



High Temperature Microwave Systems

CEM Ashing Applications Note

Application Note 001
Revision 10-01-2001

Sample Type: Wheat Flour

Summary:

This method describes the determination of ash content in wheat flour using a PHOENIX™ OR MAS-7000™ with quartz fiber ashing crucibles. See Application Note 001A for flour ashing using platinum crucibles.



Required Equipment:

PHOENIX OR MAS-7000, quartz fiber ashing crucibles, quartz fiber crucible liners (disks), absorbing crucible liners, liquid dispenser, tongs, balance capable of weighing to ± 0.1 mg.

Reagents Required:

Magnesium acetate
Ethyl alcohol (95%)

Method:

1. Prepare a solution of magnesium acetate in ethyl alcohol, 0.015:1 weight:volume. This can be done by adding 15 g of magnesium acetate to 1000 ml 95% ethyl alcohol. Be sure to filter the solution prior to use.
2. Program the PHOENIX OR MAS-7000 for 935 °C and allow the ashing furnace to reach the set temperature.
3. Program the PHOENIX OR MAS-7000 for 10 minutes.

Steps 4-8 are used to determine a blank for the ashing method.
4. Weigh a crucible lined with three disks to the nearest ± 0.1 mg. Record the weight as Figure A. See notes 1 and 2 below.

5. Place 1 absorbing liner under the top disk in the crucible and drip 3.0 mL of the magnesium acetate solution onto the top disk. The liquid should soak into the disks and be absorbed. The extra disks are used to fully absorb the liquid since no flour is being ashed. Flour will absorb most of the solution when a sample is being analyzed. See Note 3 below.
6. Place the crucible in the furnace and ash it for 10 minutes. Remove the crucible and allow it to cool in a desiccator for 2 minutes.
7. Reweigh the crucible containing the ash to the nearest ± 0.1 mg. Record the weight as Figure B.
8. Calculate the ash blank using the following equation:

$$\text{Figure B} - \text{Figure A} = \text{Ash Blank (Figure C)}$$

Note: Operator should run a minimum of 2 blanks (duplicates); these duplicates must agree within 0.0003 g (0.3 mg). If they do not meet this specification, the lab technique must be evaluated to determine where the error arises from.

To determine potential sources of errors, the first item to check is the precision of the blank. (Test method assumes blank precision is $< 0.015\%$ relative; that is, that the range of values obtained on magnesium acetate blank solution is < 0.0003 g when using a 2 g sample). To determine precision of blank, run several blanks, calculate average and standard deviation of results. The average must be less than the precision you are expecting from the results.

Eg: 2.0 g sample of flour with a 0.0003 g variation in blank value will result in a 0.015% error due to the blank alone. If blank value are excessive, identify cause. Verify by running blank with and without magnesium acetate. If magnesium acetate is the cause, find more accurate method of adding solution. The CEM pipette system, when used properly, results in $< 0.015\%$ variation in blank results.

Steps 8-13 are used to determine the ash in the flour sample.

9. Weigh a crucible lined with one disk to the nearest ± 0.1 mg. Record the weight as Figure D.

10. Place one absorbing liner in the crucible and tare the balance. Weigh 2 grams of sample to the nearest ± 0.1 mg. into the crucible. Record the weight as Figure E. Spread the sample evenly on the liner. Wet the entire sample with 3.0 mL of the magnesium acetate solution.
11. Place the crucible with sample in the furnace and ash it for 10 minutes. Remove the crucible and allow it to cool in a desiccator for 1 minute. Up to 4 samples can be placed in the furnace at one time.
12. Reweigh the crucible containing the ash to the nearest ± 0.1 mg. Record the weight as Figure F.
13. Calculate the percent ash using the following equation:

$$\% \text{ ash} = \frac{(F - C - D)}{E} \times 100$$

- 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 the soft, bristle brush provided. Quartz fiber ashing disks and absorbing liners are not reusable.
- Note 3: Delivery of the magnesium acetate solution must be precise. If operators try to dispense the solution by reading the meniscus of the dispenser, errors can be made. Available from CEM Corporation is a 3.0 mL volumetric pipette that will deliver precise amounts.

Sample: Wheat Flour

<u>Standard Ashing Procedure</u>			<u>Microwave Ashing Procedure</u>		
<u>Time</u> (mins)	<u>Temperature</u> (°C)	<u>Ash</u> (%)	<u>Time</u> (mins)	<u>Temperature</u> (°C)	<u>Ash</u> (%)
<u>Flour</u>					
60	871	0.512	10	950	0.508 0.512 0.520
<u>Durum flour</u>					
60	871	0.730	10	950	0.724 0.724 0.739
<u>Wheat flour</u>					
AACC check sample					
60	871	0.530 0.504 0.550	10	950	0.524 0.513 0.539
Average=		0.530	Average=		0.524
<u>Soft wheat flour</u>					
60	871	0.512	10	935	0.514 0.519 0.524 0.514 0.509