भारतीय मानक ब्यूरो /BUREAU OF INDIAN STANDARDS

(पूर्वी क्षेत्रीय प्रयोगशाला)/Eastern Regional Laboratory

हमारा संदर्भ/Our Ref: ERL(C)/IS 4032 : 1985 01/02/2023

विषय / Subject: Comments on method of Chemical Analysis of Hydraulic Cement

इस संबंध में प्रस्तावित बदलाव(ओं) पर विचार करने के लिए सी ई डी तथा संबंधित तकनीकी समिति द्वारा निम्नलिखित टिप्पणियों पर ध्यान दिया जाए:

क्र. सं. Sl.No	Clause no of IS	**प्रस्तावित बदलाव/ Proposed change**	**वजह/Justification**
1	*Cl*. 7.5 (Amend No. 2)	Replace the existing text with "The method for determining chloride content in **Portland Pozzolana Cement** shall be the same as described in 4.13".	Clause 7 of IS 4032, refers to chemical analysis of Portland Pozzolana Cement, accordingly clause 7.5 should be about estimation of Chloride analysis in Portland Pozzolana Cement. But clause 7.5 of Amend No. 2 to IS 4032:1985, states that the method for determining chloride content in Portland slag cement shall be the same as described in 4.13, which seems to be a typographical error.
2	*Cl.* 4.8	Add a new method in clause 4.8.2 after method 2: Method 3 (EDTA Method using EBT after removal of CaO by gravimetric method *Cl.* 4.7.1) for details please see Annexure-A.	While estimating MgO by Method 2 (EDTA method) given in Cl. 4.8.2 of IS 4032:1985 using of Thymol Phthalexone indicator for detection of end point, difficulties have been encountered in identifying the exact end point. It is seen that the change of colour at end point is not sharp which may lead to erroneous detection of end point as a result chances of error could be high for people not well conversant with the method or lacking high level of expertise. From methods specified in Indian standards for estimation of Magnesium in Water and Waste water as well as papers published in International journals (see references below) it has been observed that use of Eriochrome Black T indicator is also suitable for estimation of Magnesium, including for Cement samples. Trials have been performed on cement CRMs for PPC (BND 5052A), PSC (BND 5053), OPC (1012M) having traceability to NCCBM (Annex-B). It has been observed that the use of EBT as

			indicator gives and clear and sharp end point which is easily detectable. The results when compared to the declared value of CRMs shows that they are equally repeatable and reproducible (Annex-C). References: 1. Asrar Adil El-gray and Farough Bakheit, Mohamed Ahmed (2016), Determination of Major Oxides Percentages in Portland Cement of Some Sudanese Cement Manufactories, *American Journal of Applied Chemistry Vol 4, No 1, 2016, pp 14-17*. 2. Sufian Rasheed, Miamat Ullah and Amir Ullah (2020), Chemical analysis of some Pakistani Portland cement/clinker and their compliance with ASTM standards, *European Journal of Chemistry vol 11, No 3, 2020, pp 194-197* 3. IS 3025(Part-46), Methods of sampling and test (Physical and Chemical) for water and wastewater, Part 46 Magnesium (This method is applicable when Iron and Aluminium has not separated from the test solution).

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Annex- A

Take the filtrate set aside in *Cl* 4.7.1 (remaining after CaO precipitation) and make up the volume in a 250 ml volumetric flask. Take 50 ml or suitable aliquot of the solution in a conical flask and add 10 ml of pH 10 buffer solution (*Cl* 4.1.13) till solution pH is 10 ± 0.1 followed by addition of 5 ml of Triethanolamine (1:1 by volume, Cl 4.1.2.8).
Add 3-4 drops of Eriochrome Black T indicator and titrate with 0.01M EDTA solution till colour changes to pure blue end point free from violet tinge.

