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Methods of testing cement — Report of a test programme — Chemical analysis by x-ray fluorescence

Méthodes d'essai des ciments — Rapport d'un programme d'essais — Analyse chimique par fluorescence X

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Contents

Page

Foreword iv

Introduction v

1 Scope 1

2 Test methods 1
2.1 General arrangements 1
2.2 Test method and constituents to be determined 2

3 Samples 2
3.1 Test samples 2
3.2 Calibration standards 2
3.3 Validation materials 2
3.4 Participating laboratories 3

4 Method for analysis of results 3
4.1 Statistics 3
4.2 Definition of Q-laboratories 4

5 Results and considerations 5
5.1 Laboratory number 5
5.2 Validation results for all laboratories 5
5.3 Test results of Q-laboratories 12

6 Conclusions 26
6.1 Result of test sample analyses 26
6.2 Factors affecting conformity with the validation criteria 27
6.3 Application to other international inter-laboratory testing 27

Annex A (informative) List of participating laboratories 28

Annex B (informative) Individual results of all laboratories 30

Annex C (informative) Questionnaire results 46

Annex D (informative) Other international inter-laboratory round-robin analyses of cement 48

Annex E (informative) Acknowledgements 51

Bibliography 52

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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.

ISO/TR 12389 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

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Introduction

This Technical Report summarizes the results of inter-laboratory testing of the chemical analysis of cement by x-ray fluorescence undertaken by laboratories in Japan, in Asian countries and in Europe. This testing programme was planned and conducted by the Committee on Cement Chemistry, Japan Cement Association and extended to the members of ISO in Asia and members of CEN/TC 51/WG 15 (Revision of methods of testing cement) in Europe. A total of 42 laboratories participated.

The wet method is a longstanding technique used for chemical analysis of cement. However, since this manner of analysis is extremely time-consuming, more rapid methods have been investigated, leading to the development of chemical analysis of cement by x-ray fluorescence (XRF method). As a result, Japanese Industrial Standard JIS R 5204 was established in July 2002.

JIS R 5204 established a scheme to confirm the validity of calibration equations when the concentrations of a pair of validation beads made from certified reference materials satisfy the criteria for both the repeatability limits and accuracy limits specified in JIS R 5204. Use of this validation system improves the repeatability and accuracy of results obtained by the JIS R 5204 method.

Since an International Standard for this analysis method had not yet been established, the Japanese National Committee for ISO/TC 74 (J/TC 74) proposed the “Development of chemical analysis of cement by x-ray fluorescence” to ISO/TC 74 in June 2004. The English version of JIS R 5204 was included as the first working draft at that time.

In order to introduce JIS R 5204 to Asian members of ISO/TC 74 and to promote technical exchange among them, an inter-laboratory testing programme was organized. This inter-laboratory testing was carried out with the participation of 16 laboratories in Japan and 14 outside Japan, mostly Asian members of ISO/TC 74.

As this first part of the round-robin testing was taking place, work was in progress within CEN committee TC 51/WG 15 to produce a standard method for the chemical analysis of cement by x-ray fluorescence. The Japanese Industrial Standard JIS R 5204 was accepted by this committee and, working jointly with the Japanese co-opted member, was incorporated into the draft for ISO 29581-2. At the invitation of the Japanese Cement Association, members of CEN/TC 51/WG 15 were invited to join in the Japanese/Asian round robin and in 2005 twelve European laboratories participated. The results of their testing are included in this report.

Those laboratories that obtained analyses of JCA-CRM-1 and/or JCA-CRM-2 satisfying the criteria for both the repeatability limits and accuracy limits for all components were defined as “Q-laboratories”. A comparison of the results for “Q-laboratories” with those obtained from other inter-laboratory testing for all constituents other than CaO indicates that the variation was equal to or smaller than that of wet analysis. The variation in results for CaO in “Q-laboratories” was slightly larger than that by wet analysis. Therefore, this inter-laboratory testing demonstrates that the accuracy of results obtained by the JIS R 5204/ISO 29581-2 method is generally the same as that for the wet method.

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Methods of testing cement — Report of a test programme — Chemical analysis by x-ray fluorescence

1 Scope

This Technical Report describes the results of the inter-laboratory testing of the chemical analysis of cement by x-ray fluorescence. In the first instance, the inter-laboratory testing was carried out in Japan and in Asian countries in accordance with JIS R 5204:2002. A total of 30 laboratories, 16 in Japan and 14 outside Japan, participated in the original testing programme. A further 12 European laboratories participated in tests with the same materials in 2005 in accordance with EN 196-2, a development by CEN/TC 51/WG 15/TG 1 of JIS R 5204. The latest version, developed as ISO 29581-2, is, as of the date of publication of this Technical Report, in the process of being circulated for technical enquiry.

The test samples used were Portland cement conforming to CEM I of EN 197-1 and a mixture of Portland cement and blast furnace slag. The mixture corresponded to the composition of Portland blast furnace slag cement, class B, specified in JIS R 5211 and CEM III/A of EN 197-1. Cement reference materials for x-ray fluorescence analysis (No. 601A) are used for the calibration standards, and certified reference materials JCA-CRM-1 and JCA-CRM-2 are used as the validation materials.

Constituents analysed include SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Na_2O, K_2O, TiO_2, P_2O_5, MnO and SrO. The loss on ignition is also determined.

The ISO round robin is a method-performance study conducted under close to optimum conditions with clear calibration and measurement guidelines. This is conducive to producing “best practice” values representative of the ideal situation. However, ISO 29581-2 is intended for use under everyday conditions in laboratories that operate to “good practice”. Annex D sets out the results of some international round robins carried out by a large number of laboratories demonstrating the suitability of ISO 29581-2 as a means for comparing the everyday performance of laboratories.

2 Test methods

2.1 General arrangements

The outline of the inter-laboratory testing is shown in Table 1.

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Table 1 — Outline of the inter-laboratory testing

Test method	First part – JIS R 5204:2002 Second part – ISO 29581-1
Test samples	JCA #1 Portland cement JCA #2 Cement made by mixing Portland cement with blast furnace slag (composition corresponding to Portland blast furnace slag cement Class B specified in JIS R 5211 and CEM III/A of EN 197-1)
Calibration standards	Cement reference materials for x-ray fluorescence analysis, JCA No. 601A
Validation materials	JCA-CRM-1 Ordinary Portland cement JCA-CRM-2 Portland blast furnace slag cement
Constituents determined	SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Na_2O, K_2O, TiO_2, P_2O_5, MnO, SrO, loss on ignition (LOI)

2.2 Test method and constituents to be determined

The first phase of this inter-laboratory testing was based on JIS R 5204. The second phase of this inter-laboratory testing was based on ISO 29581-2. There were no substantial differences between the two methods. For the remainder of this report they will be referred to as “the XRF method”.

Twelve constituents: SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Na_2O, K_2O, TiO_2, P_2O_5, MnO, and SrO, determined by XRF method using glass beads, and loss on ignition were to be evaluated. Although SO_3 for Portland blast furnace slag cement was outside the scope of JIS R 5204, it was an option for Sample #2 in this testing.

3 Samples

3.1 Test samples

Two test samples were used: Portland cement (Sample #1) conforming to EN 197-1 CEM I, and a cement mixture of Portland cement and blast furnace slag (Sample #2). Sample #2 corresponded to the B-type Portland blast furnace slag cement specified in JIS R 5211 and CEM III/A of EN 197-1. Approximately 30 g of each sample was distributed.

3.2 Calibration standards

Cement reference materials for x-ray fluorescence analysis (see NOTE), provided by the Japan Cement Association, were used for the calibration standards.

Approximately 12 g of each standard of JCA No. 601A was distributed to the foreign laboratories. The only requirement was that seven or more calibration standards be used when making calibration equations in accordance with the XRF method.

NOTE JCA No. 601A is a set of 15 cement reference materials consisting of nine Portland cements and six Portland blast furnace slag cements.

3.3 Validation materials

Validation of calibration equations was specified in the XRF method. Certified reference materials JCA-CRM-1 and JCA-CRM-2, provided by the Japan Cement Association, were used as the validation materials.

Approximately 30 g of each CRM was distributed to the laboratories.

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3.4 Participating laboratories

3.4.1 Japan

An announcement of the inter-laboratory testing was sent out to members of the Japan Cement Association. In response to the announcement, 16 laboratories registered as participants in this testing programme. A list of participating laboratories is shown in Annex A.

3.4.2 Asia

An announcement of the inter-laboratory testing was sent out to Asian members registered as P-members or O-members in ISO/TC 74 (See NOTE). Sixteen laboratories responded to the announcement and expressed their desire to participate, and 14 laboratories registered for the programme. A list of participating laboratories is shown in the Annex A.

NOTE The announcement was sent out to member bodies registered in ISO/TC74, and to Cement Associations of the members; see Reference [10].

3.4.3 Europe

Invitations were issued to the members of European Standards Organization Technical Committee TC 51, *Cement and building limes*, Working Group 15, *Methods of testing cement*, Task Group 1, *Analysis by x-ray fluorescence*, to nominate participating laboratories. Twelve laboratories undertook to participate. A list of participating laboratories is shown in Annex A.

4 Method for analysis of results

4.1 Statistics

Symbols and definitions of statistical terms used in this report are shown in Table 2.

Table 2 — Definitions of the statistical terms

Statistical term	Abbreviation/symbol	Definition of the statistical term [a]
Average	Av./$\bar{x}$	$\bar{x} = \frac{\sum_{i=1}^{i=n} x_i}{n}$
Maximum value	Max.	Maximum value in the data
Minimum value	Min.	Minimum value in the data
Range	—	Range is equal to max. minus min.
Standard deviation	S.D./σ	$\sigma = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$
Coefficient of variation	C.V./C_V	$C_V = \frac{\sigma \times 100}{\bar{x}}$, expressed as a percentage

[a] n is the number of laboratories; x is the mean value of a pair of results from each laboratory.

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4.2 Definition of Q-laboratories

4.2.1 General

In this inter-laboratory testing, a Q-laboratory is one where the concentration of a pair of validation beads made from certified reference materials satisfies the criteria for both the repeatability limits and accuracy limits set out in the XRF method.

4.2.2 Validation procedure

4.2.2.1 Determination of the concentration of a pair of validation beads made from at least one certified reference material for all analysis constituents.

4.2.2.2 Check that the difference in the concentration, rounded off to three decimal places, of a pair of validation beads is within the repeatability limits obtained from Equation (1):

$$\log(y) = 0{,}48 \log(x) - 1{,}499 \quad (1)$$

where

y is the repeatability limit, as a percentage;

x is the mean value of the concentration of a pair of validation beads, as a percentage.

When x is less than 0,5 %, a limit for y of 0,020 % is applied to all values.

NOTE Equation (1) is specified in JIS R 5204.

4.2.2.3 Check that the difference between the mean values, rounded off to two decimal places, of the concentration of a pair of validation beads and the "certified values" of the certified reference materials are within the accuracy limits specified according to the level of concentration in Table 3 for each analysed constituent.

Both JCA-CRM-1 and JCA-CRM-2 are used for the validation materials in this inter-laboratory testing. However, in the XRF method, the use of just one certified reference material is permitted. Therefore, for the purpose of this inter-laboratory test, the validations are considered as satisfied if the analysis of either JCA-CRM-1 or JCA-CRM-2 meets the validation criteria. Laboratories that obtained concentrations of JCA-CRM-1 and/or JCA-CRM-2 satisfying the criteria for both the repeatability limits and accuracy limits for all constituents, were defined as "Q-laboratories" in this inter-laboratory testing. In ISO 29581-2, these are referred to as "expert" laboratories.

Table 3 — Accuracy limits for analysis validation

Level of the certified value (% absolute)	Accuracy limits for analysis validation (% absolute)
0,00 to 0,49	0,02
0,50 to 0,99	0,03
1,00 to 6,99	0,08
7,00 to 14,99	0,12
15,00 to 29,99	0,15
30,00 to 49,99	0,20
50,00 to 79,99	0,25
80,00 to 100,00	0,30

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5 Results and considerations

5.1 Laboratory number

Validation results for all laboratories and results of determination for Q-laboratories are shown in Tables B.1 to B.8. Laboratories No. 1 to No. 16 represent laboratories in Japan; Laboratories No. 101 to No. 113 represent ISO member laboratories outside Japan. Although there were 14 participating laboratories outside Japan, the report submitted by Laboratory No. 114 is mentioned only in Tables B.2, B.4, B.6 and B.8, because the laboratory carried out XRF analysis by the pellet method. Laboratories No. E1 to E12 represent laboratories participating in the European stage.

5.2 Validation results for all laboratories

The difference in concentration for each pair of JCA-CRM-1 is shown in Table 4, and that for JCA-CRM-2 is shown in Table 5. The difference between the mean values of concentration for each pair of JCA-CRM-1 and the certified values is shown in Table 6, and that for JCA-CRM-2 is shown in Table 7.

Data in the shaded cells of Table 4 and Table 5 denote results that did not satisfy the required criteria for repeatability. Data in the shaded cells of Table 6 and Table 7 represent results that did not satisfy the required criteria for accuracy limits. "-" in each table indicates that there is no report from the laboratory for that constituent.

The presence of data in the shaded cells indicates that some validation results did not satisfy the required criteria for repeatability or accuracy limits. Therefore, in determining the Q-laboratories according to the definition described in 4.2, 27 laboratories were judged as Q-laboratories.

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Table 4 — Difference in concentrations for each pair of validation beads
(All laboratories — Validation material: JCA-CRM-1)

Laboratory ref.	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	SO_3 %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	SrO %
1	0,039	0,028	0,009	0,180	0,006	0,010	0,015	0,007	0,000	0,001	0,000	-
2	0,034	0,004	0,001	0,072	0,005	0,032	0,001	0,003	0,007	0,001	0,001	0,000
3	0,012	0,005	0,017	0,018	0,012	0,028	0,013	0,002	0,003	0,001	0,000	-
4	0,080	0,010	0,013	0,020	0,001	0,009	0,002	0,004	0,004	0,002	0,000	0,000
5	0,032	0,012	0,003	0,004	0,003	0,009	0,001	0,003	0,002	0,001	0,000	0,000
6	0,024	0,027	0,002	0,083	0,039	0,012	0,011	0,007	0,008	0,002	0,002	0,000
7	0,014	0,013	0,003	0,046	0,005	0,006	0,002	0,002	0,005	0,000	0,001	0,000
8	0,040	0,006	0,004	0,115	0,010	0,008	0,007	0,001	0,008	0,001	0,000	0,000
9	0,011	0,017	0,004	0,041	0,003	0,006	0,001	0,001	0,002	0,001	0,000	0,000
10	0,028	0,035	0,003	0,012	0,017	0,017	0,007	0,001	0,003	0,000	0,000	-
11	0,012	0,006	0,008	0,061	0,014	0,018	0,012	0,010	0,004	0,005	0,001	-
12	0,011	0,006	0,006	0,073	0,005	0,024	0,007	0,002	0,011	0,000	0,003	0,001
13	0,018	0,004	0,003	0,042	0,009	0,005	0,002	0,000	0,004	0,000	0,001	0,000
14	0,080	0,029	0,006	0,112	0,006	0,001	0,002	0,018	0,013	0,010	0,001	0,000
15	0,090	0,008	0,004	0,038	0,003	0,015	0,002	0,001	0,006	0,000	0,001	0,001
16	0,007	0,011	0,004	0,014	0,003	0,008	0,008	0,001	0,005	0,002	0,000	0,000
101	0,005	0,005	0,007	0,006	0,012	0,130	0,006	0,004	0,003	0,003	-	-
102	0,017	0,058	0,004	0,005	0,007	0,001	-	0,000	-	-	-	-
103	0,020	0,020	0,010	0,060	0,170	0,020	0,010	0,010	-	-	-	-
104	0,016	0,020	0,009	0,193	0,011	0,035	0,010	0,016	0,002	0,004	0,001	0,000
105	0,084	0,052	0,008	0,024	0,001	0,006	0,003	0,001	0,011	0,003	0,000	0,001
106	0,118	0,014	0,002	0,090	0,002	0,014	0,017	0,003	-	-	-	-
107	0,006	0,211	0,008	0,045	0,033	0,032	0,005	0,006	-	0,011	0,001	-
108	0,010	0,010	0,010	0,020	0,010	0,010	0,030	0,000	-	0,000	-	-
109	0,032	0,009	0,014	0,116	0,003	0,015	-	0,002	-	-	-	-
110	0,122	0,099	0,059	0,041	0,261	0,136	0,082	0,161	-	0,022	-	-
111	0,040	0,030	0,010	0,010	0,020	0,000	0,010	0,000	0,010	0,010	0,000	0,000
112	0,110	0,010	0,000	0,020	0,090	0,010	0,000	0,000	-	-	-	-
113	0,030	0,030	0,010	0,150	0,090	0,030	0,010	0,000	0,000	0,010	0,000	0,000
E1	0,112	0,045	0,013	0,039	0,007	0,025	0,009	0,002	0,006	0,000	0,000	0,000
E2	0,001	0,053	0,008	0,016	0,019	0,021	0,002	0,005	0,002	0,003	0,000	-
E3	0,020	0,050	0,040	0,030	0,000	0,060	0,000	0,000	0,000	0,010	0,000	0,000
E4	0,034	0,012	0,009	0,038	0,008	0,003	0,005	0,002	0,000	0,003	0,001	0,000
E5	0,020	0,010	0,020	0,050	0,010	0,010	0,010	0,010	0,010	0,030	0,000	0,000
E6	0,036	0,039	0,003	0,093	0,003	0,042	0,004	0,004	0,001	0,010	0,001	-
E7	0,208	0,051	0,003	0,204	0,051	0,006	0,008	0,006	0,010	0,005	0,001	0,001
E8	0,085	0,036	0,002	0,014	0,004	0,007	0,011	0,001	0,004	0,000	0,001	0,000
E9	0,038	0,039	0,008	0,169	0,028	-	0,009	0,001	0,011	-	-	-
E10	0,010	0,010	0,020	0,050	0,020	0,000	0,000	0,000	-	-	-	-
E11	0,003	0,005	0,007	0,010	0,021	0,002	0,000	0,002	0,001	0,000	0,001	0,000
E12	0,240	0,014	0,022	0,700	0,001	0,017	0,020	0,011	0,007	0,007	0,001	0,000
Average	0,048	0,028	0,010	0,076	0,025	0,021	0,009	0,008	0,005	0,005	0,001	0,000
Max.	0,240	0,211	0,059	0,700	0,261	0,136	0,082	0,161	0,013	0,030	0,003	0,001
Min.	0,001	0,004	0,000	0,004	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000
Range	0,239	0,207	0,059	0,696	0,261	0,136	0,082	0,161	0,013	0,030	0,003	0,001
Certified analysis	20,99	5,260	2,670	65,210	2,130	2,050	0,260	0,560	0,350	0,280	0,060	0,050
Limit value	0,150	0,080	0,080	0,250	0,080	0,080	0,020	0,030	0,020	0,020	0,020	0,020

NOTE Data in shaded cells represent results that did not satisfy the required criteria for repeatability specified for "expert" laboratories for the XRF method.

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Table 5 — Difference in concentrations for each pair of validation beads
(All laboratories — Validation material: JCA-CRM-2)

Laboratory ref.	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	SO_3 [a] %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	SrO %
1	0,001	0,001	0,003	0,002	0,002	0,00	0,005	0,006	0,001	0,000	0,000	-
2	0,051	0,010	0,001	0,020	0,018	0,03	0,003	0,002	0,014	0,002	0,000	0,000
3	0,032	0,010	0,013	0,013	0,009	-	0,009	0,001	0,000	0,000	0,001	-
4	0,008	0,016	0,006	0,047	0,010	0,02	0,003	0,003	0,000	0,001	0,003	0,000
5	0,038	0,019	0,001	0,012	0,009	0,02	0,001	0,001	0,002	0,000	0,000	0,000
6	0,113	0,044	0,018	0,105	0,025	0,03	0,012	0,007	0,007	0,002	0,000	0,000
7	0,009	0,003	0,001	0,038	0,026	0,00	0,000	0,001	0,002	0,001	0,000	0,000
8	0,005	0,016	0,003	0,040	0,003	0,00	0,000	0,000	0,007	0,001	0,001	0,000
9	0,094	0,016	0,001	0,101	0,012	-	0,002	0,002	0,004	0,000	0,001	0,000
10	0,035	0,091	0,015	0,183	0,040	-	0,011	0,002	0,003	0,000	0,001	-
11	0,020	0,030	0,002	0,100	0,047	0,00	0,016	0,000	0,014	0,001	0,001	-
12	0,006	0,052	0,020	0,022	0,004	-	0,014	0,004	0,006	0,000	0,001	0,001
13	0,010	0,008	0,003	0,058	0,008	0,01	0,002	0,000	0,005	0,000	0,000	0,000
14	0,071	0,052	0,006	0,156	0,009	0,01	0,008	0,004	0,014	0,015	0,003	0,001
15	0,052	0,006	0,005	0,021	0,001	0,01	0,006	0,002	0,003	0,001	0,000	0,000
16	0,054	0,046	0,006	0,019	0,011	0,03	0,005	0,002	0,003	0,000	0,001	0,001
101	0,005	0,039	0,011	0,060	0,013	-	0,017	0,001	0,001	0,000	-	-
102	0,022	0,011	0,004	0,029	0,008	0,00	-	0,001	-	-	-	-
103	0,020	0,070	0,010	0,000	0,140	0,03	0,010	0,000	-	-	-	-
104	0,034	0,039	0,010	0,188	0,009	0,02	0,007	0,017	0,003	0,001	0,003	0,000
105	0,043	0,019	0,002	0,010	0,020	0,00	0,004	0,004	0,010	0,005	0,006	0,000
106	0,057	0,045	0,043	0,107	0,015	-	0,002	0,001	-	-	-	-
107	0,121	0,014	0,048	0,012	0,015	-	0,009	0,005	-	0,002	0,003	-
108	0,250	0,060	0,000	0,250	0,020	0,03	0,070	0,000	-	0,010	-	-
109	0,054	0,023	0,013	0,057	0,002	0,02	-	0,010	-	-	-	-
110	0,022	0,119	0,018	0,138	0,037	0,02	0,021	0,019	-	0,008	-	-
111	0,060	0,050	0,010	0,070	0,030	0,02	0,000	0,000	0,010	0,020	0,000	0,000
112	-	-	-	-	-	-	-	-	-	-	-	-
113	0,050	0,050	0,020	0,050	0,030	-	0,010	0,010	0,010	0,000	0,000	0,000
E1	0,045	0,063	0,025	0,121	0,002	0,179	0,008	0,005	0,007	0,007	0,001	0,001
E2	0,003	0,003	0,008	0,017	0,002	0,025	0,005	0,005	0,001	0,006	0,002	-
E3	0,010	0,010	0,010	0,030	0,030	0,020	0,000	0,000	0,000	0,010	0,000	0,000
E4	0,062	0,054	0,001	0,111	0,026	0,047	0,013	0,001	0,001	0,002	0,004	0,002
E5	0,040	0,030	0,010	0,120	0,100	0,030	0,015	0,000	0,003	0,010	0,001	0,002
E6	0,002	0,019	0,008	0,041	0,033	-	0,000	0,003	0,000	0,007	0,002	-
E7	0,118	0,052	0,001	0,179	0,008	0,013	0,003	0,002	0,003	0,005	0,001	0,001
E8	0,049	0,003	0,014	0,070	0,038	0,005	0,008	0,002	0,000	0,004	0,000	0,000
E9	0,063	0,025	0,006	0,082	0,008	-	-	0,001	0,016	-	-	-
E10	0,010	0,010	0,010	0,040	0,000	0,000	0,000	0,000	-	-	-	-
E11	0,086	0,030	0,006	0,052	0,009	0,003	0,008	0,001	0,000	0,001	0,000	0,001
E12	0,260	0,193	0,023	0,430	0,005	-	0,016	0,003	0,003	0,009	0,000	-
Average	0,052	0,036	0,010	0,080	0,021	0,022	0,009	0,003	0,005	0,004	0,001	0,000
Max.	0,260	0,193	0,048	0,430	0,140	0,179	0,070	0,019	0,016	0,020	0,006	0,002
Min.	0,001	0,001	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000
Range	0,259	0,192	0,048	0,430	0,140	0,179	0,070	0,019	0,016	0,020	0,006	0,002
Certified analysis	25,66	8,940	2,080	56,330	3,050	-	0,240	0,310	0,500	0,070	0,150	0,070
Limit value	0,150	0,120	0,080	0,250	0,080	-	0,020	0,020	0,030	0,020	0,020	0,020

NOTE Data in shaded cells represent results that did not satisfy the required criteria for repeatability specified for "expert" laboratories for the XRF method.

