

National Bureau of Standards Certificate of Analysis Standard Reference Material 639

Portland Cement (Cap Color is Clear)

This Standard Reference Material (SRM) is intended for use in checking chemical methods of analysis and in calibration of instrumental methods of analysis.

The value listed for a constituent is the *present best estimate* of the "true" value based on the results of a definitive analysis program carried out in the laboratories of the Portland Cement Association by N. R. Greening, W. F. Mivelaz and R. F. Crow, with W. G. Hime of Erlin, Hime Associates acting as consultant. The certified value for a constituent is not expected to deviate from the "true" value by more than ± 1 in the last significant figure reported; for a subscript figure, the deviation is not expected to be more than ± 5 . Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than those uncertainty figures.

Constituent	Percent by Weight	Constituent	Percent by Weight
CaO ^a	65.7 ₆	Na ₂ O	0.65
SiO ₂	21.6 ₁	SrO	.15
Al ₂ O ₃	4.2 ₈	P ₂ O ₅	.08
Fe ₂ O ₃	2.40	Mn ₂ O ₃	.08
SO ₃	2.4 ₈	F	.02
MgO ^a	1.2 ₆	ZnO	.01
K ₂ O	0.06	Cr ₂ O ₃	.01
TiO ₂	.32	Ign. loss	1.0 ₀
		Total ^b	100.16

^aIf the procedures of ASTM C114 are followed a small amount of CaO will remain in the MgO precipitate. In this case the uncorrected values given below for CaO and MgO should be used.

CaO 65.7₃
 MgO 1.2₈

^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).

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 B. E. Foster, R. E. Michaelis, and C. L. Stanley.

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 (Revision of certificates
 dated 2-24-77 and 6-12-74)

Stanley D. Rasberry, Chief
 Office of Standard Reference Materials

(Over)

HOMOGENEITY TESTING: Measurements were made at NBS on the portland cement supplied by the Centex Cement Corporation by S. D. Rasberry, M. M. Darr, and J. McKay by x-ray fluorescence analysis. The cement was sampled at 16 locations and two briquettes were prepared for each one. Calcium and sulfur, chosen as key elements for checking inhomogeneity, were determined by making four independent measurements on each briquette. A trend-elimination statistical design was employed in measuring the specimens. An analysis of these data indicated the homogeneity was satisfactory. Following these measurements the cement was thoroughly blended and packaged in hermetically sealed glass vials by personnel of the Cement and Concrete Research Laboratory and the Office of Standard Reference Materials.

DEFINITIVE ANALYSIS PROGRAM OF THE PORTLAND CEMENT CORPORATION: "Wet" chemical gravimetric methods were used for the major constituents CaO, SiO₂, Al₂O₃ and MgO. (The methods were essentially those of ASTM C114 using a 0.5-g sample.) Fe₂O₃ was determined by dichromate titration and SO₃ by precipitation in an acid solution after removal of the ammonium hydroxide group and silica. Atomic absorption spectroscopy techniques were used for K₂O, Na₂O, SrO, Mn₂O₃, ZnO and Cr₂O₃. Colorimetric techniques were used to determine TiO₂ and P₂O₅ while fluoride was determined by an ion selective electrode method. Loss on ignition was determined at 1000°C. In each determination duplicate measurements were made on two to ten randomly selected samples. Through the use of various techniques (colorimetric, gravimetric, potentiometric, optical emission elements were determined to be less than 0.01 wt%: Cl, Ba, Zr, B, V, Ni, Mo, Sn, Pb, Cu, and Ag. The total of 100.16% of the certified constituents in the cement corroborates the evaluation and indicates that significant biases have not been introduced.

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