

Characterization of Moisture Content of Artificial Sweeteners Using TGA

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Introduction

Pharmaceuticals and foods often contain moisture and the characterization of this moisture content is important for assessing end use properties. The moisture levels of foods and pharmaceuticals can have an impact on important characteristics including stability, shelf lifetimes, stickiness or tack, visual appearance and ability to dissolve in water. Even small differences in the level of water can have a significant effect on the properties of foods and pharmaceuticals, so it becomes important to have a high precision means of assessing moisture levels. One easy to use means of characterizing materials for moisture content is thermogravimetric analysis or TGA.

TGA measures the mass loss properties and the rate of weight loss as a function of time or temperature. The technique provides valuable information on composition, thermal stabilities, oxidative stabilities as well as kinetics or estimation of lifetimes. The measurement of water content requires a TGA with high performance capable of providing high precision levels, such as the Pyris 6 TGA from PerkinElmer Instruments.

Pyris 6 TGA

The Pyris 6 TGA is a high performance top loading thermogravimetric analyzer. TGA is one the family of techniques in the thermal analysis family and measures sample weight loss as a function of temperature and/or time.

The Pyris 6 TGA offers the following desirable features and benefits:

- High sensitivity for the detection of small weight loss transitions
- High resolution for the better separation of overlapping transitions
- Top loading balance design for ruggedness and durability
- Robust design for reliable, long term use
- Built in gas switching accessory and purge gas flow regulator for convenience and ease of use
- 45 position autosampler for reliable, unattended operation
- Pyris Player software for user friendliness and ease of use
- Ability to easily capture and export data



The autosampler featured with the Pyris 6 TGA offers state-of-the-art design with shape memory alloy grippers. This ensures reliable, long-term operation.

The built-in gas switching accessory and flow regulator is a highly desirable feature on a TGA instrument from a convenience standpoint. Many TGA experiments require the switching of an inert purge gas (e.g., nitrogen) to an oxidizing gas, such as air or oxygen. The built-in switching device makes this easy to perform. In addition, the Pyris 6 TGA provides for the control of the flow of the purge gas directly via the Pyris operating software and this is useful to ensure that the proper flow of gas is obtained and controller. This is a far more accurate and precise means of regulating and controlling the flow of the purge gas as compared to manual flow regulators. The purge gas conditions can be permanently stored

with the method making the Pyris 6 TGA a reliable instrument for multiple operators. The incorporation of the digital flow system into the Pyris 6 TGA and software minimizes the chances that the purge gas flow rate could be inadvertently altered, as can easily happen with the manual flow regulators.

Experimental

The weight loss properties of two different artificial sweeteners (Sweet 'n Low and Equal) were characterized using TGA. The following experimental conditions were used.

Experimental Conditions	
Instrument	Pyris 6 TGA
Heating rate	20 C/min
Sample mass	Approximately 7 mg
Sample container	Open ceramic pan
Purge gas	nitrogen
Temperature range	26 to 700 C

The samples of the two powdered sweeteners were taken from standard restaurant packets and analyzed without any further preparation.

Results

Displayed in Figure 1 are the TGA results generated on the Sweet 'n Low artificial sweetener. The plot shows the percent mass and the derivative (rate of mass loss) as a function of the sample temperature.

The sweetener undergoes a weight loss event beginning at 77 C and this reflects the loss of water from the sample. The total mass of water lost is 8.23% based on the well-defined weight loss step.

The sweetener starts to undergo thermal degradation at an onset temperature of 203 C. The total percentage of weight loss associated with the thermal degradation of the Sweet 'n Low material is 68.7%. There are multiple steps associated with the degradation of the sweetener as reflected by the numerous peaks occurring in the derivative signal.