[a] SO_3 for JCA-CRM-2 is outside the scope of JIS R 5204.

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Table 6 — Differences between the mean value of the concentrations of a pair of validation beads and the certified value
(All laboratories — Validation material: JCA-CRM-1)

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Mean value [a]	Diff. [b]	Mean value [a]	Diff. [b]	Mean value [a]	Diff. [b]	Mean value [a]	Diff. [b]	Mean value [a]	Diff. [b]	Mean value [a]	Diff. [b]
1	20,96	-0,03	5,27	0,01	2,61	-0,06	65,29	0,08	2,11	-0,02	2,08	0,03
2	20,92	-0,07	5,26	0,00	2,65	-0,02	65,33	0,12	2,13	0,00	2,02	-0,03
3	20,94	-0,05	5,25	-0,01	2,68	0,01	65,11	-0,10	2,14	0,01	2,08	0,03
4	20,97	-0,02	5,26	0,00	2,68	0,01	65,20	-0,01	2,14	0,01	2,08	0,03
5	20,93	-0,06	5,26	0,00	2,67	0,00	65,24	0,03	2,13	0,00	2,09	0,04
6	20,93	-0,06	5,24	-0,02	2,61	-0,06	65,17	-0,04	2,12	-0,01	2,07	0,02
7	21,00	0,01	5,27	0,01	2,66	-0,01	65,20	-0,01	2,17	0,04	2,06	0,01
8	20,92	-0,07	5,27	0,01	2,68	0,01	65,05	-0,16	2,16	0,03	2,06	0,01
9	20,91	-0,08	5,26	0,00	2,61	-0,06	65,21	0,00	2,12	-0,01	2,07	0,02
10	20,96	-0,03	5,24	-0,02	2,63	-0,04	65,32	0,11	2,11	-0,02	2,08	0,03
11	21,02	0,03	5,28	0,02	2,67	0,00	65,21	0,00	2,12	-0,01	2,07	0,02
12	20,98	-0,01	5,27	0,01	2,68	0,01	65,27	0,06	2,14	0,01	2,09	0,04
13	21,03	0,04	5,27	0,01	2,68	0,01	64,98	-0,23	2,14	0,01	2,05	0,00
14	21,00	0,01	5,28	0,02	2,72	0,05	65,40	0,19	2,15	0,02	2,07	0,02
15	21,04	0,05	5,26	0,00	2,67	0,00	65,21	0,00	2,11	-0,02	1,98	-0,07
16	21,01	0,02	5,29	0,03	2,67	0,00	65,18	-0,03	2,15	0,02	2,09	0,04
101	21,05	0,06	5,29	0,03	2,62	-0,05	65,34	0,13	2,12	-0,01	2,03	-0,02
102	21,17	0,18	5,21	-0,05	2,64	-0,03	66,29	1,08	2,13	0,00	2,43	0,38
103	20,98	-0,01	5,21	-0,05	2,64	-0,03	65,25	0,04	2,14	0,01	2,08	0,03
104	20,98	-0,01	5,24	-0,02	2,63	-0,04	64,96	-0,25	2,10	-0,03	2,08	0,03
105	21,02	0,03	5,27	0,01	2,64	-0,03	65,38	0,17	2,18	0,05	2,06	0,01
106	20,95	-0,04	5,26	0,00	2,67	0,00	65,13	-0,08	2,12	-0,01	2,06	0,01
107	21,06	0,07	5,42	0,16	2,69	0,02	64,47	-0,74	2,15	0,02	2,04	-0,01
108	20,90	-0,09	5,26	0,00	2,62	-0,05	65,17	-0,04	2,10	-0,03	2,06	0,01
109	20,98	-0,01	5,26	0,00	2,63	-0,04	65,50	0,29	2,11	-0,02	1,95	-0,10
110	20,91	-0,08	5,25	-0,01	2,61	-0,06	65,13	-0,08	2,17	0,04	1,99	-0,06
111	20,92	-0,07	4,98	-0,28	2,62	-0,05	64,68	-0,53	2,38	0,25	2,00	-0,05
112	21,00	0,01	5,86	0,60	2,58	-0,09	64,77	-0,44	2,06	-0,07	1,80	-0,25
113	22,98	1,99	5,24	-0,02	2,64	-0,03	65,28	0,07	2,02	-0,11	2,06	0,01
E1	20,91	-0,08	5,26	0,00	2,66	-0,01	65,16	-0,05	2,14	0,01	2,20	0,15
E2	20,80	-0,19	5,30	0,04	2,61	-0,06	65,00	-0,21	2,11	-0,02	2,07	0,02
E3	20,79	-0,20	5,17	-0,09	2,71	0,04	65,17	-0,04	2,27	0,14	1,98	-0,07
E4	20,93	-0,06	5,28	0,02	2,65	-0,02	65,01	-0,20	2,17	0,04	2,04	-0,01
E5	21,01	0,02	5,25	-0,01	2,65	-0,02	65,33	0,12	2,14	0,01	2,07	0,02
E6	20,95	-0,04	5,23	-0,03	2,68	0,01	65,16	-0,05	2,15	0,02	2,13	0,08
E7	21,00	0,01	5,27	0,01	2,69	0,02	65,34	0,13	2,15	0,02	2,07	0,02
E8	20,89	-0,10	5,27	0,01	2,58	-0,09	65,13	-0,08	2,14	0,01	2,07	0,02
E9	20,99	0,00	5,22	-0,04	2,68	0,01	65,21	0,00	2,15	0,02	-	-
E10	21,09	0,10	5,16	-0,10	2,61	-0,06	65,14	-0,07	2,11	-0,02	2,01	-0,04
E11	20,75	-0,24	5,27	0,01	2,67	0,00	65,52	0,31	2,17	0,04	1,99	-0,06
E12	20,88	-0,11	5,29	0,03	2,67	0,00	64,91	-0,30	2,15	0,02	2,05	0,00
Average	21,01		5,27		2,65		65,19		2,14		2,06	
Max.	22,98		5,86		2,72		66,29		2,38		2,43	
Min.	20,75		4,98		2,58		64,47		2,02		1,80	
Range	2,23		0,88		0,14		1,82		0,36		0,63	
S.D.	0,325		0,111		0,033		0,268		0,053		0,085	
C.V.	1,5		2,1		1,3		0,4		2,5		4,1	
Certified value	20,99		5,26		2,67		65,21		2,13		2,05	

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Table 6 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %	
	Mean value [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]
1	0,26	0,00	0,54	-0,02	0,34	-0,01	0,29	0,01	0,07	0,01	-	-
2	0,26	0,00	0,53	-0,03	0,35	0,00	0,28	0,00	0,07	0,01	0,04	-0,01
3	0,26	0,00	0,57	0,01	0,36	0,01	0,28	0,00	0,06	0,00	-	-
4	0,26	0,00	0,56	0,00	0,36	0,01	0,29	0,01	0,06	0,00	0,04	-0,01
5	0,26	0,00	0,56	0,00	0,36	0,01	0,28	0,00	0,06	0,00	0,04	-0,01
6	0,27	0,01	0,58	0,02	0,34	-0,01	0,29	0,01	0,06	0,00	0,04	-0,01
7	0,25	-0,01	0,56	0,00	0,36	0,01	0,28	0,00	0,06	0,00	0,04	-0,01
8	0,25	-0,01	0,56	0,00	0,35	0,00	0,28	0,00	0,06	0,00	0,04	-0,01
9	0,26	0,00	0,57	0,01	0,35	0,00	0,29	0,01	0,07	0,01	0,04	-0,01
10	0,24	-0,02	0,57	0,01	0,35	0,00	0,28	0,00	0,07	0,01	-	-
11	0,25	-0,01	0,53	-0,03	0,36	0,01	0,29	0,01	0,06	0,00	-	-
12	0,27	0,01	0,57	0,01	0,35	0,00	0,28	0,00	0,06	0,00	0,04	-0,01
13	0,26	0,00	0,56	0,00	0,36	0,01	0,29	0,01	0,06	0,00	0,04	-0,01
14	0,27	0,01	0,58	0,02	0,33	-0,02	0,30	0,02	0,06	0,00	0,04	-0,01
15	0,27	0,01	0,56	0,00	0,35	0,00	0,29	0,01	0,06	0,00	0,04	-0,01
16	0,26	0,00	0,58	0,02	0,35	0,00	0,28	0,00	0,06	0,00	0,04	-0,01
101	0,23	-0,03	0,57	0,01	0,35	0,00	0,29	0,01	-	-	-	-
102	-	-	0,60	0,04	-	-	-	-	-	-	-	-
103	0,28	0,02	0,58	0,02	-	-	-	-	-	-	-	-
104	0,24	-0,02	0,56	0,00	0,34	-0,01	0,27	-0,01	0,07	0,01	0,04	-0,01
105	0,25	-0,01	0,60	0,04	0,35	0,00	0,29	0,01	0,07	0,01	0,04	-0,01
106	0,27	0,01	0,57	0,01	-	-	-	-	-	-	-	-
107	0,25	-0,01	0,56	0,00	-	-	0,28	0,00	0,06	0,00	-	-
108	0,26	0,00	0,59	0,03	-	-	0,28	0,00	-	-	-	-
109	-	-	0,57	0,01	-	-	-	-	-	-	-	-
110	0,28	0,02	0,58	0,02	-	-	0,29	0,01	-	-	-	-
111	0,28	0,02	0,57	0,01	0,38	0,03	0,36	0,08	0,07	0,01	0,04	-0,01
112	0,19	-0,07	0,58	0,02	-	-	-	-	-	-	-	-
113	0,26	0,00	0,58	0,02	0,35	0,00	0,28	0,00	0,07	0,01	0,04	-0,01
E1	0,26	0,00	0,56	0,00	0,35	0,00	0,29	0,01	0,06	0,00	0,04	-0,01
E2	0,27	0,01	0,57	0,01	0,34	-0,01	0,29	0,01	0,07	0,01	-	-
E3	0,26	0,00	0,55	-0,01	0,35	0,00	0,30	0,02	0,07	0,01	0,05	0,00
E4	0,27	0,01	0,57	0,01	0,34	-0,01	0,29	0,01	0,07	0,01	0,04	-0,01
E5	0,27	0,01	0,58	0,02	0,35	0,00	0,28	0,00	0,07	0,01	0,04	-0,01
E6	0,13	-0,13	0,56	0,00	0,35	0,00	0,31	0,03	0,06	0,00	-	-
E7	0,27	0,01	0,56	0,00	0,36	0,01	0,28	0,00	0,06	0,00	0,05	0,00
E8	0,27	0,01	0,57	0,01	0,34	-0,01	0,28	0,00	0,07	0,01	0,04	-0,01
E9	0,26	0,00	0,56	0,00	0,34	-0,01	-	-	-	-	-	-
E10	0,29	0,03	0,57	0,01	-	-	-	-	-	-	-	-
E11	0,25	-0,01	0,57	0,01	0,34	-0,01	0,28	0,00	0,06	0,00	0,05	0,00
E12	0,25	-0,01	0,57	0,01	0,35	0,00	0,29	0,01	0,07	0,01	-	-
Average	0,26		0,57		0,35		0,29		0,06		0,04	
Max.	0,29		0,60		0,38		0,36		0,07		0,05	
Min.	0,13		0,53		0,33		0,27		0,06		0,04	
Range	0,16		0,07		0,05		0,09		0,01		0,01	
S.D.	0,027		0,015		0,010		0,015		0,005		0,003	
C.V.	10,2		2,6		2,7		5,3		8,4		6,9	
Certified value	0,26		0,56		0,35		0,28		0,06		0,05	

NOTE Data in shaded cells represent concentrations that did not satisfy the required criteria for accuracy limits for "expert" laboratories specified for the XRF method.

[a] "Mean value" is the mean value of the concentrations of a pair of validated beads.

[b] "Diff." is the difference between the mean value of the concentrations of a pair of validated beads and the certified value for the validation material.

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Table 7 — Differences between the mean value of the concentrations of a pair of validation beads and the certified value
(All laboratories — Validation material: JCA-CRM-2)

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 [a] %	
	Mean value [b]	Diff. [c]	Mean Value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]
1	25,65	-0,01	8,94	0,00	2,14	0,06	56,49	0,16	3,05	0,00	2,56	-
2	25,55	-0,11	8,92	-0,02	2,09	0,01	56,47	0,14	3,03	0,02	2,57	-
3	25,52	-0,14	8,94	0,00	2,10	0,02	56,53	0,20	2,97	-0,08	-	-
4	25,64	-0,02	8,93	-0,01	2,10	0,02	56,38	0,05	3,04	-0,01	2,58	-
5	25,61	-0,05	8,93	-0,01	2,08	0,00	56,43	0,10	3,03	-0,02	2,57	-
6	25,59	-0,07	9,00	0,06	2,14	0,06	56,56	0,23	3,06	0,01	2,53	-
7	25,69	0,03	8,95	0,01	2,07	-0,01	56,48	0,15	3,08	0,03	2,60	-
8	25,57	-0,09	8,90	-0,04	2,04	-0,04	56,15	-0,18	3,06	0,01	2,37	-
9	25,58	-0,08	8,93	-0,01	2,14	0,06	56,49	0,16	3,06	0,01	-	-
10	25,59	-0,07	8,90	-0,04	2,13	0,05	56,53	0,20	2,98	-0,07	-	-
11	25,69	0,03	8,88	-0,06	2,08	0,00	56,41	0,08	3,02	-0,03	2,61	-
12	25,68	0,02	8,94	0,00	2,08	0,00	56,43	0,10	3,04	-0,01	-	-
13	25,73	0,07	8,92	-0,02	2,08	0,00	56,24	-0,09	3,06	0,01	2,63	-
14	25,75	0,09	8,94	0,00	2,12	0,04	56,51	0,18	3,06	0,01	2,64	-
15	25,58	-0,08	8,96	0,02	2,09	0,01	56,32	-0,01	3,02	-0,03	2,60	-
16	25,68	0,02	8,96	0,02	2,08	0,00	56,52	0,19	3,04	-0,01	2,59	-
101	25,63	-0,03	8,92	-0,02	2,12	0,04	56,56	0,23	3,02	-0,03	-	-
102	25,60	-0,06	8,92	-0,02	2,13	0,05	56,66	0,33	3,07	0,02	1,32	-
103	25,66	0,00	8,96	0,02	2,16	0,08	56,53	0,20	2,99	-0,06	2,56	-
104	25,53	-0,13	8,87	-0,07	2,12	0,04	56,32	-0,01	3,03	-0,02	1,89	-
105	25,63	-0,03	8,92	-0,02	2,12	0,04	56,55	0,22	3,08	0,03	2,57	-
106	25,52	-0,14	8,94	0,00	2,07	-0,01	56,56	0,23	3,02	-0,03	-	-
107	25,57	-0,09	8,94	0,00	2,06	-0,02	56,48	0,13	2,88	-0,17	-	-
108	25,74	0,08	8,95	0,01	2,13	0,05	56,64	0,31	3,04	-0,01	2,56	-
109	25,72	0,06	8,98	0,04	2,15	0,07	56,51	0,18	3,08	0,03	2,44	-
110	25,65	-0,01	8,88	-0,06	2,07	-0,01	56,55	0,22	3,07	0,02	1,92	-
111	25,31	-0,35	8,62	-0,32	2,10	0,02	56,30	-0,03	3,24	0,19	1,27	-
112	-	-	-	-	-	-	-	-	-	-	-	-
113	25,72	0,06	8,94	0,00	2,13	0,05	56,82	0,49	3,12	0,07	-	-
E1	25,65	-0,01	8,92	-0,02	2,08	0,00	56,43	0,10	3,04	-0,01	2,38	-
E2	25,58	-0,10	8,92	-0,02	2,12	0,04	56,52	0,19	3,03	-0,02	2,58	-
E3	25,35	-0,31	8,72	-0,22	2,12	0,04	56,35	0,02	3,21	0,16	2,52	-
E4	25,59	-0,07	8,87	-0,07	2,12	0,04	56,10	-0,23	2,99	-0,06	-	-
E5	25,65	-0,01	8,93	-0,01	2,10	0,02	56,23	-0,10	3,03	-0,02	-	-
E6	25,57	-0,09	8,87	-0,07	2,10	0,02	56,42	0,09	3,05	0,00	-	-
E7	25,79	0,13	8,91	-0,03	2,09	0,01	56,45	0,12	3,04	-0,01	2,62	-
E8	25,61	-0,05	8,93	-0,01	2,15	0,07	56,40	0,07	3,04	-0,01	2,71	-
E9	25,68	0,02	8,89	-0,05	2,10	0,02	56,54	0,21	3,06	0,01	-	-
E10	25,50	-0,16	8,64	-0,30	2,13	0,05	57,10	0,77	2,97	-0,08	2,16	-
E11	25,50	-0,16	8,99	0,05	2,14	0,06	57,04	0,71	3,11	0,06	2,50	-
E12	25,65	-0,01	8,96	0,02	2,09	0,01	56,46	0,13	3,09	0,04	-	-
Average	25,61		8,91		2,11		56,49		3,05		2,40	
Max.	25,79		9,00		2,16		57,10		3,24		2,71	
Min.	25,31		8,62		2,04		56,10		2,88		1,27	
Range	0,48		0,38		0,12		1,00		0,36		1,44	
S.D.	0,097		0,079		0,028		0,191		0,059		0,376	
C.V.	0,4		0,9		1,4		0,3		1,9		-	
Certified value	25,66		8,94		2,08		56,33		3,05		-	

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Table 7 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %	
	Mean value [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]
1	0,27	0,03	0,31	0,00	0,50	0,00	0,07	0,00	0,16	0,01	-	-
2	0,24	0,00	0,32	0,01	0,51	0,01	0,07	0,00	0,16	0,01	0,07	0,00
3	0,22	-0,02	0,32	0,01	0,51	0,01	0,08	0,01	0,16	0,01	-	-
4	0,24	0,00	0,32	0,01	0,51	0,01	0,08	0,01	0,15	0,00	0,06	-0,01
5	0,24	0,00	0,32	0,01	0,50	0,00	0,08	0,01	0,15	0,01	0,07	0,00
6	0,24	0,00	0,32	0,01	0,51	0,01	0,07	0,00	0,16	0,01	0,07	0,00
7	0,23	-0,01	0,32	0,01	0,50	0,00	0,07	0,00	0,15	0,00	0,07	0,00
8	0,23	-0,01	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	0,06	-0,01
9	0,25	0,01	0,31	0,00	0,50	0,00	0,07	0,00	0,16	0,01	0,06	-0,01
10	0,23	-0,01	0,32	0,01	0,51	0,01	0,07	0,00	0,16	0,01	-	-
11	0,25	0,01	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	-	-
12	0,24	0,00	0,33	0,02	0,51	0,01	0,08	0,01	0,16	0,01	0,06	-0,01
13	0,24	0,00	0,32	0,01	0,51	0,01	0,08	0,01	0,15	0,00	0,07	0,00
14	0,25	0,01	0,33	0,02	0,48	-0,02	0,09	0,02	0,15	0,00	0,07	0,00
15	0,25	0,01	0,32	0,01	0,52	0,02	0,08	0,01	0,16	0,01	0,06	-0,01
16	0,24	0,00	0,31	0,00	0,50	0,00	0,08	0,01	0,15	0,00	0,07	0,00
101	0,21	-0,03	0,32	0,01	0,51	0,01	0,07	0,00	-	-	-	-
102	-	-	0,31	0,00	-	-	-	-	-	-	-	-
103	0,24	0,00	0,31	0,00	-	-	-	-	-	-	-	-
104	0,26	0,02	0,32	0,01	0,50	0,00	0,07	0,00	0,16	0,01	0,07	0,00
105	0,23	-0,01	0,34	0,03	0,51	0,01	0,08	0,01	0,16	0,01	0,06	-0,01
106	0,25	0,01	0,33	0,02	-	-	-	-	-	-	-	-
107	0,26	0,02	0,33	0,02	-	-	0,07	0,00	0,16	0,01	-	-
108	0,24	0,00	0,32	0,01	-	-	0,08	0,01	-	-	-	-
109	-	-	0,31	0,00	-	-	-	-	-	-	-	-
110	0,23	-0,01	0,32	0,01	-	-	0,08	0,01	-	-	-	-
111	0,27	0,03	0,33	0,02	0,52	0,02	0,02	-0,05	0,16	0,01	0,06	-0,01
112	-	-	-	-	-	-	-	-	-	-	-	-
113	0,24	0,00	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	0,06	-0,01
E1	0,25	0,01	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	0,07	0,00
E2	0,28	0,04	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	-	-
E3	0,24	0,00	0,31	0,00	0,49	-0,01	0,08	0,01	0,18	0,03	0,08	0,01
E4	0,24	0,00	0,32	0,01	0,50	0,00	0,07	0,00	0,16	0,01	0,06	-0,01
E5	0,23	-0,01	0,32	0,01	0,51	0,01	0,07	0,00	0,16	0,01	0,08	0,01
E6	0,12	-0,12	0,32	0,01	0,50	0,00	0,11	0,04	0,15	0,00	-	-
E7	0,25	0,01	0,32	0,01	0,50	0,00	0,08	0,01	0,16	0,01	0,08	0,01
E8	0,26	0,02	0,32	0,01	0,51	0,01	0,07	0,00	0,16	0,01	0,06	-0,01
E9	-	-	0,33	0,02	0,53	0,03	-	-	-	-	-	-
E10	0,26	0,02	0,34	0,03	-	-	-	-	-	-	-	-
E11	0,23	-0,01	0,33	0,02	0,50	0,01	0,08	0,01	0,16	0,01	0,08	0,01
E12	0,25	0,01	0,32	0,01	0,50	0,01	0,08	0,01	0,18	0,03	-	-
Average	0,24		0,32		0,50		0,08		0,16		0,07	
Max.	0,28		0,34		0,53		0,11		0,18		0,08	
Min.	0,12		0,31		0,48		0,02		0,15		0,06	
Range	0,16		0,03		0,05		0,09		0,03		0,02	
S.D.	0,025		0,007		0,009		0,013		0,007		0,008	
C.V.	10,3		2,4		1,8		18,0		4,5		10,7	
Certified value	0,24		0,31		0,50		0,07		0,15		0,07	

NOTE Data in shaded cells represent concentrations that did not satisfy the required criteria for accuracy limits for "expert" laboratories specified for the XRF method.

