CURRENT DATE OF REVISION
MT 500 SECTION
CHEMISTRY

Test Method No.	Title	Pages	Date of Publication or Revision
MT 501	pH, Insoluble Materials, Corrosion Rate, Percent Settleable Solids and Percent Passing a #10 Sieve of Brine Materials	1 pp.	Jun 2016
MT 502	Elemental Chemical Analysis Of Brine Materials	7 pp.	Sep 2016
MT 503	Vacant		
MT 504	Cyanide In Brine Material		Under Development
MT 505	Vacant		
MT 506	Vacant		
MT 507	Vacant		
MT 508	Vacant		
MT 509	Vacant		
MT 510	Vacant		
MT 511	Vacant		
MT 512	Vacant		
MT 513	Vacant		
MT 514	Vacant		
MT 515	Vacant		
MT 516	Vacant		
MT 517	Vacant		
MT 518	Vacant		
MT 519	Vacant		
MT 520	Determination of Wear Metals in Engine Oil		Under Development
MT 521	Determination of Red Dye #26 in Diesel Fuel	1 pp.	Sep 2016
MT 522	Vacant		
MT 523	Vacant		
MT 524	Vacant		
MT 525	Vacant		
MT 526	Moisture Analysis of PNS Category 8A, 8B, and 8C Solid Sodium Chloride	2 pp.	Jun 2016
MT 527	Vacant		
MT 528	Vacant		
MT 529	Vacant		

CURRENT DATE OF REVISION
MT 500 SECTION
CHEMISTRY

Test Method No.	Title	Pages	Date of Publication or Revision
MT 530	Determination of Chloride in Bridge Deck Cement by Ion Chromatography.		Under Development
MT 531	Vacant		
MT 532	Vacant		
MT 533	Chemical Analysis of Fly Ash and Pozzolans	9 pp.	Sep 2016
MT 534	Chemical Analysis of Portland Cement	8 pp.	Jun 2016
MT 535	Bridge Deck Crack Sealant IR Test		Under Development
MT 536	Chemical Analysis of Silica Fume	1 pp.	Jun 2016
MT 537	Chemical Analysis of Ground Granulated Blast-Furnace Slag (GGBFS)	4 pp.	Sep 2016
MT 538	Chemical Analysis of Blended Cement		Under Development
MT 539	Vacant		
MT 540	Vacant		
MT 541	Vacant		
MT 542	Vacant		
MT 543	Vacant		
MT 544	Vacant		
MT 545	Vacant		
MT 546	Vacant		
MT 547	Vacant		
MT 548	Analysis of Traffic Paint	7 pp.	Jun 2016

METHODS OF SAMPLING AND TESTING
MT 501-16
pH, INSOLUBLE MATERIAL, CORROSION RATE, PERCENT SETTLEABLE SOLIDS AND PERCENT PASSING A # 10 SIEVE OF BRINE MATERIALS
(*Montana Method*)

MT 501 is identical to the 2010 Pacific Northwest Snowfighters Snow and Ice Control Chemical Products Specifications and Test Protocols for the PNS Association of British Columbia, Colorado, Idaho, Montana and Washington (http://pnsassociation.org/wp-content/uploads/PNSSPECS.pdf) except for the following stipulations:

Test Method C

1 Perform a prescreening procedure for the Percent Total Settleable Solids methods.

1.1 Pour a well-mixed sample into a 50 ml conical bottom disposable vial to the 50 ml mark. Cap vial. Repeat for all samples. Place the vials in a freezer at the test temperature designated below for 168 hours:

- Sodium Chloride: -17.8°C ± 1°C (0°F ± 2°F)
- Magnesium Chloride: -17.8°C ± 1°C (0°F ± 2°F)
- Calcium Chloride: -29°C ± 1°C (20°F ± 2°F)

Note 1 – Ensure the samples remain free from agitation or disturbance during the entire testing duration.

1.2 At the end of the testing time (168 hours), observe samples. If any noticeable hardening or crystallization of the deicer is observed, then perform additional testing in accordance with PNS Test Method C. If the sample does not exhibit hardening or crystalizing the sample passes the test.

2 Report

2.1 Report settleable solids as a (V/V) percent ±0.1% in Site Manager.

2.2 Report solids passing through a number 10 sieve as a (V/V) percent ±0.1% in Site Manager.

METHODS OF SAMPLING AND TESTING
MT 502-16
ELEMENTAL CHEMICAL ANALYSIS OF BRINE MATERIALS
(*Montana Method*)

1 Scope

1.1 This test method describes the procedures used to analyze brine materials for the following analytes: arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), copper (Cu), phosphorus (P), lead (Pb), mercury (Hg), selenium (Se), and zinc (Zn) in brine materials. Additionally the samples are analyzed for magnesium (Mg), sodium (Na), and sulfur (S) to determine the percentage of magnesium chloride, sodium chloride, and sulfates in the samples.

1.2 This standard involves hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2 Referenced Documents

Milestone
Milestone Grease Digestion Application Note HPR-CH-16

3 Summary of Test Method

3.1 Samples are prepared using a microwave digestion technique based on Milestone Grease Digestion Application Note HPR-CH-16. Mercury is analyzed using a cold vapor technique with a vapor generation assembly (VGA) attached to a flame atomic absorption spectrometer (FAAS). All other analytes are analyzed using a inductively coupled plasma – optical emission spectrometer (ICP-OES).

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance and control for deicer materials and dust palliatives.

5 Apparatus

5.1 *Analytical Balance* – with a precision of 0.0001 g

5.2 *Microwave digestion system* – Capable of heating samples to 200°C and maintaining that temperature for at least 30 minutes. The system must use sealable vessels that prevent the escape of vapors.

5.3 *ICP-OES* – Capable of measuring analytes of interest at parts per million (ppm) levels and low parts per billion (ppb) levels

5.4 *Flame Atomic Absorption Spectrometer (FAAS)* – Equipped with a hollow cathode lamp (HCL) for measuring Hg and capable of operating with an attached VGA

5.5 *Vapor Generation Assembly (VGA)* – Capable of cold vapor techniques for detecting mercury at low ppb to high parts per trillion (ppt) levels

5.6 *Labware* – Glassware, Teflon, and Plasticware containers that have been properly cleaned and stored filled with dilute nitric acid solution (1 – 5%) for at least 2 days.

6 Reagents and Materials

6.1 Trace metal grade (TMG) hydrochloric acid (HCl), concentration of 32-38%

6.2 TMG nitric acid (HNO_3), concentration of 65-70%

6.3 Technical grade hydrogen peroxide (H_2O_2), concentration of 34-37%

6.4 Stannous chloride solution - 10% stannous chloride ($SnCl_2$) w/v and 20% TMG hydrochloric acid

6.5 1+1 hydrochloric acid – Made with TMG HCl (e.g., Add 500 ml TMG HCl to 400 ml reagent water and dilute to 1 L).

6.6 Reagent Water – Purified with a polishing system such that ASTM Type II water or better is obtained.

7 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES and VGA. Appendix A provides calibrating and standardizing specifications for a Varian Axial View ICP-OES, an Agilent Radial View ICP-OES, and Varian VGA.

8 Procedure

8.1 Tare a clean microwave digestion vessel on the analytical balance. Use a transfer pipet to dispense a portion of the sample into a digestion vessel in accordance with the table below. Record the mass.

Material Being Analyzed	Amount of material (grams)
Corrosion Inhibitor	1.2
Sodium Chloride Magnesium Chloride Calcium Chloride	7.5

Note 1 – Use a small weighing dish in place of a microwave digestion vessel when static electricity prevents a stable measurement. Transfer contents to a microwave digestion vessel; use a small amount of water to rinse the contents of the weighing dish.

8.2 In a ventilation hood, add 1 mL of hydrogen peroxide and 9 mL of nitric acid to each of the digestion vessels. Assemble the digestion vessels and all other components required for proper microwave digestion in accordance with the manufacturer's instructions and then run the digestion program. MDT's parameters for a Milestone Ethos EZ microwave digestion system are in Appendix A.

8.3 After completion of the digestion program, allow the carousel to cool before removing. The carousel may be left overnight to cool.