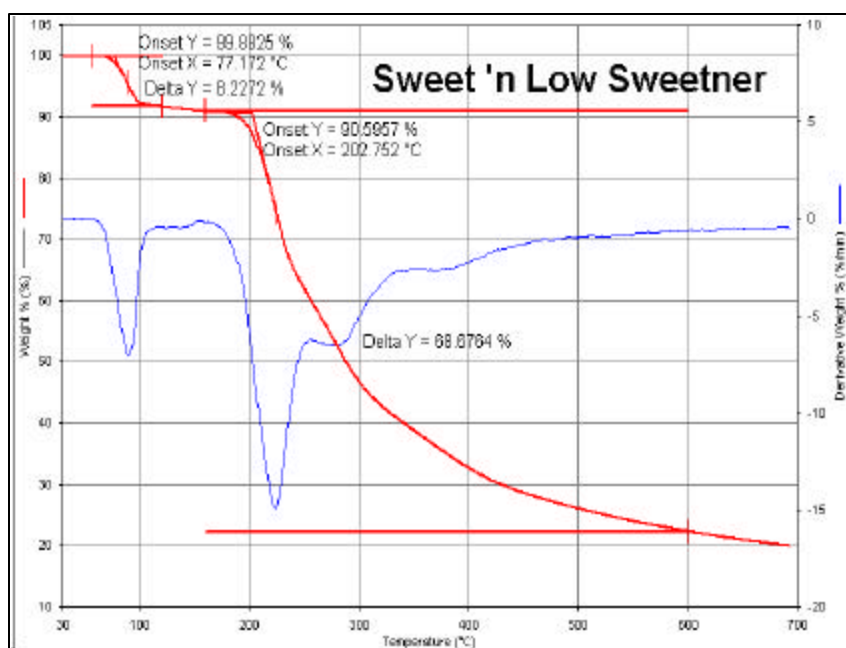


Figure 1. TGA results obtained on Sweet 'n Low artificial sweetener

The Pyris 6 provides outstanding reproducibility of the weight loss events associated with the sweetener as is demonstrated in Figure 2. This plot shows an overlay of the TGA results obtained on five experiments on the Sweet 'n Low sample.

The region of the water loss between room temperature and 200 C was enlarged to provide a more detailed analysis of the moisture content. The data was replotted to show the water loss event at magnified scaling and this is displayed in Figure 3. This plot shows an overlay of the five separate experiments performed on the Sweet 'n Low material.

The following table summarizes the values of the percent weight losses due to water

Results of Water Loss (%)	
Experiment 1	8.1636%
Experiment 2	8.0947
Experiment 3	8.2387
Experiment 4	8.1889
Experiment 5	8.2357
Average value	8.189%
Standard deviation	0.052%

The Pyris 6 TGA yields an average value of 8.189% for the water content with an excellent precision (σ) of 0.052%.

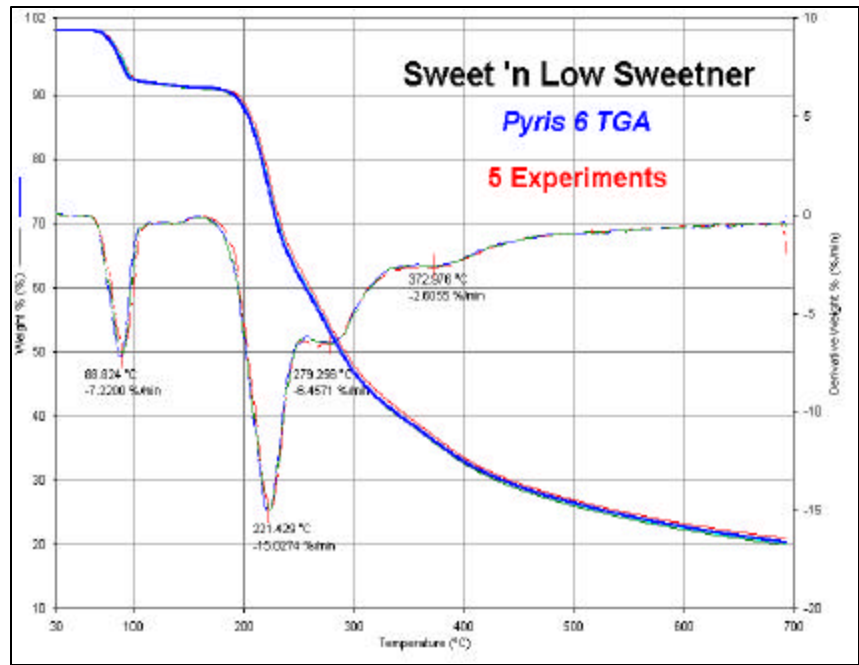


Figure 2. Overlay of five experiments performed on Sweet 'n Low

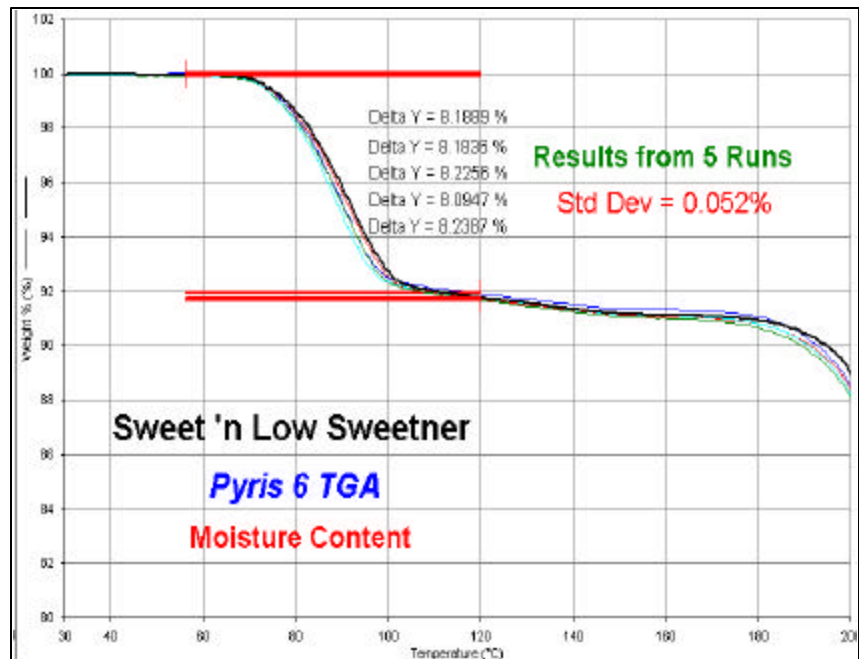


Figure 3. Enlarged view of the moisture loss of the five runs performed on Sweet 'n Low

