Ash Analysis of Milk Powder

A002.0

**Method Parameters**

Dwell Time: 10 minutes

Temperature: 935 °C

**Recommended Systems**

PHOENIX

Sample Size: 2 grams

**Equipment**

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

**Reagents**

Magnesium acetate

Ethyl alcohol (95%)

**Procedure**

1. Prepare a solution of magnesium acetate in ethyl alcohol. This can be done by adding 15g 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 3 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 ml of the alcohol 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 powder is being ashed. The powder will absorb most of the solution when a sample is being analyzed.

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:

Figure B - Figure A = Ash Blank (Figure C)

**Steps 9-13 are used to determine the ash in the milk sample.**

9. Weigh a crucible lined with 1 disk to the nearest + 0.1 mg. Record the weight as Figure D.

10. Place 1 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 ml of the alcohol magnesium acetate solution.

11. Place the crucible with sample in the furnace and ash 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:

% ash = (F - C - D) x 100

E

**Results**

Microwave Ashing Procedure

|  |  |  |  |
| --- | --- | --- | --- |
| **Sample** | **Time (min)** | **Temp. °C** | **%Ash** |
|  | 10 | 935 | 5.83 |
|  |  |  | 5.82 |
| **Avg.** |  |  | **5.83** |
| **Std.Dev.** |  |  | **0.01** |

**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.