8.4 Remove a digestion vessel and open it. Rinse the sample into a clean Teflon beaker or other suitable cleaned container. Tare an appropriate storage container on the analytical balance. Rinse the sample into the storage container. Place the storage container back onto the analytical balance and dilute with reagent water until a mass of 100.00 g ± 0.05. Record the mass to nearest 0.01 g. Repeat for all samples.

8.5 Label the sample bottles with the sample number, date, analyst initials, and as Brine Material stock solution.

8.6 Dilute the stock solutions with reagent water for the analysis of Mg, Na, and S to 1:100 by weight. Label the dilute samples with the sample number, date, analyst initials, and as Brine Material dilute solution.

8.7 Analyze the stock and dilute samples on the radial view ICP-OES, axial view ICP-OES, and VGA with FAAS, as appropriate.

9 Calculation or Interpretation of Results

9.1 Concentration Calculations

9.1.1 As, Ba, Cd, Cr, Cu, P, Pb, Se, and Zn (measured in mg/kg by the ICP-OES)

Sample concentration is calculated by multiplying the measured value by a ratio of the solution weight divided by the sample weight.

$$C = DM(\frac{L}{S})$$

Where:

C = sample concentration (mg/kg)

M = measured value (µg/kg)

L = solution mass

S = sample mass

D = dilution factor that is equal to 1 for final products or is equal to 100%/P where P is the percentage of the component of interest in the final product

Note – Concentration calculations for corrosion inhibitors requires the inclusion of a dilution factor based on the amount of inhibitor used in the product.

9.1.2 Hg (measured in µg/kg by the VGA)

Mercury concentration is calculated by multiplying the measured value by a ratio of the solution mass divided by the sample mass then divided by 1000.

$$C = \frac{ML}{1000S}$$

Where:

C = sample concentration in mg/kg

M = measured value in µg/kg

L = solution mass

S = sample mass

9.2 Percent Weight Calculations

$$P = \frac{LMNO}{10000STU}$$

Where:

P = percent weight of the desired compound

L = mass of the first solution

M = measured value of the analyte in mg/kg

N = mass of the second solution

O = molecular mass of the desired compound

S = sample mass

T = mass of the aliquot from the first solution

U = atomic mass of the measured analyte.

Elements and molecules	Atomic mass	Molecular mass
Na	22.99	
NaCl		58.44
Mg	24.31	
$MgCl_2$		95.21
S	32.066	
SO_4		96.066

10 Report

10.1 Elements As, Ba, Cd, Cr, Cu, Hg, P, Pb, Se, and Zn – report as mg/kg

10.2 $MgCl_2$, NaCl, and SO_4 – report as percent by weight of the product

APPENDIX A
ANALYTICAL INSTRUMENTATION CALIBRATION

Milestone Ethos EZ microwave digestion system

Digestion program used: motoroil.mpr

Heat and time: Heat to 200°C and hold temperature during a 30 minute interval. Follow by a 10 minute cooling period.

Rotor: SK-10

Varian Axial View ICP-OES configuration parameters for brines and non-inhibitors

Equipment Configuration

a. Nebulizer: Sea Spray
b. Spray chamber: cyclonic
c. Sample pump tubing: White/White
d. Waste pump tubing: Blue/Blue
e. Rinse Solution: 3% Nitric Acid
f. Torch: High solids with sheath gas
g. Sheath gas: Argon at 10mL/min
h. Power: 1.20 kW
i. Plasma flow: 15.0 L/min
j. Auxiliary flow: 1.50 L/min
k. Nebulizer flow: 0.70 L/min for $MgCl_2$ brines 0.80 L/min for NaCl brines
l. Replicate Read time: 10.00 s
m. Instrument stabilization delay: 15.00s
n. Sample uptake delay: 30.00s
o. Pump rate: 15 rpm
p. Rinse time: 100s
q. Fast pump: yes
r. Replicates: 3

Line Selection for Standard, Samples, and Blanks:

a. As 193.696
b. Ba 233.527
c. Cd 214.439
d. Cr 267.716
e. Cu 324.754
f. P 213.618
g. Pb 220.353
h. Se 196.026
i. Zn 213.857

Calibration Varian Axial ICP-OES

a. Type: Linear for all elements
b. Linear-Maximum percent error of 5% except Cd that can be 15% and confidence limit of 0.99%
c. Calibration standards: Prepare calibration standards and blanks so that the sample matrices are being mimicked by compensating for the content of acids and salts. Prepare a total of one standard blank and three standards with varying concentrations of each desired analyte.

Agilent Radial View ICP-OES configuration parameters for brines and inhibitors

Equipment configuration

a. Nebulizer: One Neb
b. Spray chamber: cyclonic
c. Sample pump tubing: White/White
d. Waste pump tubing: Blue/Blue
e. Rinse solution: 3% Nitric acid
f. Torch: High solids
g. Power: 1.15 kW $MgCl_2$ brines, 1.20 kW NaCl brines, and 1.10 kW for inhibitors.
h. Plasma flow: 15.00 L/min
i. Auxiliary flow: 1.50 L/min
j. Nebulizer flow: 0.80 L/min $MgCl_2$ brines, 0.75 L/min NaCl brines, and 0.70 L/min for inhibitors.
k. Viewing height: 11 mm $MgCl_2$ brines, 12 mm NaCl brines, and 9 mm for inhibitors.
l. Replicate read time: 10.00s
m. Instrument stabilization delay: 15s
n. Sample uptake delay: 30s
o. Pump rate: 15s
p. Rinse rate: 10s
q. Fast pump: yes
r. Replicates: 3

Line selection for standards, samples and blanks:

$MgCl_2$ brines

a. Mg 280.270
b. Na 589.592
c. S 181.972

NaCl brines

a. Na 589.592

Corrosion Inhibitors

b. As 188.980
c. Ba 455.403
d. Cd 226.502
e. Cr 205.560
f. Cu 324.754
g. P 213.618
h. Pb 220.353
i. Se 196.026
j. Zn 213.857

Calibration Agilent Radial ICP-OES

a. Type: Linear for all elements
b. Linear-Maximum percent error of 5% and confidence limit of 0.99%.
c. Calibration standards: Prepare calibration standards and blanks so that the inhibitor sample matrices are being mimicked by compensating for the acids and inhibitor components. For the brines only compensate for the acid content. Prepare a total of one standard blank and three standards with varying concentrations of each desired analyte.

Varian AA 240FS and VGA configuration parameters

Equipment configuration

a. Measurement time: 10.00s
b. Read delay: 115s
c. Sample tubing: Purple/black
d. Acid and reductant tubing: black/black
e. Replicates standards: 3
f. Replicates samples: 3
g. Precision % standards: 1.0
h. Precision % samples: 1.0
i. Lamp Current: Recommended current on Hg lamp
j. Slit width: 0.5R nm
k. Reslope rate: 50
l. Reslope standard: number 2

Wavelength selection for standards, samples, and blanks

a. 253.7 nm

Calibration Varian AA 240FS and VGA

a. Type: Linear
b. Calibration standards: Prepare calibration standards and blanks so that the acid content of the sample matrices are mimicked. Prepare a total of one standard blank and three standards with varying concentrations of each desired analyte.

Reagents

a. Acid: Described in 6.5
b. Reductant: Described in 6.4

METHODS OF SAMPLING AND TESTING
MT 504-16
CYANIDE IN BRINE MATERIAL

THIS PROCEDURE IS IN DEVELOPMENT

METHODS OF SAMPLING AND TESTING
MT 520-12
DETERMINATION OF WEAR METALS IN ENGINE OIL

THIS PROCEDURE IS IN DEVELOPMENT

METHODS OF SAMPLING AND TESTING
MT 521-16
METHOD OF TESTING DYED DIESEL
(*Modified ASTM D6258*)

MT 521 is identical to ASTM D6258 except for the following stipulations:

1. Add the following to Section 9.2.1:

Volume of Stock Standard/100 ml	Approximate Concentration of Working Standards
6.0 mL	18 mg/L
7.0 mL	21 mg/L

2. Add to the end of Section 9.5:

   "Also measure the amplitude difference between the peak and valley of 580 ± 20 nm and 561 ± 20 nm."

