

## Residue On Ignition (Sulfated Ash)

### Applicable Products: Carbopol<sup>®</sup>\* Polymers and Noveon<sup>®</sup>\* AA-1 Polycarbophil

#### Scope:

This procedure is used for the determination of the residual substance not volatilized from a sample of Carbopol<sup>®</sup> polymer or Noveon<sup>®</sup> AA-1 polycarbophil after ignition in the presence of sulfuric acid.

#### Abstract:

A weighed sample of the polymer is ignited, thoroughly charred and placed in a high temperature oven (600°C). After the carbon is completely consumed, the residue is cooled, weighed and the percent inorganic impurities in the organic sample is calculated.

#### Interferences:

Care must be taken to avoid moisture pick-up from the atmosphere. The absorption of moisture will alter the weight of the initial polymer sample and the residue, thus influencing the percent residue on ignition.

#### Safety Precautions:

1. Wear safety goggles and gloves and follow good laboratory practices.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. Sulfuric acid is extremely corrosive and causes serious burns. It is highly toxic and harmful by inhalation, ingestion and through skin contact. Ingestion may be fatal. Skin contact can lead to extensive and severe burns. Chronic exposure may result in lung damage and possibly cancer.
4. See all Material Safety Data Sheets (MSDS) for additional safety and handling information.

#### Apparatus:

1. Analytical balance capable of  $\pm 0.0001$  gram accuracy.
2. Muffle furnace, controlled at  $600 \pm 50^\circ\text{C}$ .
3. Silica, porcelain, platinum or quartz crucible, of suitable size.
4. Ring stand with ring.
5. Iron wire triangle with clay pipe stems.
6. Gas Bunsen burner or hot plate.
7. Desiccator with silica gel or other suitable desiccant.
8. Liquid dropper.
9. Tongs.
10. Crucible cover.

#### Reagents:

1. Concentrated Sulfuric Acid, Certified ACS grade.

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### Procedure:

1. Place a clean crucible in a 600° (± 50°) C muffle furnace for approximately 30 minutes.
2. Remove the crucible with tongs and place in a desiccator to cool.
3. When the crucible has reached room temperature, accurately weigh the crucible on the analytical balance (to the nearest 0.0001 g). Record the weight.
4. Add 1 gram of polymer to the crucible and record the weight to the nearest 0.0001 g.
5. Reweigh the crucible and its contents on the analytical balance. Record the weight to the nearest 0.0001 g.
6. Working under a well ventilated hood, moisten the sample with 1 ml concentrated sulfuric acid and heat gently with a Bunsen burner using as low a flame as possible. The polymer is to be charred thoroughly. (A ring stand supporting an iron wire triangle with clay pipe stems is useful when heating the crucible). (See Note 1.)
7. Allow the crucible to cool.
8. Moisten the charred polymer with 1 ml concentrated sulfuric acid.
9. Reheat the polymer/acid gently with an adequate flame until white fumes are no longer produced from the crucible.
10. Carefully transfer the crucible to the 600°C (+/-50°C) muffle furnace.
11. The crucible should remain in the oven until all the residue is completely incinerated (approximately two hours). Ensure that flames are not produced at any time during the procedure.
12. Remove the crucible from the muffle furnace and place cover on the crucible. Move the crucible to a dessicator to cool to room temperature.
13. When the crucible has cooled to room temperature, re-weigh to the nearest 0.0001g and record the weight.
14. Perform the % residue calculation.
15. If the amount of residue exceeds the limit specified, again moisten the residue with 1 ml of sulfuric acid, heat and ignite as before, using a 30 minute ignition period, until two consecutive weighings of the residue do not differ by more than 0.0005 g or until the percentage of residue complies with the monograph limit.

### Calculations:

$$\% \text{ Residue} = (A-B) / C \times 100$$

Where:

A = weight of crucible and residue after heating (Step 13)

B = weight of empty crucible (Step 3)

C = weight of sample (weight from Step 5 minus the weight of the empty crucible)

### Notes:

1. The procedure is written using a Bunsen burner. A hot plate capable of reaching the temperature to char the sample could be used instead of a Bunsen burner.

### References:

- *Current edition of the United States Pharmacopoeia/National Formulary (USP/NF)*
- *Current edition of the European Pharmacopoeia*