भारतीय मानक **IS 18458 : 2023**

Indian Standard

आभूषण एवं बहुमूल्य धातुओ — ईडी-एक्सआरएफ परीक्षण — परीक्षण की पद्धति

Jewellery and Precious Metals — Ed-XRF Test — Method of Test

ICS 39.060

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

November 2023 **Price Group 6**

Precious Metals Sectional Committee, MTD 10

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

ED-XRF (Energy Dispersive X-Ray Fluorescence) is an analytical technique used to determine the composition of metal alloys. A primary X-ray beam is focused on the sample causing energetic interaction. The emitted X-ray photons are detected and measured by the X-ray instrument. Each different element within the sample emits photons with characteristic energy properties and by counting these specific photons it is possible to calculate the concentrations of each element within the sample simultaneously.

ED-XRF is primarily used in the assaying and hallmarking centres to ensure the homogeneity of the lot received and to determine the presence/absence of prohibited elements.

Basically ED-XRF is a quick, reproducible and non-destructive test method. However, due to complex physical factors and interaction processes such as information depth, geometry, matrix effects and others, the representativeness of measurements, without an adequate sample preparation and/or without a matrix corrected quantification algorithm, is very poor and can lead to grave systematic errors.

For formulating this standard assistance has been drawn from ISO standard ISO 23345 : 2021 ‘Jewellery and precious metals — Non-destructive precious metal fineness confirmation by ED-XRF’. The ISO standard proposes a non-destructive method, which allows validating a declared fineness. The method has been included as Annex A to this document. However, in the foreword of the ISO standard it also mentioned that the standard is not suitable for the regulatory hallmarking application. Because of the inherent higher uncertainty associated with ED-XRF measurements, some results might be inconclusive. Also from the method specified in the ISO standard a modification has been done in this standard, wherein a destructive method has been included. In this the sample is prepared as in case of fire assay, however instead of fire assay, XRF is done on prepared sample.

The test method specified in the annexure can be used by the XRF manufacturers/R&D institutes/laboratories to design a fit-for-purpose instrument, reference material, qualified staff and written procedures to achieve the desired level of measurement uncertainty that is, ± 0.5 ppt for gold jewellery/artefacts and ± 1.0 ppt. For silver jewellery artefacts.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

JEWELLERY AND PRECIOUS METALS — ED-XRF TEST — METHOD OF TEST

1 SCOPE

This document describes a non-destructive method of test to ensure homogeneity of the major element in the lot and to detect presence of the prohibited elements, by ED-XRF (energy dispersive X-ray fluorescence).

The document also describes a nondestructive method to verify (confirm) the precious metal fineness and of finished and semi-finished jewelry item(s), considered homogeneous by ED-XRF and a destructive method wherein sample is prepared as in case of fire assay, however instead of fire assay, XRF is done on prepared sample (*see* Annex A).

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 1417 : 2016	Gold and gold alloys, jewellery/artefacts — Fineness and marking — Specification (*fourth revision*)
IS 1418 : 2009	Determination of gold in gold bullion, gold alloys and gold jewellery/artefacts — Cupellation (fire assay) method (*third revision*)
IS 2112 : 2014	Silver and silver alloys, jewellery/artefacts — Fineness and marking — Specification (*third revision*)
IS 15820 : 2009	General requirements for competence of assaying and hallmarking centres
IS 16179 (Part 1) : 2014/ISO 10576-1 : 2003	Statistical methods — Guidelines for the evaluation of conformity withspecified requirements: Part 1 General principles
IS/ISO/IEC 17025 : 2017	General requirements for the competence of testing and calibration laboratories (*second revision*)
IS/ISO 17034 : 2016	General requirements for the competence of reference material producers

3 TERMS AND DEFINITIONS

3.1 Energy-Dispersive X-Ray Fluorescence (ED-XRF) — X-ray fluorescence elemental analysis method where all elements in the sample are simultaneously excited and where the intensities of the characteristic fluorescence radiation emitted by each element are quantified by separating the differential energies specific to each element.