METHODS OF SAMPLING AND TESTING
MT 526-16
MOISTURE ANALYSIS OF PNS CATEGORY 8A, 8B, AND 8C SOLID SODIUM CHLORIDE
(*Modified ASTM E534*)

1 Scope

1.1 This method is utilized to determine the water insoluble residue of PNS specified salt for product acceptance by the Montana Department of Transportation. This method will serve as the reference method for salt with an insoluble residue of 0.0% – 10.0% by mass.

2 Referenced Documents

ASTM Standards
E534 Standard Test Methods for Chemical Analysis of Sodium Chloride

Other Standards
PNS Document - Pacific Northwest Snowfighters Snow and Ice Control Chemical Products, Specifications, and Test Protocols for the PNS Association of British Columbia, Colorado, Idaho, Montana, Oregon and Washington. http://pnsassociation.org/wp-content/uploads/PNSSPECS.pdf

3 Apparatus and Reagents

3.1 *Analytical Balance*

3.2 *Desiccator*

3.3 *Oven*

4 Procedure

4.1 Weigh 100g of salt to the nearest 0.01g (split or quartered from original sample) into a previously dried and weighed moisture dish or pan. Distribute the salt in the vessel such that it is not more than 1/4 of an inch in depth at any given location.

4.2 Dry at 110°C for 2 hours.

4.3 Cool to room temperature in a desiccator and weigh.

5 Calculation and Reporting

5.1 Calculate the percentage of moisture as follows:

$$moisture, mass\ \% = \frac{(I - F)}{I} * 100$$

where:

F = final mass of the dry salt
I = initial mass of the wet salt

5.2 Report the insoluble residue to the nearest 0.01%.

6 Validation Data

6.1 Precision and Bias

Moisture %	$\sigma_{(n-1)}$ %	n	95% CL %	Intra Laboratory $\sigma_{(n-1)}$ %	Intra Laboratory n	Intra Laboratory 95% CL %	Inter Laboratory $\sigma_{(n-1)}$ %	Inter Laboratory n	Inter Laboratory 95% CL %
0-5.0%	0.005	29	0.03	0.006	3	0.03	0.006	6	0.04
5.1%-10.0%	0.008	31	0.05	0.008	3	0.06	0.009	6	0.06

METHODS OF SAMPLING AND TESTING
MT 530-16
DETERMINATION OF CHLORIDE IN BRIDGE DECK CEMENT
BY ION CHROMATOGRAPHY

THIS PROCEDURE IS IN DEVELOPMENT

METHODS OF SAMPLING AND TESTING
MT 533-16
METHOD OF TEST FOR CHEMICAL ANALYSIS OF FLY ASH AND POZZOLANS
(Montana Method)

1 Scope

1.1 This test method describes the procedures used to determine the concentration of elemental oxides and sulfur trioxide of fly ash and pozzolans for use in Portland Cement. Three test procedures are described in this document:

1.1.1 Spectroscopic Determination of Elemental Oxides in Fly Ash and Pozzolans

1.1.2 Spectroscopic Determination of Sulfur Trioxide in Fly Ash and Pozzolans

1.1.3 Spectroscopic Determination of Silicon Dioxide in Fly Ash and Pozzolans

1.2 This test method also denotes reference test methods:

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

1.2.1 Loss on Ignition of Fly Ash and Pozzolans – ASTM C311 Sections 13 and 14

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
C150 Standard Specifications for Portland Cement
C311 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E542 Practice for Calibration of Laboratory Volumetric Apparatus
E694 Standard Specification for Laboratory Glass Volumetric Apparatus
STP 985 Rapid Methods for Chemical Analysis of Hydraulic Cement

FHWA
FHWA-RD-72-41 A New Method for Rapid Cement Analysis (Atomic Absorption Spectrophotometry)

MT Materials Manual
MT 108 Sampling and Certification of Portland Cement
MT 607 Procedure for Reducing Field Samples to Testing Size

3 Summary of Test Method

3.1 *Spectroscopic Determination of Elemental Oxides in Fly Ash and Pozzolans Summary of Test Method*
In this test method fly ash or pozzolans are dissolved in a combination of acids via a digestion in Teflon vessels secured in a microwave digestion system. The solution is diluted and analyzed by means of an Inductively Coupled Plasma – Optical Emission Spectrophotometer (ICP-OES). The following analytes are quantified as oxides: calcium, magnesium, iron, aluminum, potassium, and sodium.

3.2 *Spectroscopic Determination of Sulfur Trioxide in Fly Ash and Pozzolan Summary of Test Method*
This test method is substantially based on the publications ASTM STP 985, FHWA-RD-72-41 and the reference test method of ASTM C114. In this method, sulfur is extracted from fly ash or pozzolans using nitric acid and hydrogen peroxide and then quantified via ICP-OES analysis. This procedure is valid for the analysis and reporting of sulfur trioxide.

3.3 *Spectroscopic Determination of Silicon Dioxide in Fly Ash and Pozzolans Summary of Test Method*
This test method is substantially based on the publications ASTM STP 985 and FHWA-RD-72-41. In this method, the fly ash or pozzolan is solubilized by fusion with a mixed lithium metaborate and lithium tetraborate powder. The molten glass bead is dissolved in a weak nitric acid solution and analyzed by ICP-OES.

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance for the fly ash and pozzolan samples submitted by suppliers for inclusion on the MDT Qualified Product List as well as provide analytical information for design applications using fly ash or pozzolans.

5 Apparatus

5.1 *Inductively Coupled Plasma-Optical Emission Spectrophotometer* (ICP-OES)

5.2 *Microwave digestion system* – Capable of heating samples to 200°C and maintaining that temperature for at least 30 minutes.

5.3 *Labware* – Glassware, Teflon, and Plasticware containers that have been properly cleaned and stored filled with dilute nitric acid solution (1 – 5%) for at least 2 days.

5.4 *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.

5.5 *Muffle Furnace* – Capable of maintaining a temperature of 950°C ± 25° verified by clay pyrometric cones yearly.

6 Reagents and Materials

6.1 *Trace metal grade (TMG) Hydrochloric acid (HCl),* concentrated (32-38%)

6.2 *TMG Nitric acid (HNO_3),* concentrated (65-70%)

6.3 *Fluoroboric acid (HBF_4)*, concentrated (46-52%)

6.4 *Hydrogen Peroxide (H_2O_2)*, concentrated (30-38%)

6.5 *Reagent Water* – Purified with a polishing system such that ASTM Type II water or better is obtained.

6.6 *Filter paper* – Particle retention of 20 – 25 µm and a medium flowrate.

6.7 *Ultra-pure grade lithium borate flux* – composed of about 66% Lithium tetraborate ($Li_2B_4O_7$), 33% lithium metaborate ($LiBO_2$) and 1% lithium bromide (LiBr)

7 Sampling

7.1 Fly ash or pozzolan samples are to be collected in accordance with MT 108 and split in accordance with MT 607. A 50 mL sample should be provided to the Chemistry Lab for analysis.

SPECTROSCOPIC DETERMINATION OF ELEMENTAL OXIDES IN FLY ASH AND POZZOLANS

8 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES. Appendix A provides calibrating and standardizing specifications for a Varian Axial ICP-OES for the determination of elemental oxides.

9 Procedure

9.1 Weigh 0.1000 g ± 0.0005 g of fly ash or pozzolan onto tared waxed paper or a small weighing boat. Record the mass.

9.2 Transfer the sample to a Teflon insert for microwave digestion. Reweigh the waxed paper or weighing boat and note the residual mass from the sample. Record the residual mass and calculate the mass transferred to the Teflon insert.

9.3 In a ventilation hood, add 10 mL TMG Hydrochloric acid, 4 mL TMG Nitric acid, and 4 mL Fluoroboric acid to the Teflon insert using autopipets with disposable tips. Place the Teflon insert in the carousel.

9.4 Once all samples have been prepared, place the carousel in the microwave digestion system. Begin the digestion process (see Appendix A for MDT digestion process). After completion of the digestion process, allow the carousel to cool before removing. The carousel may be left overnight to cool.

