METHODS OF SAMPLING AND TESTING
MT 540-23
CHEMICAL ANALYSIS OF BLENDED CEMENT VIA X-RAY FLUORESCENCE
(Montana Method)

1 Scope

1.1 This test method describes the procedures used to determine the concentration of elemental oxides of Blended cement. Two test procedures are described in this document:

1.1.1 WDXRF Determination of Elemental Oxides in Blended Cement using the Pressed Pellet method.

1.1.2 WDXRF Determination of Elemental Oxides in Blended Cement using the Fusion method.

1.2 This test method also denotes reference test methods.

1.2.1 Loss on Ignition of Blended Cement – ASTM C114, Section 18

1.2.2 Insoluble Residue of Blended Cement – ASTM C114, Section 7

1.2.3 Carbon Dioxide Determination in Blended Cement – ASTM C114, Section 24

1.2.4 Sulfide Determination in Blended Cement – ASTM C114, Section 17

1.2.5 Halogen Determination in Blended Cement – No current validated method

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

C114 Standard Test Methods for Chemical Analysis of Hydraulic Cement C595/C59M Standard Specifications for Blended Hydraulic Cements
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 Blended 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 Blended Cement using Pressed Pellet Summary of Test Method*

In this test method, Blended Hydraulic cement is 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, silicon, iron, aluminum, potassium, titanium, sodium, manganese, zinc, chromium, phosphorus and sulfur.

3.2 *WDXRF Determination of Elemental Oxides in Blended Cement using Fusion Summary of Test Method*

In this test method, Blended cement is 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, silicon, iron, aluminum, potassium, titanium, sodium, manganese, zinc, chromium, phosphorus, strontium and sulfur.

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance for the Blended 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 BLENDED CEMENT 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 Blended Cement 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 Blended 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 Blended cement and 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.