[a] SO_3 determination for JCA-CRM-2 is outside the scope of JIS R 5204.

[b] "Mean value" is the mean value of the concentrations of a pair of validated beads.

[c] "Diff." is the difference between the mean value of the concentrations of a pair of validated beads and the certified value for the validation material.

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5.3 Test results of Q-laboratories

5.3.1 Differences in concentration for each pair of test samples

The differences in concentration for each pair of Sample JCA #1 and Sample JCA #2 is shown in Table 8 and Table 9.

Table 8 — Differences in concentrations for each pair of test sample
(Q-laboratories —Test sample: Sample JCA #1)

Laboratory ref.	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	SO_3 %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	SrO %
1	0,066	0,021	0,008	0,128	0,002	0,002	0,012	0,019	0,002	0,001	0,000	- [a]
2	0,015	0,008	0,003	0,066	0,005	0,030	0,003	0,001	0,002	0,004	0,002	0,000
3	0,082	0,021	0,023	0,139	0,027	0,020	0,003	0,001	0,005	0,001	0,000	-
4	0,098	0,008	0,007	0,015	0,016	0,001	0,008	0,008	0,006	0,003	0,001	0,001
5	0,008	0,006	0,002	0,027	0,006	0,014	0,001	0,001	0,000	0,001	0,000	0,000
6	0,085	0,030	0,005	0,175	0,006	0,019	0,018	0,011	0,006	0,001	0,000	0,000
7	0,016	0,004	0,003	0,062	0,013	0,001	0,005	0,000	0,000	0,001	0,001	0,000
8	0,064	0,001	0,002	0,007	0,005	0,007	0,000	0,002	0,003	0,001	0,003	0,000
9	0,010	0,001	0,003	0,062	0,000	0,007	0,000	0,001	0,003	0,003	0,000	0,000
10	0,050	0,024	0,001	0,120	0,031	0,010	0,014	0,001	0,002	0,010	0,000	-
11	0,005	0,046	0,017	0,224	0,006	0,040	0,001	0,007	0,005	0,003	0,002	-
12	0,022	0,006	0,004	0,079	0,010	0,020	0,003	0,002	0,006	0,000	0,000	0,001
13	0,028	0,010	0,001	0,083	0,006	0,007	0,002	0,001	0,001	0,001	0,001	0,000
14	0,058	0,003	0,003	0,026	0,004	0,014	0,000	0,001	0,009	0,001	0,000	0,000
15	0,056	0,035	0,018	0,199	0,022	0,030	0,011	0,003	0,003	0,002	0,002	0,000
16	0,022	0,014	0,002	0,108	0,008	0,003	0,006	0,003	0,001	0,004	0,001	0,000
104	0,102	0,035	0,040	0,135	0,010	0,029	0,005	0,019	0,007	0,004	0,001	0,000
106	0,083	0,007	0,011	0,128	0,001	0,025	0,004	0,002	-	-	-	-
108	0,020	0,000	0,000	0,060	0,010	0,010	0,000	0,000	-	0,000	-	-
109	0,034	0,000	0,003	0,053	0,008	0,012	-	0,007	-	-	-	-
E1	0,023	0,060	0,022	0,077	0,015	0,013	0,011	0,003	0,013	0,007	0,001	0,001
E4	0,079	0,020	0,003	0,066	0,007	0,026	0,006	0,004	0,002	0,000	0,000	0,004
E5	0,020	0,060	0,010	0,120	0,050	0,000	0,000	0,010	0,012	0,012	0,004	0,001
E6	0,024	0,017	0,011	0,189	0,004	0,015	0,004	0,000	0,001	0,004	0,001	-
E7	0,120	0,032	0,019	0,070	0,034	0,020	0,003	0,004	0,001	0,011	0,001	0,002
E8	0,019	0,021	0,017	0,091	0,021	0,002	0,011	0,001	0,004	0,002	0,000	0,000
E9	0,088	0,050	0,009	0,138	0,006	-	0,013	0,010	0,012	-	-	-
Average	0,049	0,033	0,011	0,101	0,018	0,012	0,006	0,004	0,006	0,005	0,001	0,002
Max.	0,120	0,060	0,022	0,189	0,050	0,026	0,013	0,019	0,013	0,012	0,004	0,004
Min.	0,019	0,000	0,000	0,060	0,004	0,000	0,000	0,000	0,000	0,000	0,000	0,000
Range	0,101	0,060	0,022	0,129	0,046	0,026	0,013	0,019	0,013	0,012	0,004	0,004
Mean analysis	20,91	5,09	3,03	65,95	1,37	2,21	0,21	0,50	0,31	0,24	0,10	0,05
Limit value	0,150	0,080	0,080	0,250	0,080	0,080	0,020	0,030	0,030	0,020	0,020	0,020

[a] "-" indicates that there was no result reported by the laboratory.

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Table 9 — Differences in concentrations for each pair of test sample
(Q-laboratories — Test sample: Sample JCA #2)

Laboratory ref.	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	SO_3 [a] %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	SrO %
1	0,014	0,011	0,002	0,077	0,002	0,006	0,000	0,003	0,001	0,000	0,000	- [b]
2	0,027	0,006	0,006	0,022	0,018	0,004	0,002	0,001	0,006	0,001	0,000	0,001
3	0,015	0,000	0,005	0,013	0,006	-	0,003	0,000	0,002	0,000	0,000	-
4	0,015	0,005	0,005	0,017	0,005	0,008	0,000	0,000	0,003	0,002	0,001	0,000
5	0,019	0,008	0,003	0,083	0,004	0,018	0,001	0,002	0,001	0,002	0,000	0,001
6	0,066	0,003	0,022	0,050	0,011	0,052	0,004	0,001	0,004	0,006	0,000	0,000
7	0,015	0,006	0,000	0,002	0,014	0,002	0,002	0,001	0,004	0,001	0,001	0,000
8	0,061	0,001	0,001	0,014	0,002	0,003	0,006	0,001	0,002	0,001	0,000	0,000
9	0,012	0,013	0,002	0,010	0,004	-	0,000	0,000	0,003	0,001	0,001	0,000
10	0,081	0,016	0,002	0,204	0,011	-	0,007	0,000	0,000	0,001	0,001	-
11	0,067	0,065	0,015	0,120	0,017	0,038	0,008	0,002	0,005	0,001	0,001	-
12	0,039	0,045	0,028	0,115	0,027	-	0,004	0,005	0,006	0,001	0,001	0,001
13	0,011	0,064	0,007	0,109	0,007	0,013	0,001	0,003	0,012	0,003	0,000	0,000
14	0,094	0,040	0,006	0,061	0,001	0,013	0,002	0,000	0,001	0,001	0,000	0,000
15	0,003	0,024	0,004	0,047	0,040	0,039	0,008	0,003	0,002	0,000	0,000	0,000
16	0,013	0,018	0,001	0,050	0,015	0,024	0,008	0,002	0,007	0,002	0,001	0,000
104	0,038	0,022	0,011	0,045	0,006	0,036	0,010	0,016	0,003	0,007	0,001	0,001
106	0,082	0,077	0,013	0,187	0,011	-	0,004	0,001	-	-	-	-
108	0,020	0,010	0,000	0,100	0,050	0,030	0,020	0,000	-	0,000	-	-
109	0,041	0,019	0,052	0,142	0,012	0,090	-	0,004	-	-	-	-
E1	0,045	0,063	0,025	0,121	0,002	0,179	0,008	0,005	0,007	0,007	0,001	0,001
E4	0,062	0,054	0,001	0,111	0,026	0,047	0,013	0,001	0,001	0,002	0,004	0,002
E5	0,040	0,030	0,010	0,120	0,100	0,030	0,015	0,000	0,003	0,010	0,001	0,002
E6	0,002	0,019	0,008	0,041	0,033	-	0,000	0,003	0,000	0,007	0,002	-
E7	0,118	0,052	0,001	0,179	0,008	0,013	0,003	0,002	0,003	0,005	0,001	0,001
E8	0,049	0,003	0,014	0,070	0,038	0,005	0,008	0,002	0,000	0,004	0,000	0,000
E9	0,063	0,025	0,006	0,082	0,008	-	-	0,001	0,016	-	-	-
Average	0,041	0,026	0,009	0,081	0,018	0,033	0,005	0,002	0,004	0,003	0,001	0,001
Max.	0,118	0,077	0,052	0,204	0,100	0,179	0,020	0,016	0,016	0,010	0,004	0,002
Min.	0,002	0,000	0,000	0,002	0,001	0,002	0,000	0,000	0,000	0,000	0,000	0,000
Range	0,116	0,077	0,052	0,202	0,099	0,177	0,020	0,016	0,016	0,010	0,004	0,002
Mean analysis	26,00	8,81	1,62	56,12	3,35	2,82	0,21	0,42	0,39	0,24	0,09	0,05
Limit value	0,150	0,120	0,080	0,250	0,080	-	0,020	0,020	0,020	0,020	0,020	0,020

NOTE Data in shaded cells represent results that did not satisfy the required criteria for repeatability specified for "expert" laboratories for the XRF method.

[a] SO_3 determination for sample JCA #2 is outside the scope of JIS R 5204.

[b] "-" indicates that there was no result reported by the laboratory.

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5.3.2 Distribution of concentrations for test samples

Each concentration obtained by Q-laboratories for Sample JCA #1 and Sample JCA #2 is shown in Table 10 and Table 11. "Mean value" represents the average of two concentrations, and "Diff." represents the difference between the "Mean value" and the average, respectively. Data in parentheses are "outliers" by the Grubbs test, which was excluded from statistic calculation of the results.

The Grubbs test specified in 7.3.4 of ISO 5725-2:1994 was carried out when analysing results. "Outlier" means 5% "outlier" by the Grubbs test and "Outliers in Q-laboratory" are shown in the histograms. However, although both the minimum of K_2O and the maximum of SrO for Sample JCA #2 were judged as "outliers", it was concluded that they were not "extreme results" and therefore, they were not excluded.

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With regard to judgment of validation for results obtained by XRF analysis using validated calibration, it would be unlikely that the analytical accuracy of the results varies significantly in the same kind of sample without a failure in one of the processes of XRF analysis. For example, both JCA-CRM-1 and Sample JCA #1 are Portland cement, and both JCA-CRM-2 and Sample JCA #2 are Portland blast furnace slag cement.

It is normal practice that when there is a statistical "outlier" it is necessary to conduct an investigation to determine the cause.

The histograms of concentrations of test samples for all constituents are shown in Figure 1 to Figure 23. The data group shown by the shaded bars indicates the data of Q-laboratories, and the white bars indicate the data of the other laboratories (non-Q-laboratories). The average and two times the standard deviation (2σ) shown in the histograms were calculated after excluding "outliers".

The concentrations obtained by Q-laboratories were generally distributed between the average $\pm 2\sigma$ for all constituents. On the other hand, the distribution of concentrations of non-Q-laboratories was wider than that of Q-laboratories, and there were some concentrations that were far from the average. As reported above, it is important for accurate analysis to use calibration equations validated in accordance with this XRF method.

Table 10 — Concentrations for sample JCA #1 — Q-laboratories

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Mean value [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]
1	20,93	-0,03	5,11	0,01	2,99	-0,04	65,97	0,08	1,36	0,00	2,23	0,02
2	20,85	-0,11	5,10	0,00	3,02	-0,01	65,95	0,06	1,36	0,00	2,19	-0,02
3	20,96	0,00	5,11	0,01	3,03	0,00	65,86	-0,03	1,34	-0,02	2,19	-0,02
4	20,88	-0,08	5,10	0,00	3,04	0,01	65,87	-0,02	1,36	0,00	2,25	0,04
5	20,90	-0,06	5,12	0,02	3,04	0,01	65,94	0,05	1,36	0,00	2,22	0,01
6	21,04	0,08	5,12	0,02	2,99	-0,04	65,91	0,02	1,34	-0,02	2,23	0,02
7	20,92	-0,04	5,10	0,00	3,03	0,00	65,76	-0,13	1,36	0,00	2,21	0,00
8	20,96	0,00	5,13	0,03	3,03	0,00	65,80	-0,09	1,38	0,02	2,21	0,00
9	20,83	-0,13	5,10	0,00	2,98	-0,05	65,89	0,00	1,36	0,00	2,23	0,02
10	20,98	0,02	5,08	-0,02	3,01	-0,02	66,10	0,21	1,36	0,00	2,22	0,01
11	21,01	0,05	5,12	0,02	3,05	0,02	65,89	0,00	1,36	0,00	(1,99) [c]	-
12	20,96	0,00	5,11	0,01	3,05	0,02	65,84	-0,05	1,34	-0,02	2,17	-0,04
13	21,08	0,12	5,12	0,02	3,06	0,03	65,68	-0,21	1,37	0,01	2,21	0,00
14	20,96	0,00	5,13	0,03	3,10	0,07	66,15	0,26	1,37	0,01	2,20	-0,01
15	21,07	0,11	5,12	0,02	3,05	0,02	65,86	-0,03	1,33	-0,03	2,16	-0,05
16	20,96	0,00	5,12	0,02	3,04	0,01	65,77	-0,12	1,38	0,02	2,21	0,00
104	20,95	-0,01	5,09	-0,01	3,00	-0,03	65,51	-0,38	1,37	0,01	2,15	-0,06
106	(20,47) [c]	-	5,05	-0,05	2,97	-0,06	(64,61) [c]	-	1,33	-0,03	2,17	-0,04
108	21,01	0,05	5,12	0,02	3,00	-0,03	65,88	-0,01	1,36	0,00	2,22	0,01
109	20,80	-0,16	5,06	-0,04	3,00	-0,03	65,88	-0,01	1,35	-0,01	2,11	-0,10
E1	20,89	-0,06	5,12	0,01	3,04	0,01	65,86	-0,03	1,36	0,00	2,30	0,09
E4	20,94	-0,01	5,10	-0,01	2,99	-0,03	65,71	-0,18	1,30	-0,06	2,21	0,00
E5	21,06	0,10	5,07	-0,03	3,02	-0,01	66,00	0,11	1,39	0,03	2,22	0,01
E6	21,10	0,14	5,10	-0,01	3,08	0,06	66,37	0,48	1,37	0,02	2,24	0,03
E7	20,86	-0,10	5,09	-0,01	3,08	0,05	65,83	-0,06	1,37	0,01	2,22	0,01
E8	20,92	-0,04	5,10	0,00	2,98	-0,04	65,91	0,02	1,37	0,01	2,22	0,01
E9	21,01	0,05	5,13	0,02	3,04	0,01	65,91	0,02	1,35	-0,01	-	-
Average	20,96		5,10		3,03		65,89		1,36		2,21	
Max.	21,10		5,13		3,10		66,37		1,39		2,30	
Min.	20,80		5,05		2,97		65,51		1,30		2,11	
Range	0,30		0,08		0,13		0,86		0,09		0,19	
S.D.	0,078		0,021		0,033		0,160		0,018		0,037	
C.V.	0,4		0,4		1,1		0,2		1,4		1,7	

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Table 10 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %	
	Mean value [a]	**Diff.** [b]	**Mean Value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]	**Mean value** [a]	**Diff.** [b]
1	0,21	0,00	(0,45) [c]	-	0,31	0,00	0,24	0,00	0,10	0,00	-	-
2	0,21	0,00	0,50	0,00	0,32	0,01	0,24	0,00	0,10	0,00	0,05	0,00
3	0,19	-0,02	0,50	0,00	0,32	0,01	0,23	-0,01	0,09	-0,01	-	-
4	0,21	0,00	0,50	0,00	0,32	0,01	0,24	0,00	0,10	0,00	0,05	0,00
5	0,21	0,00	0,49	-0,01	0,32	0,01	0,24	0,00	0,10	0,00	0,05	0,00
6	0,23	0,02	0,52	0,02	0,31	0,00	0,24	0,00	0,09	-0,01	0,04	-0,01
7	0,20	-0,01	0,49	-0,01	0,32	0,01	0,24	0,00	0,10	0,00	0,05	0,00
8	0,19	-0,02	0,49	-0,01	0,32	0,01	0,24	0,00	0,09	-0,01	0,05	0,00
9	0,21	0,00	0,50	0,00	0,31	0,00	0,24	0,00	0,10	0,00	0,04	-0,01
10	0,18	-0,03	0,50	0,00	0,32	0,01	0,24	0,00	0,10	0,00	-	-
11	0,19	-0,02	0,52	0,02	0,32	0,01	0,24	0,00	0,10	0,00	-	-
12	0,21	0,00	0,48	-0,02	0,32	0,01	0,24	0,00	0,09	-0,01	0,05	0,00
13	0,21	0,00	0,49	-0,01	0,31	0,00	0,25	0,01	0,09	-0,01	0,05	0,00
14	0,21	0,00	0,49	-0,01	0,30	-0,01	0,25	0,01	0,10	0,00	0,05	0,00
15	0,18	-0,03	0,49	-0,01	0,31	0,00	0,24	0,00	0,10	0,00	0,05	0,00
16	0,21	0,00	0,50	0,00	0,32	0,01	0,24	0,00	0,09	-0,01	0,05	0,00
104	(0,13) [c]	-	0,49	-0,01	0,31	0,00	0,23	-0,01	0,09	-0,01	0,04	-0,01
106	0,20	-0,01	0,49	-0,01	-	-	-	-	-	-	-	-
108	(0,13) [c]	-	0,49	-0,01	-	-	0,24	0,00	-	-	-	-
109	-	-	0,50	0,00	-	-	-	-	-	-	-	-
E1	0,20	-0,01	0,49	0,00	0,32	0,01	0,24	0,00	0,10	0,00	0,05	0,00
E4	0,23	0,02	0,49	-0,01	0,31	0,00	0,24	0,00	0,09	0,00	0,05	0,01
E5	0,23	0,03	0,51	0,01	0,31	0,00	0,23	-0,01	0,10	0,00	0,05	0,00
E6	(0,10) [c]	-	0,50	0,00	0,31	0,00	0,27	0,03	0,09	-0,01	-	-
E7	0,21	0,00	0,50	0,00	0,32	0,01	0,24	0,00	0,10	0,00	0,06	0,01
E8	0,21	0,00	0,50	0,00	0,31	0,00	0,24	0,00	0,10	0,00	0,05	0,00
E9	0,26	0,05	0,51	0,01	0,30	-0,01	-	-	-	-	-	-
Average	0,21		0,50		0,31		0,24		0,10		0,05	
Max.	0,26		0,52		0,32		0,27		0,10		0,06	
Min.	0,18		0,48		0,30		0,23		0,09		0,04	
Range	0,08		0,04		0,02		0,04		0,01		0,02	
S.D.	0,019		0,009		0,007		0,008		0,005		0,005	
C.V.	8,9		1,8		2,1		3,4		4,9		10,8	

[a] "Mean value" is the mean value of the concentrations of a pair of analysis beads.

[b] "Diff." is the difference between the mean value of the concentrations of a pair of analysis beads and the average for all Q-laboratories.

[c] Concentrations in parentheses are the "outliers" by the Grubbs test.

