Laboratory guidance for drying field-fresh hemp plant samples in preparation for determination of total THC on a dry weight basis

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1.0 Objective

To provide guidance for laboratories in developing standard operating procedures for processing hemp plant materials received fresh from the field (approximate moisture 70-80%) to obtain a moisture content appropriate for grinding (generally <15%) when preparing samples for determining total THC on a dry weight basis, for example when using AOAC 2018.11 or other appropriate methods.

2.0 Purpose and Scope of Document

This document offers guidance to be used when developing a procedure for drying field-fresh hemp material. The purpose of the procedure is to dry the material enough to facilitate adequate grinding and homogenization prior to analysis. This drying procedure is not quantitative and is intended only to facilitate further sample preparation. As such, this procedure does not produce any reportable data and no SMPR has been developed. This guidance is separate from procedures for reporting total THC on a dry weight basis, such as those described in OMA 2018.11.

Though no reportable data will be created by the sample drying procedure, it is important for laboratories to develop the procedure in such a way as to minimize error introduction and maintain sample integrity. The laboratory should determine and document any error

that this drying step may introduce as well as the efficacy of the procedure. This document provides guidance with that documentation.

3.0 Introduction and Background Information on drying hemp plant materials

The protocol outlined by the United States Department of Agriculture (USDA) Agricultural Marketing Service (AMS) for determination of total THC in hemp requires that hemp plant samples be delivered to the testing laboratory in a field-fresh condition. The AMS rule further outlines that testing laboratories "remove the majority of water" from hemp material prior to milling and homogenizing the material. This drying step is crucial to ensure that the material flows easily into and through the milling device and does not clump during the homogenization process, but no specific drying process is mandated and procedures must be carefully crafted in order to minimize error. Because each laboratory has different environmental conditions and equipment, laboratories will need to develop and verify their own procedures. There is no singular approach that will work for every combination of environmental conditions and equipment.

There are three generally recognized techniques for drying plant material: heat, air movement, and dehumidification. Any combination of these processes can and should be considered in the development of the drying procedure. It should be noted that applied heat may cause decarboxylation of the acid forms of cannabinoids, changing the cannabinoid profile of the material. Heat and air movement may also volatilize terpenes during the drying process. Consequently, this sample drying procedure is intended to only be used in preparing field-fresh hemp plant materials for subsequent regulatory analysis

that reports total THC through full decarboxylation of THC-A or with the approved mathematical calculation: Total THC = 0.877 * THC-A + delta9 THC.

The purpose of this drying procedure is only to dry the plant material enough that moisture does not interfere with the milling and homogenization of the material, which typically will have moisture content of approximately 15% or less. This drying step is not intended to take the material to complete dryness, and the dried material will still need to undergo additional analysis in order to determine and report total THC on a dry weight basis. **(see AOAC OMA 2018.11)**.

4.0 Drying of Hemp and Experimental Set-up Guidance

This section provides guidance on collecting data to document and verify oven temperature and drying times appropriate for processing a laboratory sample of field-fresh hemp to a moisture content appropriate for analytical sample preparation, generally less than 15%.

A sample of field-fresh hemp may arrive at the laboratory in different containers and variable quantities. Laboratory samples not in a suitable container for the oven may be transferred to a container that can withstand the drying temperature, for example a paper bag. Drying will happen more rapidly if the sample is placed in an open container or open paper bag which allows for more efficient release of moisture from the sample.

Very large samples not suitable to be dried in an oven may be subsampled. Proper subsampling procedures must be established to ensure a representative sample is acquired. Subsampling error should be assessed and included as a component of measurement uncertainty for reporting the analyte concentration in the laboratory sample.

Commented [h1]: I deleted these definitions and all of section 4.0. . We do not use any of these terms in the doc except "total THC" so there is no need to define random terms - if that seems okay to you two? If anything, we could define "total THC" and perhaps do that in one of the other sections?

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Using the container, sample quantity, and oven equipment for a given laboratory as appropriate, samples are placed in an oven set at 60 to 70ºC. Samples are periodically removed from the oven, weights are documented, and samples are placed back in the oven. Larger samples require more time to dry compared to smaller samples. Table 1 provides guidance on how often to weigh the sample when developing appropriate drying methods. The study should continue until two successive weight determinations have a percent weight change less than 5% as calculated with the equation below.

% wt change =
(Previous wt – Current wt, g) / (Previous wt, g – Container wt, g) x 100 Eq. [1]

Percent moisture in the sample is calculated by the following equation.

