METHODS OF SAMPLING AND TESTING
MT 539-23
CHEMICAL ANALYSIS OF PORTLAND 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 Portland cement. Two test procedures are described in this document:

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

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

1.2 This test method also denotes reference test methods.

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

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

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

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

1.2.5 Halogen Determination in Portland Cement – No current validated method

1.3 This standard does not purport to address all 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
C150 Standard Specifications for Portland Cement
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 Portland Cement using Pressed Pellet Summary of Test Method*

In this test method, Portland 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, strontium and sulfur.

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

In this test method, Portland 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 Portland cement submittals for suppliers on the MDT Qualified Products List as well as provide analytical information for design applications using Portland cement.

WDXRF DETERMINATION OF ELEMENTAL OXIDES IN PORTLAND 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 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 Portland 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 Portland 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 of the mixing bowl to remove any sample/wax pelletizing additive that is 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.

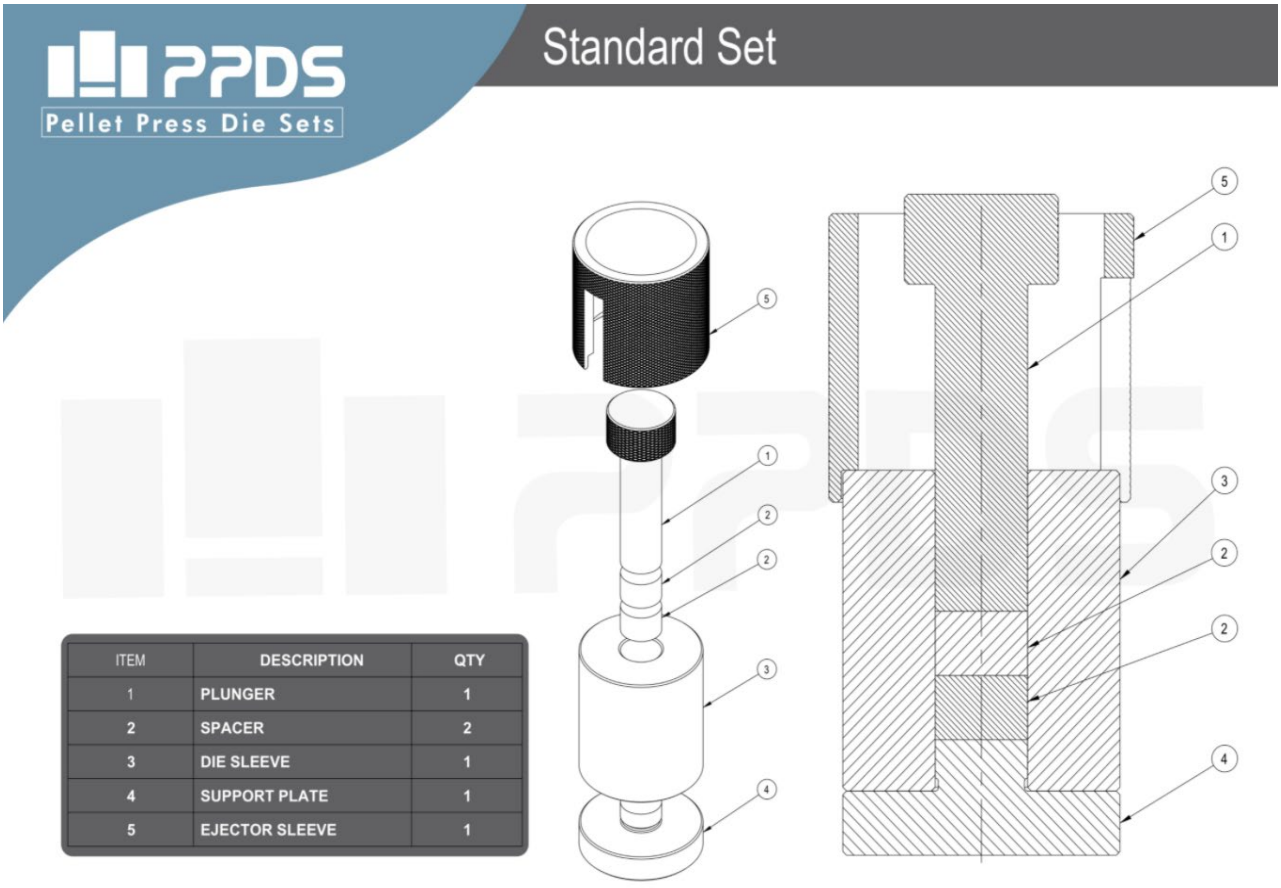


Figure 1. Die Set assembly for WDXRF

WDXRF DETERMINATION OF ELEMENTAL OXIDES IN PORTLAND 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 Portland 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 Portland Cement will be reported as shown below:

