1.0 Purpose

1.1 To determine the amount of volatile matter of any kind that is driven off under the conditions specified.

2.0 Scope

2.1 A small amount of each submitted dry sample is placed in a porcelain crucible and heated extensively until it is certain that all of the water has been driven off. By weighing and calculation it can then be determined if the full amount, or what part, of the original seven moles of water existed in the submitted sample. The monohydrate loses between 13.0% and 16.0% of its weight, the dried form loses between 22.0% and 28.0% of its weight, and the heptahydrate loses between 40.0% and 52.0% of its weight.

2.2 USP 34; Monograph: Magnesium Sulfate, and General Chapter <733>

3.0 Responsibility

3.1 Quality Associate is responsible for this procedure.

4.0 Safety Considerations

4.1 Wear safety glasses and/or goggles and heat resistant gloves when handling hot crucibles. 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

5.1 Porcelain Crucible – #C6450-4. 10 mL. capacity
5.2 Balance – Mettler Toledo X5105Du, B13929Z316
5.3 Drying Oven – Baxter Temp Con, Model # N8620-1A, 0609-3203
5.4 Muffle Furnace – Thermodyne Type 1300 120V
5.5 Spatula
5.6 Desiccator (Note: Ensure desiccant is kept fully effective by frequent replacement)
5.7 Tongs

6.0 Procedure

6.1 The empty analytical balance is first tarred to zero.

6.2 A dry porcelain crucible is placed upon the balance and its weight is recorded.

6.3 Approximately 1 gram of a magnesium sulfate sample is added to the crucible and the total weight of the sample and the crucible are recorded in the appropriate laboratory notebook.

(The weight of the sample is calculated by subtracting the prerecorded weight of the porcelain crucible)

6.4 The crucible is placed in the drying oven at a temperature of 105° C for two hours.

6.5 The crucible is removed from the drying oven using tongs and placed in a muffle furnace at 450° C for at least five hours.

6.6 The crucible is removed from the muffle furnace, again using the tongs, and placed in the desiccator to cool.

6.7 After a suitable time period the crucible is placed on the balance and the weight is recorded.

6.8 By dividing the difference between the total weights and dividing by the mass of the sample before heating the percent loss on ignition is provided (see below):

$$\frac{\text{(total weight before heating) - (total weight after heating)}}{\text{(weight of sample before heating)}}$$

6.9 Place the sample back into the desiccator for use in the USP Assay procedure.

7.0 Reference Documents

7.1 Laboratory Notebook
7.2 *USP Stability Testing Summary Worksheet* Q12-PR-100-F010

8.0 Amendment Record

Revision Number	Revision Date	Revision Author	Revision Description
0	03/08/12	SB	New Document