Title: **Determination of Oven Volatiles, Precision™ Oven**

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Release / Revision Record for SOP

Status (Initial/Revision/ Retired)	Document Revision Number	Issue/Revision Date	Revision Identification	Revision Author
Initial Release	1	04/04/2014	PPI converted to SOP.	Tammy Blake
Revision	2	07/17/2017	Revision includes sample weight for (b) (4) samples. Minor administrative changes made for clarity. Harmonization with SOP 095-3371.	Hui Liu/J. Pierotti

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A. SCOPE

1. This procedure documents the process for determining Oven Volatiles (OV) in tobacco and tobacco products (including ground tobacco, smokeless tobacco, cigarette filler, pipe tobacco, and cigar filler) as measured by the loss of compounds volatilized under testing conditions using a Precision™ model oven.
2. This procedure is applicable to cigarettes taken directly from packs (Pack OV), cigarettes that have been equilibrated for at least 48 hours under ISO conditions (Equilibrated OV).

B. DEFINITIONS

1. OV (Oven Volatiles) – The amount of volatiles lost after 3 hours in a Precision™ oven at 100 ± 0.5 °C.
2. Precision™ oven – a forced-draft, mechanical-convection oven manufactured by ThermoFisher Scientific which is used to determine OV.
3. Monitor – homogenized tobacco sample with an established target and control limits used to monitor performance of the analysis.

C. RESPONSIBILITIES

1. The designated trained analyst performing the method is responsible to follow all steps of the procedure and to document and report any procedural deviations from the method to laboratory management.
2. Personnel using this test method are responsible for conducting the analysis in a manner consistent with the safety policies of ALCS.

D. VALIDATION

1. ST-TM-440-204, “Validation of Oven Volatiles (OV), Precision Model Ovens”, September 2006, Sichi, R., Cut Filler, Expanded, Shredded, Thieved and Monitor Tobaccos.

Summary Repeatability (r) & Reproducibility (R)

Sample	r Std.	r limit (95%)	R Std.	R Limit (95%)
Monitor	0.087	0.244	0.149	0.416
Shredded	0.156	0.436	0.203	0.568
Cut Filler	0.204	0.571	0.245	0.687
Thieved Tobacco	0.169	0.473	1.215	3.402

E. EQUIPMENT AND APPARATUS

1. Equipment Requirements and Apparatus
   a. Analytical Balance, 0.0001 g readability; e.g. Mettler XP204, Mettler AX205, or equivalent.

b. Desiccator – implosion proof, and large enough to hold 20 moisture dishes with lids. Usually two desiccators are required for each oven test.

c. Weighing OV Dishes, aluminum moisture dishes with close fitting slip covers - approximately 2½" diameter x 1¼" - 2½" height, available from Dual Manufacturing, Chicago, IL; part# AD-6344.

Note: Dishes and the corresponding lids are numbered manually with non-toxic permanent ink. Newly marked dishes should be baked in an oven at 100 °C for three hours prior to use. Dishes and lids should be washed as needed to remove tobacco residue, and then renumbered and baked. Record wash and bake information on the Dish Maintenance Form.

d. Oven Gloves – insulated for working with hot surfaces.

e. Brush – Wooster Golden Glo, blended nylon and polyester 2" paint brush, or equivalent.

f. Oven – Precision™ Scientific Model 605 MP, Thermo Scientific™ Precision™ High-Performance Oven 6050, ThermoFisher Scientific.

g. Cigarette Ripper, – instrument to slit the cigarette paper lengthwise, allowing the removal of tobacco. Sodim Cigarette Ripper, manual feed, Mebtech Inc., 1404 Dogwood Way, Mebane, NC 27302, 919-563-5989 (or equivalent).

2. Instrument Setup

a. Check the oven temperature twice during each OV test: before the samples are placed in the oven and just prior to removing the samples. The temperature must be maintained at 100 ± 0.5 °C. The actual temperature of the oven is located on the display for the 605 oven series currently in use. This display has been calibrated to the temperature measuring device.

F. CHEMICALS AND REAGENTS

1. Desiccant – Drierite™, anhydrous calcium sulfate, non-indicating (white) and indicating (blue) from WA Hammond, Drierite Company, LTD, parts #13005 and #23005.

G. SAMPLE REQUIREMENTS

1. Cigarettes / Cigars

a. Pack OV – Two packs are needed for determining Pack OV. Each sample is analyzed in duplicate. Remove all cigarettes from the pack. Remove the filters if they contain carbon. Rip the cigarettes using the cigarette ripper. Collect the tobacco in a plastic tray and replace the lid. If the samples are not to be analyzed (weigh-up) within two (2) hours, remove the papers from the

plastic tray, place the filler in a glass jar with the bar code and cap with a lid. Continue with the next sample.

b. Equilibrated OV – After the cigarettes are equilibrated per SOP 095-0029, place them in jars. In a conditioned room, remove the equilibrated cigarettes from the jar, remove filters if they contain carbon, and rip cigarettes using the cigarette ripper, collecting the tobacco in the plastic tray. Remove the papers from the filler. Discard the paper. Place the filler in a glass jar and cap with a lid. Continue with the next sample.