Analyte	Reported As	Significance
Ca	CaO	XX.XX
Al	Al_2O_3	X.XX
Fe	Fe_2O_3	X.XX
Mg	MgO	X.XX
Si	SiO_2	XX.XX
Ti	TiO_2	0.XX
Cr	Cr_2O_3	0.0XX
K	K_2O	X.XX
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
Lime Content	Lime Content	X.X
C_3S	C_3S	XX.X
C_2S	C_2S	XX.X
C_3A	C_3A	X.X
C_4AF	C_4AF	XX.X
Cl-	Cl-	0.0XX

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 PORTLAND CEMENT USING PRESSED PELLET

Bruker Tiger S8 XRF 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 = 55.9 Å
f. Collimator aperture (nominal): 0.23 degrees
g. Detector: flow counter LLD = 60, ULD = 147% of nominal peak
h. Adjusted peak: 17.139 degrees 2-theta
i. Wavelength: 8.3393 Å
j. Background: 1 at 18.702 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: Intensity, (Si KA1-HR-Min) * (-0.1807)
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: 10 seconds

Equipment Configuration for Ca

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 = 235% of nominal peak
h. Adjusted peak: 45.168 degrees 2-theta
i. Wavelength: 3.3584 Å
j. Background: 1 at 47.481 degrees 2-theta
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: Al, Si
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: 10 seconds

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 = 66, ULD = 137% of nominal peak
h. Adjusted peak: 69.374 degrees 2-theta
i. Wavelength: 2.2897 Å
j. Background: None
k. Absorption correction: None

l. Intensity model: Raw peak intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 300 seconds
s. Background measurement time: NA

Equipment Configuration for Cr – Low Level

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 = 57, ULD = 148% of nominal peak
h. Adjusted peak: 69.364 degrees 2-theta
i. Wavelength: 2.2897 Å
j. Background: 1 at 70.883 degrees 2-theta
k. Absorption correction: None
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
q. Quadratic Correction: Off
r. Sample measurement time: 300 seconds
s. Background measurement time: 60 seconds

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 = 47, ULD = 164% of nominal peak
h. Adjusted peak: 57.534 degrees 2-theta
i. Wavelength: 1.936 Å
j. Background: 1 at 60.207 degrees 2-theta
k. Absorption correction: Variable alphas
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: Variable
o. Line overlap correction: None
p. Corrected Intensity offset: On
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 = 74, ULD = 136% of nominal peak
h. Adjusted peak: 136.693 degrees 2-theta

i. Wavelength: 3.7414 Å
j. Background: 1 at 139.503 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. 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): 0.23 degrees
g. Detector: flow counter LLD = 48, ULD = 147% of nominal peak
h. Adjusted peak: 20.378 degrees 2-theta
i. Wavelength: 9.893 Å
j. Background: 1 at 21.956 degrees 2-theta
k. Absorption correction: Fixed alphas (theoretical values for average standard)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 100 seconds
s. Background measurement time: 20 seconds

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 = 60, ULD = 150% of nominal peak
h. Adjusted peak: 63.005 degrees 2-theta
i. Wavelength: 2.1018 Å
j. Background: 1 at 65.353 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. 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 = 50, ULD = 138% of nominal peak
h. Adjusted peak: 24.600 degrees 2-theta
i. Wavelength: 11.91 Å
j. Background: 1 at 26.365
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 100 seconds
s. Background measurement time: 40 seconds

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 = 71, ULD = 134% of nominal peak
h. Adjusted peak: 89.430 degrees 2-theta
i. Wavelength: 6.157 Å
j. Background: 1 at 91.524 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 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): 0.23 degrees
g. Detector: flow counter LLD = 50, ULD = 150% of nominal peak
h. Adjusted peak: 109.019 degrees 2-theta
i. Wavelength: 7.1254 Å
j. Background: 1 at 110.786 degrees 2-theta
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: Fe
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

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 = 73, ULD = 132% of nominal peak
h. Adjusted peak: 75.741 degrees 2-theta
i. Wavelength: 5.3722 Å
j. Background: 1 at 78.197 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 60 seconds
s. Background measurement time: 20 seconds

Equipment Configuration for Sr

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 = 72, ULD = 135% of nominal peak
h. Adjusted peak: 25.162 degrees 2-theta
i. Wavelength: 0.87526 Å
j. Background: 1 at 28.417 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

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 = 74, ULD = 134% of nominal peak
h. Adjusted peak: 86.154 degrees 2-theta
i. Wavelength: 2.7485 Å
j. Background: 1 at 89.191 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 80 seconds
s. Background measurement time: 30 seconds