3.2 Spot — Target area on the sample where the X-ray beam strikes the surface.

3.3 Calibration Standard — Material with appropriate homogeneity whose exact composition is known and which can be used to calibrate the ED-XRF (*see* **3.1**) instrument.

3.4 Reference Material (RM) — Material produced with certified reference with appropriate homogeneity whose exact composition is known and which has been established to be fit for ED-XRF (*see* **3.1**) measurement or material certified by IS 17025 accredited laboratory with valid established method and fit for ED-XRF measurement.

3.5 Major Element — Main precious metal element of interest in the alloy whose concentration is to be verified, as well as any other element whose concentration is higher.

Example:

> In the case of gold 375 ppt alloy with 500 ppt of copper, both gold and copper are the major elements.

3.6 Minor Element — Any non-major element (*see* **3.5**) in the alloy whose concentration is at or above 1 ppt.

3.7 Trace Element — Element present in the alloy whose concentration is below 1 ppt.

NOTE — Trace elements are not taken into account for calibration. The sum of all trace elements shall be below 1 ppt.

3.8 Coating — Thin layer of covering on the surface of the item.

3.9 Prohibited Elements — Elements which should not be present in the precious metals.

NOTE — The prohibited elements are mentioned in the respective product standards, IS 1417 and IS 2112.

4 APPARATUS

4.1 ED-XRF

a) X-ray tube — adapted for precious metals analysis, preferably minimum rating 50 kV and 40 W;

b) Primary filter adapted for precious metals analysis;

c) Collimator preferably 2 collimators, at least one < 1 mm and one ≥ 1 mm;

d) Camera — with magnification of the image of the measuring area;

e) Detector Si-PIN, SDD;

f) Energy resolution — ≤ 160 eV (for K_α of Mn line), the lower eV value the better; and

g) Detectable elements — Z = 22 (Ti) to Z = 92 (U).

5 PROCEDURE

5.1 Calibration

5.1.1 *Calibration Standards*

5.1.1.1 Calibration of major element present in the alloy to be done with calibration standards with the following features shall be used:

a) Flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

b) Sufficiently thick (at least 0.5 mm); and

c) Homogeneous:

    1) Known exact composition; and

    2) Composition of major elements matching approximatively the sample to be analysed, trace elements may be ignored.

5.1.1.2 Calibration of minor/trace elements present in the alloy to be done with calibration standards with the following features shall be used:

a) Flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

b) Sufficiently thick (at least 0.5 mm); and

c) Homogeneous:

    1) Known exact composition.

NOTE — For calibration for presence of minor elements and traces elements (prohibited elements specified in IS 1417/IS 2112 such as iridium, ruthenium, osmium, cadmium, lead) the certificate of calibration provided by the supplier of XRF may be accepted till a certified reference material for each of these elements is developed. The XRF supplier shall provide the detection range of each element specified in IS 1417/ IS 2112.

5.1.2 *Method Calibration*

The ED-XRF instrument shall be specifically calibrated for each sample to be analyzed before start of testing.

5.2 Verification

5.2.1 *Reference Material*

To verify the calibration, a reference material with the following features shall be used:

a) Flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

b) Sufficiently thick (at least 0.5 mm);

c) Homogeneous;

d) Known exact composition;

e) Not being used as calibration standard;

f) Certified reference material (CRM) and materials prepared under the IS/ISO 17034 accreditation are preferred; and

g) The composition of the reference material should be matching as far as practicable that of the sample for major elements there shall be no more than 10 ppt absolute difference between the concentration in the sample and in the reference material;

NOTE — For minor elements and traces, till a reference material is developed the certificate of the manufacturer stating the capability of the XRF machine to test the prohibited elements up to a level of 0.01 percent shall be available.

5.2.2 *Method Verification*

Before each batch analysis, the calibration shall be controlled by analyzing the reference material.

The reference material shall be analyzed at least on 5 different positions. The analysis time for each replicate shall not exceed the time used for the calibration. The standard deviation obtained for the measurements in the reference material shall not exceed 2 ppt for the major element. The calibration is verified by comparing the fineness measured for the reference material (obtained by the mean value of the analyses) and its declared value. The difference, Δ_{RM}, between those two values shall not be greater than 2 ppt and will be taken into the uncertainty evaluation of the method.