9.5 Remove a digestion vessel and open it. Rinse the sample into a clean Teflon beaker. Tare a dry 500 mL plastic volumetric flask. Rinse the solution from the beaker into the plastic volumetric flask. Place the plastic volumetric flask on the balance and add reagent water to a mass of 500.00 g ± 0.05 g. Cap the plastic volumetric flask and invert it several times to homogenize the solution.

9.6 Pour some of the solution into a plastic sample bottle for a stock solution. Label the container with the sample number, date, analyst initials, and as fly ash or pozzolan stock solution.

9.7 Dilute the stock solution by dispensing 1.50 g ± 0.03 g of the solution into a 15 mL centrifuge tube and add reagent water until the mass is 15.00 g ± 0.03 g. Label the centrifuge tube with the sample number, date, analyst initials, and as fly ash or pozzolan dilute solution.

9.8 Repeat Sections 9.5 to 9.7 for all samples.

9.9 Analyze the dilute solutions on an ICP-OES.

SPECTROSCOPIC DETERMINATION OF SULFUR TRIOXIDE IN FLY ASH AND POZZOLANS

10 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES. Appendix B provides calibrating and standardizing specifications for a Agilent Radial ICP-OES for the determination of sulfur trioxide.

11 Procedure

11.1 Weigh 0.5000 g ± 0.0005 g fly ash or pozzolan directly into a dry 250 mL beaker.

Note – If a thick walled beaker is used that is too heavy for the balance, the cement can be weighed on waxed paper or a small weighing boat and transferred. Reweigh the waxed paper or weighing to account for any cement that may have stuck to it.

11.2 Add 5.0 mL of hydrogen peroxide using an autopipet to each beaker.

11.3 Bring the solution to the 100 mL mark on the beaker with reagent water.

11.4 Add 10.0 mL of nitric acid to the beaker using an autopipet.

11.5 Add a Teflon stir bar and place on a stir plate to agitate for a minimum of 60 minutes. A good stir rate is about two revolutions per second.

11.6 Quantitatively filter into a 500 mL glass volumetric flask and rinse many times with reagent water.

11.7 Bring to volume with reagent water.

11.8 Transfer the solution to a 500 mL HDPE bottle. Label with the sample number, the date, analyst initials, and analyte of interest.

11.9 Once all the samples are prepared, analyze the solutions using the ICP-OES.

SPECTROSCOPIC DETERMINATION OF SILICON DIOXIDE IN FLY ASH AND POZZOLANS

12 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES. Appendix C provides calibrating and standardizing specifications for a ICP-OES for the determination of silicon dioxide.

13 Procedure

13.1 Weigh 0.8000 g ± 0.005 g of lithium borate flux into a graphite crucible.

13.2 Tare the flux and crucible and add 0.1000 g ± 0.0005 g fly ash or pozzolan on top of the flux.

13.3 Fuse the graphite crucible(s) containing the samples in the muffle furnace at 950°C for 5 minutes. Do not fuse more than two crucibles at a time. Swirl the crucible to consolidate all residual sample that may be sticking to the sides of the crucible. Continue fusing for an additional 15 minutes at 950°C.

13.4 Prior to or during the fusion process, prepare an adequate number of Teflon beakers (at least 200 mL) by rinsing them copiously with reagent water. Add 50 mL of 1 + 24 nitric acid to the beakers. Place a Teflon stir bar in each beaker and cover each with a watch glass. Place the beakers on a stir plate near the furnace and stir at a rate of about two revolutions per second.

13.5 At the completion of the fusion process, quickly transfer the fusion bead from the graphite crucible to one of the prepared Teflon beakers. Inspect the graphite crucible for any trace of sample or fusion material; discard the sample if either are present. Replace the watch glass on the beaker and stir for a minimum of 30 minutes or until all material is dissolved. More acid may be needed to completely dissolve the fusion bead; add the same amount of acid to each sample in order to maintain the same acid content. If additional acid results in material coming out of solution, the sample is not suitable for analysis; discard the sample.

13.6 Once all the material has dissolved, prepare the stock solution by transferring the solution to a 500 mL class A volumetric flask. Bring to volume with reagent water and add nitric acid such that the concentration of nitric acid will be 15 + 485. Cover the flask with Parafilm and invert a minimum of eight times to thoroughly mix.

13.7 Transfer the stock solution to a plastic sample bottle; discard any excess solution. Label the container with the sample number, fly ash or pozzolan, Si analysis, stock solution, date, and analyst initials.

13.8 Once all the samples are prepared, analyze the stock solutions on an ICP-OES.

14 Report

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

Analyte	Report 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
K	K_2O	X.XXX
Na	Na_2O	0.XXX
S	SO_3	X.XX
LOI	LOI	X.XX
Moisture	Moisture	X.XX

15 Validation

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

APPENDIX A
SPECTROSCOPIC DETERMINATION OF ELEMENTAL OXIDES IN FLY ASH OR POZZOLANS

Varian Axial ICP-OES configuration parameters

Equipment Configuration
Nebulizer: Sea Spray
Spray chamber: cyclonic
Sample pump tubing: White/White
Waste pump tubing: Blue/Blue
Rinse solution: 3% Nitric acid
Torch: High solids with sheath gas
Sheath gas: Argon at 10mL/min
Power: 1.20 kW
Plasma flow: 15.0 L/min
Auxiliary flow: 1.50 L/min
Nebulizer flow: 0.75 L/min
Replicate read time: 6.00s
Instrument stabilization delay: 15s
Sample uptake delay: 30s
Pump rate: 15 rpm
Rinse time: 120s
Fast pump: yes
Replicates: 5

Line Selection for Standards, Samples, and Blanks: Select the best line(s) and average for each element.
Al: 396.152, 394.401, 309.271, and 257.509.
Ca: 393.366, 370.602, 318.127, 317.933, 315.887, 219.779, and 210.324.
Fe: 273.358, 261.187, 260.709, 259.940, and 238.204.
Mg: 285.213, 280.270, 279.553, and 202.582.
K: 766.491 and 769.897.
Na: 589.592 and 588.995.

Calibration Varian Axial ICP-OES
Type: Linear for all elements.
Linear-Maximum percent error of 10% and Confidence limit of 0.99%.
Calibration Standard Selection: Pick NIST or CCRL pozzolan standards that provide a range covering what would likely be expected of the samples being analyzed. Usually a minimum of four should be selected. If any samples fall outside the range of the selected standards more should be selected to expand the range.

Milestone Ethos EZ microwave digestion system

Digestion program used: portcement.mpr

Heat and time: Heat to 200°C and hold temperature during a 30 minute interval. Followed by a 10 minute cooling period

Rotor: SK-10

APPENDIX B
SPECTROSCOPIC DETERMINATION OF SULFUR TRIOXIDE IN FLY ASH OR POZZOLANS

Agilent Radial ICP-OES configuration parameters

Equipment Configuration
Nebulizer: OneNeb
Spray chamber: cyclonic
Sample pump tubing: White/White
Waste pump tubing: Blue/Blue
Rinse solution: 3% Nitric acid
Torch: High solids
Power: 1.00 kW
Plasma flow: 15.00 L/min
Auxiliary flow: 1.50 L/min
Nebulizer flow: 0.75 L/min
Viewing height: 10 mm
Replicate read time: 10.00 s
Instrument stabilization delay: 15s
Sample uptake delay: 30s
Pump rate: 15rpm
Rinse time: 10s
Fast pump: yes
Replicates: 3

Line Selection for Standards, Samples, and Blanks: Select the best line(s) and average for each element.
S : 180.669, 181.972

Calibration Agilent Radial ICP-OES
Type: Linear for all elements.
Linear-Maximum percent error of 10% and Confidence limit of 0.99%.
Calibration Standard Selection: Pick NIST or CCRL pozzolan standards that provide a range covering what would likely be expected of the samples being analyzed. Usually a minimum of four should be selected. If any samples fall outside the range of the selected standards more should be selected to expand the range.

