

ASTM Round Robin for C01.23.03 Task Group Blended Cements (with fly ash) G & H

PARTICIPATION

The ASTM C01.23.03 task group is requesting you to participate in the fourth Round Robin (RR) which will define the precision and bias of the X-ray fluorescence spectrometric (XRF) elemental analysis of hydraulic cements. Should you wish to participate please access the CCRL website to request samples be shipped to you. By making a request to CCRL on this RR you can obtain enough material to perform both fused bead and pressed pellet analyses should you choose to do so. Ask for three vials of about 30 grams on each of the RR samples for powder prep and one sample vial if sample preparation is fused disk only. The following instructions should be used for data collection and reporting.

Instructions

The ASTM C01.23.03 task group wishes to thank you for agreeing to be a part of the Round Robin (RR) which will define the precision and bias of the X-ray fluorescence spectrometric (XRF) elemental analysis of hydraulic cements. Many versions of an XRF analytical procedure exist and are used by cement producers, cement users, and commercial laboratories. Generally, all parties are interested in producing a high quality, accurate analysis for industrial use. The Cement and Concrete Laboratory (CCRL) in cooperation with the ASTM Inter-Laboratory Study (ILS) is providing samples of these Blended (with fly ash) cements. Statistical analysis of the returned data will be compiled for the task by NIST statisticians.

Method

The “10th Preliminary Draft” of “Standard Test Method for the Major and Minor Elements in Cement by X-Ray Fluorescence” produced by the task group is available by downloading from a special web-based program set up by CCRL for the specific purpose of this RR. The method is a generalized approach to analysis of hydraulic cements using XRF, and is subject to revision. The individual Standard Operating Procedures (SOP) used in your individual laboratory should fit in the generalized guidelines. Please follow the method as closely as possible so that your specific laboratory data can be utilized for comparison to all the other data.

The method has two preparation schemes for presentation to the XRF, fused disk and pressed powder. If your laboratory has the capability to do both preparation methods, please do so.

In Part 4.2.2 of the “10th Preliminary Draft,” it specifically states “*Part 2 of the method is not generally applicable to the analysis of blended cements, including those blended with silica fume, limestone, pozzolans, fly ash, slag, and any combination thereof higher than the amounts of additions found in Portland cements meeting Specification C 150.*” **For the purposes of this RR data collection only, ignore this statement and run pressed pellets, if you have the capability, for comparison with the fused disk preparation.** We need to establish the precision and truth of this statement and a comparison will determine if this statement is correct and help establish precision data on the pressed pellet mixtures by XRF.

Data Return

The data return for this RR is accomplished electronically by accessing the CCRL web site. All information required is provided for what should be reported. If you cannot prepare fused pellets, please participate in the pressed pellet RR, and conversely if you cannot prepare pressed pellets, do fused pellets. All data will be used and gratefully appreciated by the task group and task group chairman.

1. The section at the top should be completed for company and individual returning the data. No lab will be identified in the reports to the task group. The ID is necessary in case we have to contact you should data be indicate a significant outlier requiring verification of values. This is a time consuming task so please input data carefully, we hope not to go back to any lab for this reason.
2. Please give as much data on the Fusion Bead preparation as possible. Flux composition, Non-wetting agent, etc. For the Pressed Pellet, as much info as possible, Binder composition, Grinding aid and etc.
3. On Day 1, please fabricate three pellets: fusion and/or pressed pellets, and analyze each pellet twice, then report on the properly identified data sheets. On Day 2 of your choosing, make three pellets: fusion and/or pressed pellets, and run each one twice, and report results on the proper data return sheets.
4. Loss on ignition data should be run and reported each day samples are analyzed.
5. Data **shall** be reported to the CCRL web site with one more significant digit than the smallest uncertainty (or standard deviation) value of your best standard elemental value. Report determined values no later than three (3) months after receipt of samples in your laboratory.
6. The task group recognizes that ASTM C 595 blended cements require only magnesium be reported and that sulfate/sulfide are not part of XRF analysis, we wish to establish accuracy of the same suite of elements as previously requested.

The following instructions regarding calibration ranges and reporting are included for clarification purposes:

- Run both samples for apparent concentrations
- Compare the results to the end points of your calibration curves
- If your results are inside the range of your calibration curve, you may report the results to ASTM
- If your results are outside the range of your calibration curves, you must either extend your calibration curves as needed, or report the results indicating which elements are outside calibration.
- Calibrations made using the fusion technique may be extended with CRM's or certified pure chemicals (oxides)
- When extending calibration curves using pressed powder technique, for matrix matching considerations (amorphous/crystalline contents/etc.), please be advised that these cements are

blended with fly ash and each analyte determined must be from calibration curves/standards bracketing the analyte.

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