If the standard deviation for the measurements or the difference between the fineness measured for the reference material and its declared value are outside the tolerances, the calibration shall be repeated.

The reference material shall be analyzed at regular intervals under the same conditions to monitor the stability of the instrument.

5.3 Analysis

5.3.1 *Sample Preparation*

Samples to be analyzed may be cleaned (typically with alcohol, but also possibly by mechanical polishing of a very thin layer) and prepared for measurement for better accuracy and to exclude potential systematic errors. For plated articles, the surface may be slightly ground to remove the top layer.

NOTE — Repeatability and accuracy is improved if the surface to be analysed is prepared or scraped.

5.3.2 *Sample Analysis*

The sample shall be analyzed at least on 3 different positions. At least one of the analysis shall be done on solder part. The number of positions to be increased depending upon the size of articles and number of different parts soldered together.

For the major element, the standard deviation obtained for each measurement, σ_{sample}, shall not exceed 3 ppt.

If those standard deviations are higher than the tolerances, the result is not valid and the analysis has to be performed again.

Guidelines for XRF analysis for gold (PGM mixed) samples is given in Annex B.

6 CALCULATION AND EXPRESSION OF THE RESULTS

6.1 *Calculation*

The fineness of the sample, in part per thousand (ppt), is directly obtained from the ED-XRF software and shall be the mean of all measurements performed. For the major element difference Δ_{RM} as mentioned in **10.2.2** needs to be suitably incorporated in the test result. For the minor elements/trace elements the value obtained shall be directly reported.

6.2 *Uncertainty*

Uncertainty is evaluated by taking into account the standard deviations obtained on the reference material and the sample measurements, as well as the difference between the fineness measured for the reference material and its declared value (taking hence into account the uncertainty linked to the calibration).

$$U_{+} = \max\left(2 \times \sqrt{\sigma_{RM}{}^{2} + \sigma_{sample}{}^{2}} - \Delta_{\mathrm{RM}};\ 0\right)$$

$$U_{-} = \max\left(2 \times \sqrt{\sigma_{RM}{}^{2} + \sigma_{sample}{}^{2}} + \Delta_{RM};\ 0\right)$$

where

σ_{RM} = standard deviation on the reference material measurements;

σ_{sample} = standard deviation on the sample measurements; and

Δ_{RM} = difference between the fineness measured for the reference material and its declared value.

7 TEST REPORT

The test report shall include at least the following information:

a) Identification of the sample including source, date of receipt, and form of sample;

b) Method used by reference to this document;

c) Result of the test;

d) If relevant, any deviations from the method specified in this document;

e) Any unusual features observed during the determination;

f) Date of test;

g) Identification of the laboratory carrying out the test; and

h) Signature of the laboratory manager and operator.

ANNEX A

(*Clause* 1 *and Foreword*)

METHOD TO VERIFY (CONFIRM) THE PRECIOUS METAL FINENESS AND OF FINISHED AND SEMI-FINISHED JEWELRY ITEM(S), CONSIDERED HOMOGENEOUS BY ED-XRF

A-1 PRINCIPLE

A specific calibration is required for each alloy composition. This calibration is obtained from at least 3 standards whose composition is matching approximately the sample to be analyzed. A reference material is analyzed to verify the calibration. Samples can then be analyzed and the precious metal fineness verified.

This document is not suitable for any coated items the coating shall be removed by a suitable means if the fineness determination is necessary on a coated item.

A-2 APPARATUS

A-2.1 ED-XRF — with the following specifications:

a) X-ray tube adapted for precious metals analysis, minimum rating 50 kV and 40 W;

b) Primary filter adapted for precious metals analysis;

c) Collimator minimum 2 collimators, at least one < 1 mm and one ≥ 1 mm;

d) Camera with magnification of the image of the measuring area;

e) Detector SDD;

f) Energy resolution: ≤ 160 eV (for K_α of Mn line), the lower eV value the better; and

g) Detectable elements: Z = 22 (Ti) to Z = 92 (U).

