of the conical beaker with 5 ml of deionized water so that none of the sample remains on the walls. Carefully add 25 ml of aqua regia (5.10) while swirling the solution. Leave for 30 min at room temperature. Then, using a hotplate to maintain the temperature between 60 °C and 80 °C, digest the sample for 1 h.

If the mercury content of the test portion is to be determined within 6 h then proceed to 8.5. Otherwise, store the samples using the procedure described in 8.4.

### 8.4 Storage of digested solutions

Dilute the cooled solution from 8.3 to approximately 60 ml and add 10 ml of potassium permanganate solution (5.12), then cover the beaker with a watch glass or other suitable cover to minimize contamination of the sample.

When the mercury content of the sample is going to be determined, reduce the potassium permanganate by adding dropwise hydroxylamine hydrochloride solution (5.13) until the pink colour disappears, while swirling the conical beaker slowly to ensure adequate mixing.

### 8.5 Preparation of sample solutions

Dilute the sample solution from either 8.3 or 8.4 to approximately 80 ml with deionized water. If necessary, allow the solution to cool to room temperature and then dilute to 100 ml.

### 8.6 Preparation of test solutions

Prepare test solutions for each determination at least in duplicate.

Pipette 1 ml to 5 ml of the sample solution prepared in 8.5, containing between 0,1 µg and 0,5 µg of mercury, into the reaction vessel used for mercury determination. Dilute this solution to 100 ml with nitric acid solution (5.15).

### 8.7 Preparation of calibration solutions

Prepare all calibration solutions at least in duplicate.

With the mercury standard solution C 0,1 µg/ml Hg (5.18) and using Table 2 as a guide, prepare a series of calibration standards using pipettes to transfer the appropriate volumes into separate reaction vessels and dilute to 100 ml with nitric acid solution (5.15).

Table 2 — Calibrating solutions

Volume of mercury standard solution ml	Mercury content µg
1	0,1
2	0,2
3	0,3
4	0,4
5	0,5

### 8.8 Preparation of mercury calibration graph

Set up the atomic absorption spectrometer (6.4) according to the guidelines set out in 6.4. Adjust the instrument read-out scale to zero. Using vapour generation equipment, reduce the mercury in the first duplicate of the calibration solutions prepared in 8.7 with 5 ml of tin (II) chloride solution (5.11). Record the absorbance of the generated vapour with the atomic absorption spectrometer (6.4). If necessary,



flush out any residual mercury vapour from the vapour generation apparatus and check for condensed water vapour on the absorption cell.

Calculate the average absorbance for each calibration solution. Plot a graph of the average absorbance versus micrograms of mercury and repeat any errant standards.

NOTE New instruments are usually equipped with software to calculate and draw the calibration curve.

## 8.9 Determination of mercury content in test solutions

Immediately after calibrating the atomic absorption spectrometer, determine the mercury content in the test solutions prepared in 8.6.

Adjust the instrument read-out scale to zero and using vapour generation equipment reduce the mercury in the first duplicate of the test solutions prepared in 8.6 with 5 ml of tin (II) chloride solution (5.11). Record the absorbance of the generated vapour with the atomic absorption spectrometer (6.4). If necessary, flush out any residual mercury vapour from the vapour generation apparatus and check for condensed water vapour on the absorption cell. Repeat this procedure for the second duplicate.

If the difference in absorbance between the two test solutions exceeds 5 % then re-prepare the test solution as per 8.6 and repeat the mercury determination.

## 9 Expression of results

From the graph determined in 8.8 establish the micrograms of mercury found ( $F_{\text{Hg}}$ ) in the aliquot taken in 8.6 by calculating the mercury content of the sample according to Formula (1):

$$w_{\text{Hg}} = \left( \left( (F_{\text{Hg}} - B) * 100 \right) / (V_{\text{a}} * m) \right) * K \quad (1)$$

where

$w_{\text{Hg}}$  is the mercury content of the sample, in  $\mu\text{g/g}$ ;

$F_{\text{Hg}}$  is the mass of mercury found in test sample aliquot, in  $\mu\text{g}$ ;

$m$  is the mass of the test portion, in g;

$V_{\text{a}}$  is the volume of the test sample aliquot, in ml;

$B$  is the mass of mercury found in the blank test, in  $\mu\text{g}$ , corrected as follows:  $(F_{\text{Hg b}} * V_{\text{a}}) / V_{\text{a b}}$ ;

$F_{\text{Hg b}}$  is the mass of mercury found in blank test, in  $\mu\text{g}$ ;

$V_{\text{a b}}$  is the volume of blank test aliquot, in ml;

$K$  is the hygroscopic moisture conversion factor determined as follows:  $K = 100 / (100 - H)$ ;

$H$  is the hygroscopic moisture content of the sample determined by ISO 9599.

