

**CURRENT DATE OF REVISION  
MT 500 SECTION  
CHEMISTRY**

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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
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MT 504	Cyanide In Brine Material.....		Under Development
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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
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<b><u>Test Method No.</u></b>	<b><u>Title</u></b>	<b><u>Pages</u></b>	<b><u>Date of Publication or Revision</u></b>
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MT 532	Determination of Sulfate Content in Soils by Ion Chromatography .....	4 pp	Dec 2016
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		
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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:  $-17.8^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $0^{\circ}\text{F} \pm 2^{\circ}\text{F}$ )
  - Magnesium Chloride:  $-17.8^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $0^{\circ}\text{F} \pm 2^{\circ}\text{F}$ )
  - Calcium Chloride:  $-29^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $20^{\circ}\text{F} \pm 2^{\circ}\text{F}$ )

*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  $\pm 0.1\%$  in Site Manager.
  - 2.2 Report solids passing through a number 10 sieve as a (V/V) percent  $\pm 0.1\%$  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 an 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<sub>3</sub>), concentration of 65-70%
- 6.3 Technical grade hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), concentration of 34-37%
- 6.4 Stannous chloride solution - 10% stannous chloride (SnCl<sub>2</sub>) 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.

<b>Material Being Analyzed</b>	<b>Amount of material (grams)</b>
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.

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

Where:

C = sample concentration (mg/kg)

M = measured value ( $\mu\text{g}/\text{kg}$ )

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 $\mu\text{g}/\text{kg}$ 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  $\mu\text{g}/\text{kg}$

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 <sub>2</sub>		95.21
S	32.066	
SO <sub>4</sub>		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<sub>2</sub>, NaCl, and SO<sub>4</sub> – 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<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub> brines, 0.75 L/min NaCl brines, and 0.70 L/min for inhibitors.
- k. Viewing height: 11 mm MgCl<sub>2</sub> 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<sub>2</sub> 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  $580 \pm 20$  nm and  $561 \pm 20$  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:

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

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 CONCRETE**  
**BY ION CHROMATOGRAPHY**  
**(Montana Method)**

**1 Scope**

- 1.1 This method describes the procedure used to determine the concentration of acid-soluble chloride in bridge deck concrete via ion chromatography.
- 1.2 This method may involve hazardous materials, operations, and equipment. This method 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*****MT Materials Manual***

MT 114 Sampling for Chloride Content of Bridge Deck Concrete

***Other***

Method Tex-620-J, Determining Chloride and Sulfate Contents in Soil

([http://ftp.dot.state.tx.us/pub/txdot-info/cst/TMS/600-J\\_series/archives/620-0805.pdf](http://ftp.dot.state.tx.us/pub/txdot-info/cst/TMS/600-J_series/archives/620-0805.pdf))

**3 Terminology**

- 3.1 *Chromatography* – the separation and quantitative analysis of ions based on their affinity to the ion exchanger.
- 3.2 *Eluent* – A solution that transports the sample through the system and contributes to the selectivity of the separation in the ion chromatograph.

**4 Summary of Test Method**

- 4.1 The chloride ion is extracted from concrete samples, put into solution, centrifuged, filtered and injected into the Ion Chromatograph (IC). The sodium carbonate/bicarbonate eluent transports the sample through the column which is operating with a suppressed conductivity method. The ion chromatograph detects chloride ions which interface with the column and are read on the instrument's conductivity detector.

**5 Significance and Use**

- 5.1 This procedure is used to determine the concentration of the chloride ion in concrete samples collected from bridge decks. The chloride ion concentration is used to evaluate the level of corrosion and determine the maintenance requirement for bridge decks.