A-3 PROCEDURE

A-3.1 Calibration

A-3.1.1 *Calibration Standards*

To calibrate the instrument for a specific alloy, calibration standards with the following features shall be used:

a) Flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

b) Sufficiently thick (at least 0.5 mm);

c) Homogeneous;

d) Known exact composition;

e) Composition of major and minor elements matching approximatively the sample to be analyzed trace elements may be ignored a minimum of 3 standards shall be used, with the major element(s) composition covering a range up to 50 ppt; and

f) Minor elements below 5 ppt do not need to be present in the standards but shall be declared in the calibration they may be calibrated using the instrument maker's calibration.

Example:

> For the measurement of an alloy with Au = 583 ppt, the fineness of Au in the calibration standards may be 563 ppt, 583 ppt and 603 ppt. Au = 750 ppt, the fineness of Au in the calibration standards may be 730 ppt, 750 ppt, and 770 ppt. For the measurement of an alloy with Au = 916 ppt, the fineness of Au in the calibration standards may be 896 ppt, 916 ppt and 936 ppt.

A-3.1.2 *Method Calibration*

The ED-XRF instrument (*see* **A-2.1**) shall be specifically calibrated for sample of each fineness to be analyzed. Each standard shall be analyzed at least on 5 different positions. The standard deviation obtained for the measurements of each calibration standard shall not exceed 1.2 ppt for the major element.

Inter-element interference corrections should be applied.

Analysis of the calibration standards, the reference material and the samples shall be performed using the same collimator size and using the same parameters (tube voltage, current etc). The largest possible collimator compatible with the sample target area should be used.

A-3.2 Verification

A-3.2.1 *Reference Material*

To verify the calibration, a reference material with the following features shall be used:

a) Flat and clean surface of suitable size, with at least a diameter 5 times larger than the collimator used for measurement;

b) Sufficiently thick (at least 0.5 mm);

c) Homogeneous;

d) Known exact composition;

e) Not being used as calibration standard;

f) Certified reference material (CRM) and materials prepared under the IS 17034 accreditation are preferred;

g) Material certified by IS 17025 accredited laboratory with valid established method; and

h) The composition of the reference material should be matching as far as practicable that of the sample for major elements there shall be no more than 10 ppt absolute difference between the concentration in the sample and in the reference material for minor elements, that difference shall be no more than 20 ppt absolute difference trace elements may be ignored.

   Example:

   > If an alloy with a 750 ppt Au, 240 ppt Ag, 10 ppt Cu is to be analyzed, the concentrations in the reference material are between 740 ppt and 760 ppt for Au, between 260 ppt and 220 ppt for Ag and between 30 ppt and 0 ppt for Cu.

A-3.2.2 *Method Verification*

Before each batch analysis, the calibration shall be controlled by analyzing the reference material.

The reference material shall be analyzed at least on 5 different positions. The analysis time for each replicate shall not exceed the time used for the calibration. The standard deviation obtained for the measurements in the reference material shall not exceed 1.2 ppt for the major element. The calibration is verified by comparing the fineness measured for the reference material (obtained by the mean value of the analyses) and its declared value. The difference, Δ_{RM}, between those two values shall not be greater than 1.2 ppt and will be taken into the uncertainty evaluation of the method.

If the standard deviation for the measurements or the difference between the fineness measured for the reference material and its declared value are outside the tolerances, the calibration shall be repeated.

The reference material shall be analyzed at regular intervals under the same conditions to monitor the stability of the instrument.

A-3.3 Analysis

A-3.3.1 *Sample Preparation*

Samples to be analyzed may be cleaned (typically with alcohol, but also possibly by mechanical polishing of a very thin layer) and prepared for measurement for better accuracy and to exclude potential systematic errors. In case of doubt of plating on the article, the articles may be slightly grinded to remove the top layer and then checked for XRF.

Sample preparation can also be done as per IS 1418 for fire assay method and destructive testing undertaken, in this the sampling is done by scrapping/cutting in accordance with the sampling plan as described in IS 15820. For this:

a) A sample of 500 mg to one g of materials shall be taken;

b) Molten at regular temp which industry use under reducing condition is a furnace;

c) Homogenized during melting;

d) Cooling to room temperature;

e) Rolling to the thickness 0.12 mm to 0.15 mm and;

f) Scraping of surface using buffing tool with an area at least 5 times larger than the spot size; and

g) Testing the strip by XRF.