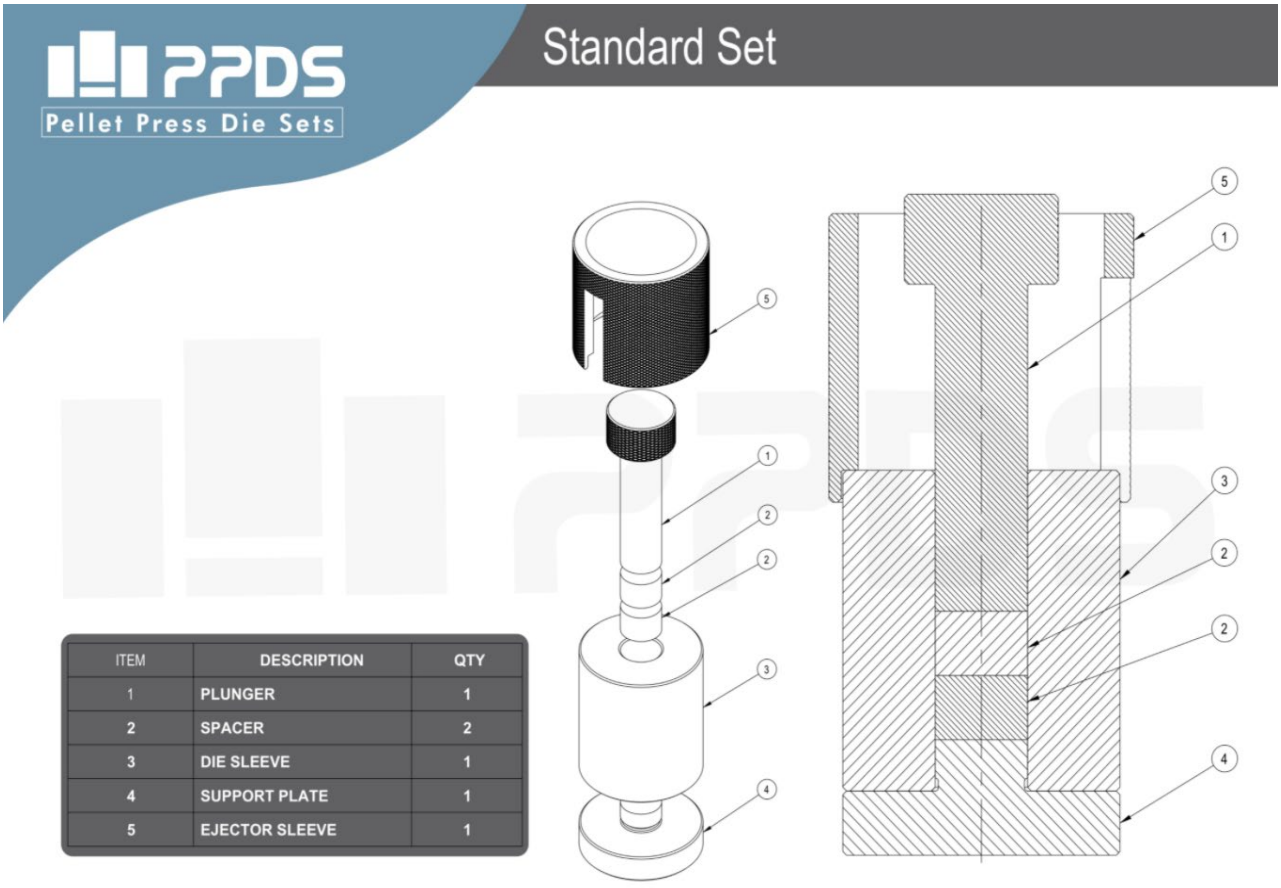

Figure 1. Die Set assembly for WDXRF

WDXRF DETERMINATION OF ELEMENTAL OXIDES IN BLENDED 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 Cement 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 Blended 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 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 Blended Cement will be reported as shown below:

Analyte	Reported As	Significance
Ca	CaO	XX.XX
Al	Al_2O_3	X.XX
Fe	Fe2O3	X.XX
Mg	MgO	X.XX
Si	SiO_2	XX.XX
Ti	TiO_2	0.XX
Cr	Cr_2O_3	0.0XX
K	K2O	X.XXX
Mn	Mn_2O_3	0.XXX
Na	Na_2O	0.XXX
P	P_2O_5	0.XXX
Zn	ZnO	0.XXX
S	SO_3	X.XX
S-	S-	X.XX
LOI	LOI	X.XX
ISR	ISR	0.XX
CO_2	CO_2	X.XX

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 BLENDED CEMENT 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 = 50, ULD = 150% of nominal peak
h. Adjusted peak: 144.673 degrees 2-theta
i. Wavelength: 8.3393 Å
j. Background: None
k. Absorption correction: None
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, -0.2853 kcps
q. Quadratic Correction: On
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, 19mA
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 = 55, ULD = 220% of nominal peak
h. Adjusted peak: 113.102 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, 23 standards
n. Alphas: Mg
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.8524 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 50 seconds
s. Background measurement time: NA

Equipment Configuration for Cr

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: scintillation counter LLD = 62, ULD = 143% of nominal peak
h. Adjusted peak: 69.391 degrees 2-theta
i. Wavelength: 2.2897 Å
j. Background: None
k. Absorption correction: None

l. Intensity model: Net Intensity
m. Minimization target: Absolute error, 15 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +1.046 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 300 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): 0.46 degrees
g. Detector: scintillation counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 57.528 degrees 2-theta
i. Wavelength: 1.936 Å
j. Background: None
t. Absorption correction: Fixed alphas (empirically adjusted values)
k. Intensity model: Net intensity
l. Minimization target: Absolute error, 24 standards
m. Alphas: Si, Ca
n. Line overlap correction: (Cr KA1-HS-Min) * (-8.552)
o. Corrected Intensity offset: On, +5.804 kcps
p. Quadratic Correction: Off
q. Sample measurement time: 40 seconds
r. Background measurement time: None

