1.0 Purpose:

The purpose of this procedure is to provide a method for testing the filter press cake in order to determine efficiency and hint at any deviations to the normal process.

2.0 Scope:

This procedure is to be performed at intervals specified by management and when a deviation to the normal process occurs.

3.0 Responsibility:

QA personnel or representative will request a sample from the material handler and will provide the material handler with a sample bag that has a blank label with spots for, at minimum, the date and the MgO railcar #.

This procedure is to be performed by any qualified laboratory personnel; a second analyst will review data for accuracy and completeness.

4.0 Safety Considerations:

Proper PPE should be worn at all times during this procedure. Including but not limited to gloves, safety goggles, and lab coat.

Safety is a condition of employment. Employees are not authorized to work in an unsafe manner and are prohibited from harming the environment of the facility or community.

5.0 Materials/Equipment:

Equipment:
- Large crucibles
- Drying oven (100 ˚ C)
- Forceps
- Desiccator
- Vacuum filter assembly including Buchner funnel, stopper, tubing and filter pump
-Whatman grade 40 (8 µm) filter paper – OR – other low retention filter paper
- Balance (0.0000g accuracy min.)
- Weigh Paper
- Spatula
- pH Meter
- 1000-ml Volumetric Flask

- 100-mL Volumetric Flask
- 100-mL Graduated Cylinder
- 250-mL Filter Flasks
- Watch Glass
- Stir bar
- Stir plate
- Class A – 100 ml burette
- Burette Stand
- 1000-µL Eppendorf Pipette and Tips
- 5-mL Eppendorf Pipette and Tips

Reagents:
- Ammonium Chloride
- DI H_20
- Eriochrome Black TS
- 0.05 M Edetate Disodium (Disodium EDTA) Volumetric Solution
- 3 N Hydrochloric Acid Solution (If Needed)
- 1 N Sodium Hydroxide Solution (If Needed)
- Lithium Metaborate
- Lithium Bromine
- Nitric Acid (conc.)

6.0 Procedure:

Solutions Preparation: *(If Needed)*

Ammonium – Ammonium Chloride Buffer Test Solution– Dissolve 67.5 g of ammonium chloride in water, add 570 ml of ammonium hydroxide, dilute with DI H2O to 1000 ml.

2% Nitric Acid (HNO_3) – Add 57.1mL of conc HNO_3 to a 2000mL volumetric flask that is half filled with DiH_2O. Dilute to volume with DiH_2O and mix well by inversion.

10% Nitric Acid (HNO_3) – Add 143mL of conc HNO_3 to a 1000mL volumetric flask that is half filled with DiH_2O. Dilute to volume with DiH_2O and mix well by inversion.

Sample Prep/Analysis:

LOD:

1. From the press cake sample received, cut a lab sample perpendicular to the cake surface weighing about 30 grams and proceed to analyze each individually as directed below.

2. Weigh a 100 mL crucible. **Record the weight (A).**
3. Place the lab sample in the crucible, weigh, and **record the weight (B)**

B − A = the weight of the sample

4 Place the crucible and sample in the 100°C oven overnight.
5. Next day, using the forceps, remove the crucible from the oven and place in the desiccator.
6. When cool, weigh the crucible containing the sample, and **record the weight (D).**

D − A ÷ (B-A) ·100% = % Total Solids in the Filter Press Cake
B − D ÷ (B-A) ·100% = % LOD of the Filter Press Cake

$MgSO_4$ Assay:

1. Grind the entire dry portion and weigh out a 0.50 g portion and **record weight (C).** Then place the portion into a suitable beaker.
2. Add exactly 50mL of DI water to the beaker, add a stir bar, and stir until dissolved.
3. Using a Whatman grade 40 (8 µm) filter paper, filter the sample through a vacuum and rinse the beaker, stir bar, and filtrate with another 50mL portion of DI water.
5. Titrate the filtrate using the following procedure *USP Assay: Magnesium Sulfate (L12-PR-100-008) (steps 7 to 14).*
6. Use the formula below to calculate % $MgSO_4$ in the Filter Press Cake:

$$\frac{\text{mL of EDTA solution} \cdot 0.006018}{C} \cdot 100\% = \%\ MgSO_4 \text{ in the Filter Press Cake (D)}$$