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 = 65, ULD = 150% of nominal peak
h. Adjusted peak: 41.817 degrees 2-theta
i. Wavelength: 1.4352 Å
j. Background: 1 at 43.006 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On
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): 0.23 degrees
g. Detector: scintillation counter LLD = 49, ULD = 132% of nominal peak
h. Adjusted peak: 65.428 degrees 2-theta
i. Wavelength: 1.4352 Å
j. Background: 2 at 65.155 degrees 2-theta and 66.176 degrees 2-theta
k. Absorption correction: None
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 20 standards
n. Alphas: none
o. Line overlap correction: None
p. Corrected Intensity offset: On
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: 10 seconds

Line Selection for Standards

a. Ca: KA1-ALT-Min
b. Al: KA1-ALT-Min
c. Fe: KA1-HS-Min
d. Mg: KA1-HR-Min
e. Si: KA1-HR-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-HS-Min
n. Sr: KA1-HS-Min
o. Cl: KA1-HR-Min

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

Bruker Tiger S8 XRF 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 = 74, ULD = 138% of nominal peak
h. Adjusted peak: 144.699 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, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -31.28 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 = 71, ULD = 234% of nominal peak
h. Adjusted peak: 113.083 degrees 2-theta
i. Wavelength: 3.3584 Å
j. Background: None
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Raw peak intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: Si
o. Line overlap correction: None
p. Corrected Intensity offset: On, -620.1 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 = 50, ULD = 147% of nominal peak
h. Adjusted peak: 69.363 degrees 2-theta
i. Wavelength: 2.2897 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: B
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.05619 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 100 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 = 56, ULD = 148% of nominal peak
h. Adjusted peak: 57.546 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, 14 standards
n. Alphas: Variable
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.4309 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 = 74, ULD = 139% of nominal peak
h. Adjusted peak: 136.651 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, -0.1314
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): 0.23 degrees
g. Detector: flow counter LLD = 47, ULD = 153% of nominal peak
h. Adjusted peak: 20.381 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: (Ca KA1-HR-Min) * (-0.002529)
p. Corrected Intensity offset: On, +0.6633 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 = 50, ULD = 147% of nominal peak
h. Adjusted peak: 62.963 degrees 2-theta
i. Wavelength: 2.1018 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, -0.7842 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 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): 0.46 degrees
g. Detector: flow counter LLD = 50, ULD = 137% of nominal peak
h. Adjusted peak: 24.609 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, 14 standards
n. Alphas: None
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.04535 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 = 72, ULD = 150% of nominal peak
h. Adjusted peak: 89.445 degrees 2-theta
i. Wavelength: 6.157 Å
j. Background: 1 at 90.994 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, -0.01465 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 70 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): 0.23 degrees
g. Detector: flow counter LLD = 50, ULD = 146% of nominal peak
h. Adjusted peak: 109.010 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, 13 standards
n. Alphas: Na
o. Line overlap correction: None
p. Corrected Intensity offset: On, -351.4 kcps
q. Quadratic Correction: On
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): 0.23 degrees
g. Detector: flow counter LLD = 67, ULD = 144% of nominal peak
h. Adjusted peak: 75.732 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, -0.2523 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 70 seconds
s. Background measurement time: NA

Equipment Configuration for Sr

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 = 139% of nominal peak
h. Adjusted peak: 25.158 degrees 2-theta
i. Wavelength: 0.87526 Å
j. Background: 1 at 23.776 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, +1.598 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 30 seconds
s. Background measurement time: 10 seconds

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 = 79, ULD = 134% of nominal peak
h. Adjusted peak: 86.176 degrees 2-theta
i. Wavelength: 2.7485 Å
j. Background: 1 at 86.176 degrees 2-theta
k. Absorption correction: Fixed alphas (empirically adjusted values)
l. Intensity model: Net intensity
m. Minimization target: Absolute error, 14 standards
n. Alphas: B, Si, Ca
o. Line overlap correction: None
p. Corrected Intensity offset: On, -0.006213 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 100 seconds
s. Background measurement time: 30 seconds

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 = 59, ULD = 146% of nominal peak
h. Adjusted peak: 41.834 degrees 2-theta
i. Wavelength: 1.4352 Å
j. Background: 1 at 43.391 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, -0.4181 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): 0.23 degrees
g. Detector: flow counter LLD = 85, ULD = 125% of nominal peak
h. Adjusted peak: 65.414 degrees 2-theta
i. Wavelength: 4.7278 Å
j. Background: 1 at 66.396 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, -0.04806 kcps
q. Quadratic Correction: Off
r. Sample measurement time: 40 seconds
s. Background measurement time: 20 seconds

Line Selection for Standards

a. Ca: KA1-HR-Min
b. Al: KA1-HS-Min
c. Fe: KA1-HS-Min
d. Mg: KA1-HR-Min
e. Si: KA1-HR-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-HR-Min
n. Sr: KA1-HS-Min
o. Cl: KA1-HR-Min

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