Determination of Moisture/Loss on Drying in Cannabis by Various Analytical Techniques

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Introduction

Moisture content in cannabis can play a vital role in several aspects of the growth and distribution of industrial hemp and recreational cannabis. Moisture content of cured cannabis is an indicator of potential microbial contamination and is a key variable used to calculate other constituent results on a dry basis. Additionally, determination of moisture content of cannabis is also required for the pressing process of extracting CBD oil from cannabis. Measurement of moisture using a gravimetric oven drying technique is difficult due to the influence of constituent properties of cannabis including, but not limited to, terpenes, cannabidiols, and THC, which may have low temperature volatility or sublimation.

Moisture determination is currently quantified as a Loss on Drying (LOD) as performed in United States Pharmacopeia (USP) Monograph 731 in conjunction with Monograph 921. The drying temperature stated in these monographs for materials of botanical origin is 105 °C. However, according to other sources for plant analysis, such as the Plant Analysis Handbook III[1], oven drying of plant matter may result in thermal decomposition of the plant tissue resulting in a false high value for moisture content when higher temperatures (such as 105 °C) are used. In addition, LOD incorporates all the components lost for a given set of conditions, including moisture and the aforementioned volatile organic compounds (VOCs) that are the result of thermal decomposition or sublimation.

The objective of this poster is to demonstrate the use of multiple techniques, including direct moisture determination in conjunction with direct carbon determination through infrared detection of H_2O and CO_2 respectively, to determine the optimal temperature for gravimetric moisture/LOD determination in cannabis. Further optimization will be explored to improve analysis time by deviating from the techniques illustrated in USP 731 and 921 while utilizing an automated thermogravimetric analyzer.

Reference

- **USP Monograph 731: Loss on Drying**
- **USP Monograph 921: Water Determination**

TGM800 Theory of Operation

The LECO TGM800 is a thermogravimetric analyzer designed to indirectly determine moisture content of materials using a mass loss on drying technique. Mass loss of the sample is measured as a function of the oven temperature by controlling the atmosphere and ventilation rate. The instrument consists of a computer, an integrated four-place balance, and a multiple sample oven that allows up to 16 samples to be analyzed simultaneously with a maximum temperature of 175 °C.

Key Features

- Supports 1.5 in (1 g nominal mass) diameter aluminum foil crucibles
- Supports 2.4 in (3 g nominal mass) diameter aluminum foil crucibles
- Analysis can be performed in air or nitrogen atmosphere
- Automated crucible and sample mass recording
- Optional fixed time or constancy method parameters

Figure 1: TGM800

Figure 2: Sample carousel using 1.5 in aluminum foil crucible (16 position)

Figure 3: Sample carousel using 2.4 in aluminum foil crucible (10 position)

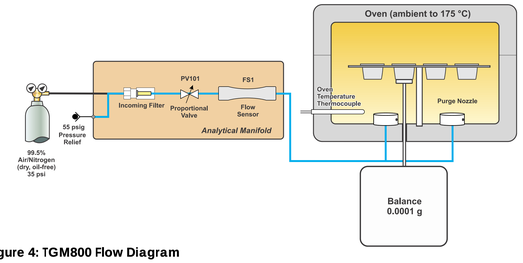


Figure 4: TGM800 Flow Diagram

RC612 Theory of Operation

The LECO RC612 is a resistance furnace multiphase carbon and moisture determinator which quantifies the carbon and water present in various organic samples and illustrates the source of several types of carbon. The RC612 features a state-of-the-art furnace control system, which allows the temperature of the furnace to be stepped and ramped from near ambient to 1100 °C, in either oxidative or reductive carrier gas. The RC612 utilizes an afterburner furnace that ensures complete combustion of volatile species released at lower primary furnace temperatures. For this application, the furnace is programmed with a two-step temperature ramping profile to separate moisture from VOCs.

Figure 5: RC612

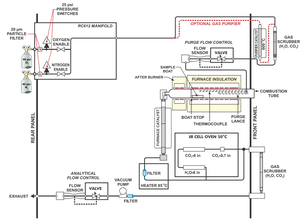


Figure 6: RC612 Flow Diagram

Background

Moisture determination in cannabis can be a critical factor in expressing accurate analytical results for other constituents on a dry basis. However, due to the presence of low temperature VOCs, obtaining a true moisture value becomes more complicated. Utilizing the LECO RC612, water can be determined directly and simultaneously with carbon allowing for the comparison of moisture and evolved VOCs. The LECO RC612 is able to perform the analysis at multiple temperatures, starting at a lower temperature and ramping to a higher temperature. This allows for the determination of the proper temperature to optimize moisture determination and minimize VOC evolution. This information allows for optimal temperature selection to be obtained and then applied to the LECO TGM800.