## 10 Procedure for obtaining the final result

Process the duplicate results for each test portion of the sample according to Figure A.1 and repeat the determination for a sample as necessary.

## 11 Precision

### 11.1 Expression of precision

The precision of this analytical method is expressed by Formulae (2) and (3):

$$S_r = 0,059 \ 4X + 0,074 \ 4 \quad (2)$$



$$S_L = 0,150\ 0X + 0,099\ 4 \quad (3)$$

where

$X$  is the concentration of mercury in the sample, in  $\mu\text{g/g}$ ;

$S_r$  is the within-laboratory standard deviation;

$S_L$  is the between-laboratory standard deviation.

## 11.2 Procedure for obtaining the final result

Calculate the quantities in [Formulae \(4\) to \(6\)](#) from the duplicate results  $X_1$  and  $X_2$  (%) and process according to [Annex A](#).

Mean of duplicates:

$$X = (X_1 + X_2) / 2 \quad (4)$$

Within-laboratory standard deviation (repeatability):

$$S_r = 0,059\ 4X + 0,074\ 4 \quad (5)$$

Repeatability limit:

$$r = 2,8S_r \quad (6)$$

## 11.3 Precision between laboratories

The precision between laboratories is used to determine the agreement between the results reported by two (or more) laboratories. It is assumed that all laboratories followed the same procedure.

Calculate the quantities in [Formulae \(7\) to \(11\)](#).

Mean of final results:

$$\mu_{1,2} = \frac{\mu_1 + \mu_2}{2} \quad (7)$$

where

$\mu_1$  is the final result, in  $\mu\text{g/g}$ , reported by laboratory 1;

$\mu_2$  is the final result, in  $\mu\text{g/g}$ , reported by laboratory 2.

Between-laboratory standard deviation:

$$S_L = 0,150\ 0X + 0,099\ 4 \quad (8)$$

Within-laboratory standard deviation:

$$S_r = 0,059\ 4X + 0,074\ 4 \quad (9)$$

Permissible tolerance:

$$P = 2,8 \left[ S_L^2 + (S_r^2 / 2) \right]^{1/2} \quad (10)$$

$$\text{Range } E = |\mu_1 - \mu_2| \quad (11)$$

where

- $\mu_1$  is the final result, in  $\mu\text{g/g}$ , reported by laboratory 1;  
 $\mu_2$  is the final result, in  $\mu\text{g/g}$ , reported by laboratory 2.

If  $E \leq P$ , the final results are in agreement.

## 11.4 Check of trueness

### 11.4.1 General

The trueness of the analytical method can be checked by applying it to a certified reference material (CRM). When the precision has been confirmed, the final laboratory result can be compared with the certified value,  $A_c$ .

Two possibilities exist, as shown in [Formula \(12\)](#) and [Formula \(13\)](#):

$$|\mu_c - A_c| \leq C \quad (12)$$

where

- $\mu_c$  is the final result, expressed as  $\mu\text{g/g}$  of mercury of the CRM;  
 $A_c$  is the certified value, expressed as  $\mu\text{g/g}$  of mercury of the CRM;  
 $C$  is a quantity, expressed as  $\mu\text{g/g}$  of mercury, depending on the type of CRM used, as defined in [11.4.2](#).

If this condition exists, the difference between the reported result and the certified value is statistically significant.

$$|\mu_c - A_c| > C \quad (13)$$

If this condition exists, the difference between the reported result and the certified value is statistically insignificant.

### 11.4.2 Type of certified reference material or reference material

The reference materials used for this purpose shall be prepared and certified in accordance with [ISO Guide 35](#).

### 11.4.3 Reference material certified or characterized by an interlaboratory test programme

The quantity  $C$  (see [11.4.1](#)), in  $5 \mu\text{g/g}$ , is given by [Formula \(14\)](#):

$$C = 2\sqrt{(S_L^2 + S_r^2 / n + S^2 \{A_c\})} \quad (14)$$

where

- $S^2\{A_c\}$  is the variance of the certified value;  
 $n$  is the number of replicate determinations.

### 11.4.4 Reference material certified or characterized by one laboratory

The quantity  $C$  (see [11.4.1](#)), in  $5 \mu\text{g/g}$ , is given by [Formula \(15\)](#):

$$C = 2\sqrt{(S_L^2 + S_r^2 / n)} \quad (15)$$



It is recommended that this type of CRM is avoided unless the particular CRM is known to have an unbiased certified value.