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Table 11 — Concentrations for Sample JCA #2 — Q-laboratories

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 [a] %	
	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]	Mean value [b]	Diff. [c]
1	26,02	0,01	8,83	0,03	1,62	0,00	56,03	-0,06	3,34	0,02	2,90	0,04
2	25,83	-0,18	8,76	-0,04	1,63	0,01	56,14	0,05	3,31	-0,01	2,90	0,04
3	25,99	-0,02	8,82	0,02	1,56	-0,06	56,25	0,16	3,29	-0,03	-	-
4	26,00	-0,01	8,78	-0,02	1,65	0,03	55,90	-0,19	3,32	0,00	2,92	0,06
5	25,95	-0,06	8,80	0,00	1,62	0,00	56,16	0,07	3,31	-0,01	2,89	0,03
6	26,16	0,15	(8,98) [d]	-	1,63	0,01	56,20	0,11	3,38	0,06	2,83	-0,03
7	25,99	-0,02	8,82	0,02	1,61	-0,01	56,06	-0,03	3,35	0,03	2,95	0,09
8	26,04	0,03	8,77	-0,03	(1,51) [d]	-	55,63	-0,46	3,34	0,02	2,68	-0,18
9	25,91	-0,10	8,77	-0,03	1,63	0,01	56,01	-0,08	3,32	0,00	-	-
10	25,94	-0,07	8,77	-0,03	1,63	0,01	56,09	0,00	3,29	-0,03	-	-
11	25,91	-0,10	8,72	-0,08	1,61	-0,01	56,08	-0,01	3,28	-0,04	2,72	-0,14
12	26,03	0,02	8,79	-0,01	1,58	-0,04	55,95	-0,14	3,33	0,01	-	-
13	26,10	0,09	8,83	0,03	1,63	0,01	55,90	-0,19	3,32	0,00	2,96	0,10
14	26,06	0,05	8,81	0,01	1,64	0,02	56,11	0,02	3,31	-0,01	2,97	0,11
15	26,15	0,14	8,87	0,07	1,64	0,02	55,82	-0,27	3,30	-0,02	2,93	0,07
16	26,02	0,01	8,80	0,00	1,61	-0,01	56,21	0,12	3,32	0,00	2,93	0,07
104	25,98	-0,03	8,78	-0,02	1,63	0,01	56,08	-0,01	3,31	-0,01	(1,80) [d]	-
106	25,97	-0,04	8,80	0,00	1,57	-0,05	55,98	-0,11	3,28	-0,04	-	-
108	26,07	0,06	8,76	-0,04	1,63	0,01	56,22	0,13	3,32	0,00	2,88	0,02
109	26,15	0,14	8,85	0,05	1,66	0,04	56,50	0,41	3,35	0,03	(1,66) [d]	-
E1	25,95	-0,06	8,84	0,04	1,62	0,00	56,10	0,02	3,32	0,00	2,52	-0,34
E4	25,87	-0,14	8,80	0,00	1,60	-0,02	(55,31) [d]	-	(3,23) [d]	-	(3,75) [d]	-
E5	26,00	-0,01	8,75	-0,05	1,60	-0,02	56,11	0,02	3,32	0,00	(2,09) [d]	-
E6	26,07	0,06	8,79	-0,01	1,64	0,02	56,53	0,44	3,36	0,04	-	-
E7	25,95	-0,06	8,76	-0,04	1,63	0,01	55,93	-0,16	3,32	0,00	2,94	0,07
E8	26,03	0,02	8,81	0,02	1,63	0,01	55,94	-0,15	3,34	0,02	2,88	0,02
E9	26,09	0,08	8,87	0,07	1,58	-0,04	56,29	0,20	3,34	0,01	-	-
Average	26,01		8,80		1,62		56,09		3,32		2,86	
Max.	26,16		8,87		1,66		56,53		3,38		2,97	
Min.	25,83		8,72		1,56		55,63		3,28		2,52	
Range	0,33		0,15		0,10		0,90		0,10		0,45	
S.D.	0,083		0,037		0,025		0,192		0,024		0,121	
C.V.	0,3		0,4		1,5		0,3		0,7		4,2	

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Table 11 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %	
	Mean value [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]	**Mean value** [b]	**Diff.** [c]
1	0,25	0,03	0,40	-0,02	0,40	0,01	0,24	0,00	0,10	0,01	-	-
2	0,23	0,01	0,43	0,01	0,39	0,00	0,24	0,00	0,09	0,00	0,05	0,00
3	0,21	-0,01	0,43	0,01	0,40	0,01	0,24	0,00	0,10	0,01	-	-
4	0,22	0,00	0,42	0,00	0,39	0,00	0,24	0,00	0,09	0,00	0,05	0,00
5	0,22	0,00	0,43	0,01	0,39	0,00	0,24	0,00	0,09	0,00	0,05	0,00
6	0,19	-0,03	0,42	0,00	0,39	0,00	0,23	-0,01	0,09	0,00	0,05	0,00
7	0,21	-0,01	0,43	0,01	0,39	0,00	0,24	0,00	0,09	0,00	0,05	0,00
8	0,22	0,00	0,43	0,01	0,40	0,01	0,24	0,00	0,09	0,00	0,05	0,00
9	0,23	0,01	0,41	-0,01	0,40	0,01	0,23	-0,01	0,10	0,01	0,05	0,00
10	0,21	-0,01	0,43	0,01	0,40	0,01	0,23	-0,01	0,10	0,01	-	-
11	0,20	-0,02	0,42	0,00	0,38	-0,01	0,24	0,00	0,09	0,00	-	-
12	0,23	0,01	0,42	0,00	0,40	0,01	0,23	-0,01	0,09	0,00	0,06	0,01
13	0,22	0,00	0,43	0,01	0,40	0,01	0,25	0,01	0,08	-0,01	0,05	0,00
14	0,22	0,00	0,43	0,01	0,38	-0,01	0,25	0,01	0,09	0,00	0,05	0,00
15	0,20	-0,02	0,43	0,01	0,38	-0,01	0,25	0,01	0,09	0,00	0,05	0,00
16	0,22	0,00	0,42	0,00	0,38	-0,01	0,24	0,00	0,09	0,00	0,05	0,00
104	0,19	-0,03	0,42	0,00	0,39	0,00	0,23	-0,01	0,09	0,00	0,05	0,00
106	0,24	0,02	0,43	0,01	-	-	-	-	-	-	-	-
108	(0,15) [d]	-	0,43	0,01	-	-	0,23	-0,01	-	-	-	-
109	-	-	0,43	0,01	-	-	-	-	-	-	-	-
E1	0,21	-0,01	0,43	0,01	0,39	-0,01	0,25	0,01	0,09	-0,01	0,05	0,00
E4	0,23	0,01	0,42	-0,01	0,39	0,00	0,23	-0,01	0,10	0,01	0,05	0,00
E5	0,23	0,01	0,43	0,01	0,40	0,01	0,23	-0,01	0,09	0,00	0,05	0,00
E6	(0,10) [d]	-	0,43	0,01	0,38	-0,01	0,27	0,03	0,08	-0,01	-	-
E7	0,23	0,01	0,43	0,00	0,39	0,00	0,24	0,00	0,09	0,00	0,06	0,01
E8	0,22	0,00	0,42	0,00	0,40	0,00	0,23	-0,01	0,10	0,00	0,05	0,00
E9	-	-	0,42	0,00	0,43	0,03	-	-	-	-	-	-
Average	0,22		0,42		0,39		0,24		0,09		0,05	
Max.	0,25		0,43		0,43		0,27		0,10		0,06	
Min.	0,19		0,40		0,38		0,23		0,08		0,05	
Range	0,06		0,03		0,05		0,04		0,02		0,01	
S.D.	0,014		0,008		0,010		0,009		0,006		0,003	
C.V.	6,6		1,8		2,6		3,9		6,4		5,8	

[a] SO_3 for sample JCA #2 is outside the scope of JIS R 5204.

[b] "Mean value" is the mean value of the concentrations of a pair of analysis beads.

[c] "Diff." is the difference between the mean value of the concentrations of a pair of analysis beads and the average for all Q-laboratories.

[d] Concentrations in parentheses are the "outliers" by the Grubbs test.

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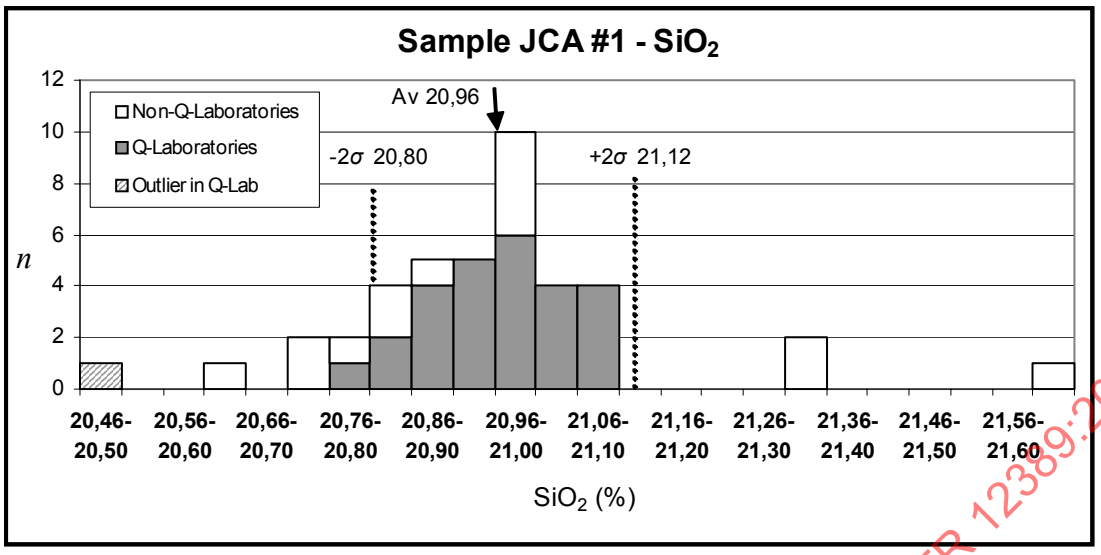

Figure 1 — The histogram of concentrations (Sample JCA #1, constituent SiO_2)

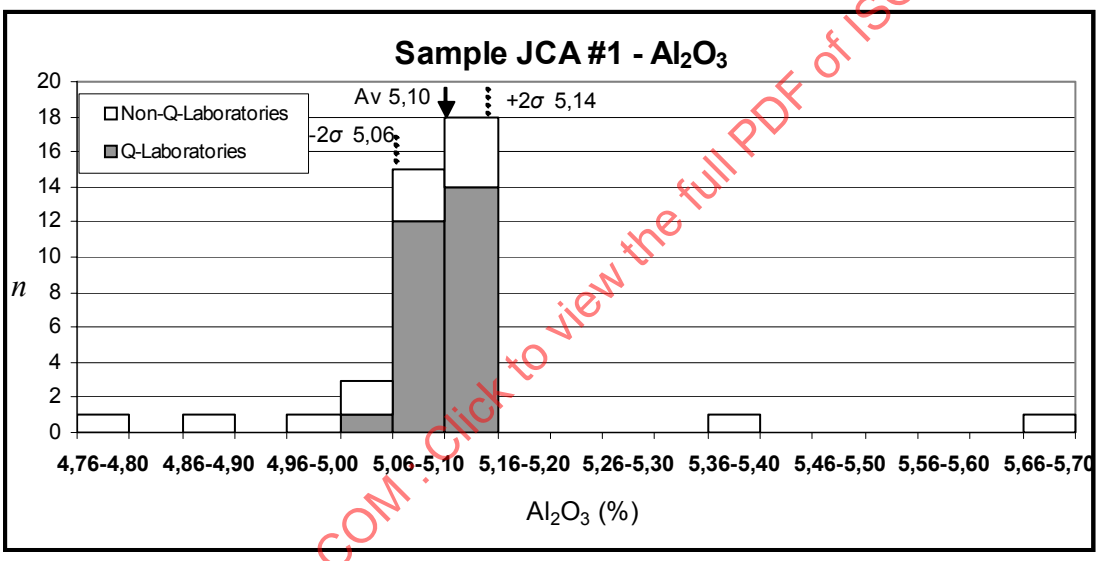

Figure 2 — The histogram of concentrations (Sample JCA #1, constituent Al_2O_3)

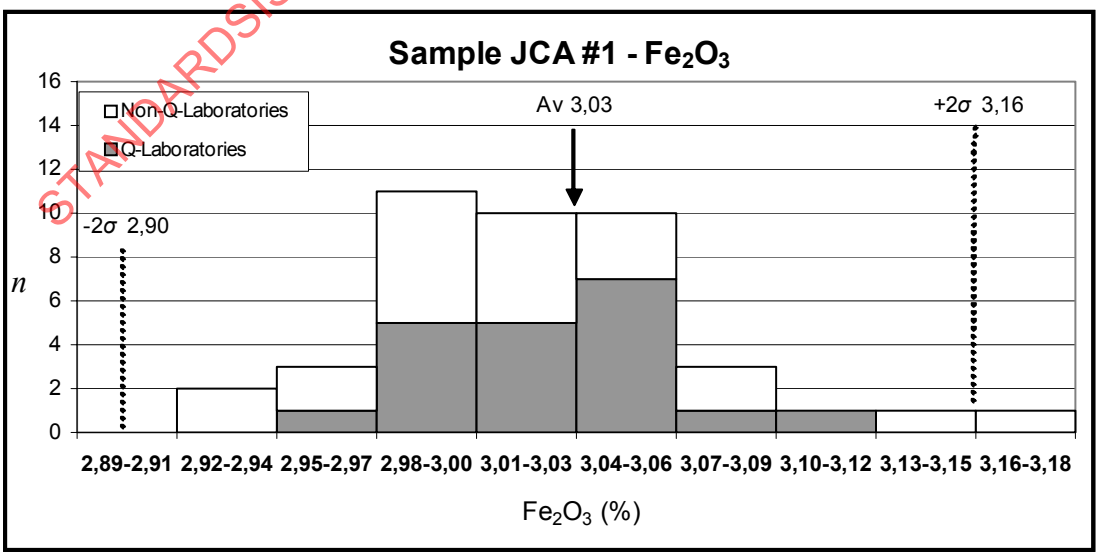

Figure 3 — The histogram of concentrations (Sample JCA #1, constituent Fe_2O_3)

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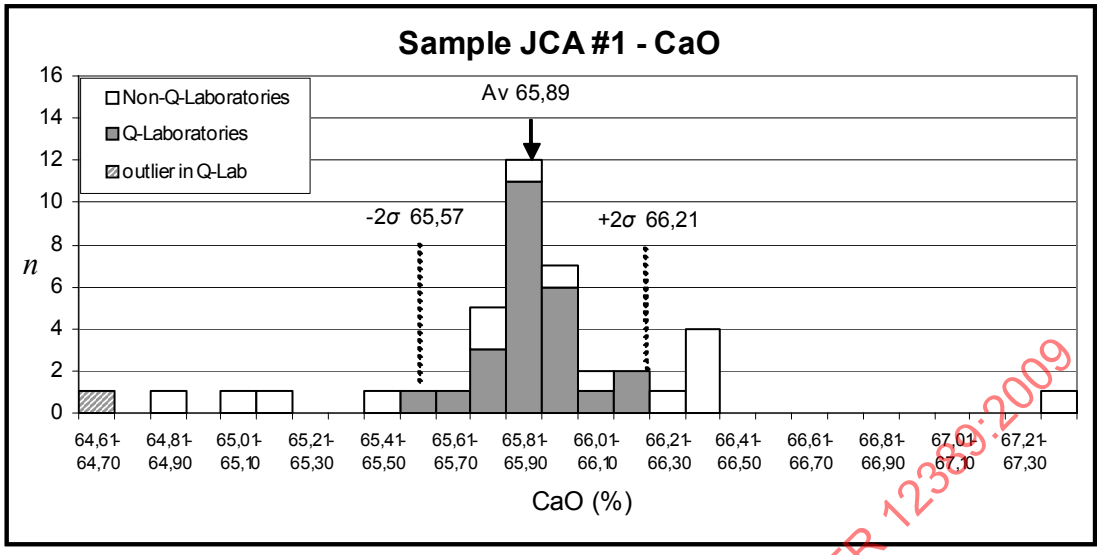

Figure 4 — The histogram of concentrations (Sample JCA #1, constituent CaO)

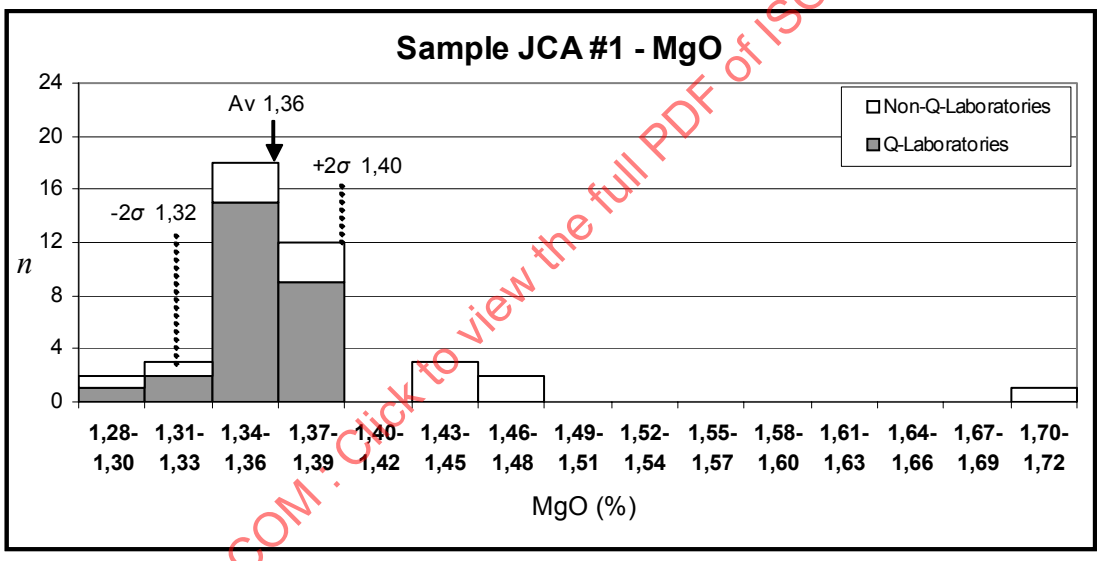

Figure 5 — The histogram of concentrations (Sample JCA #1, constituent MgO)

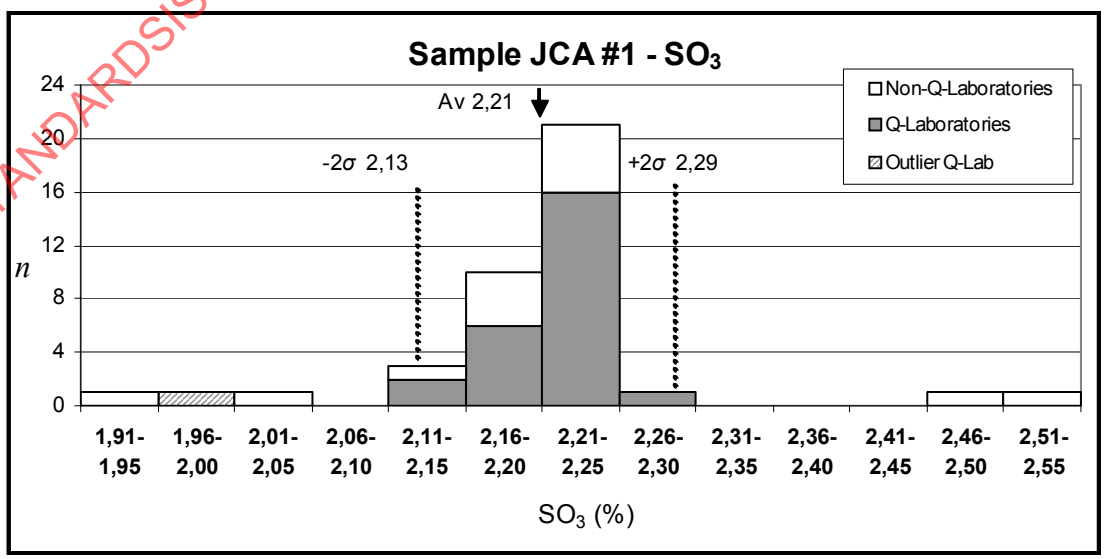

Figure 6 — The histogram of concentrations (Sample JCA #1, constituent SO_3)

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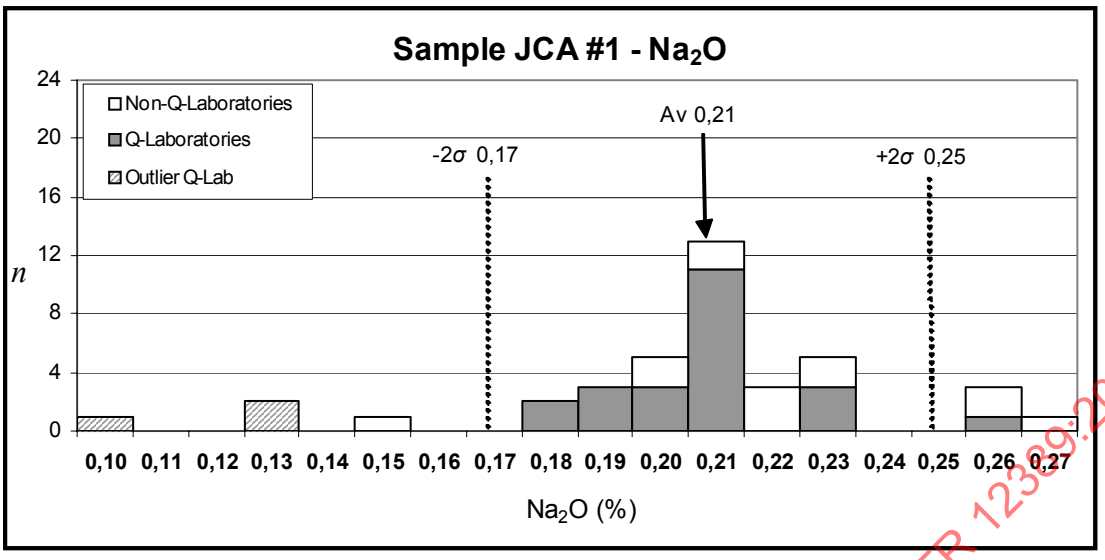

Figure 7 — The histogram of concentrations (Sample JCA #1, constituent Na_2O)