Calculations —
Calculate the percentage of MgO as given below:

1 ml of 0.01 M EDTA = 0.4032 mg of MgO

$$\text{Magnesium oxide (MgO) percent} = \frac{0.04032 \times 5^* \times V}{W}$$

where,
V = Volume of EDTA used in this titration in ml and W = Weight of the sample in g.

*Note: Considering 50 ml of aliquot pipetted from 250 ml test solution.

भारतीय निर्देशक द्रव्य

BHARATIYA NIRDESHAK DRAVYA

(INDIAN REFERENCE MATERIAL)

Certificate

BND® 5052A

PORTLAND POZZOLANA CEMENT STANDARD (CHEMICAL PARAMETERS)

Certificate Number: BND® 5052A/312

This Bharatiya Nirdeshak Dravya, BND®5052A, an Indian Certified Reference Material (CRM), produced by National Council for Cement and Building Materials (NCB), India and it is intended to use as a primary standard for calibration of instruments and validation of method for the characterization of the measurand for the analysis of Portland Pozzolana Cement (PPC). One set of BND®5052A consists of 4 sealed vials, each containing approximately 5g of powdered PPC. The analysis of LOI, MgO, SO_3, IR and Cl have been determined as per IS 4032:1985. Estimation of alkalies have been done as per NCB standard procedure, MS-13-2010 (validated method).

Certified Value: The parameters have been analysed as w/w of the total material and reported in percent. The certified value is determined by National Council for Cement and Building Materials and further verified by CSIR-National Physical Laboratory, New Delhi, National Metrology Institute (NMI) of India. The associated measurement uncertainty was calculated at 95% confidence level with coverage factor k=2, considering major sources of uncertainty including measurement replication, instrument background, mass taken, possible heterogeneity and stability factors according to ISO GUM Guide [1] and ISO Guide 35 [2] as given in Table 1.

Table1: Certified values and associated measurement uncertainties of chemical parameters of PPC

CONSTITUENT	CERTIFIED VALUE (PERCENT BY WEIGHT)	EXPANDED UNCERTAINTY (Coverage Factor k=2)
LOI	1.25	±0.02
MgO	2.27	±0.05
SO_3	1.91	±0.02
IR	24.5	±0.38
Na_2O	0.49	±0.04
K_2O	0.77	±0.03
Cl	0.012	±0.002

Development of BND® 5052A has been carried out at NCB, as per ISO 17034:2016 [3] and testing is performed with compliance as per ISO/IEC 17025:2017 [4]. The certified values of the parameter given in the certificate are the best estimate of true value within the stated uncertainties. The certification of the BND® 5052A is also complying the requirements of ISO Guide 31 [5].

Nature of Material: Non-hazardous

Traceability: The BND®5052A is the metrological traceable to SI units through Indian national measurement standards at CSIR-National Physical Laboratory. All measurements have been carried out to establish traceability through an unbroken chain of comparisons, having stated uncertainty.

Expiration of Certification: The certification of BND®5052A is valid, within the measurement uncertainty specified, until **03 January 2025**, provided the BND is handled and stored in accordance with instructions given in this certificate. This certification is nullified if the BND® is damaged, contaminated or otherwise modified.

Maintenance of Certification: NCB will monitor the certified values of BND®5052A over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of certification, NCB will notify the purchaser.

Instructions for Storage and Use and Precautions for Handling:

1. BND® 5052A CRM should be used in controlled conditions, using calibrated equipment, by trained personnel.
2. The CRM is to be dried at 105±5°C for two hours before use.
3. After drying, the material should be protected from atmospheric moisture and used immediately. Leftover portion in a vial, if any, should not be used at a later date.
4. The material should be stored under non-humid conditions to avoid any ingress of moisture/CO_2.
5. Record of usage of CRM and the results must be maintained.

