



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1888

Portland Cement (Cap Color is Purple)

This Standard Reference Material (SRM) is intended primarily for use in checking chemical methods of analysis and in calibration of instrumental methods for analysis of cements and materials of a similar matrix. SRM 1888 consists of three sealed vials of Portland cement, each containing approximately 5 grams.

The value listed for a constituent is the present best estimate of the "true" value based on the results of analyses performed at NIST and of a "definitive analysis" (1) program carried out by personnel of Construction Technology Laboratories, Inc. The uncertainty listed with each certified value is two standard deviations of the value.

| Constituent | Percent by Weight | Constituent | Percent by Weight |
|--------------------------------|---------------------------|--------------------------------|-------------------|
| CaO | 63.78 ^a ± 0.14 | SrO | 0.07 ± 0.01 |
| SiO ₂ | 20.86 ± .06 | P ₂ O ₅ | 0.085 ± 0.010 |
| Al ₂ O ₃ | 5.35 ± .05 | Mn ₂ O ₃ | 0.025 ± 0.006 |
| Fe ₂ O ₃ | 3.18 ± .02 | F | (0.02) |
| SO ₃ | 3.16 ± .13 | Cl | (0.015) |
| MgO | 0.71 ^a ± .09 | ZnO | (0.01) |
| K ₂ O | 0.56 ± .01 | Cr ₂ O ₃ | (0.01) |
| TiO ₂ | 0.29 ± .02 | LOI | 1.79 |
| Na ₂ O | 0.14 ± .02 | Total ^b | (100.05) |

Values in parentheses are not certified, but are presented for use as Information Only.

^aIf the gravimetric reference procedures of ASTM C 114 are followed, a small amount of CaO will remain in the MgO precipitate and SrO will remain in the CaO precipitate. When this procedure was used on this SRM the uncorrected values given below for CaO and MgO were obtained. (These values are not certified, but are provided for informational purposes.)

| | |
|-----|---------|
| CaO | (63.91) |
| MgO | (0.71) |

^bA correction has been made for the amount of fluoride present. This correction, which was subtracted from the gross total, was determined by multiplying the percent fluoride by the ratio of the atomic weight of oxygen to the molecular weight of fluorine (0.421). Correction of the total for the small amount of chloride was negligible (<0.01%).

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

Gaithersburg, Md 20899
September 7, 1989

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

HOMOGENEITY TESTING AND DEFINITIVE ANALYSIS PROGRAM (CONSTRUCTION TECHNOLOGY LABORATORIES, INC.): Homogeneity testing was performed by x-ray fluorescence on eighteen samples of the SRM selected by stratified random sampling. Both major (Ca and Si) and minor (Al and Fe) constituents were measured. Analysis of the data revealed no significant differences between samples, demonstrating the homogeneity of the material. "Wet" chemical gravimetric methods were used for determination of the major constituent elements Ca, Si, Al and Mg. (The methods were essentially those of ASTM C 114 using a 0.5-g sample.) Fe was determined by dichromate titration and S by precipitation in an acid solution after removal of the ammonium hydroxide group and silica. Atomic absorption spectroscopy was used for K, Na, Sr, Mn, Zn and Cr. Colorimetry was used to determine Ti and P while fluoride was determined by an ion selective electrode method. Loss on ignition (LOI) was determined at 1000 °C. In each determination, duplicate measurements were made on two to ten randomly selected samples. Summations of $100 \pm 0.1\%$ of the certified constituents in the cement are considered a good indication that the major and minor elemental data are internally consistent.

HOMOGENEITY TESTING AND COMPOSITIONAL ANALYSIS (NIST): Randomly selected samples (six) of this SRM were analyzed by x-ray fluorescence by P. Pella and G. Sleater of the Gas and Particulate Science Division. These measurements verified the homogeneity of the SRM. These measurements were also used to help establish the chemical composition of the SRM. Samples of the cement were also prepared by lithium metaborate-tetraborate fusion and analyzed by flame atomic absorption spectrometry (Al, Na) and inductively-coupled plasma emission spectrometry (Mg, Mn) by L. Wood, T. Rush, M. Epstein, and R.L. Watters, Jr., of the Inorganic Analytical Research Division. R. Paule (statistician with the National Measurement Laboratory) provided statistical analysis of the certification data obtained from the analytical programs at NIST and CTL.

ACKNOWLEDGEMENTS: The selection and preparation of this material and the coordination of the technical measurements leading to certification were under the direction of H.M. Kanare of the Chemical/Physical Research Department, Construction Technology Laboratories (CTL), Inc., Skokie, Illinois. A number of CTL personnel participated in various aspects of this project. The cements were selected and obtained with the assistance of M. Lee. The cements were processed by M. Lee, H. McCoy, C. Setterdahl, and J. Heinz. The packaging was performed by J. Heinz, H. McCoy, C. Setterdahl, A. Malen, L. Hills, K. Tylor, and T. Behr. Homogeneity testing and chemical analyses by XRF were performed by L. Hills, A. Malen, and J. Heinz. The definitive chemical analyses were carried out by R. Crow.

CAUTION: To obtain the most accurate results by x-ray fluorescence methods of analysis, the user should compare his samples to the particular SRM that is most nearly the same in overall chemical composition. Alternatively, interelement effect calibration procedures may be adopted to minimize biases.

USE AND STORAGE: The powder is extremely hygroscopic and the following procedure should be used. Samples should be used as immediately after opening as possible. The vial should be recapped immediately and stored in a desiccator over magnesium perchlorate or phosphorous pentoxide. When a sample is used after storage in a previously opened vial, the LOI for that sample should be determined in accordance with ASTM C 114 and the weight of the sample corrected for any additional moisture above the certified LOI value.

References:

1. The term "definitive analysis" is used in this certificate in a special sense to describe the careful, thorough analysis of these samples by the methods and procedures described by R.K. Kirby and H.M. Kanare in NIST Special Publication 260-110 (February, 1988) entitled "Portland Cement Chemical Composition Standards (Blending, Packaging and Testing)". The more general use of the term is described and defined in an article by J.P. Cali and W.P. Reed entitled "The Role of the National Bureau of Standards Standard Reference Materials in Accurate Trace Analysis" (pp 41-63) in NBS Special Publication 422, Vol. 1 (1976), Accuracy in Trace Analysis: Sampling, Sample Handling, Analysis.