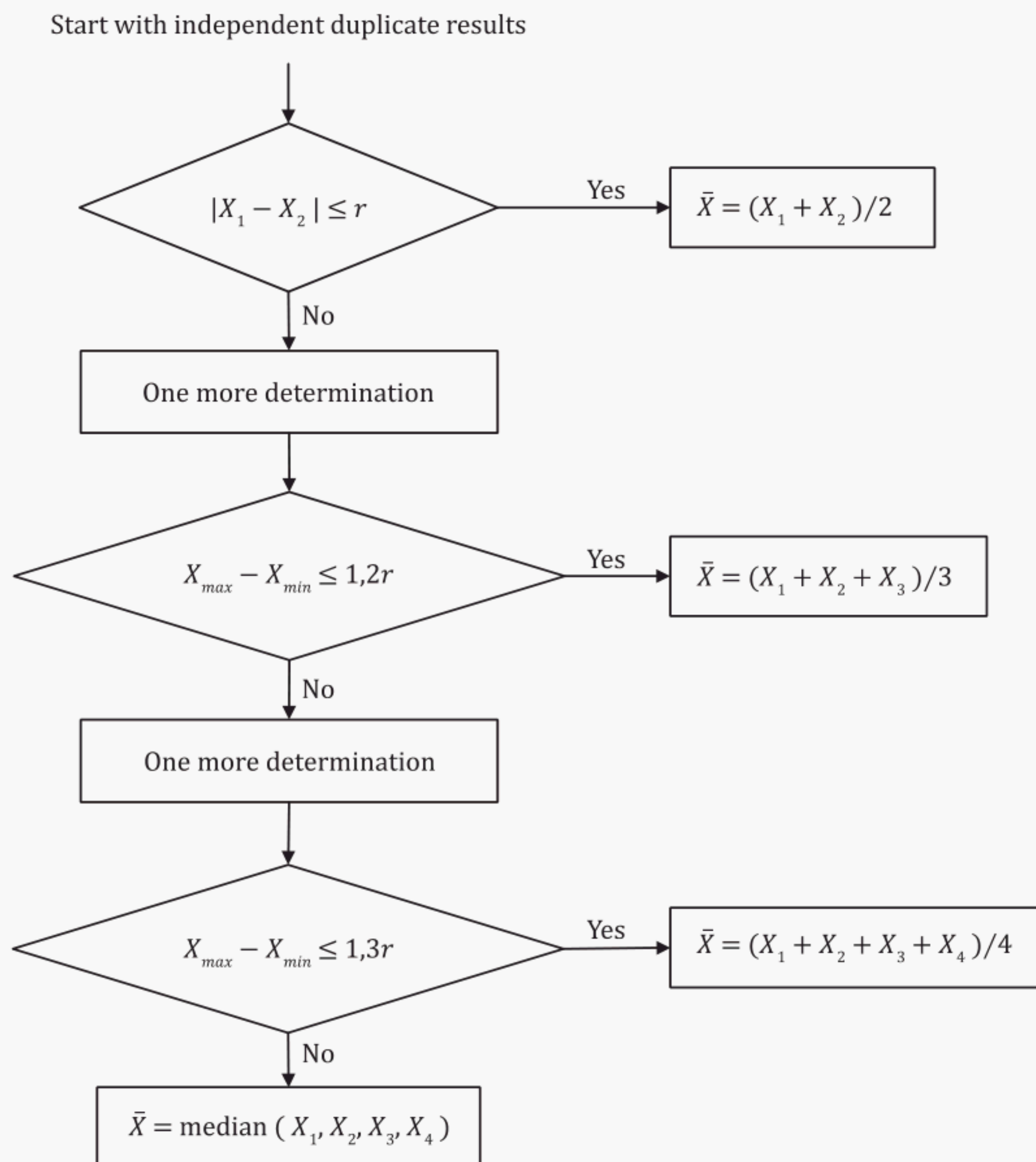
## **12 Test report**

The test report shall contain the following information:

- a) sample identification;
- b) date of issue of the test report;
- c) a reference to this document, i.e. [ISO 13546:2021](#);
- d) the mercury content of the sample, in  $\mu\text{g/g}$ ;
- e) any characteristics noticed during the determination and any operations not specified in this document which have possibly influenced the results, for either the test sample or the CRM.

## Annex A (normative)

### Procedure for obtaining the final results



**Figure A.1 — Flowsheet of the procedure for the acceptance of analytical values for the test sample**



## Bibliography

- [1] ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

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