

## Phoenix

Sample Type: Carbon Black

Summary:

This method describes the determination of ash content in carbon black using a Microwave Muffle Furnace, PHOENIX OR MAS-7000.

Required Equipment:

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

Method:

1. Program the PHOENIX OR MAS-7000 for 90 minutes and 550 °C. Allow the ashing furnace to reach the set temperature.
2. Weigh a crucible lined with 1 disk to the nearest  $\pm 0.1$  mg. Record the weight as Figure A. See notes 1 and 2 below.
3. Weigh 2.0 gram(s) of sample to the nearest  $\pm 0.1$  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 90 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  $\pm 0.1$  mg. Record the weight as Figure C.

6. Calculate the percent ash using the following equation:

$$\% \text{ Ash} = \frac{C - A}{B} \times 100$$

A=Weight of crucible and disk

B=Weight of sample

C=Weight of ashed sample, crucible and disk

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  $\pm 0.001\%$ .

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.

### MICROWAVE APPLICATION DATA

Sample: Carbon Black

#### Standard Ashing Procedure

<u>Time</u> (mins)	<u>Temperature</u> (°C)	<u>Ash</u> (%)
960	550	<0.50
960	550	0.57

#### Microwave Ashing Procedure

<u>Time</u> (mins)	<u>Temperature</u> (°C)	<u>Ash</u> (%)
90	550	0.235
		0.255
		0.255
90	550	0.524
		0.539
		0.564

Note: Exert care in moving samples in and out of ashing furnace. Very light sample material located in the furnace near the front may be disturbed by air being drawn in around the furnace door. You may try placing an additional disk on top of the sample to help avoid losing sample. It has also been determined that higher temperatures can reduce the time from 90 minutes to 60 minutes or less. It may be advantageous to your customer to try higher temperatures of 750 °C and reduce the time required.