APPENDIX C
SPECTROSCOPIC DETERMINATION OF SILICON DIOXIDE IN FLY ASH OR POZZOLANS

Agilent Radial ICP-OES configuration parameters

Equipment Configuration
Nebulizer: OneNeb
Spray chamber: cyclonic
Sample pump tubing: White/White
Waste pump tubing: Blue/Blue
Rinse solution: 3% Nitric acid
Torch: High solids
Power: 1.10 kW
Plasma flow: 15.00 L/min
Auxiliary flow: 1.50 L/min
Nebulizer flow: 0.75 L/min
Viewing height: 10 mm
Replicate read time: 10.00 s
Instrument stabilization delay: 15s
Sample uptake delay: 45s
Pump rate: 15rpm
Rinse time: 10s
Fast pump: yes
Replicates: 3

Line Selection for Standards, Samples, and Blanks: Select the best line(s) and average for each element.
Si : 212.412

Calibration Agilent Radial ICP-OES
Type: Linear for all elements.
Linear-Maximum percent error of 10% and Confidence limit of 0.99%.
Calibration Standard Selection: Pick NIST or CCRL pozzolan standards that provide a range covering what would likely be expected of the samples being analyzed. Usually a minimum of four should be selected. If any samples fall outside the range of the selected standards more should be selected to expand the range.

METHODS OF SAMPLING AND TESTING
MT 534-16
CHEMICAL ANALYSIS OF PORTLAND CEMENT
(Montana Method)

1 Scope

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

1.1.1 Spectroscopic Determination of Elemental Oxides in Portland Cement

1.1.2 Spectroscopic Determination of Sulfur Trioxide in Portland Cement

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 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
C150 Standard Specifications for Portland Cement
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E542 Practice for Calibration of Laboratory Volumetric Apparatus
E694 Standard Specification for Laboratory Glass Volumetric Apparatus
STP 985 Rapid Methods for Chemical Analysis of Hydraulic Cement

FHWA
FHWA-RD-72-41 A New Method for Rapid Cement Analysis (Atomic Absorption Spectrophotometry)

MT Materials Manual
MT 108 Sampling and Certification of Portland Cement
MT 607 Procedure for Reducing Field Samples to Testing Size

3 Summary of Test Method

3.1 *Spectroscopic Determination of Elemental Oxides in Portland Cement Summary of Test Method*
In this test method, Portland cement is dissolved in a combination of acids via a microwave digestion system. The solution is then diluted and analyzed by an Inductively Coupled Plasma – Optical Emission Spectrophotometer (ICP-OES). The following analytes are quantified as oxides: calcium, magnesium, silicon, iron, aluminum, potassium, titanium, sodium, manganese, zinc, chromium, and phosphorus.

3.2 *Spectroscopic Determination of Sulfur Trioxide in Portland Cement Summary of Test Method* This test method is substantially based on the publications STP 985, FHWA-RD-72-41, and ASTM C114. In this method, sulfur is extracted from Portland cement using nitric acid and hydrogen peroxide and then quantified via ICP-OES analysis. This procedure is valid for the analysis and reporting of sulfur trioxide.

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 Product List as well as provide analytical information for design applications using Portland cement.

5 Apparatus

5.1 *Inductively Coupled Plasma-Optical Emission Spectrophotometer* (ICP-OES)

5.2 *Microwave digestion system* – Capable of heating samples to 200°C and maintaining that temperature for at least 30 minutes.

5.3 *Labware* – Glassware, Teflon, and Plasticware containers that have been properly cleaned and stored filled with dilute nitric acid solution (1 – 5%) for at least 2 days.

5.4 *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.

6 Reagents and Materials

6.1 Trace metal grade (TMG) hydrochloric acid (HCl) – Any commercially available brand at a concentration of 32-38%.

6.2 TMG nitric acid (HNO_3) – Any commercially available brand at a concentration of 65-70%.

6.3 Fluoroboric acid (HBF_4) – Any commercially available brand at a concentration of 46-54%.

6.4 Hydrogen Peroxide (H_2O_2) – Any commercially available brand at a concentration of 30-38%.

6.5 Reagent Water – Purified with a polishing system such that ASTM Type II water or better is obtained.

6.6 Filter paper – Particle retention of 20 – 25 µm and a medium flowrate.

7 Sampling

7.1 Cement samples are to be collected in accordance with MT 108 and split in accordance with MT 607. A 50 mL sample should be provided to the Chemistry Lab for analysis.

SPECTROSCOPIC DETERMINATION OF ELEMENTAL OXIDES IN PORTLAND CEMENT

8 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES. Appendix A provides calibrating and standardizing specifications for an Agilent Radial ICP-OES for the determination of elemental oxides.

9 Procedure

9.1 Weigh 0.2000 g ± 0.0005 g Portland cement onto tared waxed paper or small weighing boat. Record the mass.

9.2 Transfer the sample to a Teflon insert for microwave digestion. Reweigh the waxed paper or weighing boat and note the residual mass from the sample. Record the residual mass and calculate the mass transferred to the Teflon insert.

9.3 In a ventilation hood, add 10 mL TMG Hydrochloric acid, 4 mL TMG Nitric acid, and 4 mL Fluoroboric acid to the Teflon insert using autopipets with disposable tips. Place the Teflon insert in the carousel.

9.4 Assemble all components required for proper microwave digestion according to the manufacturer's instructions and then run the digestion. MDT's parameters for a Milestone Ethos EZ microwave digestion system are in Appendix A.

9.5 After completion of the digestion process, allow the carousel to cool before removing. The carousel may be left overnight to cool.

9.6 Remove the digestion vessel and open it. Rinse the sample into a clean Teflon beaker or other suitable cleaned container. Tare an appropriate storage container on the analytical balance. Rinse the sample into the storage container. Place the storage container back onto the analytical balance and dilute with reagent water until a mass of 100.00 g ± 0.05. Record the mass to nearest 0.01 g.

9.9 Label the sample bottle with the sample number, date, analyst initials, and as Portland cement stock solution.

9.10 Using the stock solution, prepare a dilute solution for the analysis of silicon and calcium oxides. In a centrifuge tube, dilute the stock solution at a ratio of 1:5 with reagent water. Label the centrifuge tube with the sample number, date, analyst initials, and as Portland cement dilute solution.

9.11 Once all the samples have been prepared, analyze the dilute and stock solutions on an ICP-OES.

SPECTROSCOPIC DETERMINATION OF SULFUR TRIOXIDE IN PORTLAND CEMENT

10 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the ICP-OES. Appendix B provides calibrating and standardizing specifications for an Agilent Radial ICP-OES for the determination of sulfur trioxide.

11 Procedure

11.1 Weigh 0.5000 g ± 0.0005 g Portland cement directly into a dry 250 mL beaker.

Note – If a thick walled beaker is used that is too heavy for the balance, the cement can be weighed on waxed paper or a small weighing boat and transferred. Reweigh the waxed paper or weighing to account for any cement that may have stuck to it.

11.2 Add 5.0 mL of hydrogen peroxide using an autopipet to the beaker.

11.3 Bring the solution to the 100 mL mark on the beaker with reagent water.

11.4 Add 10.0 mL of nitric acid to the beaker using an autopipet.

11.5 Add a Teflon stir bar and place on a stir plate to agitate for a minimum of 60 minutes. A good stir rate is about two revolutions a second.

11.6 Quantitatively filter the sample into a 500 mL glass volumetric flask rinsing many times with reagent water.

11.7 Bring to volume with reagent water.

11.8 Transfer the solution to a 500 mL HDPE bottle. Label with the sample number, the date, analyst initials, and analyte of interest.

