

	NEW YORK STATE DEPARTMENT OF TRANSPORTATION MATERIALS BUREAU ALBANY, NY 12232-0861	Test Method No. NY 711-24C Issue Date: October 2013 Subject Code: 7.42-5
	TEST METHOD	
SUBJECT: WDXRF Method for Chemical Analysis of Oxides in Supplementary Cementitious Materials (SCM)		
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1. SCOPE

- 1.1 This test method covers the chemical analysis of Supplementary Cementitious Materials (SCM) by Wave Dispersive X-ray Fluorescence for the oxides in the ranges indicated in Table 1.

The methods are based on the “Rapid Methods of Chemical Analysis of Hydraulic Cement” ASTM STP 985, and “Chemical Analysis of Hydraulic Cement” AASHTO T-105 and were modified and developed by New York State Department of Transportation, Instrumental Analysis-Inorganic Unit of the Chemistry Laboratory.

A powdered SCM sample is blended with a Lithium Borate flux and fused to form a bead. The beads are analyzed by WDXRF in a vacuum environment, and the concentration is measured based upon the wavelength of the spectral line of the desired element.

Table 1

Ranges of Concentrations for Elements for Chemical Analysis

Element	Concentration Range, % Oxide
Silicon (SiO ₂)	66 to 74
Aluminum (Al ₂ O ₃)	2 to 5
Iron (Fe ₂ O ₃)	0 to 3

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards:

T-105 Standard Method of Test for Chemical Analysis of Hydraulic Cement
M-85 Standard Specifications for Portland Cement

2.2 ASTM Standards:

STP 985 Rapid Methods for Chemical Analysis of Hydraulic Cement

C-311-11b Standard Test Method for Sampling and Testing of Fly Ash or Natural Pozzolans for use in Portland-Cement Concrete.

2.3 Katanx K1 Prime User's Manual

2.4 ARL OPTIM'X Users Manual

3. METHODS

3.1 The methods appear in the order shown in Table 2.

Table 2 – Order of Methods

METHOD	SECTION
Sample & Standard Preparation	7
Fe ₂ O ₃	8
Al ₂ O ₃	8
SiO ₂	8

4. INTERFERENCES AND LIMITATIONS

4.1 These procedures were developed primarily for the analysis of SCM. Limitations are noted in the procedure for specific constituents.

5. APPARATUS AND MATERIALS

- 5.1 Wave Dispersive X-ray Fluorescence Spectrometer: Power supply 50 watts (50 kV max. or 2 mA max), Goniometer with: Fixed collimator (medium angular admittance), Crystal changer with 3 crystals fitted: PET, AX06, and LiF200 and 2 detectors fitted: Flow Proportional Counter and Scintillation Counter.
- 5.2 Gas cylinders of P10 gas: 90% Ar (48), 10% (±5%) of CH₄(35) and Helium (46) with a two-gauge, two stage pressure reducing regulators compatible with the gas required.
- 5.3 Analytical Balance meeting requirements of AASHTO T-105, Section 4.2.
- 5.4 Weighing dish.
- 5.6 Automatic Fluxer with platinum crucible and bead mold

6. REAGENTS

- 6.1 Spex Sample Prep Flux™ Lithium Borates containing 34.83% Li₂B₄O₇, 64.67% LiBO₂, 0.5% LiBr

NOTE 1: The text of this method is most applicable to the use of an ARL OPTIM'X with UniQuant® and Katanax K1 Prime Automatic Fluxer. Laboratories using instruments other than ARL and Katanax should utilize

the method to the fullest extent possible.

7. PREPARATION OF SAMPLE

7.1 Preparation of Beads— Weigh 5.000 grams of Flux into tared platinum crucible and record weight. Tare crucible containing flux, weigh 1.000 gram of SCM directly into platinum crucible, record weight. Using a weigh dish, weigh 5 .000 grams of Flux and record weight. Add flux to the platinum crucible containing flux and sample. Carefully place platinum crucible in to Automatic Fluxer. Fuse sample utilizing desired method.

7.2 Analyze for Fe_2O_3 , Al_2O_3 , SiO_2 on WDXRF using OptiQuant method. Report in SM/LIMS

8. PROCEDURE FOR Fe_2O_3 , Al_2O_3 , SiO_2 ,

8.1 Samples are prepared in accordance with ARL OPTIM'X User Manual AA83612, Chapter 4 Samples, Thermo Scientific, 2002.

9. PROBLEMS ENCOUNTERED

9.2 Interferences may arise due to scatter, absorption or other elements; therefore standardization is based upon internal calibration and standards for the WDXRF Instrument. Calibration discrepancies can be resolved with drift correction which is performed utilizing Setting-Up Samples provided by the manufacturer.

10. CALCULATIONS

10.1 The WDXRF's software program gives a direct reading of the concentrations in percent.