A second artificial sweetener (Equal) was analyzed using the same conditions as for the Sweet 'n Low substance. The TGA results generated for the Equal sweetener are displayed in Figure 4. The results for the Equal sweetener are similar to those of the Sweet 'n Low, but there are some differences that the high performance Pyris 6 TGA was able to observe. The water loss of the Equal sweetener (7.99%) is slightly (but significantly) less than that of the Sweet 'n Low (8.19%). In addition, the thermal degradation of the Equal substance occurs at higher temperatures. This may be better seen in an overlay plot where the TGA results from the two sweeteners are displayed. The overlay plot is shown in Figure 5. The Pyris 6 TGA is able to detect well defined differentiations between the two similar sweeteners.

Summary

The water loss and thermal degradation properties of two artificial sweeteners (Sweet 'n Low and Equal) were studied using the high performance, top loading Pyris 6 TGA. The instrument yielded excellent precision ($\sigma = 0.052\%$) on the moisture content of the sweeteners. The Pyris 6 TGA offers ease of use, reliability, robustness and excellent performance for the high precision moisture and weight loss measurements of pharmaceuticals and food materials.

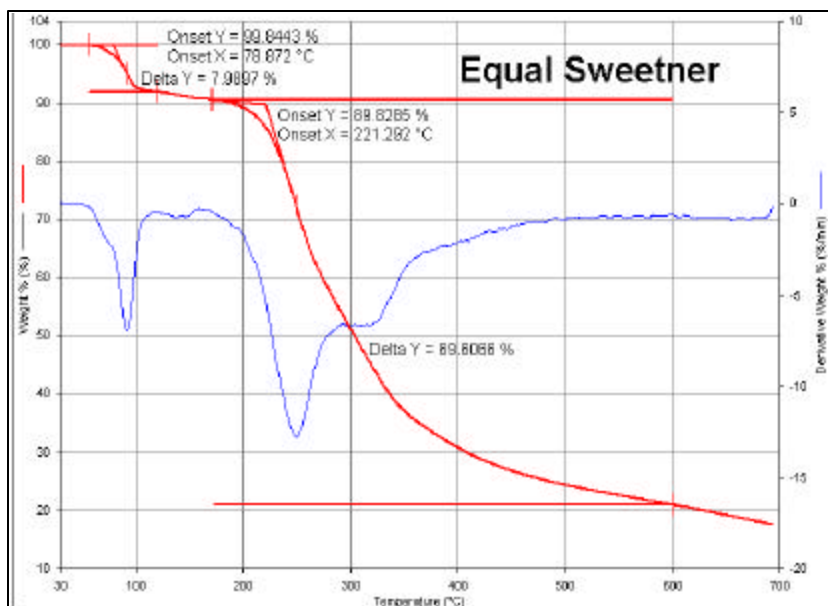


Figure 4. TGA results obtained on Equal sweetener

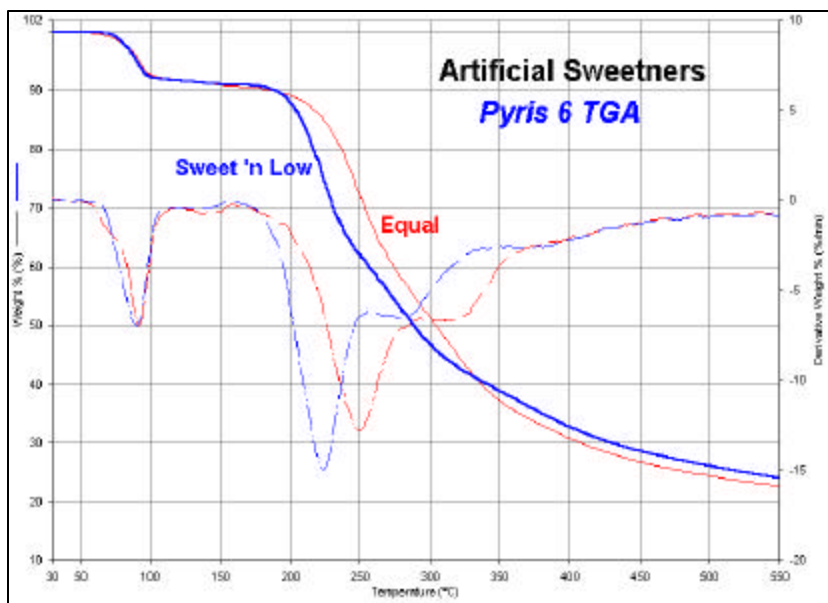


Figure 5. Overlay of TGA results generated on Sweet 'n Low and Equal artificial sweeteners

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