METHODS OF SAMPLING AND TESTING
MT 541-23
CHEMICAL ANALYSIS OF FLY ASH AND POZZOLANS VIA X-RAY FLOURESCENCE
(Montana Method)

1 Scope

1.1 This test method describes the procedures used to determine the concentration of elemental oxides of fly ash and pozzolans. Two test procedures are described in this document.

1.1.1 WDXRF Determination of Elemental Oxides in fly ash and pozzolans using the Pressed Pellet method.

1.1.2 WDXRF Determination of Elemental Oxides in fly ash and pozzolans using the Fusion method.

1.2 This test method also denotes reference test methods.

1.2.1 Loss on Ignition of Pozzolan and Fly Ash Cement – ASTM C311, Sections 13 and 14

1.2.2 Moisture of Fly Ash and Pozzolans – ASTM C311 Sections 11 and 12

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Referenced Documents

ASTM

C311 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
E29 Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E542 Standard Practice for Gravimetric Calibration of Laboratory Volumetric Apparatus
E694 Standard Specification for Laboratory Glass Volumetric Apparatus STP985 Rapid Methods for Chemical Analysis of Hydraulic Cement

MT Materials Manual

MT 607 Procedure for Reducing Field Samples to Testing Size

3 Summary of Test Method

3.1 *WDXRF Determination of Elemental Oxides in Fly Ash or Pozzolans using Pressed Pellet Summary of Test Method*

In this test method, fly ash or pozzolans are mixed with a grinding, blending, and pelletizing additive and then pressed into a pellet using a 32 mm Die Set and Press. The pellet is then analyzed by S8 Tiger Wave Dispersive X-Ray Fluorescence Analyzer (WDXRF). The following analytes are quantified as oxides: calcium, magnesium, iron, aluminum, potassium, silicon, sodium, and sulfur.

3.2 *WDXRF Determination of Elemental Oxides in Fly Ash and Pozzolans using Fusion Summary of Test Method*

In this test method, fly ash or pozzolans are mixed with a Lithium based flux, fused in a platinum crucible, and then poured into a platinum mold to form a glass bead. The bead is cooled at room temperature. The pellet is then analyzed by S8 Tiger Wave Dispersive X-Ray Fluorescence Analyzer (WDXRF). The following analytes are quantified as oxides: calcium, magnesium, iron, aluminum, potassium, silicon, sodium, and sulfur.

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance for the fly ash or pozzolan cement submittals for suppliers on the MDT Qualified Products List as well as provide analytical information for design applications using Blended cement.

WDXRF DETERMINATION OF ELEMENTAL OXIDES IN FLY ASH AND POZZOLANS USING PRESSED PELLET

5 Apparatus

5.1 Wave Dispersive X-Ray Fluorescence (WDXRF) Analyzer

5.2 32 mm Pellet Press Die Set

5.3 Pellet Press – Capable of maintaining 10,000 pounds of pressure for 4 minutes

5.4 Analytical Mill (i.e., IKA A11 Basic S001)

5.5 Analytical Balances – For the initial weighing of samples and standards, a balance with a precision of 0.0001 g should be used. For weighing material over 210 g, a balance with a precision of 0.01 g should be used.

6 Reagents and Materials

6.1 Grinding, blending, and pelletizing additive - Powder wax form (i.e., Chemplex Spectroblend)

7 Sampling

7.1 Fly and pozzolan samples are to be split in accordance with MT 607. A 50 mL sample should be provided to the Chemistry Lab for analysis.

8 Calibration and Standardization

8.1 Follow manufacturer's specifications for calibrating, standardizing and drift correcting the WDXRF. Appendix A provides calibration and standardization specifications for a Bruker Tiger S8 WDXRF for the determination of elemental oxides.

9 Procedure

9.1 Weigh 5.0000 g ± 0.05 g fly ash or pozzolan cement onto tared waxed paper or small weighing boat. Record the mass.

9.2 Weigh 1.2500g ± 0.05 g wax powder pelletizing agent onto tared waxed paper or small weighing boat. Record the mass.

9.3 Transfer the fly ash or pozzolan cement and the wax powder pelletizing agent to the grinder/mixer cup of the analytical mill.

9.4 Mix the sample for 30 seconds in the analytical mill.

9.5 Scrape down the sides, and along the bottom of the mixing cup and around the blade to remove any sample/wax pellet that became stuck to the sides.

9.6 Repeat 9.4 and 9.5 three more times for a total of 2 minutes. At the end of the last mixing time scrape down the sides.

9.7 Assemble the die set according to Figure 1. Pour the sample/pelletizing additive mixture into the assembled die set.

9.8 Press the sample for 4 minutes at approximately 10,000 pounds.

9.9 Release the press and place the ejector sleeve on the die set and place it in the press to remove the pellet from the die set.