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 = 50, ULD = 150% of nominal peak
h. Adjusted peak: 136.681 degrees 2-theta
i. Wavelength: 3.7414 Å
j. Background: 1 at 138.878 degrees 2-theta
u. Absorption correction: Variable alphas
k. Intensity model: Net intensity
l. Minimization target: Absolute error, 24 standards
m. Alphas: None
n. Line overlap correction: None
o. Corrected Intensity offset: On, -0.2407 kcps
p. Quadratic Correction: Off
q. Sample measurement time: 40 seconds
r. Background measurement time: 20 seconds

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 = 68, ULD = 157% of nominal peak
h. Adjusted peak: 20.384 degrees 2-theta

i. Wavelength: 9.893 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 25 standards
n. Alphas: None
o. Line overlap correction: (Na KA1-HS-Min) * (-1.035)
p. Corrected Intensity offset: On, +0.683 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 50 seconds
s. Background measurement time: None

Equipment Configuration for Mn

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: scintillation counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 62.999 degrees 2-theta
i. Wavelength: 2.1018 Å
j. Background: 1 at 65.468 degrees 2-theta
v. Absorption correction: Fixed alphas (empirically adjusted values)
k. Intensity model: Net intensity
l. Minimization target: Absolute error, 24 standards
m. Alphas: Mg, Ca
n. Line overlap correction: (Cr KA1-HS-Min) * (-0.2186)
o. Corrected Intensity offset: On, +0.03243 kcps
p. Quadratic Correction: Off
q. Sample measurement time: 40 seconds
r. Background measurement time: 20 seconds

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 = 71, ULD = 145% of nominal peak
h. Adjusted peak: 24.629 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, 24 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.06942 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 100 seconds
s. Background measurement time: None

Equipment Configuration for P

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 = 77, ULD = 139% of nominal peak
h. Adjusted peak: 89.470 degrees 2-theta
i. Wavelength: 6.157 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: Mg
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.1273 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: None

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 = 66, ULD = 150% of nominal peak
h. Adjusted peak: 109.007 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, 23 standards
n. Alphas: Al, Fe
o. Line overlap correction: None
p. Corrected Intensity offset: On, +183.6 kcps
q. Quadratic Correction: On
r. Sample measurement time: 30 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 = 68, ULD = 141% of nominal peak
h. Adjusted peak: 75.740 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, 25 standards
n. Alphas: Fe
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.1217 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 100 seconds
s. Background measurement time: None

Equipment Configuration for Ti

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 = 83, ULD = 132% of nominal peak
h. Adjusted peak: 86.168 degrees 2-theta
i. Wavelength: 2.7485 Å
j. Background: None
t. Absorption correction: Fixed alphas (empirically adjusted values)
k. Intensity model: Net intensity
l. Minimization target: Absolute error, 25 standards
m. Alphas: Ca
n. Line overlap correction: None
o. Corrected Intensity offset: On, -0.7824 kcps
p. Quadratic Correction: Off
q. Sample measurement time: 80 seconds
r. Background measurement time: None

Equipment Configuration for Zn

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: scintillation counter LLD = 50, ULD = 148% of nominal peak
h. Adjusted peak: 41.824 degrees 2-theta
i. Wavelength: 1.4352 Å
j. Background: 1 at 42.820 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 24 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.4159 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

Line Selection for Standards

a. Ca: KA1-HS-Min
b. Al: KA1-HS-Min
c. Fe: KA1-HS-Min
d. Mg: KA1-HS-Min
e. Si: KA1-HS-Min
f. Ti: KA1-HS-Min
g. Cr: KA1-HS-Min
h. K: KA1-HS-Min
i. Mn: KA1-HS-Min
j. Na: KA1-HS-Min
k. P: KA1-HS-Min
l . Zn: KA1-HS-Min
m. S: KA1-HR-Min
n. Cl: KA1-HR-Min
o. Cr: KA1-HS-Min