**6 Apparatus**

- 6.1 *Metrohm Basic Ion Chromatograph Plus 883* equipped with:
- 6.1.1 Autosampler
- 6.1.2 Metrosep A Supp 150/4.0 Column
- 6.1.3 Conductivity Detector
- 6.1.4 MagIC Net Software
- 6.2 *Centrifuge*

## 7 Reagents and Materials

- 7.1 *Reagent Water* – Purified with a polishing system such that ASTM Type II water or better is obtained.
- 7.2 *Suppressor Regenerant Solution*, 0.1 Molar Sulfuric Acid ( $H_2SO_4$ )
- 7.3 *Suppressor Rinse Solution*, 99.9% MeOH
- 7.4 *Neutralizing Solution*, 0.5 M Sodium Hydroxide (aqueous)  
Add 500mL of reagent water to a 750 or 1000mL glass beaker and slowly add 9.99 g of solid NaOH pellets to the reagent water and mix.
- 7.5 *Eluent*, 3.2 M Sodium Carbonate,  $Na_2CO_3$ /1.0 M Sodium Bicarbonate,  $NaHCO_3$  (aqueous); (stock eluent solution, A Supp 5 Eluent 100x, ordered from Metrohm). Shelf life of eluent is 2 days.
- 7.6 *Nitric Acid ( $HNO_3$ )*, concentrated Trace Metal Grade, 65-75%
- 7.7 *Standard Stock Solution*, Dionex Five Anion Standard, chloride concentration of 30 ppm
- 7.7 *Glassware*: 50 mL beakers, 100mL volumetric flasks for dilution
- 7.8 *Auto-pipettes*
- 7.9 *Plastic Syringes*, 30 mL non-sterile
- 7.10 *Syringe filters* - particle retention of 0.2  $\mu m$
- 7.11 *Dry plastic bottles* that have been stored filled with a dilute  $HNO_3$  acid solution (1 – 5%) prior to use
- 7.12 *Disposable IC vials*, 11 ml

## 8 Sampling

- 8.1 Bridge deck concrete samples are collected in accordance with [MT 114](#) and pulverized in a Bico pulverizer with plates spaced between 1 and 2 mm.

## 9 Calibration and Standardization

- 9.1 *IC Instrumentation Settings*:
- 9.1.1 Flow rate on IC: 0.7 mL/min
- 9.1.2 Sample Loop: 20  $\mu L$
- 9.1.3 Run time: 20 minutes
- 9.2 *Calibration Curve Determination*
- 9.2.1 Prepare standards from a five anion standard. The Dionex Five Anion Standard with a chloride concentration of 30 ppm is commonly used.
- 9.2.2 Weigh indicated masses from the 5 anion standard bottle into a dry plastic bottle (See Table 1). Record weights to the thousandth decimal place.
- 9.2.3 Dilute with reagent water for a target total mass of 50 g ( $\pm 0.001$ )

Table 1: Standard calibration solutions and masses of components

Concentration, ppm	Mass of 5 anion standard, g	Mass of solution, g
0.5	0.834	50.000
1.0	1.667	50.000
2.0	3.334	50.000
4.0	6.667	50.000
5.0	8.334	50.000
10.0	16.668	50.000

Note 1 - The masses in Table 1 above have been developed for the Dionex Five Anion Standard which has a chloride concentration of 30 ppm, if another standard is used the weights in the table are not applicable

9.2.4 Determine concentration of each standard and enter into the IC software.

## 10 Procedure

### 10.1 Preparation of Controls

10.1.1 For each sequence to be analyzed on the IC, prepare and analyze a quality control (QC) sample. Use a QC sample with a concentration which is mid-range of the calibration curve. For example, chloride standard preparations range from 0.5 ppm to 10 ppm. A QC sample with a target of approximately 3 ppm is sufficient. Using a Portland cement with a qualified standard of 0.127% is an acceptable QC sample. Determine percent recovery per Section 11.5 below. Analysis is considered acceptable with a percent recovery on the quality control sample of  $\pm 10\%$ .

### 10.2 Sample Preparation and Analysis

10.2.1 Weigh 0.25 g of sample into a 50 – 60 mL beaker using a balance with an accuracy of  $\pm 0.0001$  g. Record the sample weight to be used in final calculation ( $W_s$ ). Add 1 mL of reagent water to the beaker to liquefy the cement sample.

