Ash content determination of various samples

Wet Digester B-440: Sulfated ash determination of various samples

1. Introduction

The wet digestion is used to remove all carbonous material from the sample. The remaining inorganic content can be weighed out, resulting in the sulfated ash, or then be used for further elemental analysis [1]. The digestion is performed using concentrated sulfuric acid while heating the mixture. The heat is ramped automatically using the BUCHI Wet Digester B-440. Once the sulfuric acid has digested the sample and is evaporated, the sample is eventually ashed in a muffle oven to remove any remaining organic material.

2. Experimental

Equipment: Wet Digester B-440, K-415 TripleScrub^{ECO}, Muffle oven

Samples: Various samples, listed in Table 1.

Procedure: Sample is weighed into a ceramic crucible. Depending on the application, an additive is added to the crucible. After 30 minutes reaction time, the mixture is heated up according to Table 1.

Table 1: Parameters for the digestion with the Wet Digester B-440

Sample	Additive	Volume additive [mL]	Step 1	Step 2	Step 3	Step 4	Step 5	Final ashing in muffle oven
Lactose	H_2SO_4 98%	1	130 °C 15 min	200 °C 35 min	250 °C 15 min	350 °C 15 min	500 °C 20 min	600 °C 2 h
Crystal sugar	H_2SO_4 98%	1	105 °C 20 min	110 °C 15 min	115 °C 15 min	250 °C 20 min	500 °C 10 min	600 °C 2 h
Modified starch	-	-	400 °C 30 min	500 °C 15 min	600 °C xx[a] min			550 °C 2 h
Modified starch	H_2O_2 30%	1	150 °C 5 min	400 °C 5 min	600 °C xx[a] min			550 °C 2 h
Wheat flour	-	-	400 °C 30 min	500 °C 15 min	600 °C xx[a] min			550 °C 2 h
Wheat flour	H_2O_2 30%	1	150 °C 5 min	400 °C 5 min	600 °C xx[a] min			550 °C 2 h

[a] Time was set to maximum (99 min) and stopped manually

3. Results

The determined ash content is presented in Table 2.

Table 2: Determined ash content in various samples

Sample	Sample amount [g]	Result
Lactose	**1**	**0.02 - 0.04%**
Crystal sugar	**1**	**0.02 - 0.03%**
Modified starch (without H_2O_2)	**3**	**0.38% (±0.01%)**
Modified starch (with H_2O_2)	**3**	**0.35% (±0.01%)**
Wheat flour (without H_2O_2)	**2**	**0.49% (±0.04%)**
Wheat flour (with H_2O_2)	**2**	**0.49% (±0.07%)**

4. Conclusion

With simple operation and high sample throughput, the determination of the ash content by use of the Wet Digester provides reliable and repeatable results.

5. References

[1] Badran, M. et al. Assessment of wet acid digestion methods for ICP-MS determination of trace elements in biological samples by using multivariate statistical analysis, Journal of Elementology, 179-189, 2018.

For more information, please refer to buchi.com.