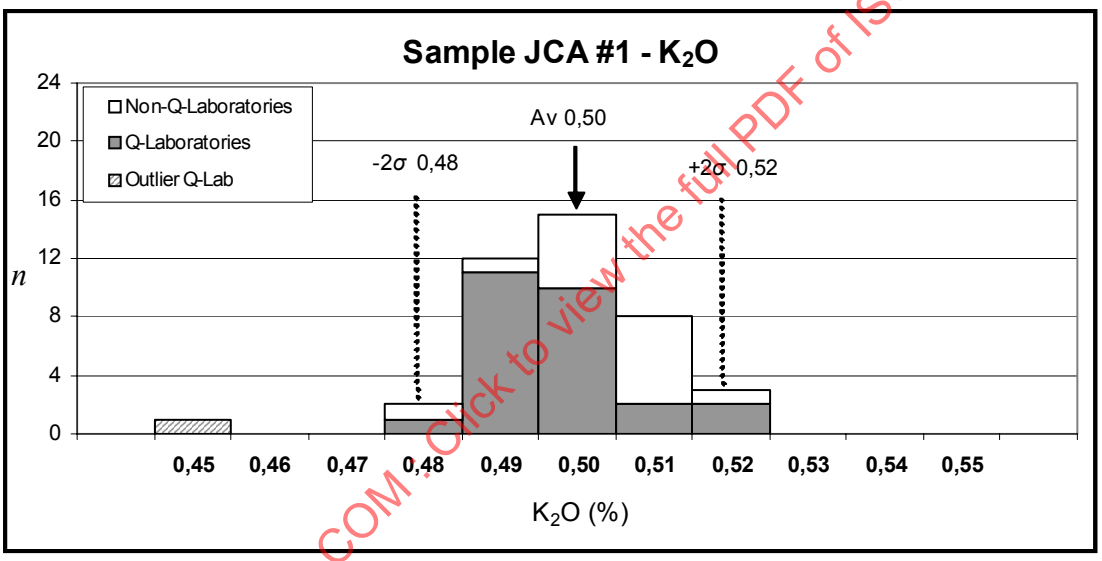

Figure 8 — The histogram of concentrations (Sample JCA #1, constituent K_2O)

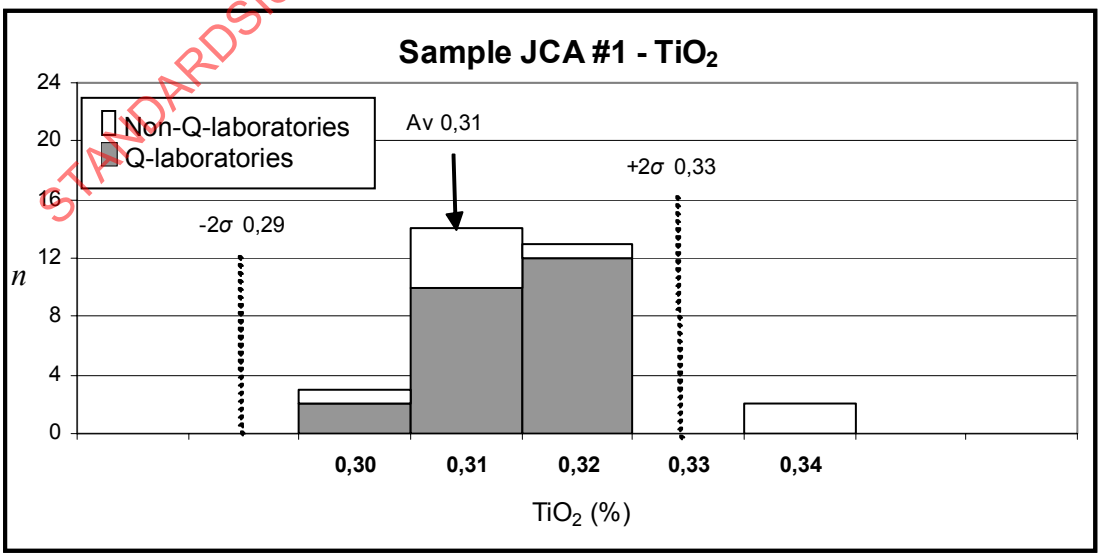

Figure 9 — The histogram of concentrations (Sample JCA #1, constituent TiO_2)

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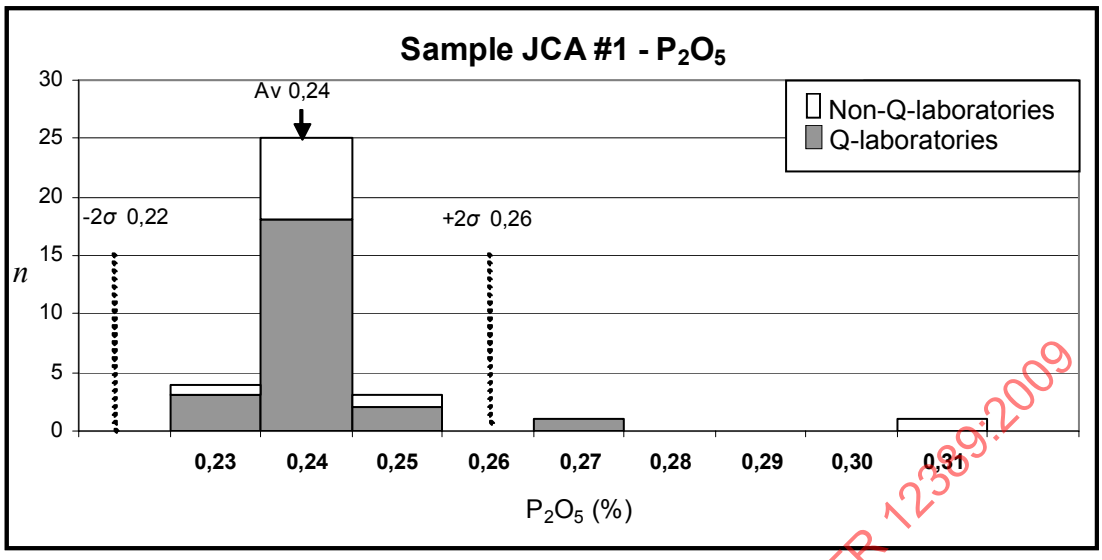

Figure 10 — The histogram of concentrations (Sample JCA #1, constituent P_2O_5)

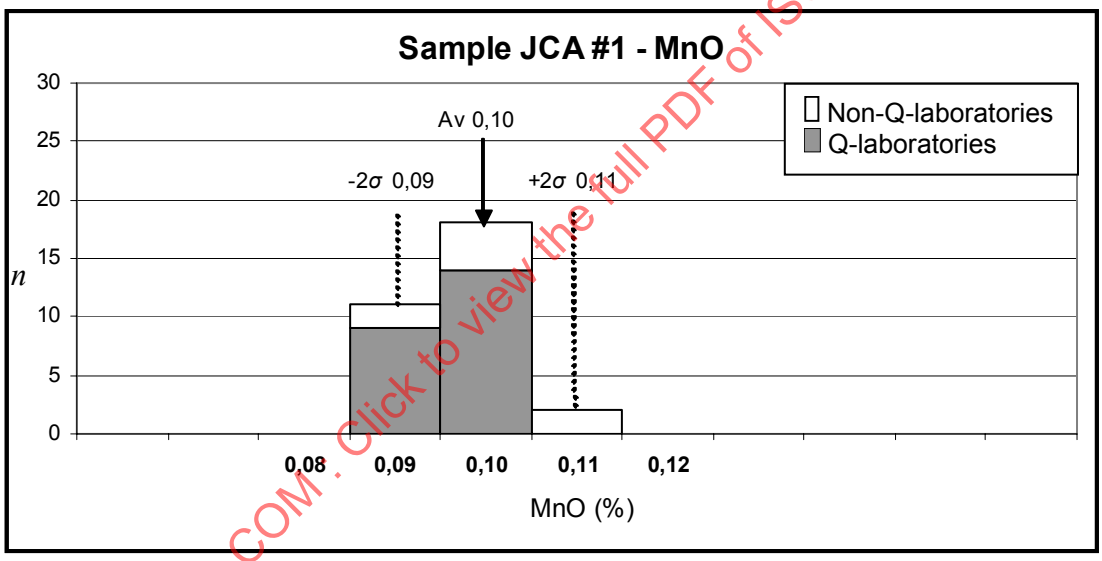

Figure 11 — The histogram of concentrations (Sample JCA #1 – constituent MnO)

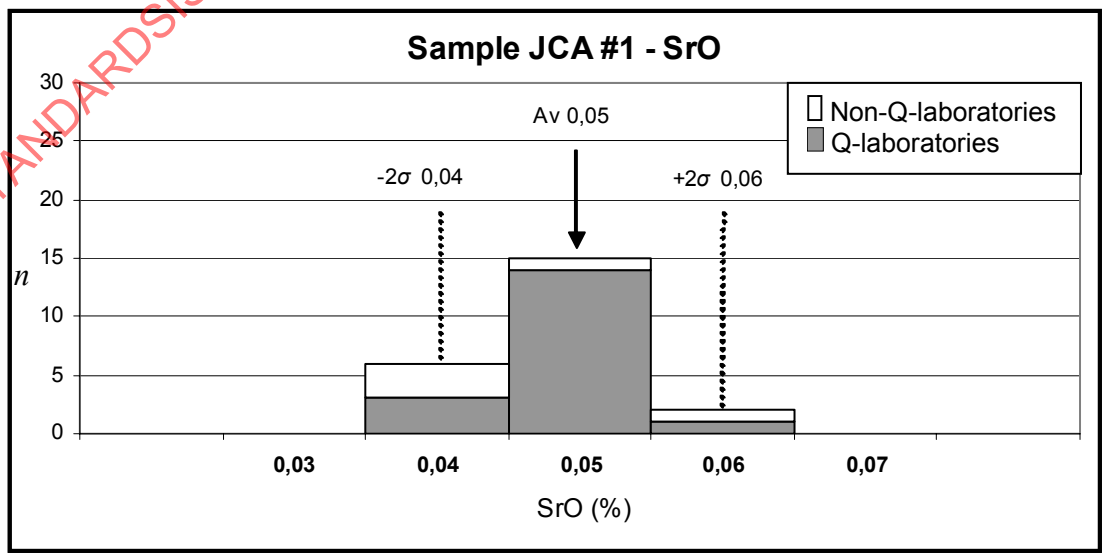

Figure 12 — The histogram of concentrations (Sample JCA #1, constituent SrO)

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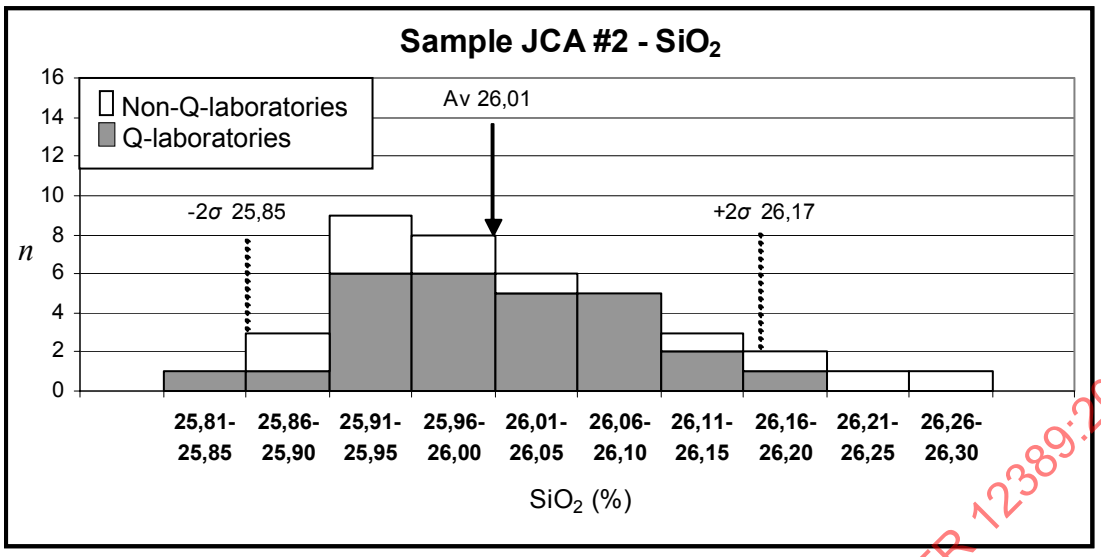

Figure 13 — The histogram of concentrations (Sample JCA #2, constituent SiO_2)

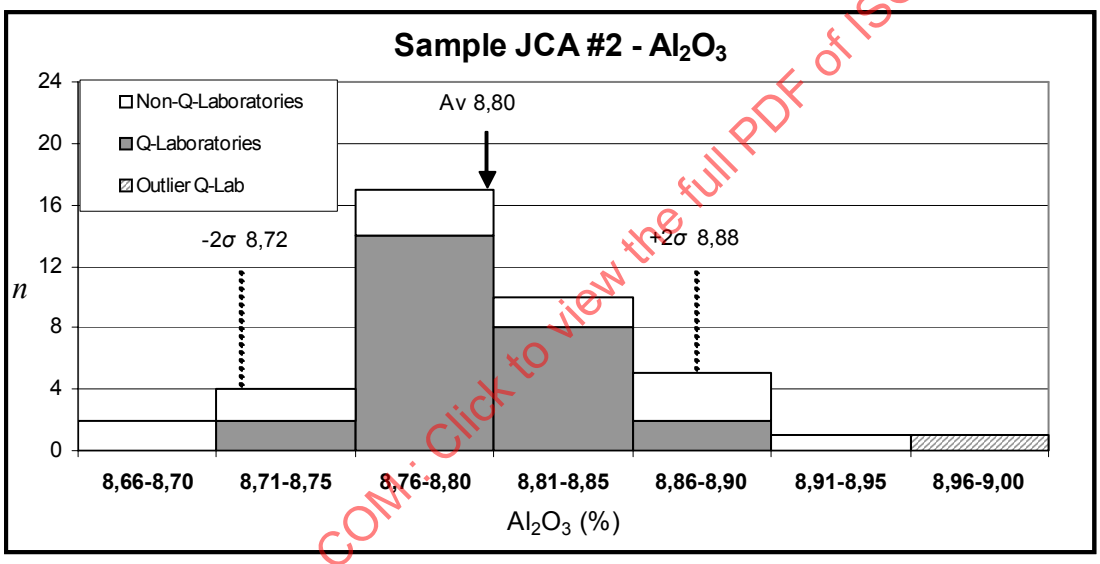

Figure 14 — The histogram of concentrations (Sample JCA #2, constituent Al_2O_3)

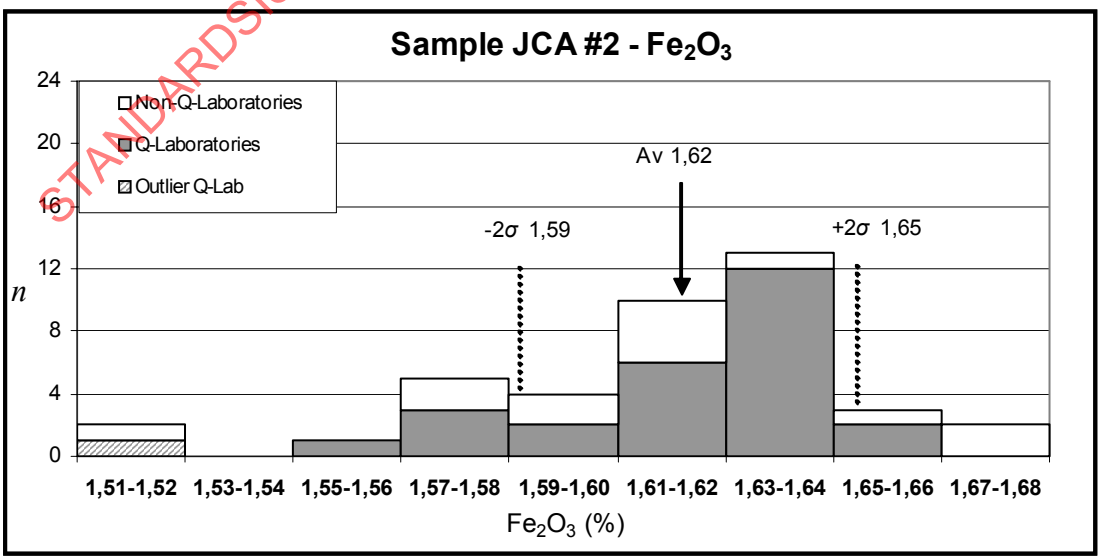

Figure 15 — The histogram of concentrations (Sample JCA #2, constituent Fe_2O_3)

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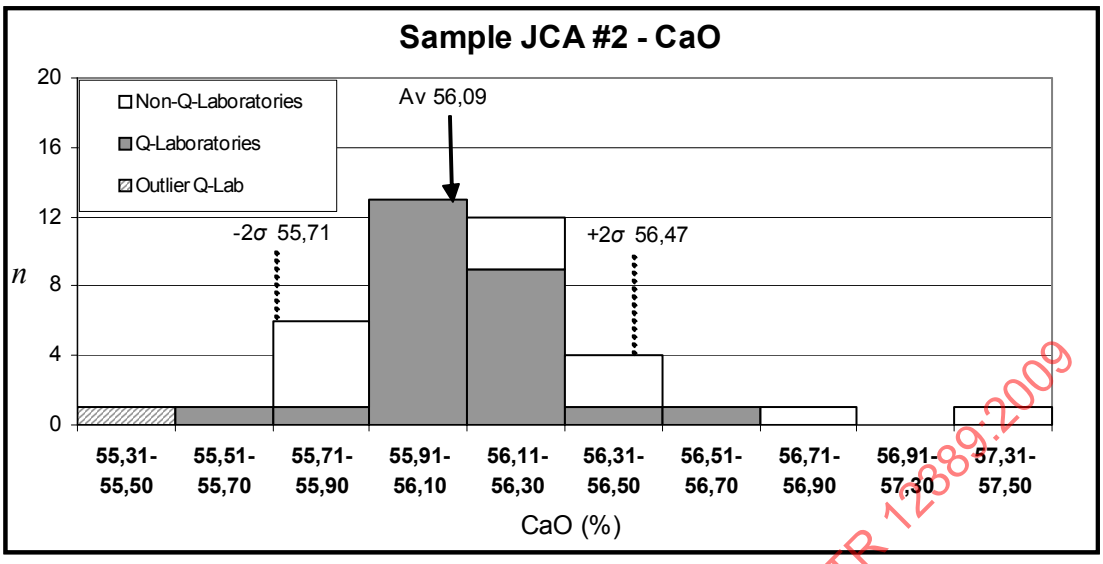

Figure 16 — The histogram of concentrations (Sample JCA #2, constituent CaO)

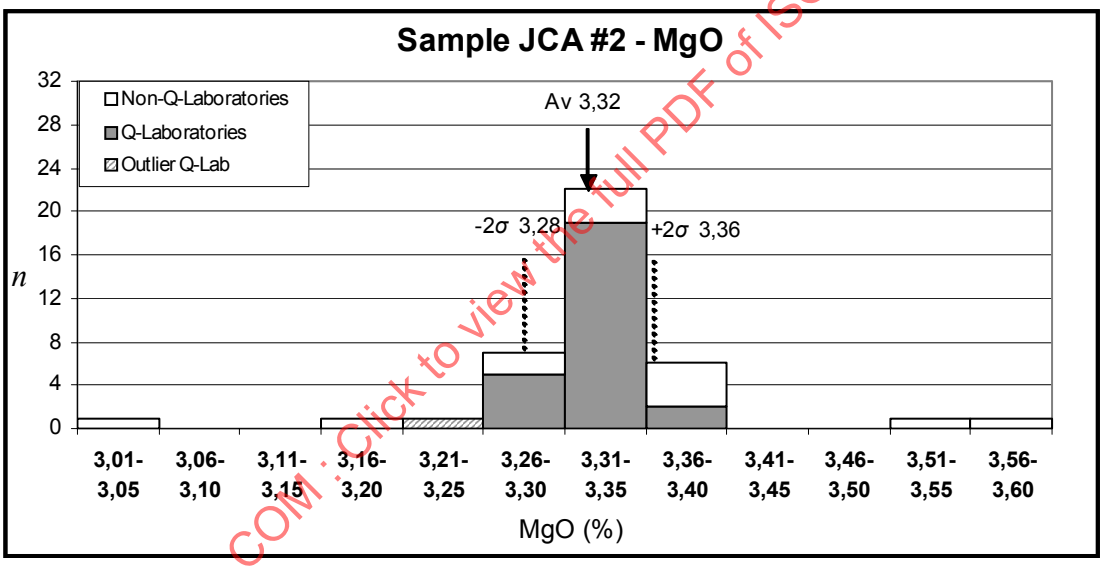

Figure 17 — The histogram of concentrations (Sample JCA #2, constituent MgO)

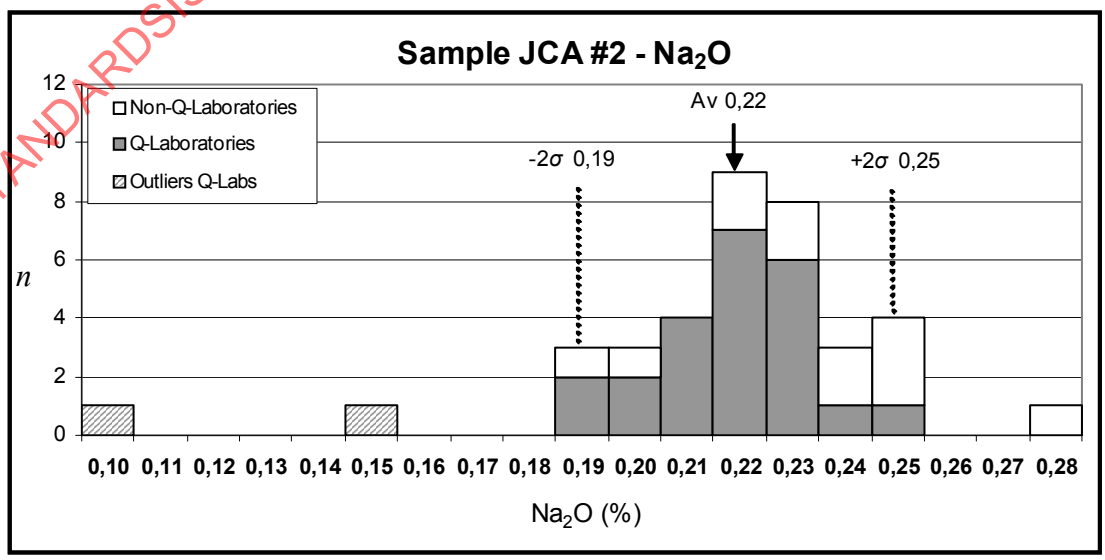

Figure 18 — The histogram of concentrations (Sample JCA #2, constituent Na_2O)