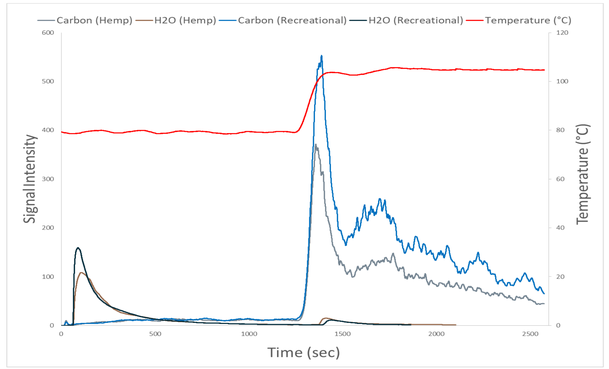
Methodology

RC612

Using information obtained from the Plant Analysis Handbook III[1] in conjunction with USP Monographs 731 and 921, two temperatures were selected for the determination of moisture. It was advised that determination of moisture below 80 °C will not sufficiently liberate water from the plant tissue, while temperatures above 80 °C have increased thermal decomposition of the plant tissue.

The 80 °C temperature was compared to the existing method of 105 °C with direct IR detection of carbon (as CO_2) and water. The results of this comparison are illustrated in Plot 1.

Plot 1: Recreational and Hemp Cannabis



The data presented in Plot 1 illustrates that the evolution of carbon attributed to VOCs at 80 °C is minimal compared to the carbon at 105 °C for both recreation and hemp cannabis. The water peak at 105 °C is attributed to hydrogen in the VOCs combusting to form water as the water peak coincides with the carbon peak. These plots indicate that the determination of moisture at 80 °C represents a closer approximation to the true moisture content of cannabis.

TGM800

Utilizing the information from the RC612, a method was created for the analysis of cannabis plant tissue for the determination of moisture at 80 °C. Several sample types were selected for analysis as shown below:

- Ø **Recreational Cannabis**
- Ø **Cured Hemp Cannabis**
- Ø **Uncured Hemp Cannabis**

Sample Preparation

All cannabis samples were ground using a hand tobacco grinder for the cured samples, and a knife mill for the wet (uncured) samples. The wet samples were prepared immediately prior to analysis to minimize moisture loss during sample preparation. After preparation, any noticeable seeds were removed to improve accuracy and precision.

Sample Results

The following tables display the results from the LECO TGM800 determining moisture at 80 °C and 105 °C until constant mass is obtained.

Table 1: Recreational Cannabis

Name	Initial Mass (g)	Moisture @ 80 °C (%)	LOD @ 105 °C (%)
Recreational Cannabis	1.0141	8.11	11.27
	1.0251	7.68	11.23
	1.0631	8.12	11.52
	1.0079	8.27	11.23
	1.0303	8.15	11.68
	Average	**8.07**	**11.39**
	Standard Deviation	**0.22**	**0.20**

Table 2: Cured Hemp Cannabis #1

Name	Initial Mass (g)	Moisture @ 80 °C (%)	LOD @ 105 °C (%)
Cured Hemp #1	1.0056	7.28	9.85
	1.0584	7.53	9.35
	1.0357	7.14	9.55
	1.0224	7.25	10.00
	1.0142	7.27	9.55
	Average	**7.30**	**9.66**
	Standard Deviation	**0.14**	**0.26**

Table 3: Cured Hemp Cannabis #2

Name	Initial Mass (g)	Moisture @ 80 °C (%)	LOD @ 105 °C (%)
Cured Hemp #2	1.0925	7.60	10.12
	1.0109	7.44	10.56
	1.0675	7.68	10.67
	1.0459	7.21	10.43
	1.0935	7.12	10.43
	Average	**7.41**	**10.44**
	Standard Deviation	**0.24**	**0.21**

Table 4: Uncured Hemp Cannabis #1

Name	Initial Mass (g)	Moisture @ 80 °C (%)	LOD @ 105 °C (%)
Uncured Hemp #1	1.0312	74.0	74.7
	1.0802	74.2	74.7
	1.0073	74.2	74.9
	1.0026	73.8	74.4
	1.1079	73.8	74.5
	Average	**73.99**	**74.64**
	Standard Deviation	**0.19**	**0.22**

Table 5: Uncured Hemp Cannabis #2

Name	Initial Mass (g)	Moisture @ 80 °C (%)	LOD @ 105 °C (%)
Uncured Hemp #2	1.0252	74.3	75.1
	1.2081	73.8	74.6
	1.1355	73.8	74.7
	1.1024	73.9	74.6
	1.0195	73.8	74.7
	Average	**73.94**	**74.74**
	Standard Deviation	**0.22**	**0.21**

Conclusion

Utilizing the 80 °C temperature from the Plant Analysis Handbook III[1] and subsequent information obtained from the analysis of cannabis on the LECO RC612, a precise, close approximation of the true moisture in cannabis flower and plant tissue may be obtained using thermogravimetric analysis. Analysis of cannabis above 80 °C results in the evolution and decomposition of VOCs contributing to a high moisture bias as shown in the data tables above. The LECO TGM800 is able to analyze up to 16 samples simultaneously, resulting in high throughput with accurate and repeatable results. Furthermore, utilizing the TGM800 mass constancy option, analysis time is reduced from five hours, as stated in USP 921, to approximately two hours.

References

1. G. Bryson, H. Mills, D. Sasseville, J. Jones Jr., and A. Barker, *Plant Analysis Handbook III*. Athens, Georgia: Micro-Macro Publishing, Inc., 2014