Ash Analysis of Pharmaceuticals, Bulk

AW108.0

**Method Parameters**

Dwell Time: 10 minutes

Temperature: 800°C

**Recommended Systems**

PHOENIX AirWave

Sample Amount: **(Note 1)**

**Equipment**

PHOENIX Airwave, appropriate crucible (porcelain, fused silica, platinum, CEM Quartz Fiber crucible, or evaporating dish), tongs, gloves, brush, desiccator, balance capable of weighing to + 0.1 mg.

**Procedure**

1. Weigh the appropriate amount **(Caution: See Note 1)** of sample into the pre-ashed **(Note** **3)** evaporating dishes or crucibles.
2. Wet the sample with a sufficient amount of H2SO4.
3. With the furnace temperature at 175oC or less **(Caution: See Note 2)**, place the evaporating dishes or crucibles into the furnace chamber, replace the furnace door allowing no gaps and close the microwave cavity door.
4. In the Quick Test Mode, program the Dwell Time = 10 minutes, Set Point = 800oC. Press the **Start/Stop** button and then the **Time** button.
5. At the end of the Dwell Time, remove the crucible and let it cool in a desiccator then weigh and calculate % sulfated ash.
6. Program the Set Point = 175° C. Press the **Start/Stop** button and the furnace chamber will cool to the starting temperate of 175oC.

% Ash = C - A x 100

B

A=Weight of crucible

B=Weight of sample

C=Weight of ashed sample; crucible

**Note 1:** This is a reference procedure for running no more than 50 grams. Sample sizes and times may need adjustment for specific products. For larger samples, multiple samples, or other types of pharmaceutical products contact CEM Corporation at Tel: 800-726-3338.

**Note 2:** Never place samples into the furnace chamber when the temperature is above 175oC or the ignition point of the product being ashed, whichever is the lowest. If the results are lower than certified values, a ramping procedure should be employed.

**Note 3:** Crucibles should be cleaned and fired at 800 °C for 30 minutes, and stored in a desiccator until used.