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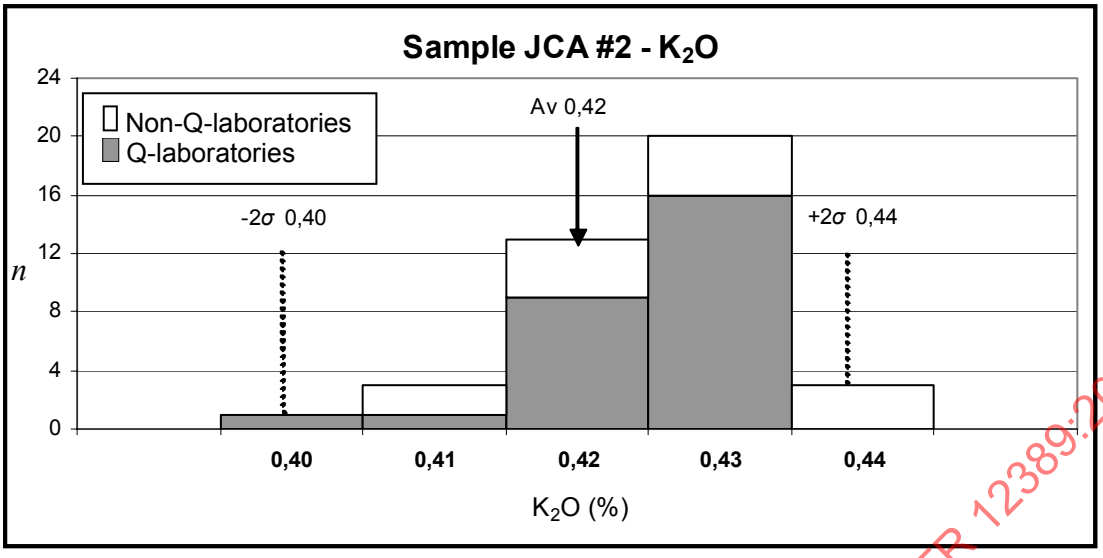

Figure 19 — The histogram of concentrations (Sample JCA #2, constituent K_2O)

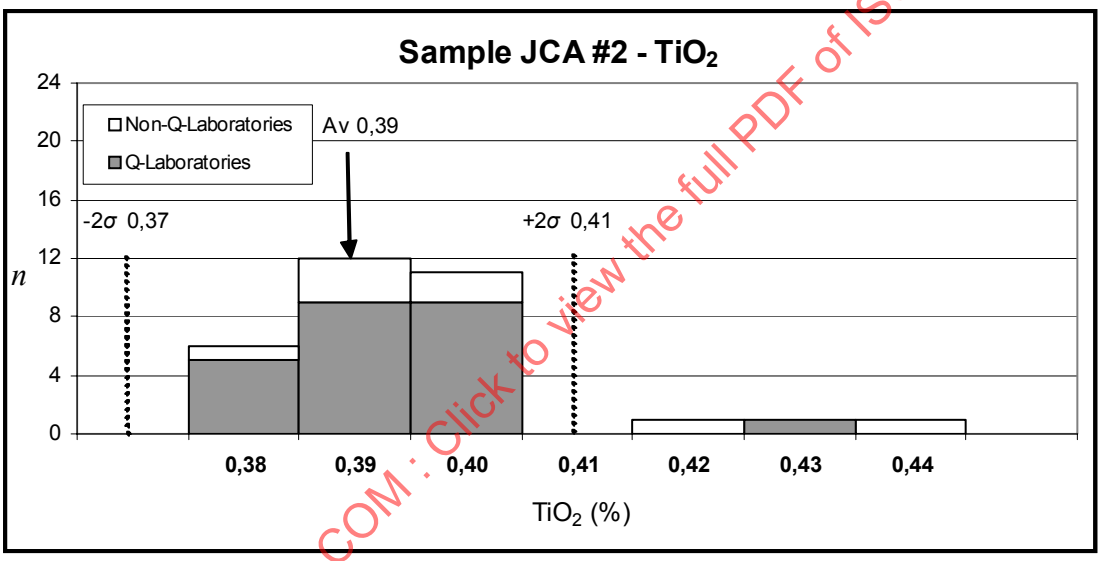

Figure 20 — The histogram of concentrations (Sample JCA #2, constituent TiO_2)

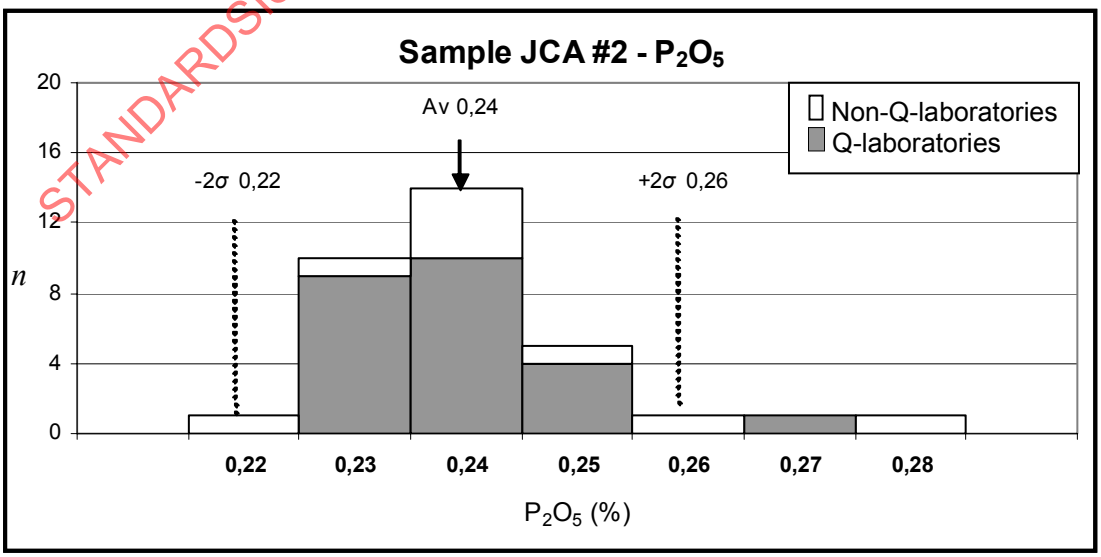

Figure 21 — The histogram of concentrations (Sample JCA #2, constituent P_2O_5)

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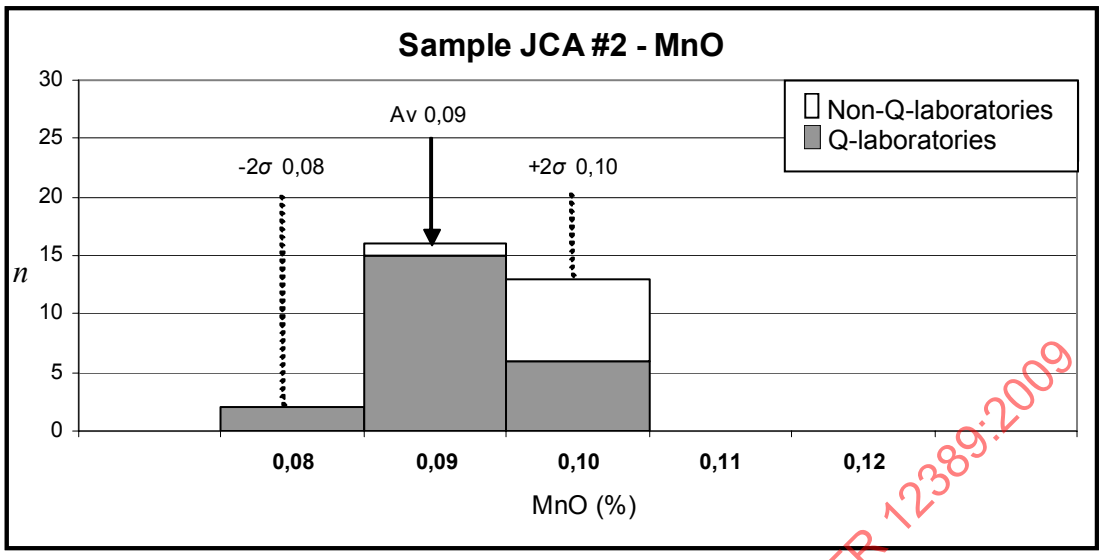

Figure 22 — The histogram of concentrations (Sample JCA #2, constituent MnO)

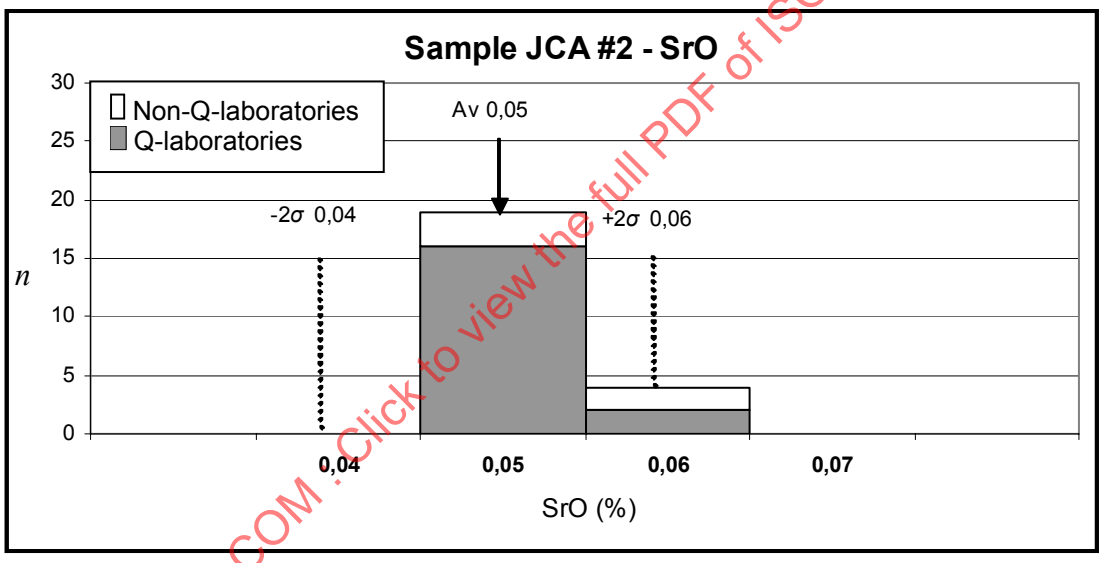

Figure 23 — The histogram of concentrations (Sample JCA #2, constituent SrO)

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6 Conclusions

6.1 Result of test sample analyses

The average and the standard deviations of reproducibility of test samples obtained from the inter-laboratory testing by x-ray fluorescence are shown in Table 12.

For comparison, standard deviations of reproducibility obtained from the wet-chemistry methods in Inter-laboratory testing, 2004-OC in accordance with JIS R 5202 are also shown in Table 12.

Inter-laboratory testing, 2004-OC was conducted by the Japan Cement Association and the standard deviation of the reproducibility was calculated using only data from the laboratories of cement companies in Japan. Most of these laboratories participated in the inter-laboratory testing by x-ray fluorescence.

NOTE An "Inter-laboratory testing, xxxx-OC" has been conducted by the Japan Cement Association since 1948. "xxxx" stands for the year in which the inter-laboratory testing was conducted.

Table 12 — Comparison between standard deviation obtained by Q-laboratories in this testing and that obtained from data of the wet method in Inter-laboratory testing, 2004-OC

Constituent	Sample	Q-laboratories this inter-laboratory testing		Inter-laboratory testing 2004-OC	
		Average %	Reproducibility S.D. %	Reproducibility S.D. %	Number of laboratories
SiO_2	Sample #1	20,96	0,078	0,129	22
	Sample #2	26,01	0,083		
Al_2O_3	Sample #1	5,10	0,021	0,068	22
	Sample #2	8,80	0,037		
Fe_2O_3	Sample #1	3,03	0,033	0,049	21
	Sample #2	1,62	0,025		
CaO	Sample #1	65,89	0,160	0,117	22
	Sample #2	66,09	0,192		
MgO	Sample #1	1,36	0,018	0,042	22
	Sample #2	3,32	0,024		
SO_3	Sample #1	2,21	0,037	0,039	27
	Sample #2	(2,86) [a]	(0,121) [a]		
Na_2O	Sample #1	0,21	0,019	0,014	23
	Sample #2	0,22	0,014		
K_2O	Sample #1	0,50	0,009	0,009	23
	Sample #2	0,42	0,008		
TiO_2	Sample #1	0,31	0,007	0,012	22
	Sample #2	0,39	0,010		
P_2O_5	Sample #1	0,24	0,008	0,027	22
	Sample #2	0,24	0,009		
MnO	Sample #1	0,10	0,005	0,007	22
	Sample #2	0,09	0,006		
SrO	Sample #1	0,05	0,005	-	-
	Sample #2	0,05	0,003		

[a] Concentrations in parentheses are the "outliers" by the Grubbs test.

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The standard deviations of Q-laboratories are almost equal to or less than those of Inter-laboratory testing, 2004-OC, except for CaO. Therefore, the accuracy of XRF analysis according to the XRF method is considered to be generally equal to that of wet analysis.

For CaO, a few laboratories obtained results that are not within the average ± 2 times the standard deviation, even if the results are not "outliers". Factors affecting the determination of CaO should be investigated further.

As reported in 5.3.2, there are some "outliers", even in the analyses by Q-laboratories. The principle of the method assumes that extreme results should not arise, provided that the calibration used for sample analysis satisfied the criteria for both repeatability limits and accuracy limits. The possibility of error factors in these analyses should be investigated.

6.2 Factors affecting conformity with the validation criteria

The XRF method requires that, when analysing a sample by XRF analysis, it is necessary to satisfy the criteria for repeatability limits and accuracy limits for certified reference materials. This validation is essential for accurate analysis.

Some laboratories were unable to satisfy the validation criteria in this inter-laboratory testing.

In these cases, it is recommended that those laboratories re-check general matters for chemical analysis, such as weighing of samples, performance of spectrometer and general facilities for accurate XRF analysis.

The following matters should also be checked:

a) bead preparation conditions, such as fusion temperature, flux-to-sample ratio;

b) repeatability of bead preparation;

c) procedure for preparing ignited sample;

d) agreed basis for calculating results, e.g., as-received or loss-free;

e) procedure for establishing calibration equations, such as range of contents or application of correction for inter-element effect;

f) measurement conditions for light elements, especially Na_2O, MgO and Al_2O_3.

6.3 Application to other international inter-laboratory testing

ISO 29581-2 is intended for use under everyday conditions in laboratories that operate to "good practice". Annex D sets out the results of some international round robins carried out by a large number of laboratories demonstrating the suitability of ISO 29581-2 as a means for comparing the everyday performance of laboratories.

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Annex A
(informative)

List of participating laboratories

A.1 Japanese laboratories:

Ube-Mitsubishi Cement Research Institute Corporation, Ube Center

Ube-Mitsubishi Cement Research Institute Corporation, Kurosaki Center

Ube-Mitsubishi Cement Research Institute Corporation, Saitama Center

Ube-Mitsubishi Cement Research Institute Corporation, Sendai Concrete Center

Nippon Steel Blast-Furnace Slag Cement Co., Ltd., Production and Technical Dept., Quality Assurance Group

Sumitomo Osaka Cement Co., Ltd., Production and Technical Dept., Analytical Center

Sumitomo Osaka Cement Co., Ltd., Cement/Concrete Research Laboratory, Cement Chemistry Research Group

Sumitomo Osaka Cement Co., Ltd., Cement/Concrete Research Laboratory, Cement Technology Group

Taiheiyo Cement Corporation, Ofunato Plant, Production Dept., Quality Assurance Section

Taiheiyo Cement Corporation, Kamiiso Plant, Production Dept., Quality Assurance Section

Taiheiyo Cement Corporation, Oita Plant(Saiki), Production Dept., Quality Assurance Section

Taiheiyo Consultant Co., Ltd., Quality Assurance Dept., Chemical Analysis Group

Chuken Consultant Co., Ltd., Kanto Branch, Technical Dev., Material Analytical Section

Tokuyama Corporation, Cement Manufacturing Dept., Technical Section 1

Tosoh Corporation, Cement and Energy Manufacturing Dept., Cement Testing Div.

Japan Cement Association, R & D Laboratory, Basic Research on Cement/Environment Group

A.2 ISO member laboratories – Outside Japan:

China, People's Republic of – China Building Materials Academy

Asia Cement Co., Ltd.

Hanil Cement Co., Ltd.

Hyundai Cement Co., Ltd., Danyang Plant

Hyundai Cement Co., Ltd., Yongwol Plant

Lafarge Halla Cement Corporation

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	Ssangyong Cement Industrial Co., Ltd.
	Yongwol Plant
	Sungshin Cement Co., Ltd.
Korea, Republic of –	Tong Yang Cement Corporation, Samchok Plant
Lithuania, Republic of –	Prustitas Co., Ltd.
Philippines, Republic of the –	Cement Manufactures Association/APO Cement Corporation
	Union Cement Corporation, Lugait Plant
Vietnam, Socialist Republic of –	Butson Cement Company
	National Cement Association

A.3 CEN member laboratories:

Italy –	Italcementi Group, Bergamo
France –	Three laboratories co-ordinated by ATILH, Paris
Germany –	VDZ, Research Institute of the Cement Industry, Duesseldorf
	HeidelbergCement Technology Center GmbH, Leimen
Belgium –	CRIC, Brussels
Turkey –	Turkish Cement Manufacturing Association, Ankara
United Kingdom –	Castle Cement Technical Centre, Clitheroe
Netherlands –	ENCI B.V. Vestiging, Maastricht
Spain –	IECA, Madrid
Czech Republic –	Research Institute for Binding Materials, Brno
Poland –	IMMB, Krakow

NOTE One CEN member laboratory was unable to meet the validation criteria and withdrew. It was subsequently discovered that the x-ray tube was unstable.

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Annex B
(informative)

Individual results of all laboratories

Tables B.1 to B.8 list the results of all laboratories participating in the test programme: Chemical analysis of cement by x-ray fluorescence.

Table B.1 — Individual results for all laboratories — JCA-CRM-1

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
1	20,982 20,943	20,962	5,287 5,259	5,273	2,619 2,610	2,614	65,197 65,377	65,287	2,117 2,111	2,114	2,083 2,073	2,078
2	20,902 20,936	20,919	5,257 5,253	5,255	2,647 2,648	2,648	65,370 65,298	65,334	2,134 2,129	2,132	2,005 2,037	2,021
3	20,933 20,945	20,939	5,244 5,249	5,246	2,691 2,674	2,682	65,115 65,097	65,106	2,143 2,131	2,137	2,089 2,061	2,075
4	21,006 20,926	20,966	5,260 5,250	5,255	2,670 2,683	2,676	65,188 65,208	65,198	2,135 2,137	2,136	2,074 2,083	2,078
5	20,914 20,946	20,930	5,254 5,266	5,260	2,668 2,671	2,670	65,243 65,247	65,245	2,136 2,133	2,134	2,085 2,094	2,090
6	20,945 20,921	20,933	5,250 5,223	5,236	2,608 2,606	2,607	65,126 65,209	65,168	2,142 2,103	2,122	2,061 2,073	2,067
7	20,996 21,010	21,003	5,273 5,260	5,266	2,663 2,660	2,662	65,177 65,223	65,200	2,163 2,168	2,166	2,063 2,057	2,060
8	20,945 20,905	20,925	5,272 5,266	5,269	2,681 2,677	2,679	65,103 64,988	65,046	2,161 2,151	2,156	2,054 2,062	2,058
9	20,912 20,901	20,906	5,266 5,249	5,258	2,613 2,609	2,611	65,227 65,186	65,206	2,117 2,120	2,118	2,076 2,070	2,073
10	20,942 20,970	20,956	5,219 5,254	5,236	2,631 2,628	2,630	65,331 65,319	65,325	2,122 2,105	2,114	2,067 2,084	2,076
11	21,027 21,015	21,021	5,280 5,274	5,277	2,677 2,669	2,673	65,241 65,180	65,210	2,127 2,113	2,120	2,083 2,065	2,074
12	20,978 20,989	20,984	5,264 5,270	5,267	2,690 2,664	2,677	65,310 65,237	65,274	2,147 2,142	2,144	2,098 2,074	2,086
13	21,042 21,024	21,033	5,265 5,269	5,267	2,680 2,677	2,678	64,999 64,957	64,978	2,137 2,146	2,142	2,056 2,051	2,054
14	21,040 20,960	21,000	5,296 5,267	5,282	2,716 2,722	2,719	65,341 65,453	65,397	2,154 2,148	2,151	2,067 2,068	2,068
15	21,086 20,996	21,041	5,260 5,252	5,256	2,674 2,670	2,672	65,192 65,230	65,211	2,109 2,106	2,108	1,976 1,991	1,984
16	21,004 21,011	21,008	5,297 5,286	5,292	2,671 2,667	2,669	65,190 65,176	65,183	2,153 2,156	2,154	2,088 2,096	2,092
101	21,050 21,055	21,052	5,288 5,293	5,290	2,623 2,616	2,620	65,348 65,342	65,345	2,122 2,110	2,116	1,962 2,092	2,027
102	21,162 21,179	21,170	5,183 5,241	5,212	2,638 2,634	2,636	66,288 66,283	66,286	2,127 2,134	2,130	2,429 2,430	2,430
103	20,990 20,970	20,980	5,200 5,220	5,210	2,640 2,650	2,645	65,280 65,220	65,250	2,230 2,060	2,145	2,070 2,090	2,080
104	20,989 20,973	20,981	5,248 5,228	5,238	2,631 2,622	2,626	65,060 64,867	64,964	2,093 2,104	2,098	2,051 2,096	2,074
105	20,978 21,062	21,020	5,242 5,294	5,268	2,642 2,634	2,638	65,396 65,372	65,384	2,176 2,175	2,176	2,064 2,058	2,061