   NOTE — Repeatability and accuracy is improved if the surface to be analyzed is prepared or scraped.

A-3.3.2 *Sample Analysis*

The sample shall be analyzed at least on 3 different positions. Each position is analyzed with a minimum of 3 replicates. The analysis time for each replicate shall be equal to the time used for the reference material.

For the major element, the standard deviation obtained for each set of replicates shall not exceed 1.2 ppt and the standard deviation obtained for each measurement, σ_{sample} shall not exceed 1.2 ppt.

If those standard deviations are higher than the tolerances, the result is not valid and the analysis has to be performed again.

A-3.4 Calculation and Expression of the Results

A-3.4.1 *Calculation*

The fineness of the sample, in part per thousand (ppt), is directly obtained from the ED-XRF software and shall be the mean of all measurements performed.

A-3.4.2 *Uncertainty*

Uncertainty is evaluated by taking into account the standard deviations obtained on the reference material and the sample measurements, as well as the difference between the fineness measured for the reference material and its declared value (taking hence into account the uncertainty linked to the calibration).

$$U_{+} = \max\left(2 \times \sqrt{\sigma_{RM}^{2} + \sigma_{sample}^{2}} - \Delta_{RM};\ 0\right)$$

$$U_{-} = \max\left(2 \times \sqrt{\sigma_{RM}^{2} + \sigma_{sample}^{2}} + \Delta_{RM};\ 0\right)$$

where

σ_{RM} = standard deviation on the reference material measurements;

σ_{sample} = standard deviation on the sample measurements; and

Δ_{RM} = difference between the fineness measured for the reference material and its declared value.

A-3.4.3 *Result Interpretation*

Fineness validation of the sample is based on IS 16179 (Part 1). If the result falls in the inconclusive range, the measurement shall be performed again. Should the final result still be inconclusive and below the declared fineness, a destructive analysis as per IS 1418 allowing for higher precision may be performed.

The ranges are shown in Fig. 1 and defined as:

Assurance of non-conformity	$F_{measured} < F_{declared} - U_{+}$
Inconclusive range	$F_{declared} - U_{+} \leq F_{measured} \leq F_{declared} + U_{-}$
Assurance of conformity	$F_{measured} > F_{declared} + U_{-}$

where

$F_{measured}$ = measured fineness of the sample

$F_{declared}$ = declared fineness by the manufacturer

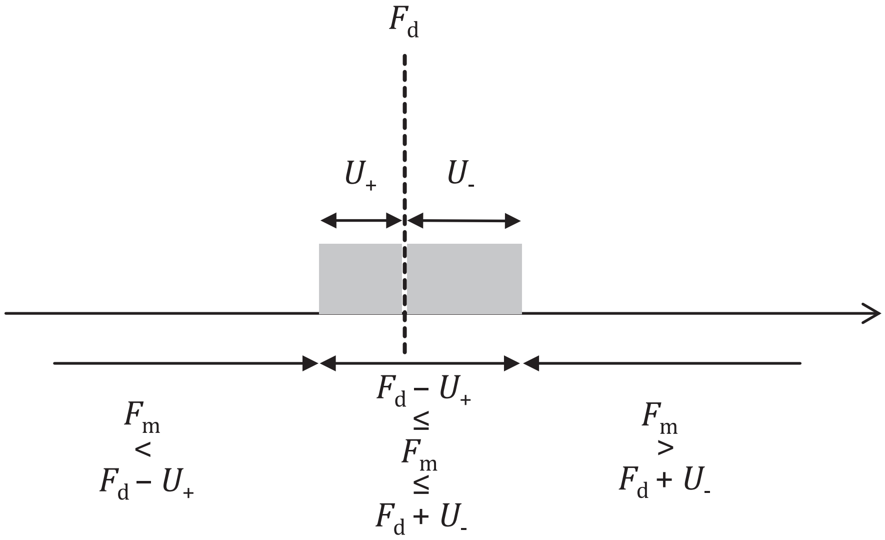

FIG. 1 RANGES FOR RESULT INTERPRETATION

A-3.4.4 If approved by the inspecting authority**,** this can be used as a quantitative method provided if the internal method is accredited to IS/ISO/IEC 17025 and the measurement uncertainty is equal to or better than that of already accepted methods. This means that the measurement uncertainty of the X-ray spectrometric method should not exceed the following limits:

a) Gold ± 0.5 ppt; and
b) Silver ± 1.0 ppt.