References:

1. JCGM 100.2008-Evaluation of measurement data- Guide to the expression of uncertainty in measurement.
2. ISO Guide 35-Reference Materials - General and statistical principles for certifications;.
3. ISO 17034:2016 - General Requirements for the competence of Reference Material Producers.
4. ISO/IEC 17025:2017- General Requirements for the competence of testing and calibration laboratories.
5. ISO Guide 31 - Reference Materials - Contents of certificate, labels and accompanying documentation.

Date of Certification: 04/01/2020

Document Version: BND/NPL/NCB/2020/2.0

Certificate issued by

Accredited as per ISO 17034:2016 vide Certificate No. RC-1016

Dr R P Pant
Head, BND Division
CSIR-National Physical Laboratory
Dr K S Krishnan Marg
New Delhi-110012

P N Ojha
Head of Centre-CQC
National Council for Cement and Building Materials
Ballabgarh-121004, Haryana

- In case of any query regarding this BND® please contact Dr. R. P. Pant, Head (BND Division), CSIR-NPL (NMI of India), New Delhi-110012.
- This certificate shall not be reproduced without written approval from CSIR-NPL, New Delhi.
- Responsibility for issue and release of this certificate lie with NCB.

For Further Information, Please Contact: National Council for Cement and Building Materials, 34 Km. Stone, Delhi-Mathura Road (NH-2), Ballabgarh, Haryana – 121004, India; phone +91-129-4192239/303, +91-129-2242051, +91-129-4192222; Fax +91-129-2242100, +91-129-2246175 Email: ncb.cqc@gmail.com, cqcb@ncbindia.com, nccbm@ncbindia.com, website: www.ncbindia.com

भारतीय निर्देशक द्रव्य

INDIAN REFERENCE MATERIAL

(BHARATIYA NIRDESHAK DRAVYA, BND)

BND Certificate

BND® 5053

PORTLAND SLAG CEMENT STANDARD

(CHEMICAL PARAMETERS)

Certificate Number: BND® 5053/1.0/19

This Bharatiya Nirdeshak Dravya, BND® 5053, is an Indian Certified Reference Material of Portland Slag Cement Standard produced by National Council for Cement and Building Materials (NCB), India. It is intended to be used as a primary standard for calibration of instruments and validation of method for characterization of the measurand for analysis of Portland Slag Cement. One set of BND® 5053 consists of 4 sealed vials, each containing approximately 5g of powdered Portland Slag Cement. The estimation of Loss on Ignition (LOI), SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO, SO_3, Insoluble Residue (IR), Cl and Sulphide Sulphur (S) has been carried out as per IS 4032: 1985. The estimation of Mn_2O_3, TiO_2, P_2O_5 has been carried out as per IS 12423:1988. The analysis of Alkalis has been carried out as per NCB standard procedure, MS-13-2010 [1]. The parameters have been analysed as w/w of the total material and reported in percent. The certified value is assigned by National Council for Cement and Building Materials and further ascertained by CSIR-National Physical Laboratory, New Delhi, National Measurement Institute (NMI) of India. The certified value of Portland Slag Cement along with the associated uncertainty was calculated at 95% confidence level with coverage factor k=2, considering major sources of uncertainty including measurement replication, instrument background, mass taken, possible heterogeneity and stability factors according to ISO GUM Guide 100:2008 [2] and ISO Guide 35:2017 [3] as given in Table 1.

Table1: Certified value

CONSTITUENT (PERCENT BY WEIGHT)	CERTIFIED VALUE	EXPANDED UNCERTAINTY k=2
LOI	3.13	±0.12
SiO_2	25.12	±0.14
Fe_2O_3	2.48	±0.09
Al_2O_3	9.42	±0.12
CaO	51.40	±0.14
MgO	2.73	±0.21
SO_3	3.12	±0.07
IR	0.19	±0.09
Cl	0.013	±0.007
S	0.13	±0.03
Na_2O	0.38	±0.02
K_2O	0.49	±0.05
TiO_2	0.75	±0.05
Mn_2O_3	0.20	±0.04
P_2O_5	0.05	±0.01

Traceability: The BND® 5053 is the metrological traceable to SI units through national measurement standards at CSIR-National Physical Laboratory (National Measurement Institute of India). All measurements have been carried out to establish traceability through an unbroken chain of measurements having stated uncertainty.