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Table B.1 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
1	0,272 0,257	0,264	0,541 0,548	0,544	0,344 0,344	0,344	0,287 0,286	0,286	0,069 0,069	0,069	- -	-	99,835
2	0,264 0,263	0,264	0,533 0,536	0,534	0,356 0,349	0,352	0,283 0,284	0,284	0,066 0,065	0,066	0,044 0,044	0,44	99,853
3	0,266 0,253	0,260	0,570 0,568	0,569	0,355 0,358	0,356	0,279 0,278	0,278	0,063 0,063	0,063	- -	-	99,711
4	0,260 0,258	0,259	0,560 0,564	0,562	0,358 0,354	0,356	0,288 0,286	0,287	0,063 0,063	0,063	0,043 0,043	0,043	99,879
5	0,262 0,263	0,262	0,560 0,563	0,562	0,357 0,355	0,356	0,283 0,284	0,284	0,064 0,064	0,064	0,044 0,044	0,044	99,901
6	0,265 0,276	0,270	0,581 0,588	0,584	0,341 0,349	0,345	0,287 0,285	0,286	0,066 0,064	0,065	0,042 0,042	0,042	99,725
7	0,253 0,255	0,254	0,566 0,564	0,565	0,359 0,354	0,356	0,282 0,282	0,282	0,065 0,064	0,064	0,044 0,044	0,044	99,922
8	0,244 0,251	0,248	0,562 0,561	0,562	0,352 0,344	0,348	0,283 0,282	0,282	0,061 0,061	0,061	0,043 0,043	0,043	99,677
9	0,264 0,265	0,264	0,575 0,574	0,574	0,345 0,347	0,346	0,285 0,286	0,286	0,069 0,069	0,069	0,041 0,041	0,041	99,752
10	0,232 0,239	0,236	0,568 0,567	0,568	0,347 0,350	0,348	0,281 0,281	0,281	0,069 0,069	0,069	- -	-	99,839
11	0,242 0,254	0,248	0,523 0,533	0,528	0,353 0,357	0,355	0,283 0,288	0,286	0,063 0,062	0,062	- -	-	99,854
12	0,264 0,271	0,268	0,572 0,570	0,571	0,346 0,357	0,352	0,285 0,285	0,285	0,063 0,066	0,064	0,043 0,044	0,044	100,016
13	0,264 0,266	0,265	0,559 0,559	0,559	0,354 0,358	0,356	0,291 0,291	0,291	0,064 0,063	0,064	0,044 0,044	0,044	99,731
14	0,267 0,269	0,268	0,586 0,568	0,577	0,341 0,328	0,334	0,302 0,292	0,297	0,064 0,065	0,064	0,045 0,045	0,045	100,202
15	0,274 0,272	0,273	0,560 0,559	0,560	0,354 0,348	0,351	0,291 0,291	0,291	0,064 0,065	0,064	0,043 0,042	0,042	99,853
16	0,263 0,255	0,259	0,577 0,576	0,576	0,351 0,356	0,354	0,284 0,282	0,283	0,063 0,063	0,063	0,043 0,043	0,043	99,976
101	0,229 0,223	0,226	0,569 0,573	0,571	0,347 0,344	0,346	0,288 0,285	0,286	- -	-	- -	-	99,879
102	- -	-	0,596 0,596	0,596	- -	-	- -	-	- -	-	- -	-	100,460
103	0,270 0,280	0,275	0,580 0,570	0,575	- -	-	- -	-	- -	-	- -	-	99,160
104	0,239 0,249	0,244	0,556 0,572	0,564	0,343 0,341	0,342	0,276 0,272	0,274	0,066 0,065	0,066	0,042 0,042	0,042	99,517
105	0,248 0,251	0,250	0,599 0,598	0,598	0,352 0,341	0,346	0,291 0,294	0,292	0,067 0,067	0,067	0,042 0,041	0,042	100,142

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Table B.2 — Individual results for all laboratories — JCA-CRM-1

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
106	20,890 21,008	20,949	5,251 5,265	5,258	2,673 2,675	2,674	65,088 65,178	65,133	2,123 2,125	2,124	2,058 2,072	2,065
107	21,055 21,061	21,058	5,526 5,315	5,420	2,687 2,695	2,691	64,449 64,494	64,472	2,167 2,134	2,150	2,055 2,023	2,039
108	20,91 20,90	20,905	5,25 5,26	5,255	2,62 2,61	2,615	65,18 65,16	65,170	2,11 2,10	2,105	2,07 2,06	2,065
109	20,992 20,960	20,976	5,256 5,265	5,260	2,636 2,622	2,629	65,555 65,439	65,497	2,109 2,112	2,110	1,939 1,954	1,946
110	20,852 20,974	20,913	5,202 5,301	5,252	2,581 2,640	2,610	65,154 65,113	65,134	2,302 2,041	2,172	1,925 2,061	1,993
111	20,94 20,90	20,920	4,97 5,00	4,985	2,62 2,63	2,625	64,67 64,68	64,675	2,39 2,37	2,380	2,00 2,00	2,000
112	20,95 21,06	21,005	5,86 5,87	5,865	2,58 2,58	2,580	64,78 64,76	64,770	2,10 2,01	2,055	1,81 1,80	1,805
113	22,960 22,990	22,975	5,260 5,230	5,245	2,650 2,640	2,645	65,210 65,360	65,285	2,070 1,980	2,025	2,080 2,050	2,065
114	21,33 21,98	21,655	5,09 5,27	5,180	2,94 2,84	2,890	65,45 66,07	65,760	2,16 1,85	2,005	2,30 2,17	2,235
E1	20,965 20,853	20,909	5,282 5,237	5,260	2,669 2,656	2,663	65,141 65,180	65,161	2,138 2,145	2,142	2,212 2,187	2,200
E2	20,790 20,803	20,797	5,269 5,322	5,296	2,601 2,609	2,605	65,080 64,919	65,000	2,104 2,123	2,114	2,078 2,057	2,068
E3	20,780 20,800	20,790	5,140 5,190	5,165	2,730 2,690	2,710	65,180 65,150	65,165	2,270 2,270	2,270	1,950 2,010	1,980
E4	20,947 20,913	20,930	5,269 5,281	5,275	2,658 2,649	2,654	65,025 64,987	65,006	2,174 2,168	2,171	2,033 2,036	2,035
E5	21,020 21,000	21,010	5,250 5,240	5,245	2,640 2,660	2,650	65,350 65,300	65,325	2,130 2,140	2,135	2,070 2,060	2,065
E6	20,963 20,927	20,945	5,213 5,252	5,233	2,680 2,683	2,682	65,112 65,205	65,159	2,151 2,154	2,153	2,104 2,146	2,125
E7	20,892 21,100	20,996	5,244 5,295	5,270	2,689 2,686	2,688	65,444 65,240	65,342	2,177 2,126	2,152	2,062 2,068	2,065
E8	20,852 20,937	20,895	5,250 5,286	5,268	2,576 2,578	2,577	65,140 65,126	65,133	2,142 2,138	2,140	2,062 2,069	2,066
E9	21,010 20,972	20,991	5,203 5,242	5,223	2,681 2,673	2,677	65,293 65,124	65,209	2,134 2,162	2,148	- -	-
E10	21,080 21,090	21,085	5,150 5,160	5,155	2,600 2,620	2,610	65,110 65,160	65,135	2,100 2,120	2,110	2,010 2,010	2,010
E11	20,753 20,756	20,755	5,263 5,268	5,266	2,670 2,663	2,667	65,528 65,518	65,523	2,157 2,178	2,168	1,989 1,991	1,990
E12	21,000 20,760	20,880	5,293 5,279	5,286	2,678 2,656	2,667	65,260 64,560	64,910	2,150 2,151	2,151	2,060 2,043	2,052

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Table B.2 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
106	0,282 0,265	0,274	0,569 0,566	0,568	- -	-	- -	-	- -	-	- -	-	99,045
107	0,247 0,252	0,250	0,563 0,557	0,560	- -	-	0,273 0,284	0,278	0,061 0,060	0,060	- -	-	98,978
108	0,240 0,270	0,255	0,59 0,59	0,590	- -	-	0,28 0,28	0,280	- -	-	- -	-	99,240
109	- -	-	0,569 0,571	0,570	- -	-	- -	-	- -	-	- -	-	98,988
110	0,240 0,322	0,281	0,662 0,501	0,582	- -	-	0,276 0,298	0,287	- -	-	- -	-	99,224
111	0,280 0,290	0,285	0,57 0,57	0,570	0,38 0,39	0,385	0,35 0,36	0,355	0,07 0,07	0,070	0,04 0,04	0,040	99,290
112	0,190 0,190	0,190	0,58 0,58	0,580	- -	-	- -	-	- -	-	- -	-	98,850
113	0,260 0,250	0,255	0,580 0,580	0,580	0,350 0,350	0,350	0,280 0,290	0,285	0,070 0,070	0,070	0,040 0,040	0,040	101,820
114	0,710 0,710	0,710	0,54 0,54	0,540	- -	-	- -	-	- -	-	- -	-	100,975
E1	0,253 0,262	0,258	0,565 0,563	0,564	0,351 0,357	0,354	0,292 0,292	0,292	0,062 0,062	0,062	0,044 0,044	0,044	99,906
E2	0,274 0,272	0,273	0,572 0,567	0,570	0,340 0,342	0,341	0,287 0,284	0,286	0,068 0,068	0,068	- -	-	99,415
E3	0,260 0,260	0,260	0,550 0,550	0,550	0,350 0,350	0,350	0,300 0,290	0,295	0,070 0,070	0,070	0,050 0,050	0,050	99,655
E4	0,267 0,272	0,270	0,572 0,574	0,573	0,344 0,344	0,344	0,291 0,288	0,290	0,067 0,066	0,067	0,041 0,041	0,041	99,654
E5	0,270 0,260	0,265	0,570 0,580	0,575	0,340 0,350	0,345	0,260 0,290	0,275	0,070 0,070	0,070	0,040 0,040	0,040	100,000
E6	0,124 0,128	0,126	0,564 0,560	0,562	0,348 0,347	0,348	0,313 0,303	0,308	0,059 0,060	0,060	- -	-	99,698
E7	0,270 0,278	0,274	0,561 0,567	0,564	0,361 0,351	0,356	0,281 0,286	0,284	0,063 0,062	0,063	0,054 0,053	0,054	100,105
E8	0,273 0,262	0,268	0,571 0,572	0,572	0,341 0,345	0,343	0,284 0,284	0,284	0,067 0,068	0,068	0,042 0,042	0,042	99,654
E9	0,251 0,260	0,256	0,562 0,563	0,563	0,332 0,343	0,338	- -	-	- -	-	- -	-	97,403
E10	0,290 0,290	0,290	0,570 0,570	0,570	- -	-	- -	-	- -	-	- -	-	98,965
E11	0,249 0,249	0,249	0,571 0,569	0,570	0,340 0,341	0,341	0,280 0,280	0,280	0,064 0,063	0,064	0,050 0,050	0,050	99,920
E12	0,263 0,243	0,253	0,572 0,561	0,566	0,355 0,348	0,352	0,288 0,281	0,285	0,071 0,070	0,071	- -	-	99,471

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Table B.3 — Individual results for all laboratories — JCA-CRM-2

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
1	25,651 25,652	25,652	8,943 8,944	8,944	2,137 2,140	2,138	56,491 56,489	56,490	3,055 3,053	3,054	2,588 2,557	2,558
2	25,573 25,522	25,548	8,915 8,925	8,920	2,087 2,088	2,088	56,482 56,462	56,472	3,022 3,040	3,031	2,553 2,586	2,570
3	25,535 25,503	25,519	8,942 8,932	8,937	2,107 2,094	2,100	56,538 56,525	56,532	2,977 2,968	2,972	- -	-
4	25,633 25,641	25,637	8,924 8,940	8,932	2,104 2,098	2,101	56,406 56,359	56,382	3,035 3,045	3,040	2,569 2,590	2,580
5	25,595 25,633	25,614	8,921 8,940	8,930	2,083 2,082	2,082	56,424 56,436	56,430	3,026 3,035	3,030	2,579 2,559	2,569
6	25,651 25,538	25,594	9,027 8,983	9,005	2,152 2,134	2,143	56,617 56,512	56,564	3,073 3,048	3,060	2,544 2,510	2,527
7	25,695 25,686	25,690	8,948 8,951	8,950	2,071 2,072	2,072	56,462 56,500	56,481	3,097 3,071	3,084	2,602 2,603	2,602
8	25,568 25,563	25,566	8,909 8,893	8,901	2,040 2,043	2,042	56,167 56,127	56,147	3,059 3,056	3,058	2,367 2,366	2,366
9	25,537 25,631	25,584	8,923 8,939	8,931	2,138 2,139	2,138	56,444 56,545	56,494	3,049 3,061	3,055	- -	-
10	25,568 25,603	25,586	8,947 8,856	8,902	2,137 2,122	2,130	56,437 56,620	56,528	2,956 2,996	2,976	- -	-
11	25,697 25,677	25,687	8,892 8,862	8,877	2,084 2,082	2,083	56,457 56,357	56,407	3,044 2,997	3,020	2,603 2,608	2,606
12	25,678 25,672	25,675	8,965 8,913	8,939	2,070 2,090	2,080	56,440 56,418	56,429	3,039 3,043	3,041	- -	-
13	25,736 25,726	25,731	8,929 8,921	8,925	2,083 2,086	2,084	56,271 56,213	56,242	3,053 3,061	3,057	2,632 2,624	2,628
14	25,787 25,716	25,752	8,965 8,913	8,939	2,125 2,119	2,122	56,428 56,584	56,506	3,062 3,053	3,058	2,641 2,635	2,638
15	25,610 25,558	25,584	8,968 8,962	8,965	2,087 2,092	2,090	56,327 56,306	56,316	3,019 3,018	3,018	2,605 2,596	2,600
16	25,712 25,658	25,685	8,937 8,983	8,960	2,072 2,078	2,075	56,533 56,514	56,524	3,046 3,035	3,040	2,604 2,577	2,590
101	25,631 25,636	25,634	8,942 8,903	8,922	2,124 2,113	2,118	56,586 56,526	56,556	3,013 3,026	3,020	- -	-
102	25,589 25,611	25,600	8,918 8,929	8,924	2,131 2,127	2,129	56,644 56,673	56,658	3,077 3,069	3,073	1,320 1,323	1,322
103	25,650 25,670	25,660	8,930 9,000	8,965	2,150 2,160	2,155	56,530 56,530	56,530	3,060 2,920	2,990	2,540 2,570	2,555
104	25,550 25,516	25,533	8,885 8,846	8,866	2,123 2,113	2,118	56,411 56,223	56,317	3,033 3,024	3,028	1,880 1,900	1,890
105	25,653 25,610	25,632	8,911 8,930	8,920	2,125 2,123	2,124	56,557 56,547	56,552	3,073 3,093	3,083	2,568 2,571	2,570

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Table B.3 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
1	0,273 0,268	0,270	0,309 0,315	0,312	0,505 0,504	0,504	0,074 0,074	0,074	0,162 0,162	0,162	- -	-	100,158
2	0,242 0,245	0,244	0,322 0,320	0,321	0,500 0,514	0,507	0,073 0,075	0,074	0,155 0,155	0,155	0,069 0,068	0,68	99,998
3	0,223 0,214	0,218	0,323 0,324	0,324	0,506 0,506	0,506	0,075 0,075	0,075	0,160 0,159	0,160	- -	-	97,343
4	0,240 0,237	0,238	0,320 0,317	0,318	0,506 0,506	0,506	0,076 0,075	0,076	0,151 0,154	0,152	0,065 0,065	0,065	100,027
5	0,241 0,240	0,240	0,320 0,321	0,320	0,504 0,502	0,503	0,075 0,075	0,075	0,155 0,155	0,155	0,069 0,069	0,069	100,017
6	0,246 0,234	0,240	0,320 0,313	0,316	0,515 0,508	0,512	0,074 0,072	0,073	0,155 0,155	0,155	0,066 0,066	0,066	100,255
7	0,231 0,231	0,231	0,322 0,323	0,322	0,502 0,504	0,503	0,074 0,073	0,074	0,154 0,154	0,154	0,068 0,068	0,068	100,231
8	0,234 0,234	0,234	0,323 0,323	0,323	0,496 0,503	0,500	0,075 0,076	0,076	0,157 0,158	0,158	0,063 0,063	0,063	99,434
9	0,252 0,254	0,253	0,310 0,312	0,311	0,504 0,500	0,502	0,074 0,074	0,074	0,163 0,162	0,162	0,065 0,065	0,065	97,569
10	0,232 0,221	0,226	0,324 0,326	0,325	0,508 0,505	0,506	0,074 0,074	0,074	0,158 0,159	0,158	- -	-	97,411
11	0,257 0,241	0,249	0,319 0,319	0,319	0,512 0,498	0,505	0,076 0,077	0,076	0,156 0,155	0,156	- -	-	99,985
12	0,236 0,250	0,243	0,330 0,326	0,328	0,504 0,510	0,507	0,076 0,076	0,076	0,157 0,156	0,156	0,063 0,064	0,064	97,538
13	0,241 0,243	0,242	0,320 0,320	0,320	0,503 0,508	0,506	0,080 0,080	0,080	0,152 0,152	0,152	0,069 0,069	0,069	100,036
14	0,252 0,244	0,248	0,329 0,325	0,327	0,492 0,478	0,485	0,094 0,079	0,086	0,153 0,156	0,154	0,070 0,071	0,070	100,385
15	0,253 0,247	0,250	0,320 0,322	0,321	0,521 0,518	0,520	0,081 0,082	0,082	0,155 0,155	0,155	0,065 0,065	0,065	99,966
16	0,244 0,239	0,242	0,310 0,312	0,311	0,504 0,501	0,502	0,075 0,075	0,075	0,155 0,154	0,154	0,066 0,067	0,066	100,224
101	0,217 0,200	0,208	0,323 0,324	0,324	0,506 0,505	0,506	0,074 0,074	0,074	- -	-	- -	-	97,362
102	- -	-	0,309 0,310	0,310	- -	-	- -	-	- -	-	- -	-	98,016
103	0,240 0,250	0,245	0,310 0,310	0,310	- -	-	- -	-	- -	-	- -	-	99,410
104	0,266 0,259	0,262	0,331 0,314	0,322	0,502 0,499	0,500	0,067 0,066	0,066	0,158 0,155	0,156	0,066 0,066	0,066	99,124
105	0,226 0,230	0,228	0,345 0,341	0,343	0,518 0,508	0,513	0,086 0,081	0,084	0,164 0,158	0,161	0,065 0,065	0,065	100,275

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Table B.4 — Individual results for all laboratories — JCA-CRM-2

Laboratory ref.	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
106	25,547 25,490	25,518	8,962 8,917	8,940	2,047 2,090	2,068	56,510 56,617	56,564	3,009 3,024	3,016	- -	-
107	25,510 25,631	25,570	8,944 8,930	8,937	2,033 2,081	2,057	56,456 56,468	56,462	2,885 2,870	2,878	- -	-
108	25,870 25,620	25,745	8,980 8,920	8,950	2,130 2,130	2,130	56,770 56,520	56,645	3,050 3,030	3,040	2,570 2,540	2,555
109	25,752 25,698	25,725	8,996 8,973	8,984	2,154 2,141	2,148	56,542 56,485	56,514	3,076 3,074	3,075	2,454 2,430	2,442
110	25,663 25,641	25,652	8,940 8,821	8,880	2,082 2,064	2,073	56,482 56,620	56,551	3,054 3,091	3,072	1,910 1,932	1,921
111	25,280 25,340	25,310	8,650 8,600	8,625	2,110 2,100	2,105	56,340 56,270	56,305	3,230 3,260	3,245	1,260 1,280	1,270
112	- -	-	- -	-	- -	-	- -	-	- -	-	- -	-
113	25,690 25,740	25,715	8,960 8,910	8,935	2,140 2,120	2,130	56,790 56,840	56,815	3,110 3,140	3,125	- -	-
114	25,460 25,310	25,385	6,540 6,600	6,570	2,570 2,460	2,515	56,570 56,740	56,655	2,160 3,140	2,650	2,310 2,160	2,235
E1	25,667 25,635	25,651	8,914 8,919	8,917	2,082 2,079	2,081	56,470 56,398	56,434	3,030 3,040	3,035	2,385 2,384	2,385
E2	25,515 25,595	25,555	8,915 8,921	8,918	2,119 2,111	2,115	56,440 56,593	56,517	3,032 3,035	3,034	2,592 2,569	2,581
E3	25,330 25,370	25,350	8,700 8,730	8,715	2,130 2,100	2,115	56,330 56,370	56,350	3,210 3,200	3,205	2,540 2,500	2,520
E4	25,538 25,636	25,587	8,861 8,872	8,867	2,121 2,125	2,123	56,073 56,121	56,097	2,984 2,997	2,991	- -	-
E5	25,630 25,670	25,650	8,940 8,920	8,930	2,100 2,100	2,100	56,210 56,250	56,230	3,030 3,030	3,030	- -	-
E6	25,532 25,598	25,565	8,870 8,879	8,875	2,094 2,096	2,095	56,360 56,480	56,420	3,043 3,061	3,052	- -	-
E7	25,769 25,808	25,789	8,930 8,881	8,906	2,094 2,095	2,095	56,416 56,491	56,454	3,059 3,025	3,042	2,698 2,716	2,618
E8	25,612 25,606	25,609	8,928 8,938	8,933	2,154 2,141	2,148	56,391 56,404	56,398	3,042 3,043	3,043	2,160 2,160	2,707
E9	25,724 25,639	25,682	8,919 8,869	8,894	2,091 2,116	2,104	56,434 56,643	56,539	3,041 3,075	3,058	- -	-
E10	25,470 25,530	25,500	8,620 8,650	8,635	2,120 2,130	2,125	57,080 57,110	57,095	2,970 2,960	2,965	2,619 2,617	2,160
E11	25,519 25,472	25,496	8,989 8,996	8,993	2,137 2,137	2,137	57,040 57,038	57,039	3,125 3,089	3,107	2,503 2,504	2,504
E12	25,620 25,680	25,650	9,002 8,919	8,961	2,091 2,083	2,087	56,250 56,660	56,455	3,077 3,099	3,088	- -	-

