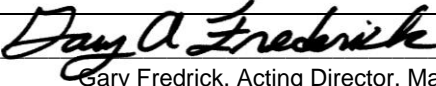
	NEW YORK STATE DEPARTMENT OF TRANSPORTATION MATERIALS BUREAU ALBANY, NY 12232-0861 TEST METHOD	Test Method No. NY 711-23C Issue Date: February 2013 Subject Code: 7.42-5
SUBJECT: WDXRF Screening for Sulfur in Ground Granulated Blast Furnace Slag		
APPROVED:  Gary Fredrick, Acting Director, Materials Bureau		Supersede: n/a Dated:

1. SCOPE

- 1.1 This test method covers the chemical analysis of Ground Granulated Blast Furnace Slag (GGBFS) by Wave Dispersive X-ray Fluorescence for Sulfur content.

The methods are based on the "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 cement sample is milled with a cellulose binder and pressed to form a pellet. The pellets 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.

2. REFERENCE DOCUMENTS:

- 2.1 AASHTO T-105 Method of Test for Chemical Analysis of Hydraulic Cement
- 2.2 *ARL OPTIM'X Users Manual*
- 2.3 UniQuant[®] 5 User Manual, chapters 6-7.

3. INTERFERENCES AND LIMITATIONS

- 3.1 These procedures were developed primarily for the analysis of GGBFS. Limitations are noted in the procedure for specific constituents

4. METHODS

- 4.1 The methods appear in the order shown in Table 2.

Table 2 – Order of Methods

METHOD	SECTION
Sample Preparation	7
Sulfur	8

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 Simultaneous analysis of Magnesium and Sodium with curved AX06 multilayer crystal and sealed detector (no gas required).
- 5.3 Gas cylinders of P10 gas: 90% Ar (48), 10% ($\pm 5\%$) of CH₄(35) and Helium (46) with a two-gauge, two stage pressure reducing regulators compatible with the gas required.
- 5.4 Analytical Balance meeting requirements of AASHTO T-105, Section 4.2.
- 5.5 Weighing dish.
- 5.6 Mixer/Mill capable of grinding to analytical fineness
- 5.7 Tungsten carbide milling vessel and beads
- 5.8 Manual Hydraulic Pellet Press capable of 40,000lbs of pressure.
- 5.9 40mm Die set for Pellet Press.
- 5.10 Adjustable-Volume Pipette, volume range 0.5-5ml.
- 5.11 Desiccators provided with desiccant preferably with a color change indicator to show depletion. Calcium Chloride is not a satisfactory desiccant for this type of analysis

6. REAGENTS

- 6.1 Spex Sample Prep PrepAid™ 3642 Cellulose Binder.
- 6.2 Vertrel.®

NOTE 1: The text of this method is most applicable to the use of an ARL OPTIM'X with UniQuant®. Laboratories using instruments other than ARL should utilize the method to the fullest extent possible

7. PREPARATION OF AND SAMPLE

- 7.1 Preparation of Pellets_Weigh 12.000 grams of Ground Granulated Blast Furnace Slag into a weighing dish, add 1.200 grams of Cellulose binder. Carefully with a small brush, pour contents of weighing dish into the Tungsten carbide grinding vessel. In a fume hood, pipette 0.5 ml Vertrel® into the vessel. Place vessel in Mixer/Mill. Grind for 2

minutes. Transfer ground materials to a clean sheet of paper with a stiff brush, brushing out the inside of the Tungsten Grinding vessel, lids and gaskets. Mix the sample repeatedly on the paper by pulling up alternate corners of the paper to ensure homogeneity. Transfer the mixed sample to a prepared aluminum cup so that it is heaping. Tap the cup on the lab bench to ensure a level surface. Place bottom mold, shiny side up, in die, followed by the prepared sample and top mold, shiny side down and finally the plunger. Place the die in the pellet press and press to reach 30,000 lbs, hold for 15 - 20 seconds maintaining pressure. Gently and slowly turn the release valve. Remove die from press and take off bottom mold replacing it with sample cup. Place die back into the press and increase pressure until the plunger drops assuring sample has been discharged into sample cup. Remove sample pellet carefully, only touching sides and place in a desiccator until ready for analysis

7.2 Analyze for S on WDXRF using OptiQuant method. Report in SM/LIMS.

7.3 Samples are prepared and analyzed in accordance with ARL OPTIM'X User Manual AA83612, Chapter 4 Samples, Thermo Scientific, 2002 and UniQuant[®] User Manual, chapters 6-7.

8. PROCEDURE FOR SULFUR

8.1 Samples are prepared in accordance with ARL OPTIM'X User Manual AA83612, Chapter 4 Samples, Thermo Scientific, 2002. Samples are prepared and analyzed in accordance with ARL OPTIM'X and UniQuant[®] 5 User Manual, chapters 6-7.

9. PROBLEMS ENCOUNTERED

9.1 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.