11.9 Once all the samples are prepared, analyze the solutions using the ICP-OES.

12 Calculation or Interpretation of Results

12.1 Portland Cement Potential Phase Composition Calculations

12.1.1 Refer to ASTM C150 Annex A1.

12.2 Portland Cement Limestone Content Calculation

12.2.1 Refer to ASTM C150 Annex A2.

12.3 Equivalent Alkalis Calculation for Portland Cement

12.3.1 Refer to ASTM C150 Section 4 Table 2.

13 Report

13.1 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.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
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
Alkalinity	Alkalinity	X.XX
Cl-	Cl-	0.0XX
F-	F-	0.XX

14 Validation

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

APPENDIX A
SPECTROSCOPIC DETERMINATION OF ELEMENTAL OXIDES IN PORTLAND CEMENT

Milestone Ethos EZ microwave digestion system

Digestion program used: portcement.mpr

Heat and time: Heat to 200°C and hold temperature during a 30 minute interval. Followed by a 10 minute cooling period

Rotor: SK-10

Agilent Radial ICP-OES configuration parameters

Equipment Configuration for Ca and Si

a. Nebulizer: One Neb
b. Spray chamber: cyclonic
c. Sample pump tubing: Gray/Gray
d. Waste pump tubing: Blue/Blue
e. Rinse solution: 5% Nitric acid
f. Torch: High solids
g. Power: 1.20 kW
h. Plasma flow: 15.00 L/min
i. Auxiliary flow: 1.50 L/min
j. Nebulizer flow: 0.60 L/min
k. Viewing height: 10 mm
l. Replicate read time: 1.00 s
m. Instrument stabilization delay: 15 s
n. Sample uptake delay: 30 s
o. Pump rate: 15 rpm
p. Rinse time: 10 s
q. Fast pump: yes
r. Replicates: 8

Equipment Configuration for all other analytes

a. Nebulizer: One Neb
b. Spray chamber: cyclonic
c. Sample pump tubing: White/White
d. Waste pump tubing: Blue/Blue
e. Rinse solution: 5% Nitric acid
f. Torch: High solids
g. Power: 1.15 kW
h. Plasma flow: 15.00 L/min
i. Auxiliary flow: 1.50 L/min
j. Nebulizer flow: 0.75 L/min
k. Viewing height: 8 mm
l. Replicate read time: 1.00 s
m. Instrument stabilization delay: 15 s
n. Sample uptake delay: 45 s
o. Pump rate: 15 rpm
p. Rinse time: 40 s
q. Fast pump: yes
r. Replicates: 5

Line Selection for Standards, Samples, and Blanks: Select the best line(s) and average for each element.

a. Ca : 210.324, 219.779, 315.887, 317.933, 318.127, 370.602, 393.366
b. Al: 309.271
c. Fe: 261.187
d. Mg: 280.270
e. Si: 185.005, 185.185, 250.690, 251.611, 251.920, 252.411, 252.851, 288.158
f. Ti: 334.941
g. Cr: 267.716
h. K: 766.491
i. Mn: 257.610
j. Na: 589.592
k. P: 213.618
l. Zn: 213.857

Calibration Agilent Radial ICP-OES

a. Type: Linear for all elements.
b. Linear-Maximum percent error of 10% and Confidence limit of 0.99%.
c. Calibration Standard Selection: Pick NIST or CCRL Portland cement standards that provide a range covering what would likely be expected of the samples being analyzed. Usually a minimum of four should be selected. If any samples fall outside the range of the selected standards more should be selected to expand the range.

APPENDIX B
SPECTROSCOPIC DETERMINATION OF SULFUR TRIOXIDE IN PORTLAND CEMENT

Agilent Radial ICP-OES configuration parameters

Equipment Configuration

a. Nebulizer: OneNeb
b. Spray chamber: cyclonic
c. Sample pump tubing: White/White
d. Waste pump tubing: Blue/Blue
e. Rinse solution: 5% Nitric acid
f. Torch: High solids
g. Power: 1.45 kW
h. Plasma flow: 15.00 L/min
i. Auxiliary flow: 1.50 L/min
j. Nebulizer flow: 0.55 L/min
k. Viewing height: 6 mm
l. Replicate read time: 3.00 s
m. Instrument stabilization delay: 15 s
n. Sample uptake delay: 30 s
o. Pump rate: 15 rpm
p. Rinse time: 10 s
q. Fast pump: yes
r. Replicates: 3

Line Selection for Standards, Samples, and Blanks: Select the best line(s) and average for each element

a. S : 180.669, 181.972

Calibration Agilent Radial ICP-OES

a. Type: Linear for all elements.
b. Linear-Maximum percent error of 10% and Confidence limit of 0.99%.
c. Calibration Standard Selection: Pick NIST or CCRL Portland cement standards that provide a range covering what would likely be expected of the samples being analyzed. Usually a minimum of four should be selected. If any samples fall outside the range of the selected standards more should be selected to expand the range.

METHODS OF SAMPLING AND TESTING
MT 535-16
BRIDGE DECK CRACK SEALANT IR TEST

THIS PROCEDURE IS IN DEVELOPMENT

METHODS OF SAMPLING AND TESTING
MT 536-16
CHEMICAL ANALYSIS OF SILICA FUME
(Montana Method)

1 Scope

1.1 This method describes the procedures to be used for determining chemical analytes in silica fume as recommended by AASHTO M 307 Standard Specification for Silica Fume Used in Cementitious Mixtures.

1.2 Spectroscopic Determination of Silicon Dioxide in Silica Fume – MT 533 Chemical Analysis of Fly Ash and Pozzolans

1.3 Moisture Content of Silica Fume – ASTM C311 sections 11 and 12

1.4 Loss on Ignition (LOI) of Silica Fume – ASTM C311 sections 13 and 14

1.5 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
C311 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

AASHTO
M 307 Standard Specification for Silica Fume Used in Cementitious Mixtures

MT Materials Manual
MT 108 Sampling and Certification of Portland Cement
MT 533 Chemical Analysis of Fly Ash and Pozzolans
MT 607 Procedure for Reducing Field Samples to Testing Size

3 Significance and Use

3.1 This procedure is primarily used to provide quality assurance for the silica fume submittals for suppliers on the MDT Qualified Product List as well as provide analytical information for design applications using silica fume.

4 Report

4.1 Data Reporting and Retention for Silica Fume will be reported as shown below:

Analyte	Report As	Significance
Si	SiO_2	XX.XX
Moisture	Moisture	X.XX
LOI	LOI	X.XX

5 Validation

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

METHODS OF SAMPLING AND TESTING
MT 537-16
CHEMICAL ANALYSIS OF GROUND GRANULATED BLAST-FURNACE SLAG
(Montana Method)

1 Scope

1.1 This method describes the procedures to be used for determining chemical analytes of ground granulated blast-furnace slag (GGBFS) as recommended by AASHTO M 302 Slag Cement for Use in Concrete Mortars.

1.1.1 This method includes the procedures for the determination of chloride by Ion Chromatography (IC) and the following reference methods.

1.2 This test method also denotes reference test methods:

1.2.1 Spectroscopic Determination of Sulfur Trioxide – MT 533 Chemical Analysis of Fly Ash and Pozzolans

1.2.2 Sulfide Determination – Reference method in ASTM C114 Section 17.2.

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

AASHTO
M 302 Slag Cement for Use in Concrete Mortars

MT Materials Manual
MT 108 Sampling and Certification of Portland Cement
MT 533 Chemical Analysis of Fly Ash and Pozzolans
MT 607 Procedure for Reducing Field Samples to Testing Size

3 Summary of Test Method

3.1 Chloride ions are extracted from GGBFS with nitric acid. The resulting sample solutions are analyzed on an Ion Chromatograph.

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance for the GGBFS submittals by suppliers for inclusion on the MDT Qualified Product List as well as provide analytical information for design applications using GGBFS.

5 Apparatus

5.1 *Ion Chromatograph (IC)*

5.2 *Analytical Balance* – A balance with a precision of 0.0001 g should be used.

6 Reagents and Materials

6.1 *Trace metal grade (TMG) Nitric acid (HNO_3)*, concentrated (65-70%)

6.2 *Sodium Hydroxide (NaOH)*, concentration of 0.7 Molar

6.2 *Reagent Water* – Purified with a polishing system such that ASTM Type II water or better is obtained.

6.3 *Syringe filters* - particle retention of 0.2 µm

6.4 *Litmus paper* - capable of indicating basicity or acidity

7 Sampling

7.1 GGBFS samples are to be collected in accordance with MT 108 and split in accordance with MT 607. A 50 mL sample should be provided to the Chemistry Lab for analysis.

