

Title: Determination of Volatiles in Smokeless Tobacco by Mechanical Convection Oven	Control #: ST-TM-440-254	Revision #: 2.2
	Approval Date: 12/16/2013	Effective Date: 12/16/2013
Test Method Owner: J.A. Sampson, ALCS RD&E Analytical Technical Services		

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NOTICE
This method may involve the use of hazardous substances and/or equipment. The user must not assume that all of the safety issues associated with its use have been described. Prior to use of this method, the user is responsible for establishing appropriate safety and health practices and determining the applicability of regulatory requirements.
The employee performing this method must be trained according to the safety guidelines specific to the job task and area of assignment. The employee must use all appropriate safety equipment referenced by the facility's safety guidelines. Copies of Material Safety Data Sheets (MSDS) are available from the Altria Safety Management intranet site, facility safety department or the area supervisor.

A. Scope

1. This test method describes a procedure to determine the weight-percent Oven Volatiles (OV) of smokeless tobacco products. It is specific to oven volatiles measured in a forced air oven at 100°C for 3 hours. Data is reported as % oven volatiles (OV).

B. Definitions

1. % Oven Volatiles – Gravimetric result based on a tobacco sample and measured by % weight loss in a forced air oven at three hours and 100 °C.
2. Pouch – Portion pack consisting of tobacco encased in porous paper.
3. OV dish – Round aluminum can with lid to be used for loose tobacco drying.
4. OV screen – Square wire gauze to be used for pouch drying alternative.

C. Responsibilities

1. Laboratory management shall ensure that personnel performing this method have demonstrated competence and documented proficiency.
2. Laboratory personnel are responsible for performing testing and documenting information as defined in this method. Any significant deviations from this method are to be documented and reported to laboratory management.

D. Equipment and Apparatus

1. Equipment and Apparatus Required
 - a. Balance - Electronic, analytical, resolution to 0.0001 g. (Refer to Balance Model List in [Attachment I](#))
 - b. Forced-Air convection oven capable of maintaining 100 °C +/- 1 °C. (Refer to Oven Model List in [Attachment II](#).)
 - c. Desiccant – Commercially available drying agent. (e.g., t.h.e®, Cat # EM-DX-0013-3, VWR Scientific). Refer to Desiccant Use in [Attachment III](#).

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- d. Desiccators - implosion proof and capable of holding approximately 24 aluminum moisture dishes.
 - e. Gloves
 - 1) Insulating type for working with hot surfaces at 212°F (100°C), (local laboratory or safety equipment supply company).
 - 2) Laboratory grade gloves, disposable for use during sample weighing.
 - f. Numbered aluminum moisture dishes with close fitting slip over covers approximate size: 3½" diameter x 2" height or equivalent (e.g., Model #AD9051, Dual Manufacturing).
 - g. Numbered wire gauze screens (5" X 5") (e.g., Model #15585B, Cat # JCO160205X5, VWR Scientific)
- 2. Instrument Setup
 - a. If power has been interrupted or balance is off, turn on balance and allow it to equilibrate for 1 hour. Follow the manufacturer's procedure for internal calibration and work instructions for verification. If the balance is already on, proceed with verification. If oven is off, turn oven on and allow it to equilibrate at 100°C +/-1°C for 1 hour prior to use. Verify that the oven is at 100°C ± 1°C if already powered on. Insure that the oven exhaust vent on top is open. Oven shelves should be place on bottom and middle positions to allow ample room for loading.
- 3. Instrument Maintenance
 - a. N/A
- E. Chemicals and Reagents
 - 1. N/A
- F. Sample Requirements
 - 1. USSTMC process samples are sufficiently homogeneous that result may be reported from single sample measurements.
 - 2. Samples are to be collected in accordance with appropriate sampling procedures. Refer to Work Instructions relating to the appropriate process step when sampling.
- G. Test Procedure
 - 1. Sample Handling
 - a. Samples should be stored in appropriate containers and transported to the lab for analysis within 24 hours. If samples will not be analyzed within 24 hours, they should be stored at 4°C for up to 7 days.
 - 2. Calibration
 - a. Verify that the balance calibration is current. Perform balance verification according to site-specific instructions. If verification weight fails, refer to manufacturer's instructions and perform a calibration. If the verification weight

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fails again, notify supervisor and do not use the balance. Verify that the oven is within the required temperature specifications. If oven temperature cannot be maintained, notify supervisor and do not use the oven. Eight-hour checks should be performed at least yearly or after any oven maintenance.

3. Analysis

- a. Determine the tare weight of the OV dish or OV screen
 - 1) Record the Identification (Typically a Laboratory Information Management System (LIMS) ID.
 - 2) Clear the weighing surface of any extraneous material and zero the balance by depressing the tare button. If a screen will be used in the next step, 3, a pedestal (e.g. an empty plastic can) should be used to isolate the screen from the balance pan. If this is the case, place the pedestal on the balance pan insuring that it is not touching the balance pan guard. Then, zero the balance by depressing the tare button.
 - 3) Place the empty OV dish and its cover, or alternatively an OV screen, which may be used for pouch material, on the balance.
 - 4) Record the tare weight and associate it with the can/screen number and Sample ID.
 - 5) Zero the balance with can or screen in place.
- b. Determine the sample "target wet weight" from the following:
 - 1) $5.0 \pm 0.5\text{g}$ for loose tobacco material.
 - 2) Pouch samples may be handled in different ways according to the facility needs.
 - a) Weigh the entire contents of 1 can (~15 pouches) into a can or on to a screen.
 - b) Weigh the number of pouches needed to obtain a sample weight of approximately 5g.
- c. Load the OV dish:
 - 1) If possible, gently mix the tobacco in the original OV sample container without opening the container.
 - 2) Load the dish or screen with tobacco from the OV sample container.
- d. Record the weight displayed on the balance.
 - 1) Discard any tobacco removed from dish for weight adjustment. Do not return to sample container.
 - 2) Minimize time during weight adjustment, as tobacco may pick up or lose moisture due to atmosphere.

