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

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Foreword

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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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.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*.

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.