8 Calibration and Standardization

Follow manufacturer's specifications for calibrating and standardizing the IC. Appendix A provides calibrating and standardizing specifications for a Metrohm 883 Basic IC plus for the determination of chloride.

9 Procedure

9.1 Weigh 0.2500 g ± 0.0005 g GGBFS into a 50 mL beaker. Record the mass.

9.2 Gently tap the materials to thinly spread it over the bottom of the beaker.

9.3 Mix 1 mL of reagent water and 200 µL of TMG Nitric Acid.

9.3 Add the acid/water mixture to the sample in a hood and gently break apart clumps using a glass rod.

9.4 Set the mixture on a warm hot plate for five minutes, intermittently grinding any clumps so the slag completely dissolves.

9.5 Remove the sample from the hot plate; let it cool for 10 minutes.

9.6 Add 1 mL of 0.7 M sodium hydroxide to the sample. Check the pH with litmus paper; continue adding sodium hydroxide or nitric acid until the pH is neutral.

9.7 Transfer the sample to a 100 mL volumetric flask and bring to volume with reagent water.

9.8 Using a syringe filter, transfer a sufficient amount of the sample to a vial for the IC.

9.9 Analyze the sample on the IC.

10 Report

10.1 Data Reporting and Retention for GGBFS will be reported as shown below:

Analyte	Report As	Significance
S	SO_3	X.XX
S-	S-	X.XX
Cl-	Cl-	0.0XX

11 Validation

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

APPENDIX A
CHLORIDE DETERMINATION IN GGBFS USING ION CHROMATOGRAPHY

Metrohm 883 Basic IC Plus configuration parameters

Equipment Configuration
Run time: 20 minutes
Injection volume: 20µL
Eluent: 3.2 mM sodium carbonate and 1.0 mM sodium bicarbonate

METHODS OF SAMPLING AND TESTING
MT 538-12
CHEMICAL ANALYSIS OF BLENDED CEMENT

THIS PROCEDURE IS IN DEVELOPMENT

METHODS OF SAMPLING AND TESTING
MT 548-16
METHOD OF TEST FOR ANALYSIS OF TRAFFIC PAINT
(*Montana Method*)

1. Scope

1.1 This method describes the procedures for analyzing physical and chemical properties of traffic paint samples including:

1.1.1 Color and Contrast Ratio – Modified ASTM D2805 and E1347

1.1.2 Viscosity – ASTM D562

1.1.3 Density – Modified ASTM D1475

1.1.4 Freeze-Thaw – primarily Modified ASTM D562 and D2243

1.1.5 Static Heat Stability

1.1.6 Bleeding Ratio – primarily Modified ASTM D868

1.1.7 Skinning and Lumps

1.1.8 Settling

1.1.9 Skinning

1.1.10 FTIR Spectra Verification – Modified ASTM D7588

1.1.11 Cracking

1.1.12 Determination of Heavy Metals

1.2 This standard involves hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2 Referenced Documents

ASTM Standards

D562 Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer
D660 Standard Test Method for Evaluating Degree of Checking of Exterior Paints
D661 Standard Test Method for Evaluating Degree of Cracking of Exterior Paints
D823 Standard Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels
D868 Standard Practice for Determination of Degree of Bleeding of Traffic Paint
D1475 Standard Test Method for Density of Liquid Coatings, Inks, and Related Products
D2243 Standard Test Method for Freeze-Thaw Resistance of Water-Borne Coatings
D2805 Standard Test Method for Hiding Power of Paints by Reflectometry
D7588 Standard Guide for FTIR Fingerprinting of a Non-Aqueous Liquid Paint as Supplied in the Manufacturer's Container
E77 Standard Test Method for Inspection and Verification of Thermometers
E1347 Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry

3 Summary of Test Methods

3.1 The Static Heat test involves storing paint at an elevated temperature for a week then conducting a viscosity test on the paint.

3.2 The Skinning and Lumps test involves storing paint at ambient conditions then straining the paint with a 100 mesh screen.

3.3 The Settling test involves centrifuging paint to produce a separation of the paint components.

3.4 The Skinning test involves partially filling a container with paint and inspecting it after two days to see if the paint has formed a skin.

3.5 The Cracking test involves casting a paint film over asphalt saturated felt and examining the film for cracks after it has dried.

3.6 For the determination of heavy metals, the paint is analyzed for the presence of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), lead (Pb), mercury (Hg), and tin (Sn). Paint samples are prepared through a microwave digestion system using nitric and fluoroboric acids. The samples are then analyzed for mercury with a cold vapor technique using a vapor generation assembly (VGA) outfitted onto a flame atomic absorption spectrometer (FAAS) and for the remainder of the analytes by an inductively coupled plasma optical emission spectrometer (ICP-OES).

4 Significance and Use

4.1 This procedure is primarily used to provide quality assurance for traffic paint used within the Montana Department of Transportation's oversight.

5 Apparatus

5.1 *Analytical Balance* – Capable of measuring to 0.0001 g.

5.2 *Microwave digestion system* – Capable of heating samples to 200°C and maintaining that temperature for at least 10 minutes. The system must use sealable vessels that prevent the escape of vapors.

5.3 *ICP-OES* – Capable of measuring trace elements to low parts per billion (ppb) levels.

5.4 *FAAS* – Outfitted with a hollow cathode lamp (HCL) for measuring Hg and capable of being outfitted with a VGA.

5.5 *VGA* – Capable of cold vapor techniques for detecting mercury at low ppb to high parts per trillion (ppt) levels.

5.6 *Plasticware* – Suitable for trace element analysis. Properly cleaned and stored filled with dilute nitric acid solution (1 – 5%) for at least 2 days.

5.7 *Centrifuge* – Capable of a centrifugal force of 1112 Newtons.

5.8 *Asphalt Felt Paper* – 15 pound saturated asphalt felt paper.

5.9 *Cans* – Pint sized, lined and unlined.

5.10 *Screens* – 100 mesh screen capable of accommodating paint.

5.11 *Leneta 5C form* – an opacity chart containing a white section and a black section paint films can be applied to.

5.12 *Oven* – Capable of maintaining 60°C ± 1°C.

6 Reagents and Materials

6.1 Trace metal grade (TMG) hydrochloric acid (HCl) – Any commercially available brand at a concentration of 32-38%.

6.2 TMG nitric acid (HNO_3) – Any commercially available brand at a concentration of 63-70%.

6.3 Fluoroboric acid (HBF_4) – Any commercially available brand at a concentration of 46-54%.

6.4 Stannous chloride ($SnCl_2$) solution – 10% stannous chloride w/v and 20% TMG hydrochloric acid.

6.5 1+1 hydrochloric acid – Made with TMG HCl (e.g., Add 500 ml TMG HCl to 400 ml reagent water and dilute to 1 L).

6.6 *Reagent Water* – Purified with a polishing system such that ASTM Type II water or better is obtained.

7 Calibration and Standardization

7.1 Follow manufacturer's instructions for calibrating and standardizing the ICP-OES. Appendix A provides parameters for a Varian Axial ICP-OES used by MDT.

7.2 Follow manufacturer's instructions for calibrating and standardizing the FAAS and VGA. Appendix A provides parameters for a Varian FAAS used by MDT.

7.3 Check the calibration of the oven every 6 month with a thermometer verified in accordance with ASTM E77.

PROCEDURES

8 Color and Contrast Ratio

8.1 The paint shall be tested in accordance with ASTM D2805 and ASTM E1347 except for the following stipulations.

8.1.1 Rescind ASTM D2805 Sections 7.1.3 through 7.8 and replace with "15 mil films shall be cast on Leneta 5C opacity charts or equivalent and dried for a minimum of 2 hours." Use a colorimeter to determine coordinates per ASTM E1347. Use Y, x, and y coordinates with illuminant and observer settings of C and 2° when collecting readings. Record Y, x and y coordinates. Use these coordinates to calculate contrast ratio via ASTM D2805.