APPENDIX B
WDXRF DETERMINATION OF ELEMENTAL OXIDES IN BLENDED CEMENT USING FUSION

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 = 52, ULD = 150% of nominal peak
h. Adjusted peak: 144.677 degrees 2-theta
i. Wavelength: 8.3393 Å
j. Background: 1 AT 140.556 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, --0.1054 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 50 seconds
s. Background measurement time: 20 seconds

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 = 70, ULD = 234% of nominal peak
h. Adjusted peak: 113.116 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, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -89.31 kcps
q. Quadratic Correction: On
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for Cr

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: scintillation counter LLD = 50, ULD = 145% of nominal peak
h. Adjusted peak: 69.367 degrees 2-theta
i. Wavelength: 2.2897 Å
j. Background: None
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity

m. Minimization target: Absolute error, 13 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.1925 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): 0.46 degrees
g. Detector: scintillation counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 57.534 degrees 2-theta
i. Wavelength: 1.936 Å
j. Background: 1 at 59.703 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +5.078 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: 10 seconds

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 = 143% of nominal peak
h. Adjusted peak: 136.648 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, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.2396
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 = 65, ULD = 160% of nominal peak
h. Adjusted peak: 20.402 degrees 2-theta
i. Wavelength: 9.893 Å

j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.2929 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: NA

Equipment Configuration for Mn

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: scintillation counter LLD = 54, ULD = 150% of nominal peak
h. Adjusted peak: 63.002 degrees 2-theta
i. Wavelength: 2.1018 Å
j. Background: 1 at 64.592 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.07747 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

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 = 70, ULD = 137% of nominal peak
h. Adjusted peak: 24.595 degrees 2-theta
i. Wavelength: 11.91 Å
j. Background: None
k. Absorption correction: Fixed alphas (theoretical values for average standard)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.1205 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for P

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 = 73, ULD = 134% of nominal peak
h. Adjusted peak: 89.476 degrees 2-theta
i. Wavelength: 6.157 Å
j. Background: None
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.02595 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): 0.23 degrees
g. Detector: flow counter LLD = 50, ULD = 146% of nominal peak
h. Adjusted peak: 109.017 degrees 2-theta
i. Wavelength: 7.1254 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -6.211 kcps
q. Quadratic Correction: On
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for SO3

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 = 70, ULD = 144% of nominal peak
h. Adjusted peak: 75.727 degrees 2-theta
i. Wavelength: 5.3722 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.04943 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 60 seconds
s. Background measurement time: NA

Equipment Configuration for Ti

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 = 81, ULD = 127% of nominal peak
h. Adjusted peak: 86.169 degrees 2-theta
i. Wavelength: 2.7485 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, +0.03345 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 80 seconds
s. Background measurement time: NA

Equipment Configuration for Zn

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: scintillation counter LLD = 64, ULD = 143% of nominal peak
h. Adjusted peak: 41.810 degrees 2-theta
i. Wavelength: 1.4352 Å
j. Background: 1 at 42.831 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.3309 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

Equipment Configuration for Cl

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 = 48, ULD = 128% of nominal peak
h. Adjusted peak: 65.363 degrees 2-theta
i. Wavelength: 4.7278 Å
j. Background: None
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: none
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.28 kcps
q. Quadratic Correction: Off

r. Sample measurement time: 50 seconds
s. Background measurement time: NA

Line Selection for Standards

a. Ca: KA1-HS-Min
b. Al: KA1-HS-Min
c. Fe: KA1-HS-Min
d. Mg: KA1-HS-Min
e. Si: KA1-HS-Min
f. Ti: KA1-HR-Min
g. Cr: KA1-HS-Min
h. K: KA1-HS-Min
i. Mn: KA1-HS-Min
j. Na: KA1-HS-Min
k. P: KA1-HS-Min
l. Zn: KA1-HS-Min
m. S KA1-HS-Min
n. Cl KA1-HS-Min