A-3 TEST REPORT

The test report shall include at least the following information:

a) Identification of the sample including source, date of receipt, and form of sample;
b) Method used by reference to this document;
c) Result of the test conforming, non-conforming or inconclusive result;
d) If relevant, any deviations from the method specified in this document;
e) Any unusual features observed during the determination;
f) Date of test;
g) Identification of the laboratory carrying out the test; and
h) Signature of the laboratory manager and operator.

ANNEX B

(*Clause* 5.3.2)

GUIDELINES FOR XRF ANALYSIS FOR GOLD (PGM MIXED) SAMPLES

B-1 Normally Ir, Ru and Os belongs to the platinum group member of metals. When mixed with gold, these elements never go in alloyed state and disperse in the gold unevenly so optimum sampling should be considered for comprehensive analysis.

B-2 However, accuracy of XRF analysis depends on attenuation-enhancement correction procedure used to correct matrix effects caused by concomitant elements and the reference standards used in calibration procedures.

B-3 Using XRF by a suitable sampling procedure it’s possible to detect the presence of Ir, Ru and Os though to accurately predict the percentage of these elements is still difficult as its dispersed in the metal and do not form a solid solution.

B-4 The rational approach to minimize the chances of error in detection of iridium, is to draw a sampling plan spreading predominantly at center and bottom side of sample at multiple location.

B-5 In case of ruthenium containing samples, chances of detection are higher on top side because being lighter Ru will try to be more on top surface.

B-6 Being heavier, Ir and Os have more probability of being at bottom side, but still possibility of detection is unpredictable. By increasing no of analysis at multiple spots, detection probability can be increased.

B-7 In case of one or two traceable metal contains less than the 10 ppt and the same metal is not calibrated by machine there is a possibility of showing other metal due to interference effect. For example, if galium metal present in alloy less than 10 ppt and the same metal is not calibrated by XRF machine there is the possibility of showing other metal like Iridium. In such cases the graph analysis and kilo electron volt analysis will support to confirm the metal.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Precious Metals Sectional Committee, MTD 10

Organization	*Representative(s)*
Geological Survey of India, New Delhi	Shri Paravjeet Singh **(*Chairperson*)**
All India Gems and Jewellery Trade Federation, Mumbai	Shri D. D. Karel Shri Suresh I. Dhruv (*Alternate*)
Association of Gold Refineries and Mints, New Delhi	Shri Anil C. Kansara
Bhartiya Swarnkar Sangh, Jaipur	Shri Duli Chand Karel Shri Prem Kumar Soni (*Alternate*)
CGR Metalloys Private Limited, Kochi	Shri James Jose Shri Joseph K. James (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	Dr C. J. Shishoo
Consumer Guidance Society of India, Mumbai	Dr Sitaram Dixit Dr M. S. Kramath (*Alternate*)
CSIR − Indian Institute of Toxicology Research, Lucknow	Shri R. C. Murthy
CSIR − National Physical Laboratory, New Delhi	Dr N. Vijayan
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Gem and Jewellery Export Promotion Council, Mumbai	Shri Sabyasachi Ray
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MMTC − PAMP India Private Limited, New Delhi	Shri Pankaj Shri Ankur Goyal (*Alternate*)

Organization	*Representative(s)*
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World Gold Council, New Delhi	SHRI P. R. SOMASUNDARAM
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Member Secretary
SHRI SHIV PRAKASH
SCIENTIST 'D'/JOINT DIRECTOR
(METALLURGICAL ENGINEERING), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

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