10.2.2 In a fume hood, Add 100  $\mu$ L of concentrated  $\text{HNO}_3$  to the cement paste and swirl gently to mix well.  $\text{CO}_2$  gas liberates from the reaction between the cement constituents and nitric acid. Wait roughly 10 minutes for the extraction to occur.

10.2.3 Neutralize the solution by adding approximately 1.5 mL of 0.5 M NaOH (2-4 mL may be needed). Test with litmus paper strips to make sure the solution is neutral.

10.2.4 Transfer the neutral solution into a 100 mL volumetric flask (V), rinsing the beaker to transfer the solids from the beaker to the flask. Dilute to the fill line with reagent water.

10.2.5 Pour approximately 10 mL of the solution into a plastic syringe equipped with a 0.2  $\mu$ m nylon filter tip. Filter the solution into a plastic ion chromatography test tube.

Note 2 – If the turbidity of the sample is such that light cannot pass through it, then centrifuge the sample at 500 rpm for roughly 15 minutes in a plastic nunc tube prior to pouring the sample into the IC vial centrifuge.

10.6 Analyze the sample and the QC sample simultaneously on the IC to determine the chloride concentration. Shelf life of the sample is one week.

## 11 Calculation or Interpretation of Results

### 11.1 Dilution Factor

$$DF = \frac{V_f}{V_i}$$

Where:

$DF$  = Dilution factor

$V_f$  = Final volume (diluent and aliquot)

$V_i$  = initial volume

#### 11.1.1 Dilution Factor for Concrete Sample

The dilution factor ( $DF$ ) for the concrete sample is the volume of the volumetric flask used in Section 10.2.4 (i.e., 100 mL).

### 11.2 Chloride Concentration in Concrete

$$C_{cl} = \frac{(C_{ic} * DF)}{W_s}$$

Where:

$C_{cl}$  = Concentration of the chloride in the original concrete, ppm

$C_{ic}$  = Concentration of the chloride as determined by the IC, ppm

$DF$  = Dilution factor

$W_s$  = Weight of the sample, g

11.2.1 Example:  $C_{ic} = 2.78 \text{ mg/L (or ppm)}$   
 $DF = 100$   
 $W_s = 0.2529 \text{ g}$

$$C_{cl} = (2.78 * 100) / 0.2529 = 1099 \text{ ppm}$$

### 11.3 Percent Chloride in Concrete

$$\%Cl = \frac{C_{cl}}{10,000}$$

11.3.1 Example:  $C_{cl} = 1099 \text{ ppm (from above)}$

$$\%Cl = 1099 / 10,000 = 0.1099\%$$

### 11.4 Pounds of Chloride per cubic yard of Concrete

$$P_c = \frac{(C_{cl} * D_c)}{1,000,000}$$

Where:

$P_c$  = Pounds of chloride per cubic yard of concrete

$C_{cl}$  = Concentration of chloride ions in the original concrete sample, ppm

$D_c$  = Density of concrete (4,000 lb/cy)

11.5 *Percent Recovery of Quality Control Sample*

$$\% \text{ Recovery} = [C_{IC(QC)} * DF / W_{QC} / 10,000 / C_{QC}] * 100\%$$

Where:

$C_{IC(QC)}$  = Concentration of the Quality Control Sample as determined by the IC, ppm

$C_{QC}$  = Known value of the quality control sample

DF = Dilution factor

$W_{QC}$  = Weight of the Quality Control Sample, g

11.5.1 *Example:*

$$C_{IC(QC)} = 3.15 \text{ ppm}$$
$$DF = 100$$
$$W_{QC} = 0.2529 \text{ g}$$
$$C_{QC} = 0.127\%$$

$$\% \text{ Recovery} = [3.15 * 100 / 0.2529 / 10,000 / 0.127] * 100 = 98.07\%$$

**12 Report**

- 12.1 Enter chloride analysis results in the following location:  
<X:\CHEMISTRYLAB\Bridge Chlorides 20XX.xlsx>



**METHODS OF SAMPLING AND TESTING**  
**MT 532-16**  
**DETERMINATION OF SULFATE CONTENT IN SOILS BY ION CHROMATOGRAPHY**