9 Viscosity

9.1 The paint shall be tested in accordance with ASTM D562.

10 Density

10.1 The paint shall be tested in accordance with ASTM D1475 including the following modification.

10.1.1 Add "Stir the sample until homogeneous." at the beginning of ASTM D1475 Section 9.1.

11 Freeze-Thaw

11.1 The paint shall be tested in accordance with ASTM D2243. Repeat procedures for 3 freeze-thaw cycles (ASTM D2243 Section 7.3). MDT does not require a control sample as described in ASTM D2243 Section 7.1.

11.2 After completion of the freeze-thaw procedures, visually examine the sample and note any signs of livering, hard settling, coagulating, lumps or coarse particles. Then determine the viscosity of the freeze-thaw sample per ASTM D562. Compare the original viscosity to the freeze-thaw viscosity and determine if there was an increase or decrease in viscosity.

12 Static Heat Stability

12.1 Pour paint into a pint container to within 6.4 mm (0.25 inches) of the top. Close the container, seal it with tape, and place the container in an oven maintained at 60°C ± 1°C (140°F ± 2°F) for 7 days.

12.2 After 7 days, remove the container from the oven. Equilibrate the paint at standard conditions and gently stir the paint for a minimum of 5 minutes. Visually examine the sample and note any signs of livering, hard settling, coagulating, lumps or coarse particles. Then determine the viscosity of the static heat sample per ASTM D562. Compare the original viscosity to the static heat viscosity and determine if there was an increase or decrease in viscosity.

13 Bleeding Ratio

13.1 The paint shall be tested in accordance with ASTM D868, except in section 7.4.1.1, replace the CIE L* value with the reflectance coordinate Y.

14 Skinning and Lumps

14.1 Fill a pint container ¾ full and seal it tightly. Allow the container and sample to sit for 72 hours at ambient laboratory conditions then strain the paint through a 100 mesh screen. Note if any lumps or skin is retained on the screen.

15 Settling

15.1 Fill a centrifuge tube with paint and revolve it for 2 hours at a speed producing a centrifugal force of 1112 Newtons (250 pound-force). Note the amount of separation of the components; there shouldn't be more than 13 mm of separation.

16 Skinning

16.1 Fill a lined paint can ¾ full with paint and seal tightly. Invert the can momentarily, and then turn it upright. Place the can in an area where the temperature is 21°C to 32°C for 48 hours. Do not agitate or disturb the sample. After 48 hours, open the can and inspect the paint for the presence or absence of a skin.

17 FTIR Spectra Verification

17.1 The paint shall be tested in accordance with ASTM D7588. Compare the acquired absorbance spectrum to a previously measured spectrum or one provided by the manufacturer. Note any significant differences between the spectra. For multicomponent paints, determine a spectrum for each component separately.

18 Cracking

18.1 The film shall be cast with a 380 micron (15 mil) drawdown blade on 15-pound saturated asphalt felt paper. Allow the film to dry for 24 ours then examine the paint for cracking. Cracking is understood to be any break extending through the paint film to the surface it is applied to. Partial breakthroughs known as checking are not categorized as cracking and are not addressed in this test. Consult ASTM D660 and ASTM D661 to better understand definitions of cracking and checking.

19 Heavy Metals Determination

19.1 Procedure

19.1.1 Weigh 0.50 g of paint or a paint component into a Teflon insert of a high pressure sample rotor system for microwave digestion. Make note of the mass to 0.0001 g.

19.1.2 In a properly functioning ventilation hood add 8 mL TMG nitric acid and 2 mL of fluoroboric acid to the Teflon inserts. Place the Teflon insert in the carousel.

19.1.3 Assemble all components required for proper microwave digestion according to the manufacturer's instructions and then run the digestion. MDT's parameters for a Milestone Ethos EZ microwave digestion system are in Appendix A.

19.1.4 Following the manufacturer's instructions, remove and cool the digestion vessel and open it. Rinse the sample into a clean Teflon beaker or other suitable cleaned container. Tare an appropriate storage container on the analytical balance. Rinse the sample into the storage container. Place the storage container back onto the analytical balance and dilute with reagent water to a mass of 100 g. Record the mass to nearest 0.01 g.

Note – If titanium dioxide is present in the sample, the sample should be filtered with a syringe filter prior to analyzing in order to remove small particles that could block the nebulizer.

19.1.5 Once all the samples have been prepared, analyze them on an ICP-OES and a FAAS outfitted with a VGA.

19.2 Calculation or Interpretation of Results

19.2.1 For elements Sb, As, Cd, Cr, Co, Pb, and Sn measured in mg/kg on the spectrometer, the sample concentration is calculated by multiplying the measured value by the ratio of the solution weight and then dividing by the sample weight.

19.2.2 For Hg measured in µg/kg on the spectrometer, the sample concentration (mg/kg) is calculated by multiplying the measured value by the ratio of the solution weight then dividing by the sample weight then dividing by 1000.

19.3 Report

19.3.1 Elements Sb, As, Cd, Cr, Co, Pb, Hg, and Sn are to be reported in the units mg/kg.

APPENDIX A

Varian Axial ICP-OES configuration parameters

Equipment Configuration

a. Nebulizer: Sea Spray
b. Spray chamber: cyclonic
c. Sample pump tubing: White/White
d. Waste pump tubing: Blue/Blue
e. Rinse Solution: 3% Nitric Acid
f. Torch: High solids with sheath gas
g. Sheath gas: Argon at 10mL/min
h. Power: 1.40 kW
i. Plasma flow: 15.0 L/min
j. Auxiliary flow: 1.50 L/min
k. Nebulizer flow: 0.55 L/min
l. Replicate Read time: 10.00 s
m. Instrument stabilization delay: 15.00s
n. Sample uptake delay: 30.00s
o. Pump rate: 15 rpm
p. Rinse time: 100s
q. Fast pump: yes
r. Replicates: 3

Line Selection for Standard, Samples, and Blanks

a. As 188.980, 193.696
b. Cd 214.439
c. Co 230.786, 231.160
d. Cr 267.716
e. Pb 220.353
f. Sb 217.582, 231.146
g. Sn 189.925, 283.998

Calibration Varian Axial ICP-OES

a. Type: Linear for all elements
b. Linear-Maximum percent error of 5% except Cd that can be 15% and confidence limit of 0.99%
c. Calibration standards: Prepare calibration standards and blanks so that the sample matrices are being mimicked by compensating for the content of acids in the case of analyzing the amine component of two component paints. Prepare a total of one standard blank and three standards with varying concentrations of each desired analyte. For other paint types or components standard additions will need to be made by adding 3 differing spikes to a known portion of sample solution kept at a constant portion of each solution followed by a dilution with no spike.

An example for a stock solution taken to 500 g for standard addition spikes is as follows:

Analyte	Starting Concentration (mg/kg)	Mass Added (g)	Standard Concentration (mg/kg)
As	1000	0.5	1.0
Cd	1000	0.01	0.02
Co	1000	0.5	1.0
Cr	1000	0.1	0.2
Pb	1000	0.5	1.0
Sb	1000	0.5	1.0
Sn	1000	0.5	1.0

40 mL of TMG nitric acid and 10 mL of fluoroboric acid would added to keep the analytes stable.

Varian AA 240FS and VGA configuration parameters

Equipment configuration

a. Measurement time: 10.00s
b. Read delay: 115s
c. Sample tubing: Purple/black
d. Acid and reductant tubing: black/black
e. Replicates standards: 3
f. Replicates samples: 3
g. Precision % standards: 1.0
h. Precision % samples: 1.0
i. Lamp Current: Recommended current on Hg lamp
j. Slit width: 0.5R nm
k. Reslope rate: 50
l. Reslope standard: number 2

Wavelength selection for standards, samples, and blanks

a. 253.7 nm

Calibration Varian AA 240FS and VGA

a. Type: Linear
b. Calibration standards: Prepare calibration standards and blanks so that the acid content of the sample matrices are mimicked. However do not compensate for hydrogen peroxide as it seems to keep the analysis from working properly. Prepare a total of one standard blank and three standards with varying concentrations of each desired analyte

Milestone Ethos EZ microwave digestion system

Heat and time: Heat to 200°C and hold temperature during a 20 minute interval. Followed by a 10 minute cooling period

Microwave power: 1200W (500 W for 3 vessels or less)

Rotor: SK-10