9.10 Label the side not being analyzed on the WDXRF with the sample ID.

9.11 Repeat steps 9.1 to 9.10 for additional samples.

9.12 Once all the samples have been prepared, analyze the pellets on the S8 Tiger WDXRF.

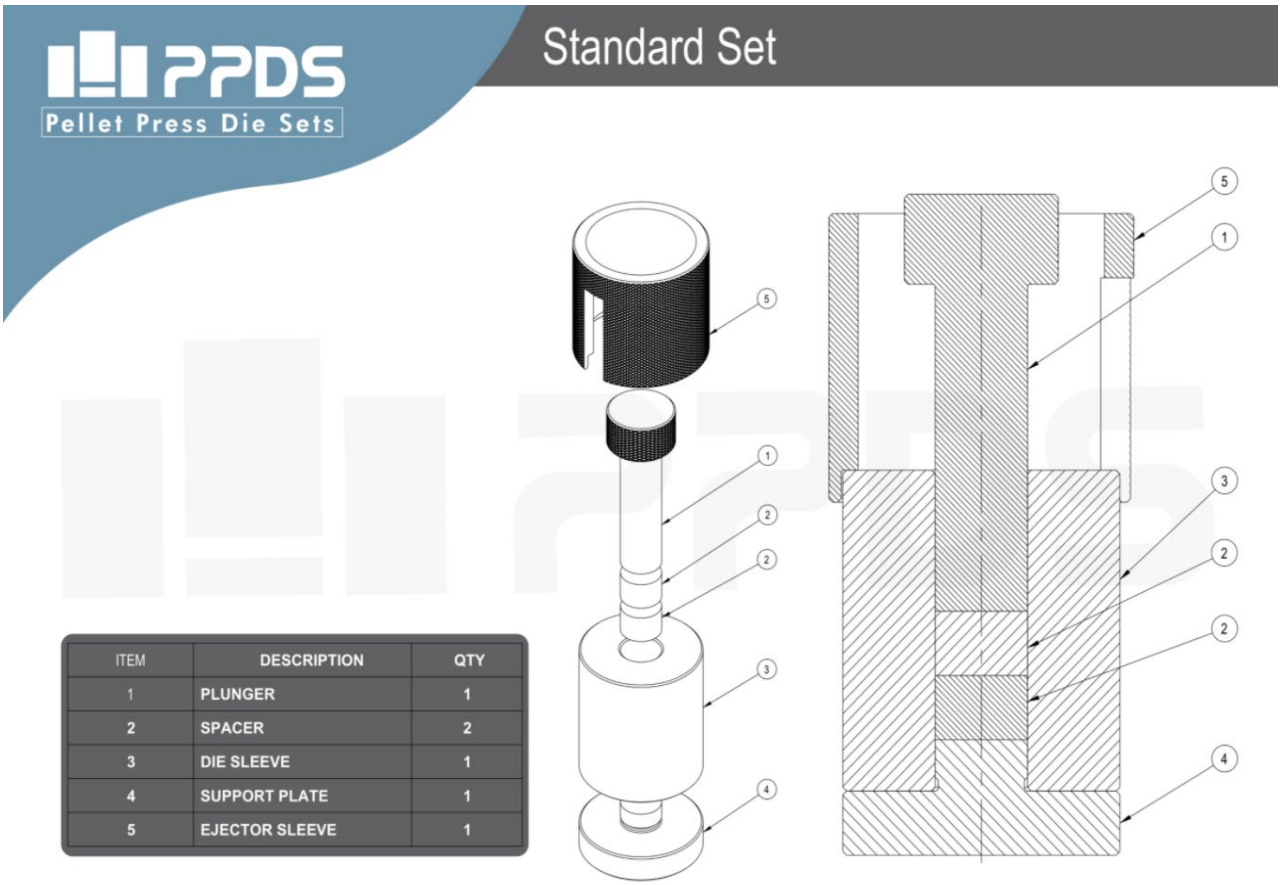


Figure 1. Die Set assembly for WDXRF

WDXRF DETERMINATION OF ELEMENTAL OXIDES IN POZZOLAN CEMENT USING FUSION

10 Apparatus

10.1 Wave Dispersive X-Ray Fluorescence (WDXRF) Analyzer

10.2 Platinum crucible

10.3 32 mm platinum mold

10.4 Analytical Balances – For the initial weighing of samples and standards, a balance with a precision of 0.0001 g should be used. For weighing material over 210 g, a balance with a precision of 0.01 g should be used.