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Table B.4 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
106	0,253 0,251	0,252	0,328 0,329	0,328	- -	-	- -	-	- -	-	- -	-	96,686
107	0,252 0,261	0,256	0,332 0,337	0,334	- -	-	0,075 0,073	0,074	0,159 0,162	0,160	- -	-	96,728
108	0,270 0,200	0,235	0,320 0,320	0,320	- -	-	0,080 0,070	0,075	- -	-	- -	-	99,695
109	- -	-	0,307 0,317	0,312	- -	-	- -	-	- -	-	- -	-	99,200
110	0,220 0,241	0,230	0,331 0,312	0,322	- -	-	0,086 0,078	0,082	- -	-	- -	-	98,783
111	0,270 0,270	0,270	0,330 0,330	0,330	0,530 0,520	0,525	0,010 0,030	0,020	0,160 0,160	0,160	0,060 0,060	0,060	98,225
112	- -	-	- -	-	- -	-	- -	-	- -	-	- -	-	-
113	0,240 0,230	0,235	0,330 0,320	0,325	0,510 0,500	0,505	0,080 0,080	0,080	0,160 0,160	0,160	0,060 0,060	0,060	98,085
114	0,660 0,660	0,660	0,280 0,270	0,275	- -	-	- -	-	- -	-	- -	-	96,945
E1	0,249 0,248	0,249	0,323 0,326	0,325	0,498 0,504	0,501	0,075 0,080	0,078	0,156 0,156	0,156	0,068 0,068	0,068	99,877
E2	0,303 0,265	0,284	0,320 0,317	0,319	0,503 0,503	0,503	0,076 0,077	0,077	0,160 0,161	0,161	- -	-	100,061
E3	0,240 0,240	0,240	0,310 0,310	0,310	0,490 0,490	0,490	0,080 0,080	0,080	0,180 0,180	0,180	0,080 0,080	0,080	99,635
E4	0,236 0,239	0,238	0,316 0,319	0,318	0,506 0,503	0,505	0,070 0,069	0,070	0,164 0,160	0,162	0,058 0,059	0,059	97,014
E5	0,220 0,240	0,230	0,310 0,320	0,315	0,500 0,510	0,505	0,070 0,070	0,070	0,160 0,160	0,160	0,070 0,080	0,075	97,295
E6	0,118 0,114	0,116	0,319 0,322	0,321	0,495 0,495	0,495	0,108 0,116	0,112	0,145 0,145	0,145	- -	-	97,195
E7	0,256 0,240	0,248	0,317 0,318	0,318	0,497 0,502	0,500	0,075 0,077	0,076	0,156 0,156	0,156	0,082 0,080	0,081	100,280
E8	0,271 0,257	0,264	0,315 0,316	0,316	0,509 0,505	0,507	0,075 0,074	0,075	0,160 0,160	0,160	0,066 0,060	0,063	100,221
E9	- -	-	0,328 0,327	0,328	0,532 0,521	0,527	- -	-	- -	-	- -	-	97,130
E10	0,260 0,260	0,260	0,340 0,340	0,340	- -	-	- -	-	- -	-	- -	-	99,080
E11	0,238 0,228	0,233	0,329 0,329	0,329	0,503 0,505	0,504	0,079 0,080	0,080	0,157 0,157	0,157	0,075 0,075	0,075	100,652
E12	0,262 0,240	0,251	0,324 0,324	0,324	0,502 0,505	0,504	0,071 0,085	0,078	0,175 0,175	0,175	- -	-	97,572

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Table B.5 — Individual results for all laboratories — Sample JCA #1

Laboratory ref.	Loss on ignition	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
		Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
1	1,97	20,896 20,962	20,929	5,103 5,124	5,114	2,984 2,992	2,988	65,905 66,033	65,969	1,360 1,362	1,361	2,233 2,231	2,232
2	1,98	20,845 20,860	20,852	5,100 5,108	5,104	3,022 3,019	3,020	65,920 65,986	65,935	1,355 1,360	1,358	2,203 2,173	2,188
3	2,02	20,916 20,998	20,957	5,102 5,123	5,112	3,020 3,043	3,032	65,792 65,931	65,862	1,328 1,355	1,342	2,179 2,199	2,189
4	1,98	20,826 20,924	20,875	5,091 5,099	5,095	3,047 3,040	3,044	65,875 65,860	65,868	1,368 1,352	1,360	2,247 2,246	2,246
5	1,96	20,898 20,906	20,902	5,125 5,119	5,122	3,040 3,038	3,039	65,931 65,958	65,944	1,362 1,368	1,365	2,223 2,209	2,216
6	2,04	21,086 21,001	21,044	5,136 5,106	5,121	2,994 2,989	2,992	65,993 65,818	65,906	1,342 1,348	1,345	2,236 2,217	2,226
7	1,98	20,930 20,914	20,922	5,101 5,105	5,103	3,032 3,035	3,034	65,725 65,787	65,756	1,368 1,355	1,362	2,215 2,214	2,214
8	1,98	20,989 20,925	20,957	5,132 5,131	5,132	3,032 3,030	3,031	65,802 65,795	65,798	1,378 1,373	1,376	2,206 2,213	2,210
9	2,01	20,829 20,839	20,834	5,097 5,096	5,096	2,981 2,978	2,980	65,863 65,925	65,894	1,357 1,357	1,357	2,228 2,235	2,232
10	2,05	20,956 21,006	20,981	5,064 5,088	5,076	3,011 3,010	3,010	66,160 66,040	66,100	1,380 1,349	1,364	2,211 2,221	2,216
11	1,95	21,008 21,003	21,006	5,142 5,096	5,119	3,056 3,039	3,048	66,004 65,780	65,892	1,364 1,358	1,361	1,971 2,011	1,991
12	1,96	20,946 20,968	20,957	5,111 5,105	5,108	3,046 3,050	3,048	65,803 65,882	65,842	1,339 1,349	1,344	2,176 2,156	2,166
13	1,97	21,092 21,064	21,078	5,115 5,125	5,120	3,064 3,063	3,064	65,726 65,643	65,684	1,374 1,368	1,371	2,202 2,209	2,206
14	2,01	20,930 20,988	20,959	5,128 5,131	5,130	3,097 3,094	3,096	66,161 66,135	66,148	1,372 1,376	1,374	2,207 2,193	2,200
15	2,00	21,038 21,094	21,066	5,102 5,137	5,120	3,037 3,055	3,046	65,763 65,962	65,862	1,323 1,345	1,334	2,147 2,177	2,162
16	1,96	20,973 20,951	20,962	5,130 5,116	5,123	3,039 3,037	3,038	65,826 65,718	65,772	1,382 1,374	1,378	2,215 2,212	2,214
101	-	21,009 20,924	20,966	5,104 5,095	5,100	2,989 2,976	2,982	66,017 65,836	65,926	1,355 1,338	1,346	2,458 2,476	2,467
102	2,00	21,306 21,338	21,322	5,147 5,145	5,146	3,052 3,053	3,052	67,380 67,387	67,384	1,382 1,386	1,384	2,514 2,520	2,517
103	2,04	20,990 21,000	20,995	5,050 5,050	5,050	3,000 3,020	3,010	66,020 65,990	66,005	1,440 1,500	1,470	2,240 2,240	2,240
104	1,74	21,001 20,899	20,950	5,107 5,072	5,090	3,017 2,977	2,997	65,574 65,439	65,506	1,371 1,361	1,366	2,169 2,140	2,154
105	2,00	20,777 20,694	20,736	5,107 5,062	5,084	2,967 2,965	2,966	65,120 65,061	65,090	1,389 1,389	1,389	2,200 2,185	2,192

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Table B.5 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
1	0,214 0,202	0,208	0,463 0,444	0,454	0,308 0,310	0,309	0,241 0,242	0,242	0,097 0,097	0,097	- -	-	99,903
2	0,211 0,208	0,210	0,500 0,499	0,500	0,314 0,316	0,315	0,241 0,237	0,239	0,096 0,094	0,095	0,048 0,048	0,048	99,882
3	0,191 0,188	0,190	0,499 0,500	0,500	0,320 0,315	0,318	0,232 0,233	0,232	0,094 0,094	0,094	- -	-	99,828
4	0,213 0,205	0,209	0,496 0,504	0,500	0,320 0,326	0,323	0,239 0,242	0,240	0,095 0,096	0,096	0,048 0,047	0,048	99,904
5	0,210 0,211	0,210	0,491 0,490	0,490	0,316 0,316	0,316	0,238 0,239	0,238	0,096 0,096	0,096	0,049 0,049	0,049	99,987
6	0,236 0,218	0,227	0,521 0,510	0,516	0,317 0,311	0,314	0,244 0,243	0,244	0,092 0,092	0,092	0,045 0,045	0,045	100,072
7	0,205 0,200	0,202	0,494 0,494	0,494	0,319 0,319	0,319	0,238 0,237	0,238	0,096 0,097	0,096	0,048 0,048	0,048	99,788
8	0,193 0,193	0,193	0,495 0,493	0,494	0,318 0,315	0,316	0,237 0,238	0,238	0,092 0,095	0,094	0,046 0,046	0,046	99,885
9	0,209 0,209	0,209	0,504 0,505	0,504	0,307 0,310	0,308	0,238 0,241	0,240	0,097 0,097	0,097	0,044 0,044	0,044	99,795
10	0,170 0,170	0,170	0,499 0,499	0,500	0,317 0,317	0,316	0,247 0,237	0,242	0,097 0,097	0,097	- -	-	100,079
11	0,194 0,193	0,194	0,522 0,515	0,518	0,320 0,315	0,318	0,236 0,239	0,238	0,097 0,095	0,096	- -	-	99,781
12	0,205 0,208	0,206	0,484 0,486	0,485	0,317 0,323	0,320	0,235 0,235	0,235	0,091 0,091	0,091	0,053 0,054	0,054	99,856
13	0,212 0,214	0,213	0,493 0,494	0,494	0,313 0,312	0,312	0,246 0,245	0,246	0,094 0,095	0,094	0,049 0,049	0,049	99,931
14	0,213 0,213	0,213	0,493 0,492	0,492	0,300 0,309	0,304	0,246 0,247	0,246	0,095 0,095	0,095	0,050 0,050	0,050	100,307
15	0,187 0,176	0,182	0,490 0,493	0,492	0,311 0,314	0,312	0,244 0,246	0,245	0,097 0,095	0,096	0,047 0,047	0,047	99,964
16	0,205 0,211	0,208	0,503 0,506	0,504	0,319 0,318	0,318	0,240 0,236	0,238	0,093 0,094	0,094	0,048 0,048	0,048	99,897
101	0,162 0,142	0,152	0,500 0,499	0,500	0,311 0,308	0,310	0,239 0,235	0,237	- -	-	- -	-	99,986
102	- -	-	0,517 0,515	0,516	- -	-	- -	-	- -	-	- -	-	101,321
103	0,260 0,260	0,260	0,510 0,510	0,510	- -	-	- -	-	- -	-	- -	-	99,540
104	0,135 0,130	0,132	0,503 0,484	0,494	0,313 0,306	0,310	0,234 0,230	0,232	0,092 0,093	0,092	0,044 0,044	0,044	99,367
105	0,215 0,215	0,218	0,508 0,508	0,510	0,309 0,309	0,310	0,240 0,233	0,237	0,096 0,094	0,095	0,043 0,043	0,043	99,870

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Table B.6 — Individual results for all laboratories — Sample JCA #1

Laboratory ref.	Loss on ignition	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
		Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
106	2,06	20,512 20,429	20,470	5,058 5,051	5,054	2,979 2,968	2,974	64,670 64,542	64,606	1,334 1,335	1,334	2,180 2,155	2,168
107	2,02	21,354 21,305	21,330	5,367 5,356	5,362	3,173 3,142	3,158	66,389 66,335	66,362	1,438 1,459	1,448	2,255 2,224	2,240
108	1,95	21,000 21,020	21,010	5,120 5,120	5,120	3,000 3,000	3,000	65,910 65,850	65,880	1,360 1,370	1,365	2,220 2,230	2,225
109	2,00	20,783 20,817	20,800	5,065 5,065	5,065	2,998 2,995	2,996	65,852 65,905	65,878	1,346 1,354	1,350	2,113 2,101	2,107
110	-	20,590 20,631	20,610	4,781 4,941	4,861	2,930 2,973	2,952	65,161 65,083	65,122	1,292 1,351	1,322	2,211 2,170	2,190
111	-	20,730 20,710	20,720	4,770 4,760	4,765	2,980 2,970	2,975	64,690 64,700	64,695	1,680 1,740	1,710	2,060 2,040	2,050
112	-	20,860 20,900	20,880	5,650 5,710	5,680	2,920 2,920	2,920	64,820 65,000	64,910	1,300 1,280	1,290	1,990 1,850	1,920
113	2,03	20,780 20,850	20,815	5,160 5,110	5,135	3,010 3,030	3,020	65,400 65,540	65,470	1,440 1,420	1,430	2,230 2,160	2,195
114	-	20,690 21,340	21,015	5,020 5,210	5,115	3,220 3,450	3,335	66,110 66,580	66,345	1,080 1,010	1,045	2,380 2,310	2,345
E1	-	20,882 20,905	20,894	5,147 5,087	5,117	3,029 3,050	3,040	65,821 65,898	65,860	1,353 1,368	1,361	2,308 2,295	2,302
E2	2,00	20,814 20,886	20,850	5,111 5,103	5,107	2,995 3,013	3,004	65,725 65,924	65,825	1,361 1,361	1,361	2,230 2,214	2,222
E3	-	20,950 21,020	20,985	5,040 5,030	5,035	3,120 3,130	3,125	66,280 66,180	66,230	1,450 1,460	1,455	2,110 2,110	2,110
E4	-	20,903 20,982	20,943	5,085 5,105	5,095	2,992 2,995	2,994	65,675 65,741	65,708	1,303 1,296	1,300	2,193 2,219	2,206
E5	1,96	21,070 21,050	21,060	5,100 5,040	5,070	3,010 3,020	3,015	65,940 66,060	66,000	1,410 1,360	1,385	2,220 2,220	2,220
E6	2,12	21,087 21,111	21,099	5,105 5,088	5,097	3,076 3,087	3,082	66,274 66,463	66,369	1,376 1,372	1,374	2,235 2,250	2,243
E7	2,02	20,918 20,798	20,858	5,076 5,108	5,092	3,088 3,069	3,079	65,865 65,793	65,828	1,385 1,351	1,368	2,228 2,208	2,218
E8	2,07	20,929 20,910	20,920	5,113 5,092	5,103	2,992 2,975	2,984	65,865 65,956	65,911	1,380 1,359	1,370	2,217 2,215	2,216
E9	1,94	21,054 20,966	21,010	5,152 5,102	5,127	3,031 3,040	3,036	65,981 65,843	65,912	1,344 1,350	1,347	- -	-
E10	-	20,600 20,630	20,615	5,000 5,000	5,000	2,960 2,970	2,965	65,680 65,720	65,700	1,370 1,370	1,370	2,220 2,220	2,220
E11	2,04	20,737 20,831	20,784	5,094 5,118	5,106	3,019 3,019	3,019	66,257 66,333	66,295	1,434 1,434	1,434	2,160 2,158	2,159
E12	1,99	21,050 20,860	20,955	5,118 5,089	5,104	3,037 3,029	3,033	65,980 65,460	65,720	1,357 1,354	1,356	2,221 2,191	2,206

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Table B.6 (*continued*)

Laboratory ref.	Na_2O %		K_2O %		TiO_2 %		P_2O_5 %		MnO %		SrO %		Total
	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	
106	0,201 0,205	0,203	0,491 0,489	0,490	- -	-	- -	-	- -	-	- -	-	97,299
107	0,265 0,275	0,270	0,510 0,510	0,510	- -	-	0,237 0,237	0,237	0,081 0,091	0,086	- -	-	101,003
108	0,130 0,130	0,130	0,490 0,490	0,490	- -	-	0,240 0,240	0,240	- -	-	- -	-	99,460
109	- -	-	0,504 0,497	0,500	- -	-	- -	-	- -	-	- -	-	98,696
110	0,243 0,220	0,232	0,490 0,511	0,500	- -	-	0,230 0,238	0,234	- -	-	- -	-	98,023
111	0,250 0,260	0,255	0,500 0,500	0,500	0,350 0,340	0,345	0,320 0,300	0,310	0,100 0,100	0,100	0,040 0,040	0,040	98,465
112	0,230 0,230	0,230	0,510 0,510	0,510	- -	-	- -	-	- -	-	- -	-	98,340
113	0,210 0,200	0,205	0,510 0,510	0,510	0,340 0,340	0,340	0,240 0,240	0,240	0,100 0,100	0,100	0,040 0,040	0,040	99,500
114	0,640 0,650	0,645	0,440 0,420	0,430	- -	-	- -	-	- -	-	- -	-	100,275
E1	0,195 0,206	0,201	0,493 0,496	0,495	0,326 0,313	0,320	0,241 0,248	0,245	0,095 0,096	0,096	0,048 0,049	0,049	99,975
E2	0,208 0,207	0,208	0,498 0,499	0,499	0,312 0,311	0,312	0,240 0,236	0,238	0,097 0,097	0,097	- -	-	99,721
E3	0,220 0,220	0,220	0,480 0,470	0,475	0,310 0,310	0,310	0,250 0,250	0,250	0,110 0,110	0,110	0,060 0,060	0,060	100,365
E4	0,234 0,228	0,231	0,490 0,494	0,492	0,305 0,307	0,306	0,238 0,238	0,238	0,092 0,092	0,092	0,052 0,056	0,054	99,658
E5	0,234 0,234	0,234	0,510 0,500	0,505	0,315 0,303	0,309	0,234 0,222	0,228	0,102 0,098	0,100	0,046 0,047	0,047	100,173
E6	0,105 0,101	0,103	0,496 0,497	0,496	0,313 0,315	0,314	0,273 0,269	0,271	0,090 0,091	0,090	- -	-	100,538
E7	0,214 0,211	0,213	0,495 0,499	0,497	0,318 0,319	0,319	0,244 0,233	0,239	0,099 0,100	0,100	0,061 0,063	0,062	99,871
E8	0,204 0,215	0,210	0,501 0,502	0,502	0,309 0,305	0,307	0,240 0,238	0,239	0,096 0,096	0,096	0,045 0,045	0,045	99,900
E9	0,258 0,271	0,265	0,501 0,511	0,506	0,308 0,296	0,302	- -	-	- -	-	- -	-	97,504
E10	0,220 0,210	0,215	0,510 0,510	0,510	- -	-	- -	-	- -	-	- -	-	98,595
E11	0,202 0,201	0,202	0,496 0,497	0,497	0,302 0,303	0,303	0,237 0,235	0,236	0,093 0,093	0,093	0,054 0,054	0,054	100,181
E12	0,227 0,186	0,207	0,496 0,490	0,493	0,317 0,315	0,316	0,234 0,243	0,239	0,107 0,104	0,106	- -	-	99,732

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Table B.7 — Individual results for all laboratories — Sample JCA #2

Laboratory ref.	Loss on ignition	SiO_2 %		Al_2O_3 %		Fe_2O_3 %		CaO %		MgO %		SO_3 %	
		Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean	Results	Mean
1	0,09	26,015 26,029	26,022	8,827 8,838	8,832	1,623 1,625	1,624	56,064 55,987	56,026	3,340 3,338	3,339	2,901 2,895	2,898
2	0,21	25,839 25,812	25,826	8,768 8,762	8,765	1,629 1,623	1,626	56,130 56,152	56,141	3,318 3,300	3,309	2,903 2,907	2,905
3	0,63	25,987 26,002	25,994	8,816 8,816	8,816	1,556 1,561	1,558	56,244 56,257	56,250	3,288 3,294	3,291	- -	-
4	0,18	25,990 26,005	25,998	8,781 8,786	8,784	1,643 1,648	1,646	55,904 55,887	55,896	3,325 3,320	3,322	2,920 2,912	2,916
5	0,19	25,955 25,936	25,946	8,805 8,797	8,801	1,624 1,621	1,622	56,205 56,122	56,164	3,310 3,314	3,312	2,879 2,897	2,888
6	0,12	26,196 26,130	26,163	8,986 8,983	8,984	1,638 1,616	1,627	56,229 56,179	56,204	3,382 3,371	3,376	2,857 2,805	2,831
7	0,11	25,999 25,984	25,992	8,820 8,826	8,823	1,607 1,607	1,607	56,058 56,060	56,059	3,359 3,345	3,352	2,949 2,947	2,948
8	0,14	26,014 26,075	26,044	8,774 8,775	8,774	1,509 1,510	1,510	55,662 55,636	55,629	3,345 3,343	3,344	2,682 2,685	2,684
9	0,16	25,913 25,901	25,907	8,773 8,760	8,766	1,627 1,629	1,628	56,014 56,004	56,009	3,320 3,324	3,322	2,890 2,887	2,888
10	0,30	25,979 25,898	25,938	8,766 8,782	8,774	1,628 1,626	1,627	56,191 55,987	56,089	3,286 3,297	3,292	2,898 2,908	2,903
11	0,14	25,942 25,875	25,908	8,683 8,748	8,716	1,621 1,606	1,614	56,137 56,017	56,077	3,272 3,289	3,280	2,704 2,742	2,723
12	0,12	26,048 26,009	26,028	8,771 8,816	8,794	1,594 1,566	1,580	56,012 55,897	55,954	3,343 3,316	3,330	- -	-
13	0,11	26,099 26,110	26,104	8,861 8,797	8,829	1,625 1,632	1,628	55,848 55,957	55,902	3,312 3,319	3,316	2,958 2,971	2,964
14	0,12	26,111 26,017	26,064	8,832 8,792	8,812	1,637 1,643	1,640	56,077 56,138	56,108	3,313 3,312	3,312	2,978 2,965	2,972
15	0,08	26,152 26,149	26,150	8,879 8,855	8,867	1,634 1,638	1,636	55,843 55,796	55,820	3,316 3,276	3,296	2,906 2,945	2,926
16	0,11	26,025 26,012	26,018	8,791 8,809	8,800	1,611 1,612	1,612	56,236 56,186	56,211	3,308 3,323	3,316	2,946 2,922	2,934
101	-	26,035 26,012	26,024	8,786 8,773	8,780	1,613 1,608	1,610	56,202 56,175	56,188	3,306 3,295	3,300	2,515 2,392	2,454
102	0,08	25,904 25,941	25,922	8,750 8,721	8,736	1,597 1,596	1,596	56,245 56,270	56,258	3,339 3,348	3,344	1,619 1,617	1,618
103	0,13	26,010 25,970	25,990	8,790 8,820	8,805	1,640 1,650	1,645	56,020 56,020	56,020	3,430 3,380	3,405	2,890 2,900	2,895
104	0,00	25,998 25,960	25,979	8,766 8,788	8,777	1,638 1,627	1,632	56,102 56,057	56,080	3,310 3,304	3,307	1,814 1,778	1,796
105	0,11	26,149 26,174	26,162	8,796 8,841	8,818	1,620 1,628	1,624	56,355 56,380	56,368	3,363 3,380	3,372	2,911 2,897	2,904

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