**1 Scope**

- 1.1 This method describes the procedures used to determine water-soluble sulfate in soils through the use of the ion chromatograph (IC).
- 1.2 This method was developed as an alternative to the sulfate analysis in [MT 232](#) and was validated June 2016.
- 1.3 This method may involve hazardous materials, operations, and equipment. This method 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**

C1580 Standard Test Method for Water-Soluble Sulfate in Soil

**MT Materials Manual**

MT 232 Soil Corrosion Test

**Other**

TX DOT, Tex-620-J, Determining Chloride and Sulfate Contents in Soil  
([http://ftp.dot.state.tx.us/pub/txdot-info/cst/TMS/600-J\\_series/archives/620-0805.pdf](http://ftp.dot.state.tx.us/pub/txdot-info/cst/TMS/600-J_series/archives/620-0805.pdf))  
Validation of Sulfate Method on the Ion Chromatograph, MDT, June 2016.

**3 Summary of Test Method**

- 3.1 Soils are dissolved in water and analyzed on the ion chromatograph. This method is substantially based on ASTM C1580 and the Texas Department of Transportation Method Tex-620-J. The method of analysis can be used for both solid soil and water samples. The samples are dissolved or diluted in water, filtered and injected into the IC instrument. A calibration curve is prepared on the IC using a five anion standard. Sulfate results are obtained in ppm as determined by the IC.

**4 Significance and Use**

- 4.1 This procedure is used to determine sulfate content in soil samples for pipe corrosion evaluation and soil survey samples.

**5 Apparatus**

- 5.1 *Metrohm Basic Ion Chromatograph Plus 883* equipped with:
- 5.1.1 Autosampler
- 5.1.2 Metrosep A Supp 150/4.0 Column
- 5.1.3 Conductivity Detector
- 5.1.4 MagIC Net Software

**6 Reagents and Materials**

- 6.1 *Reagent Water* – Reverse Osmosis (RO) water purified with a polishing system such that ASTM Type II water or better is obtained.
- 6.2 *Suppressor Regenerant Solution*, 0.1M Sulfuric Acid, H<sub>2</sub>SO<sub>4</sub> (aqueous)

- 6.3 *Suppressor Rinse Solution*, 99.9% MeOH
- 6.4 *Eluent*, 3.2 M Sodium Carbonate, Na<sub>2</sub>CO<sub>3</sub>/1.0 M Sodium Bicarbonate, NaHCO<sub>3</sub> (aqueous); (stock eluent solution, A Supp 5 Eluent 100x, ordered from Metrohm and kept in-house).
- 6.5 *Standard Stock Solution*, Dionex Five Anion Standard, Sulfate concentration of 150 mg/L
- 6.6 *Glassware*: 250 mL beakers and large glass centrifuge tubes
- 6.7 *Class A pipettes*, for secondary dilution
- 6.8 *Plastic Syringes*, 30 mL non-sterile
- 6.9 *0.20 μm nylon filters*, non-sterile

## 7 Sampling

- 7.1 Samples are prepared in accordance with [MT 232](#). A 3 gram (g) sample is required for analysis.

## 8 Calibration and Standardization

### 8.1 IC Instrumentation Settings

- 8.1.1 Flow rate on IC: 0.7 mL/min
- 8.1.2 Sample Loop: 20 μL
- 8.1.3 Run time: 20 minutes

### 8.2 Calibration Curve Determination

- 8.2.1 Prepare six standards based on the indicated concentrations and masses in Table 1 using the Dionex Five Anion Standard. The values for the sulfate calibration curve range from approximately 2.5 ppm and 50 ppm.
- 8.2.2 Weigh the standard into a plastic bottle. Record weights to the thousandth decimal place.
- 8.2.3 Dilute the standard to a target total mass of 50 (±0.001g) with reagent water. Record the final weight.