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- 3) When sample weight is within the $\pm 0.5\text{g}$ weight limit, record the weight ("wet weight") on the worksheet to the nearest 0.0001g and place cover on the dish.
- e. Repeat steps a - d (above) for the next OV sample and continue until all samples have been loaded.
- f. Before opening the door to place the samples in the oven, assure oven is at proper operating temperature
- g. Remove the cover from each dish, place it upside down under the dish, and place the dish on the oven shelf. If screens are used, place them uncovered directly on the oven shelf.
- h. When the oven is loaded:
 - 1) Close oven Door.
 - 2) Start an elapsed time clock with an audible alarm to sound at the end of the three hour and 5 minute drying period.
 - 3) Alternatively, a label may be attached to the oven door with the removal time noted.
- i. The samples must be removed from the oven after 3 hours and 5 minutes (± 5 minutes).
- j. Using gloves transfer the dishes from the oven to the desiccators with lids replaced on top if using cans. Ensure the desiccator is sealed.
- k. Close oven door and remove time tag attached to the oven door if applicable.
- l. Allow the OV dishes to cool to room temperature (this should take approximately 30 minutes to 1 hour). Samples are to be removed and weighed as described below as soon as practical after they have cooled.
- m. Weigh each dish with its corresponding lid:
 - 1) Clear the weighing surface of OV dishes and any extraneous material.
 - 2) Zero the balance by depressing the tare button, if necessary. If a screen will be used in the next step, 3, a pedestal (e.g. an empty plastic can) should be used to isolate the screen from the balance pan. If this is the case, center the pedestal on the balance pan insuring that it is not touching the balance pan guard. Then, zero the balance by depressing the tare button.
 - 3) Place the dish and cover, or screen, on the balance and record the "dry weight" on the worksheet.
 - 4) Repeat steps (1-3) above for each dish.
- n. Discard the contents of the OV dishes in accordance with facility instructions. Remove any tobacco clinging to the OV dishes. It is generally not necessary or desirable to wash the OV dishes; a gentle wipe with a gloved finger or brush is usually sufficient to remove the tobacco materials. If a heavy residue is left in

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the dish, however, it will be necessary to wash the dish with a mild soap solution and thoroughly dry it before using the dish again.

4. Calculations and Reporting

- a. The percent OV of each sample is obtained by calculating the dry weight percent of the sample and subtracting from 100%.

$$\% \text{ OV} = 100 - (\text{Dry Weight of sample}) \div (\text{Wet Weight of sample})$$

Where:

Dry Weight of sample = (dry weight of container + sample) – (dry weight of container)

$$\text{Example: } 100 - ((62.3769 - 60.1293) \div (4.9947) * 100) = 55.00 \%$$

5. Quality Control and Acceptance Criteria

- a. Document all maintenance and calibrations for the balances and ovens used. Verify and record oven temperature. Log any desiccant replacement. Verify desiccant is within useful life per log and [Attachment III](#).

H. Related Documents

1. Validation Report Rev 02. 2011. Supplemental Validation of "Determination of Oven Volatiles in Moist Smokeless Tobacco".
2. Validation Plan Rev 02. 2011. Supplemental Validation of "Determination of Oven Volatiles in Moist Smokeless Tobacco".
3. Validation Report Rev 01. 2010. Determination of Oven Volatiles in Moist Smokeless Tobacco.
4. Validation Plan Rev 01. 2010. USSTMC % Oven Volatiles.

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I. Attachments

Attachment I

Validated Balance List

The following balances have been included in the validation study:

Nashville

Mettler Toledo AX204

Hopkinsville

Mettler Toledo AX204

Mettler Toledo XP204

Franklin Park

Denver Instrument AA-160

Fisher Scientific S-400

Balances used for %OV analysis should show practical equivalence to one of these listed balances.

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Attachment II

Validated Oven List

Validated oven list: The following ovens have been included in the validation study:

Nashville

Oven#1: Precision Scientific, Precision Model STM 135

Oven#2: Precision Scientific, Thelco Model 130 DM

Oven#3: Barnstead Lab Line, Imperial V Model 3488M

Oven#4: Barnstead Lab Line, Imperial V Model 3488M

Hopkinsville

Oven#1: Precision Scientific, Precision Model STM 135

Oven#2: Barnstead Lab Line, Imperial V Model 3488M

Franklin Park

Oven#2: Lindberg M01450SA

Oven#3: Thermolyne Oven Series 9000

Ovens used for %OV analysis should yield practically equivalent values relative to one of the listed ovens. Schuirmann's TOST will test for practical equivalence.

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Attachment III

Desiccant Use

Commercially available desiccant beads should be placed in ovens and dried each week for a minimum of 2 hours. The desiccant will be replaced at a minimum of 6 months and the end date for the shelf life noted in a logbook and on the desiccator.