10.5 Furnace - Capable of reaching 950ºC

11 Reagents and Materials

11.1 Fusion Flux (66.67% Lithium Tetraborate, 32.83% Lithium Metaborate, 0.5% Lithium Bromide)

12 Sampling

12.1 Fly ash and pozzolan samples are to be split in accordance with MT 607. A 50 mL sample should be provided to the Chemistry Lab for analysis.

13 Calibration and Standardization

13.1 Follow manufacturer's specifications for calibrating, standardizing and drift correcting the WDXRF. Appendix B provides calibration and standardization specifications for a Bruker Tiger S8 WDXRF for the determination of elemental oxides.

14 Procedure

14.1 Weigh 1.5000 g ± 0.05 g fly ash or pozzolan cement into a small beaker or plastic cup. Record the mass.

14.2 Weigh 7.5000g ± 0.05 g Fusion Flux into a small beaker or plastic cup. Record the mass.

14.3 Mix the cement sample and Fusion Flux together and pour the mixture into the platinum crucible.

14.4 Place the crucible and mold into a furnace preheated to 550ºC.

14.5 Change the furnace temperature to 950ºC.

14.6 5 minutes after the furnace reaches 950ºC, mix the sample/flux mixture at a 45º angle.

14.7 Continue to fuse the sample for 5 minutes, mix the sample/flux mixture at a 45º angle.

14.8 Continue the fusing process for 10 minutes, mix the sample again at a 45º angle.

14.9 Continue to fuse the sample for an additional 5 minutes.

14.10 Remove the mold from the furnace and place it on a heat resistant surface. Remove the crucible from the furnace and quickly, but carefully pour the molten sample into the mold.

14.11 Allow the sample to cool for 25 minutes at room temperature.

14.12 Once cool, remove the sample from the mold and label the rounded side of the bead with a

sticker.

14.13 Repeat steps 14.1 to 14.12 for additional samples.

14.14 Once all the samples have been prepared, analyze the fusion beads on the S8 Tiger WDXRF.

15 Report

Data Reporting for fly ash and pozzolans will be reported as shown below:

Analyte	Reported As	Significance
Ca	CaO	XX.XX
Al	Al_2O3	X.XX
Fe	Fe_2O_3	X.XX
Mg	MgO	X.XX
Si	SiO_2	XX.XX
K	K_2O	0.XX
Na	Na_2O	0.0XX
S	SO_3	X.XXX
LOI	LOI	0.XXX
Moisture	Moisture	0.XXX

16 Validation

For validation data quality control information consult ASTM C114 and ensure all instruments meet its conditions.

APPENDIX A
WDXRF DETERMINATION OF ELEMENTAL OXIDES IN FLY ASH AND POZZOLANS USING PRESSED PELLET

Bruker Tiger S8 configuration parameters

Equipment Configuration for Al

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 68, ULD = 141% of nominal peak
h. Adjusted peak: 144.680 degrees 2-theta
i. Wavelength: 8.3393 Å
j. Background: None
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 21 standards
n. Alphas: Ca
o. Line overlap correction: None
p. Corrected Intensity offset: On, -3.3128 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: NA

Equipment Configuration for Ca

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 78, ULD = 230% of nominal peak
h. Adjusted peak: 113.109 degrees 2-theta
i. Wavelength: 3.3584 Å
j. Background: none
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 21 standards
n. Alphas: Fe
o. Line overlap correction: None
p. Corrected Intensity offset: On, +45.71 kcps
q. Quadratic Correction: on
r. Sample measurement time: 40 seconds
s. Background measurement time: NA

Equipment Configuration for Fe

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: scintillation counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 57.521 degrees 2-theta
i. Wavelength: 1.936 Å
j. Background: none

k. Absorption correction: Variable alphas
l. Intensity model: net intensity
m. Minimization target: absolute error, 23 standards
n. Alphas: none
o. Line overlap correction: none
p. Corrected Intensity offset: on, -17.8 kcps
q. Quadratic Correction: off
r. Sample measurement time: 30 seconds
s. Background measurement time: NA

Equipment Configuration for K

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 75, ULD = 136% of nominal peak
h. Adjusted peak: 136.655 degrees 2-theta
i. Wavelength: 3.7414 Å
j. Background: none
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 23 standards
n. Alphas: Ca
o. Line overlap correction: none
p. Corrected Intensity offset: on, 0.1985 kcps
q. Quadratic Correction: off
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for Mg

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 55.9 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 153% of nominal peak
h. Adjusted peak: 20.381 degrees 2-theta
i. Wavelength: 9.893 Å
j. Background: none
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 25 standards
n. Alphas: K
o. Line overlap correction: (Al KA1-HS) * (-0.01484), (Ca KA1-HS) * (+0.008641)
p. Corrected Intensity offset: on, -1.37 kcps
q. Quadratic Correction: off
r. Sample measurement time: 50 seconds
s. Background measurement time: none

