Ash Analysis of Resin (Black Solid Hydrocarbon)

A014.0

**Method Parameters**

Dwell Time: 15 minutes

Temperature: 950 °C

**Recommended Systems**

PHOENIX

Sample Size:2 grams

**Equipment**

PHOENIX OR MAS-7000, quartz fiber ashing crucibles, quartz fiber crucible liners (disks), tongs, gloves, brush, desiccator, balance capable of weighing to + 0.1 mg.

**Sample Preparation**

**Procedure**

1. Program the PHOENIX OR MAS-7000 for 30-60 minutes and 925 °C. Allow the ashing furnace to reach the set temperature.

2. Weigh a crucible lined with 1 disk to the nearest + 0.1 mg. Record the weight as Figure A. See notes 1 and 2 below.

3. Weigh 2 grams of sample to the nearest + 0.1 mg. into the crucible and spread evenly on the disk. Record the weight as Figure B.

4. Place the crucible with sample in the furnace and ash for 15 minutes. Remove the crucible and allow it to cool in a desiccator for 1 minute.

5. Reweigh the crucible containing the ash to the nearest + 0.1 mg. Record weight as Figure C.

6. Calculate the percent ash using the following equation:

% Ash = C - A x 100

B

A=Weight of crucible and disk

B=Weight of sample

C=Weight of ashed sample; crucible and disk

**Results**

Standard Ashing Procedure

|  |  |  |
| --- | --- | --- |
| **Time (min)** | **Temp. °C** | **% Ash** |
| Note 3 | 950 | <0.5 |
| Note 3 | 950 | <0.5 |
| Note 3 | 950 | <0.2 |

Microwave Ashing Procedure

|  |  |  |  |
| --- | --- | --- | --- |
| **Sample** | **Time (min)** | **Temp. °C** | **%Ash** |
| #1 | 15 | 950 | 0.529 |
|  |  |  | 0.524 |
|  |  |  | 0.584 |
| **Avg.** |  |  | **0.546** |
| **Std.Dev.** |  |  | **0.033** |
|  |  |  |  |
| #2 | 15 | 950 | 0.434 |
|  |  |  | 0.434 |
|  |  |  | 0.429 |
| **Avg.** |  |  | **0.432** |
| **Std.Dev.** |  |  | **0.003** |
|  |  |  |  |
| #3 | 20 | 950 | 0.275 |
|  |  |  | 0.275 |
|  |  |  | 0.270 |
| **Avg.** |  |  | **0.273** |
| **Std.Dev.** |  |  | **0.003** |

**Note 1**:Quartz fiber ashing crucibles and disks should be pre-ashed for 10 minutes before they are used for sample ashing to insure results are accurate to + 0.001%.

**Note 2**: Quartz fiber ashing crucibles may be reused until small holes or cracks begin to appear. The crucibles should then be discarded. Used quartz fiber ashing crucibles should be cleaned before reusing by brushing out all ash particles with a soft, bristle brush. Quartz fiber ashing disks are not reusable.

**Note 3:** Standard method calls for a 10 g sample heated to ignition and continue to maintain heat until all signs of volatile evolution are no longer visible over a Bunsen burner. Muffle to constant weight ± 0.001 g at 950 °C.