Quantification of Unreacted Matter:

1. Accurately weigh ~50mg of the dry grinds out onto a sheet of weigh paper.
2. Using the KATANA fluxer, flux the 50mg with 1.0g of **Lithium Metaborate**, and 250mg of **Lithium Bromine** into 100mL of 10% HNO_3.
   (Note: Ensure the sample is entirely in solution before continuing.)
3. Prepare a series of ICP standards in the following concentrations:

Standard ID	Ca Conc.	Fe Conc.	Mg Conc.
STD 1	0	0 ppm	0 ppm
STD 2	2 ppm	1 ppm	5 ppm
STD 3	4 ppm	2 ppm	12.5 ppm
STD 4	6 ppm	3 ppm	20 ppm

4. Using the method labeled Mud Analysis in the SALSA ICP software, initiate the ICP as directed in *USP ICP-OES Analysis (L13-PR-100-057)* .
5. Place standards in positions 8-11. Align the torch using the *Auto Align* function and STD 4. Repeat the *Auto Align* function until the window shows a good saturation and a *dx/dy* of 0/0.
6. Prepare the sample by diluting the fluxed solution by 1:10. *(Example: Pipette 10mL into a 100mL volumetric flask and q.s. with 2% HNO_3)*
7. Under the **Method→QC Automation** tab ensure that standards 1-4 and QC1 are selected. Continue to the **Analysis** tab and create a new analysis.
8. Continue to the **Sequence** pane and start the sequence.
9. After the standards have run check the calibration curve under **Methods→Elemental Selection→Calibration.** *(Note: The line for each element should be relatively linear and should have a Rho value near 1.0)*
10. If the calibration curve and the QC pass then proceed otherwise use the **Auto Align** function again and re-establish a new calibration curve. *(Note: To determine if the QC passes go to the **Analysis→Results** tab. If beside the concentrations of the elements there is an **(L)** or **(H)** then the QC failed and must be re-run, if there is no such character then continue.)*
11. Go back to the **Method→QC Automation** tab and uncheck the standards and the first QC. Continue to the **Sequence** pane and check the number of boxes = to the number of samples to be analyzed. Give each sample a name that includes the date the sample was taken. In the weight cell enter 0.050 and in the volume cell enter 1000. *(Note: The values entered are the values from the sample prep i.e.- {[50mg/100mL] x [10mL/100mL]} if the sample was prepped differently then the numbers will change.)*
12. Click the **Update Sequence** button and then **Run Sequence**. There should be a QC standard run every 5 samples and at the end of the run.
13. Once the run is complete go to the **Analysis** pane again and go to the **Repots** tab. Select all of the injections from the first QC down. Then click the **Load Report** button. Select **Simple SBD** report and the **Format 2** button. Click **Print** and the **Printer** button. Ensure the printer has paper loaded.
14. The report will show values in the concentration cell. These values are reported in ppm so to convert to % the value will need to be divided by 10,000.
15. To get % Mg^{+2} the Mg associated with the $MgSO_4$ must be subtracted using the calculation:

$$\frac{\boldsymbol{Mg\ (ppm)}}{\boldsymbol{10000}} - \boldsymbol{(D * 0.2020)} = \%\ \boldsymbol{Mg^{+2}}$$

7.0 Reference Documents:

USP Assay: Magnesium Sulfate (L12-PR-100-008)
USP ICP-OES Analysis (L13-PR-100-057)
Analysis of Filter Cake (L15-PR-100-F064)

8.0 Change Information:

Update of entire document