Nature of Material: Non-hazardous

Homogeneity: Ten vials of BND® 5053 were selected and homogeneity assessment was carried out within and between these bottles as per ISO Guide 35:2017 [3].

Instructions for Storage, Usage and Precautions for Handling:

1. BND® 5053 should be used in controlled conditions, using calibrated equipment, by trained personnel.
2. The BND® is to be dried at 105±5°C for two hours before use. The minimum quantity of the sample is recommended for the analysis as given in the standard as per the standard method.
3. After drying, the material should be protected from atmospheric moisture and used immediately. Leftover portion in a vial, if any, should not be used at a later date.
4. The material should be stored under non-humid conditions to avoid any ingress of moisture/CO_2.

Expiration of Certification: The certification of BND® 5053 is valid, within the measurement uncertainty specified, until **31st December 2026**, provided the BND is handled and stored in accordance with instructions given in this certificate. This certification is nullified if the BND is damaged, contaminated or otherwise modified.

Maintenance of Certification: NCB will monitor the certified values of BND® 5053 over the period of its certification. If substantial technical variation is observed that deviate the certified value before the expiration of validity, NCB will notify the customer.

Disclaimer: Development of BND® 5053 has been carried out at NCB, accredited as per ISO 17034:2016 [4] and testing is performed with compliance as per ISO/IEC 17025:2017 [5]. The certification of the BND® 5053 is also complying to the requirements of ISO Guide 31: 2015 [6]. However, it assumes no liability with respect to or for damages resulting from the misuse of any information, material, apparatus, method or process disclosed in this certificate or any warranties with respect to the material safety. The certified values of the parameters given in this certificate are the best estimates of true values within the stated uncertainties.

References:

1. MS-13-2010- NCB monograph for analysis of Alkalis.
2. JCGM 100.2008- Evaluation of measurement data- Guide to the expression of uncertainty in measurement
3. ISO Guide 35:2017- Reference Materials - General and statistical principles for certifications.
4. ISO 17034:2016- General Requirements for the competence of Reference Material Producers.
5. ISO/IEC 17025:2017- General Requirements for the competence of testing and calibration laboratories.
6. ISO Guide 31-2015- Reference Materials - Contents of certificate, labels and accompanying documentation.

Date of Certification: 01/01/2022

Document Version: BND/CSIR-NPL/NCB/2022/1.0

Accredited as per ISO 17034:2016 vide Certificate No. RC-1016

Certificate issued by:

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- In case of any query regarding this BND® 5053 please contact Dr Nahar Singh, Head (BND Division), CSIR-NPL (NMI of India), New Delhi-110012.
- This certificate shall not be reproduced without written approval from CSIR-NPL, New Delhi.
- Responsibility for issue and release of this certificate lie with NCB.

For Further Information, Please Contact: National Council for Cement and Building Materials, 34 Km. Stone, Delhi-Mathura Road (NH-2), Ballabgarh, Haryana – 121004, India; Phone +91-129-4192239/303, +91-129-2242051, +91-129-4192222; Email: ncb.cqc@gmail.com, cqcb@ncbindia.com, nccbm@ncbindia.com; website: www.ncbindia.com

INVOICE-324

NATIONAL COUNCIL FOR CEMENT AND BUILDING MATERIALS

Centre for Quality Management, Standards and Calibration Services

CERTIFICATE OF ANALYSIS

ORDINARY PORTLAND CEMENT STANDARD
CRM-1012M

Date: 12TH February 2018
Certificate No.: OPC/1012M/108

The Certified Reference Material **CRM-1012M** has been developed by National Council for Cement and Building Materials (NCB), India, for evaluating proficiency of analysts, evaluating/comparing various test methods and calibration of equipment for analyzing minor constituents, for analysis of Ordinary Portland Cement and material of similar matrix. This **CRM-1012M** is packed in 4 sealed vials, each containing approximately 5g of powdered Ordinary Portland Cement.