% moisture =
(Current wt, g – Last wt, g) / (Current wt, g – Container wt, g) x 100 Eq. [2]

Example data for 140 and 400 g samples in closed paper bags at 60oC are shown in Table 2. The percent moisture is plotted versus time to construct moisture-loss curves (Figure 1).

Table 1. Suggested Periodicity for collecting weights to determine moisture-loss curve for field moist hemp.

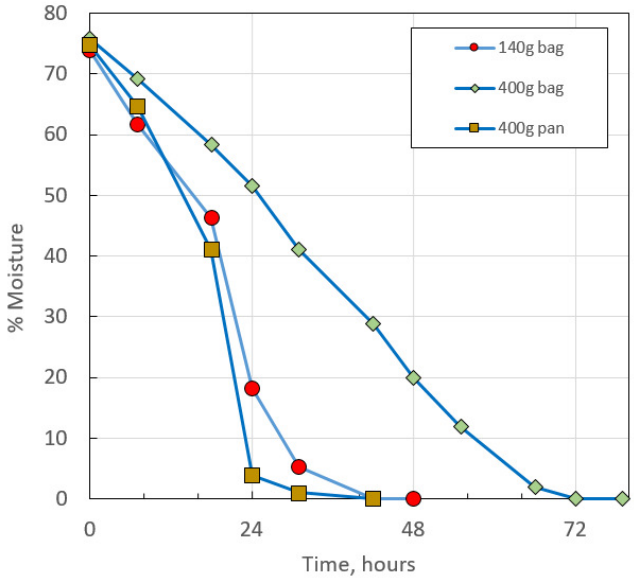
Sample weight (g)	Weighing periodicity
< 100 g	6 / day
100- 500 g	3 / day
> 500 g	1 / day

Table 2. Example Data for 140 and 400 g field fresh hemp in closed paper bags or open pan dried at 60°C.

Time, h	***Weight, g***	***% weight change[1]***	***% Moisture[1]***
---------- 140 g bag ----------			
0	161		74
7	117	27	62
18	90	23	46
24	67	26	18
31	61	9	5
42	59	3	0
48	59	0	0
---------- 400 g bag ----------			
0	420		76
7	334	22	70
18	254	26	59
24	222	14	52
31	186	18	42
42	158	17	30
48	143	11	21
55	132	9	13
66	121	10	3
72	119	2	1
79	118	1	0
---------- 400 g pan ----------			
0	932		75
7	820	29	65
18	708	40	41
24	643	39	4
31	640	3	1
42	639	1	0

[1] Percent weight change and percent moisture determined from Eqs. 1 and 2, respectively. Container weight was 23 g for bags and 540 g for pan.

Figure 1. Example Moisture-loss curves for field fresh hemp at 60°C for 140 and 400 g samples in closed paper bags or pan.



Equation 1 and the moisture-loss curves in Figure 1 assume no moisture in the final weighed sample. Although the sample may have variable moisture content when removed from the oven, the material may absorb moisture from the air due to ambient

conditions which can vary widely prior to grinding and even during grinding or milling. The amount of moisture absorbed depends on the humidity level to which the material is exposed. When determining an adequate time to dry hemp prior to grinding, careful evaluation of the appropriate time for drying is critical, and laboratories may target a moisture level of approximately 10% or less using the moisture-loss curves. Targeting this level should ensure samples are below 15% moisture in most analytical sample sizes. For the examples in Figure 1, 56-60 hours drying time was adequate for 400 g in a closed paper bag at 60ºC; 32 hour drying time was adequate for 140 g in closed paper bag at 60ºC; and 24 hr drying time is adequate for 400 g in open pan at 60ºC. Curves should be generated for multiple samples to provide assurance on the appropriate drying time. Studies should be conducted under conditions that reflect routine laboratory operations.

The goal of drying fresh hemp samples is to reduce the moisture content of the plant material to a level appropriate for grinding the material into a powdered analytical sample. This condition is apparent when plant material disintegrates readily when crushed. As samples are withdrawn from the oven using the conditions and determined time from the previous analysis, samples should be inspected by feel. If samples do not readily crumble on handling, the sample is not dry enough and should be dried for a longer period of time.

This guidance is intended to assist laboratories in developing and verifying standard operating procedures for processing field fresh hemp to a dried state appropriate for grinding and preparation of an analytical sample. To report percent total THC on a dry weight basis, an additional and separate analytical determination of moisture must be

made on a test portion of the sample taken at the same time a test portion is analyzed for total THC, in accordance with validated methods (see OMA 2018.11).

Ryan Randolph wording: Drying conditions should be verified under conditions that are representative of routine sample processing, with consideration given to oven capacity and various sample locations within the oven"?

7.0 Additional Resources

8.0 References