*Table 1: Standard calibration solutions and masses of components*

Concentration, ppm	Mass of 5 anion standard, g	Mass of solution, g
2.5	0.834	50.000
5	1.667	50.000
10	3.334	50.000
20	6.667	50.000
25	8.334	50.000
50	16.668	50.000

*Note 1 - The masses in Table 1 above have been developed for the Dionex Five Anion Standard which has a sulfate concentration of 150 ppm, if another standard is used the weights in the table are not applicable*

- 8.2.4 Inject the six prepared standards and utilize the instrumentation on the IC to develop a calibration curve.

## 9 Procedure

### 9.1 Preparation of Controls

For each sequence to be analyzed on the IC, prepare a Quality Control (QC) sample in the target range of approximately 20 ppm. Use a qualified soil standard with a known concentration of 1630 ppm ( $\pm 43.7$  ppm) to prepare the control. Determine percent recovery per Section 10.4 below. Analysis is considered acceptable with a percent recovery on the control sample of  $\pm 10\%$ .

### 9.2 Sample Preparation and Analysis

9.2.1 Weigh 3 grams of sample from the remaining material used for the conductivity preparation in [MT 232](#) into a 250 ml glass beaker. Record the weight to the thousandth decimal place ( $W_s$ ).

9.2.2 Add 150 mL of reagent water and mix with a stir bar for an hour.

9.2.3 Pour approximately 20 mL of the liquid extract into a syringe with attached 0.20  $\mu\text{m}$  nylon filter. Filter this extract directly into plastic ion chromatography test tube and cap.

9.2.4 Analyze the sample and the QC sample simultaneously on the IC to determine the sulfate concentrations.

*Note 2 – A secondary dilution may be necessary if concentration falls outside of the range determined by the calibration curve.*

## 10 Calculation or Interpretation of Results

### 10.1 Dilution Factor

$$DF = \frac{V_f}{V_i}$$

Where:

$DF$  = Dilution factor

$V_f$  = Final volume (diluent and aliquot)

$V_i$  = initial volume

#### 10.1.1 Dilution Factor for Secondary Dilution

$$DF = \frac{V_d}{V_p}$$

Where:

$DF$  = Dilution factor

$V_d$  = Volume of the flask used for the dilution, mL

$V_p$  = Volume of the pipette used to make the dilution, mL

## 10.2 Concentration of sulfate ion in the original soil sample

$$C_{SO_4} = (C_{IC} * DF) / W_s$$

Where:

$C_{SO_4}$  = Concentration of the sulfate in the original soil sample, ppm

$C_{IC}$  = concentration of sulfate ion in the sample run through the IC, ppm

DF = Dilution factor

$W_s$  = Weight of the sample, g

10.2.1 Example:  $C_{IC} = 2.948$  mg/L (or ppm)  
 $DF = 150$   
 $W_s = 3.0041$  g

$$C_{SO_4} = (2.948 * 150) / 3.0041 = 147.2 \text{ ppm}$$

## 10.3 Percent Sulfate in Soil

$$\% SO_4 = C_{SO_4} / 10,000$$

## 10.4 Percent Recovery Quality Control Sample

$$\% \text{ Recovery} = [C_{IC(QC)} * DF / W_{QC} / 10,000 / C_{QC}] * 100\%$$

Where:

$C_{IC(QC)}$  = Concentration of the soil standard sample as determined by the IC, ppm

$C_{QC}$  = Known value of the soil standard (soil standard typically is 1630 ppm  $SO_4$ ), ppm

DF = Dilution factor

$W_{QC}$  = Weight of the quality control sample, g

10.4.1 Example:  $C_{IC(QC)} = 31.96$  ppm  
 $DF = 50$   
 $W_{QC} = 1.004$  g  
 $C_{QC} = 1630$  ppm

$$\% \text{ Recovery} = [31.96 * 50 / 1.004 / 1630] * 100 = 98.07\%$$

## 11 Report

The concentration of the sulfate will be recorded in the soil corrosion spreadsheets and the Sitemanager data base as percent sulfate.