Traceability, Certified Value and Uncertainty: The **CRM-1012M** is metrologically traceable to SI units of measurement. Each constituent has been analysed as g/g of the total material and reported in percent. The certified value and expanded uncertainty of each constituent, estimated with known sources of bias, is given below:

CONSTITUENT	CERTIFIED VALUE (PERCENT BY WEIGHT)	EXPANDED UNCERTAINTY (Coverage Factor k=2)
LOI	2.51	±0.07
SiO_2	20.81	±0.24
Fe_2O_3	4.30	±0.06
Al_2O_3	4.59	±0.08
CaO	59.94	±0.23
MgO	4.34	±0.20
SO_3	1.87	±0.03
IR	2.23	±0.06
Na_2O	0.44	±0.03
K_2O	0.57	±0.03
Mn_2O_3	0.10	±0.014
TiO_2	0.25	±0.01
Cl	0.032	±0.004

The expanded uncertainty of the certified value for a constituent is estimated according to the Guide to the expression of Uncertainty in Measurement (GUM) at 95.45% confidence level. The uncertainty includes the measurement variability among the participating laboratories and material inhomogeneity, latter controlled through statistical means. The value listed for LOI, SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO, SO_3, IR, Na_2O, K_2O and Cl is the best estimate of the true value based on the inter-laboratory comparison (ILC) results of 17 laboratories, and for minor constituents, it is based on the results of analytical programme carried out in three NCB laboratories. The analysis of major constituents, alkalis and Cl has been carried out as per IS 4032:1985. Estimation of Mn_2O_3 and TiO_2 has been done as per IS 12423:1988.

Instructions for Use:

1. CRM should be used in controlled conditions, using calibrated equipment, by trained personnel.
2. The CRM is to be dried at 105±5°C for two hours before use.
3. After drying, the material should be protected from atmospheric moisture and used immediately. Leftover portion in a vial, if any, should not be used at a later date.
4. The material should be stored under non-humid conditions to avoid any ingress of moisture/CO_2.
5. Record of usage of CRM and the results must be maintained.

Nature of Material: Non-hazardous

Expiry Date of CRM: This **CRM-1012M** with all its values and uncertainty is developed in **February 2018** and valid upto **31st January 2023** in sealed condition. The CRM will be **monitored** over the period of its certification.

Certification: NCB has provided only one set of material against the Certificate No. mentioned at the top.

Date of release: 04.09.18

Dr. S K Breja
Joint Director & Head
Centre for Quality Management,
Standards and Calibration Services

For Further information, please contact: National Council for Cement and Building Materials,
34KM Stone, Delhi Mathura Road (NH-2), Ballabgarh-121004, phone: 0129-2242051, 4192222, 4192239 (D)
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Annexure C

Data of results of MgO with CRMs

S.No	CRM	Certified Value, percent by weight	Observed Value, percent by weight	Tested By
1	PPC – BND 5052A	2.27 ± 0.05	2.2202	RNP
2	PPC – BND 5052A	2.27 ± 0.05	2.2215	RNP
3	PPC – BND 5052A	2.27 ± 0.05	2.2265	RNP
4	PPC – BND 5052A	2.27 ± 0.05	2.2215	RNP
5	PSC – BND 5053	2.73 ± 0.21	2.6181	RNP
6	PSC – BND 5053	2.73 ± 0.21	2.6145	RNP
7	PSC – BND 5053	2.73 ± 0.21	2.8077	RNP
8	OPC - 1012M	4.34 ± 0.20	4.3071	SSG
9	OPC - 1012M	4.34 ± 0.20	4.2302	RNP
10	OPC - 1012M	4.34 ± 0.20	4.4052	RNP