Equipment Configuration for Na

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 55.9 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak

h. Adjusted peak: 24.618 degrees 2-theta
i. Wavelength: 11.91 Å
j. Background: 1 at 27.230
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 25 standards
n. Alphas: K
o. Line overlap correction: none
p. Corrected Intensity offset: on, +0.08056 kcps
q. Quadratic Correction: off
r. Sample measurement time: 60 seconds
s. Background measurement time: 30 seconds

Equipment Configuration for Si

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 109.004 degrees 2-theta
i. Wavelength: 7.1254 Å
j. Background: none
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 22 standards
n. Alphas: Al, K
o. Line overlap correction: none
p. Corrected Intensity offset: on, +8.5 kcps
q. Quadratic Correction: off
r. Sample measurement time: 40 seconds
s. Background measurement time: none

Equipment Configuration for SO_3

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: flow counter LLD = 49, ULD = 142% of nominal peak
h. Adjusted peak: 75.738 degrees 2-theta
i. Wavelength: 5.3722 Å
j. Background: none
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: net intensity
m. Minimization target: absolute error, 23 standards
n. Alphas: Mg
o. Line overlap correction: (Fe KA1-HR) * (+0.003027)
p. Corrected Intensity offset: on, -2.137 kcps
q. Quadratic Correction: on
r. Sample measurement time: 60 seconds
s. Background measurement time: none

Line Selection for Standards

a. Ca: KA1-HS-Min
b. Al: KA1-HS-Min
c. Fe: KA1-HR-Min
d. Mg: KA1-HS-Min
e. Si: KA1-HS-Min
f. K: KA1-HS-Min
g. Na: KA1-HS-Min
h. S: KA1-HS-Min

APPENDIX B
XRF FUSION DETERMINATION OF ELEMENTAL OXIDES IN FLY ASH AND POZZOLANS

Bruker Tiger S8 configuration parameters

Equipment Configuration for Al

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 59, ULD = 147% of nominal peak
h. Adjusted peak: 144.673 degrees 2-theta
i. Wavelength: 8.3393 Å
j. Background: None
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 17 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.971 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 50 seconds
s. Background measurement time: NA

Equipment Configuration for Ca

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: flow counter LLD = 60, ULD = 232% of nominal peak
h. Adjusted peak: 113.083 degrees 2-theta
i. Wavelength: 3.3584 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 17 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.32 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 50 seconds
s. Background measurement time: NA

Equipment Configuration for Fe

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: scintillation counter LLD = 57, ULD = 150% of nominal peak
h. Adjusted peak: 57.522 degrees 2-theta
i. Wavelength: 1.936 Å
j. Background: None
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity

m. Minimization target: Absolute error, 17 standards
n. Alphas: Variable alphas
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.3622 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: NA

Equipment Configuration for K

a. Mask: 28 mm
b. Mode: Vacuum with seal, 50kV, 20mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 4.026 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 136.630 degrees 2-theta
i. Wavelength: 3.7414 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 16 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.05864
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: NA

Equipment Configuration for Mg

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 55.9 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 40, ULD = 160% of nominal peak
h. Adjusted peak: 20.366 degrees 2-theta
i. Wavelength: 9.893 Å
j. Background: 19.033 degrees 2-theta and 23.122 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 17 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.1882 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: NA

Equipment Configuration for Na

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 55.9 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 24.612 degrees 2-theta
i. Wavelength: 11.91 Å

j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 17 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.01355 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for Si

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 109.022 degrees 2-theta
i. Wavelength: 7.1254 Å
j. Background: None
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 17 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -12.62 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 80 seconds
s. Background measurement time: NA

Equipment Configuration for SO_3

a. Mask: 28 mm
b. Mode: Vacuum with seal, 30kV, 33mA
c. Filter: None
d. Be: 75um
e. Crystal (nominal): 2d = 8.752 Å
f. Collimator aperture (nominal): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 75.748 degrees 2-theta
i. Wavelength: 5.3722 Å
j. Background: None
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity
m. Minimization target: absolute error, 16 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.02588 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 80 seconds
s. Background measurement time: NA

Line Selection for Standards

a. Ca: KA1-HR-Min
b. Al: KA1-HS-Min
c. Fe: KA1-HR-Min
d. Mg: KA1-HS-Min
e. Si: KA1-HS-Min
f. K: KA1-HR-Min
g. Na: KA1-HR-Min
h. S: KA1-HS-Min

PAGE INTENTIONALLY LEFT BLANK