

PREMIER MAGNESIA - GILES CHEMICAL

COMPANY PROCEDURE

USP Loss On Ignition (LOI): Magnesium Sulfate Heptahydrate

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Revision: 00 Effective Date: 03/08/2012

Author: Stephen Ballew Procedure Number: QA-LAB-36



Safety:

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

Personnel responsible:

Lab

Purpose:

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

Background Information:

Epsom Salt crystals forming in a cooled solution of magnesium sulfate develop in the "heptahydrate" form. This, means that the molecules includes not only MgSO₄, but seven moles of water for each mole of MgSO₄. Each molecule of Epsom Salt in crystalline form is expressed therefore as MgSO₄ • 7H₂O. The water molecules however, are not tightly bound, and can be readily driven off by subjecting the salt to rather mild heat conditions starting about 40 °C. Such a temperature, and higher, may be encountered during the final drying process. Hence it become necessary to check the product from to make sure that none of the H₂O has been inadvertently driven off, and final yield diminished thereby. Extensive heating also results in crystal damage and degradation, resulting in the formation of dust.

Scope:

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.

Test Method:

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

Equipment:

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

Procedure:

- 1. The empty analytical balance is first tarred to zero.
- 2. A dry porcelain crucible is placed upon the balance and its weight is recorded.



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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.

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

- 4. The crucible is placed in the drying oven at a temperature of 105° C for two hours.
- 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. The crucible is removed from the muffle furnace, again using the tongs, and placed in the desiccator to cool.
- 7. After a suitable time period the crucible is placed on the balance and the weight is recorded.
- 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):

(total weight before heating) - (total weight after heating) (weight of sample before heating)

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



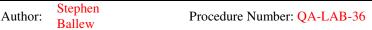
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00	03/08/2012	03/08/2012	Stephen Ballew	Deborah Durbin	Jason Bumgarner	New Document