Ash Analysis of Petroleum Products (FCC Feed) Sulfated Ash

AW113.0

(This procedure covers the determination of ash, sulfated ash and preparation of petroleum products for AAS determination of: Fe, Ni, V, Cu, Na, & K.)

**Method Parameters**

Dwell Time: 5-90 minutes

Temperature: 100-775°C

**Recommended Systems**

PHOENIX AirWave

Sample Amount: up to 30 grams

**Equipment**

PHOENIX AirWave,100 mL to 150 mL porcelain, fused silica, or platinum crucible, tongs, gloves, desiccator, balance capable of weighing to + 0.1 mg. H2SO4

**Procedure**

1. In the Program Mode program the following:

 **Stage** **Temp°C**  **Ramp(min)** **Dwell(min) Hold**

 1 100 00:10 00:10 n **(See Note 4)**

 2 275 00:15 00:30 y **(See Note 3)**

 3 300 00.00 00:05 n

 4 425 01:20 00:10 n

 5 775 00:20 01:30 n

1. Weigh up to 30 grams **(Caution: See Note 1)** of sample into the pre-ashed crucibles.
2. With the furnace temperature at 100oC or less **(Caution: See Note 2)**, place the crucibles into the furnace chamber, replace the Ashing Furnace Insert Door allowing no gaps, close the microwave cavity door, press the **Start/Stop** button.
3. When the Stage 2 Temperature has been reached and the **“HOLD”** stops flashing and is continuously displayed, remove the crucibles, allow to cool. **(Caution: See Note 3).** For procedures not requiring a sulfated ash, eliminate the **“HOLD”** in Stage.
4. Replace the Ashing Furnace Insert Door, close the microwave cavity door, Press the **Start/Stop** button to allow the furnace to maintain the stage set point temperature.
5. After the samples have cooled sufficiently, 10 to 15 minutes, wet the ash with a sufficient amount of H2SO4. To sulfate the ash, place the crucibles into the furnace chamber, replace the Ashing Furnace Insert Door allowing no gaps, close the microwave cavity door, press the **Start/Stop** button to continue the program.
6. At the end of the Stage 5 Dwell Time, remove the crucibles, allow to cool in a desiccator before recording the finial weight and calculate % sulfated ash or preparation for further analysis.
7. Allow the furnace chamber to cool to the starting temperate of 100oC or less.

% Ash = C - A x 100

 B

A=Weight of crucible

B=Weight of sample

 C=Weight of ashed sample; crucible

**Note 1**: Crucibles should be pre-ashed for 10 minutes before they are used for sample ashing to insure results are accurate to + 0.001%. This procedure is for running 4 samples, of no more than 30 grams each. For larger samples, multiple samples, or other types of petroleum products contact CEM Corporation at +1 800-726-3331.

**Note 2:** Never place samples into the furnace chamber when the temperature is above 100oC or any temperature that the sample will begin to burn, whichever is the lowest.

**Note 3:** Open the furnace door slowly. If the sample is still flaming or not well carbonized extend the Stage 2 dwell time.

**Note 4:** The Stage 1 Dwell Time may be reduced or eliminated depending on the amount of water in the sample.

**Note 5:** This procedure is a reference point for sample ashing using CEM Microwave Ashing System and may require modifications or changes to obtain the required results on specific samples.