**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<sub>3</sub>)*, concentrated (65-70%)

6.3 *Fluoroboric acid (HBF<sub>4</sub>)*, concentrated (46-52%)

6.4 *Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>)*, 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<sub>2</sub>B<sub>4</sub>O<sub>7</sub>), 33% lithium metaborate (LiBO<sub>2</sub>) 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.

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**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  $0.1000 \text{ g} \pm 0.0005 \text{ g}$  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  $500.00 \text{ g} \pm 0.05 \text{ g}$ . 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  $1.50 \text{ g} \pm 0.03 \text{ g}$  of the solution into a 15 mL centrifuge tube and add reagent water until the mass is  $15.00 \text{ g} \pm 0.03 \text{ g}$ . 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.

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**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  $0.5000 \text{ g} \pm 0.0005 \text{ g}$  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.

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**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  $0.8000 \text{ g} \pm 0.005 \text{ g}$  of lithium borate flux into a graphite crucible.
- 13.2 Tare the flux and crucible and add  $0.1000 \text{ g} \pm 0.0005 \text{ g}$  fly ash or pozzolan on top of the flux.
- 13.3 Fuse the graphite crucible(s) containing the samples in the muffle furnace at  $950^{\circ}\text{C}$  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  $950^{\circ}\text{C}$ .
- 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 <sub>2</sub> O <sub>3</sub>	X.XX
Fe	Fe <sub>2</sub> O <sub>3</sub>	X.XX
Mg	MgO	X.XX
Si	SiO <sub>2</sub>	XX.XX
K	K <sub>2</sub> O	X.XXX
Na	Na <sub>2</sub> O	0.XXX
S	SO <sub>3</sub>	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<sub>3</sub>) – Any commercially available brand at a concentration of 65-70%.
- 6.3 Fluoroboric acid (HBF<sub>4</sub>) – Any commercially available brand at a concentration of 46-54%.
- 6.4 Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) – 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.

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**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  $0.2000 \text{ g} \pm 0.0005 \text{ g}$  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  $100.00 \text{ g} \pm 0.05$ . 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.

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**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  $\pm$  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.

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**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 <sub>2</sub> O <sub>3</sub>	X.XX
Fe	Fe <sub>2</sub> O <sub>3</sub>	X.XX
Mg	MgO	X.XX
Si	SiO <sub>2</sub>	XX.XX
Ti	TiO <sub>2</sub>	0.XX
Cr	Cr <sub>2</sub> O <sub>3</sub>	0.0XX
K	K <sub>2</sub> O	X.XXX
Mn	Mn <sub>2</sub> O <sub>3</sub>	0.XXX
Na	Na <sub>2</sub> O	0.XXX
P	P <sub>2</sub> O <sub>5</sub>	0.XXX
Zn	ZnO	0.XXX
S	SO <sub>3</sub>	X.XX
S-	S-	X.XX
LOI	LOI	X.XX
ISR	ISR	0.XX
CO <sub>2</sub>	CO <sub>2</sub>	X.XX
Lime Content	Lime Content	X.X
C <sub>3</sub> S	C <sub>3</sub> S	XX.X
C <sub>2</sub> S	C <sub>2</sub> S	XX.X
C <sub>3</sub> A	C <sub>3</sub> A	X.X
C <sub>4</sub> AF	C <sub>4</sub> AF	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 <sub>2</sub>	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<sub>3</sub>)*, 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 <sub>3</sub>	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: 20 $\mu$ L

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  $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$ .

## 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 ( $\text{HNO}_3$ ) – Any commercially available brand at a concentration of 63-70%.
- 6.3 Fluoroboric acid ( $\text{HBF}_4$ ) – Any commercially available brand at a concentration of 46-54%.
- 6.4 Stannous chloride ( $\text{SnCl}_2$ ) 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  $2^{\circ}$  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  $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$  ( $140^{\circ}\text{F} \pm 2^{\circ}\text{F}$ ) 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  $\frac{3}{4}$  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  $\frac{3}{4}$  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  $21^{\circ}\text{C}$  to  $32^{\circ}\text{C}$  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 hours 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  $\mu\text{g}/\text{kg}$  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