

Sulfated Ash of Various Sweeteners and Syrups

Introduction

Tight specifications around the amount of sulfated ash that can be present in sugars and syrups are maintained to ensure quality. Most standards specify that ash content cannot exceed 0.015% in refined granulated sugar. Various research for some sugar products points to a correlation between ash content and color; color being one of the most important perceptions of quality by consumers.¹ While some ash may be added during processing of sugar variants, ash can also result from processing equipment erosion, improper handling, and insufficient washing. The Phoenix BLACK™ Sulfated Ash system provides a safe, automated, and reliable alternative to traditional sulfated ashing techniques, while meeting all muffle furnace requirements.

Traditional Methods

In order to produce a uniform ash and facilitate the destruction of organic matter, sugars and syrups are ignited in the presence of sulfuric acid. The residue on ignition (ROI), reported as sulfated ash, is used as a quantification of the amount of inorganic impurities and for preparation for specific elemental analyses. It is often referred to as wet ashing, acid-facilitated oxidation, or sulfated ashing.

Traditional techniques require Bunsen burners, heat lamps, hot plates, acid resistant hoods, and muffle furnaces continuously held at high temperature. As operators are directly exposed to hot surfaces and open flames, these techniques pose significant safety risks. Extensive training, oversight and cleaning are all required by laboratory personnel, limiting the amount of time and focus that can be dedicated to other important testing. The risk of experimental error is also high with traditional methods, because of the numerous manual steps required. Due to the time and energy required to heat traditional muffle furnaces, they are held at high temperatures constantly, leaving lab personnel susceptible to burns and accidents. Temperate uniformity, control, accuracy, and precision also continue to be a concern.

Phoenix BLACK Sulfated Ash Technology

The Phoenix BLACK Sulfated Ash is a muffle furnace that meets all microwave and standard muffle furnace requirements listed in various ASTM, USP, AOAC, FDA, ISO, DIN, and industry standards,

including AOAC 900.02C. The system is easily customizable, contains a library of preprogrammed methods, can ash up to 15 samples at a time, and is a faster alternative to traditional methods. The silicon carbide heating element is encased in a quartz-fiber furnace, which maintains a uniform temperature throughout the cavity. The Phoenix BLACK Sulfated Ash is compatible with all crucible types, including platinum, quartz, quartz-fiber, and ceramic. All temperature measurements are performed using dual NIST traceable thermocouples. The exterior of the furnace is cool enough to touch, even when the interior of the Phoenix BLACK Sulfated Ash is in excess of 1000 °C. Upon method completion, active ventilation rapidly cools the furnace and an exterior cooling fan ensures that the furnace can quickly be taken back to room temperature.

All sulfated ashing steps can be performed in the Phoenix BLACK Sulfated Ash, eliminating the need for additional equipment and minimizing operator oversight and handling. Acid is added to the sample in the crucible defined in the chosen standard, and placed directly into the Phoenix BLACK Sulfated Ash furnace. A lower temperature sulfating/ashing step, with active vapor scrubbing, is automatically completed prior to the high-temperature dry ashing of the sample.

Complete control of the system allows users to program a pause step prior to the dry ashing step, if desired, to remove and weigh the sulfated crucible. The Phoenix BLACK Sulfated Ash neutralizes toxic acid vapor through a series of scrubbers for a more environmentally sound, laboratory-friendly solution with no emission of hazardous fumes. The system also helps laboratories comply with ISO 14000 standards to improve environmental management efforts and to eliminate greenhouse gases.

The workstation option allows quantification for document control, via an external printer and connected balance. It automatically determines the % ROI, % LOI or % Ash. Once generated, sample information cannot be modified, aiding in 21 CFR Part 11 compliance. All weighing and method information is included in the data file, as well as the final result, user-entered sample ID, signature, date, and time stamps indicating when the sample was ashed. All methods are stored automatically and the system provides the security of a password lock to reduce the possibility of erroneous method modification. Data is easily exported via a data bridge for even easier data management and storage.

Analysis

To demonstrate the accuracy and precision of the system, the sugar substitute, molasses, maple syrup, and lactose powder were ashed utilizing 50 mL and 100 mL CEM Quartz-Fiber crucibles, caps, and covers following AOAC 900.02C. All crucibles, caps, and covers were pre-ashed and stored in a desiccator prior to use. Approximately 10 mL of 10% (by weight) sulfuric acid was used to wet the 3.40 to 5.00 g samples. A CEM cover was placed on top of the samples and an additional ~5.0 mL of sulfuric acid was added to wet the cover. The samples were introduced into the Phoenix BLACK below 100 °C and the method was started. After two hours, the samples were removed and the percent sulfated ash was calculated.

Results

All of the tests were completed in significantly less time than traditional muffle furnace methods require and matched expected results (see **Table 1**). It should be noted that the time for the conventional muffle furnace does not include additional operator actions, such as using a hot plate or Bunsen burner, which may require an excess of 30 minutes of operator time, prior to insertion of the sample into the muffle furnace. Pre-ashing or charring requires constant attention, as many technicians experience bubbling and overflow. The Phoenix BLACK Sulfated Ash automates the sulfating step, reducing the amount of operator attention, maintaining more consistency, and creating a safer laboratory environment.

Table 1. Sulfated Ash in Various Sweeteners and Syrups

Sample	1 (% Ash)	2 (% Ash)	3 (% Ash)	4 (% Ash)	5 (% Ash)	6 (% Ash)	7 (% Ash)	Average (% Ash)	STDEV
Sugar Substitute	0.140	0.123	0.152	0.152	0.130	N/A	N/A	0.139	0.013
Molasses A	2.236	2.241	2.546	2.243	2.494	2.537	2.289	2.369	0.148
Molasses B	2.338	2.447	2.434	2.211	N/A	N/A	N/A	2.358	0.109
Maple Syrup	0.760	0.788	0.772	0.765	0.786	N/A	N/A	0.774	0.012
Lactose Powder	0.136	0.126	0.157	0.133	0.085	0.112	0.123	0.125	0.030

Conclusion

The Phoenix BLACK Sulfated Ash meets the requirements of AOAC 900.02C and ICUMSA Method GS3, as well as various ASTM, USP, AOAC, FDA, ISO, DIN and other industry standards. The system provides a simple, automated method of analysis with a high degree of accuracy that requires minimal operator input and oversight. The safety, customizability, and automated nature of the system make it very operator-friendly and contributes to increased lab efficiency.

References

¹ McKee, M.; Triche, R.; Richard, C. Color and Ash – Is there a relationship between them? *Sugar Processing Research Institute, Inc - American Society of Sugar Beet Technologists* [Online] **2017** <https://bsdf-assbt.org/wp-content/uploads/2017/03/McKee-M.pdf> (accessed January 12, 2023).

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