Note: Equilibrated cigarettes expire 10 days after the time and date of entry into the hotpack.

2. Other tobacco material and tobacco related articles – plastic bags or bottles containing sample are provided to the lab by the Analytical Operations group. Sample information can be found in LIMS.

H. PROCEDURE

1. Calibration

a. Verify the balance calibration in accordance with SOP 095-9483, “Calibration Verification.”

b. The oven calibration for temperature and air flow settings is coordinated in accordance with the IM&TE schedule according to ST-TM-440-201 “Installing and Calibrating Mechanical Convection Ovens”.

c. Desiccant:

1) The desiccant used is a combination of non-indicating (white desiccant) and indicating (blue desiccant). The blue desiccant must be visible and inspected for saturation prior to using the desiccator. If the majority of the indicating desiccant has turned from blue to any shade of purple, replace the desiccant. (Desiccant in shades of pink indicates an extreme state of saturation.) Record the date of desiccant replacement on each desiccator.

2) Use the following amounts when replacing the desiccant: combine approximately one quart of non-indicating (white) desiccant and approximately three tablespoons of indicating (blue) desiccant in each desiccator.

2. Sample Handling

a. The testing area should be maintained at 22 ± 2.0 °C. Verify the balance calibration, check the oven temperature, and check the desiccators.

b. Determine the sample "target wet weight" by adding one of the following sample weights to the tare weight of the dish:

   1) 10.0 ± 0.5 g for cigarette and cigar samples, unless otherwise specified.

   2) 5.0 ± 0.5 g for smokeless tobacco, unless otherwise specified.

   3) 2.0 ± 0.5 g for [redacted] (b) (4) coded projects, unless otherwise specified.

c. Duplicate 10-g monitor aliquots are analyzed in each oven batch to monitor the performance of the ovens. Open the can, empty the contents into a plastic bag, and thoroughly mix the filler. The monitor must be used within same day of opening. The monitor dishes are in positions 15-16 for the first set of dishes (1-40) and 55-56 for the second set of dishes (41-80), etc. (see Figure 1).

Note: Alternative weights may be appropriate in the following cases: limited sample quantity, sample type, customer request. Consult laboratory management or the receiving analyst under these circumstances.

3. Create an Oven Batch in LIMS

   a. Prepare monitor dishes and enter weights in LIMS.

   b. Prepare the test sample(s) and enter weights in LIMS.

      1) Enter the first dish number, always starting with the lowest odd-numbered dish.

      2) Ensure OV dish and balance weighing surface are clean.

      3) Zero the balance and place an empty OV dish with lid on the balance tray to obtain the container weight. Tare the balance.

      4) Gently mix the tobacco in the sample container by inverting the plastic tray or jar several times, prior to filling the dish with the appropriate amount of sample.

      5) Place the lid on the dish when removing it from the balance.

Note: If LIMS is out of operation, the steps listed above may be performed manually using the Form 099-3107 OV Manual Calculation Sheet.

4. Analysis

Caution: Always wear protective gloves (insulating type) when transferring dishes in and out of the oven. Portable sleeves are available for operators who desire to use them.

a. Loading Samples in the Oven

1) Every oven load must consist of exactly 40 dishes. If less than forty dishes are required for all samples to be tested, load additional empty dishes with the lids on into the oven so that there are forty dishes in the oven during the three hour drying period. It is not necessary to enter empty dishes into the computer, because absence of a weight on the batch indicates that the dish was empty.

2) Before opening the door to place the samples in the oven, ensure that the oven is at the proper operating temperature of 100 ± 0.5 °C and record the actual oven temperature on the Oven Log Sheet. If the oven is not at the target temperature, allow the oven to come to temperature before placing any samples in the oven. If the oven fails to come to temperature, notify lab management. Ovens must be free of debris as this can affect air flow which will affect the measurement of OV.

3) Remove the lid from a dish containing the sample, place it upside down under the dish, and place the dish on the appropriate oven shelf tray according to the following arrangement (Figure 1). Space dishes evenly across the width and depth of the shelf assuring they are not touching either side of the oven or each other. Load the top shelf first to minimize the loss of samples in the event of a spill. If a spill does occur, that dish must not be used for computing the OV. Record any spills, including sample data, in LIMS.

Top Shelf

39	29	19	9
37	27	17	7
35	25	15	5
33	23	13	3
31	21	11	1

---Oven Door---

Bottom Shelf

2	12	22	32
4	14	24	34
6	16	26	36
8	18	28	38
10	20	30	40

---Oven Door---

Figure 1. OV Dish Layout

4) Once the oven is loaded, close the door. Using the laboratory clock, record the time the oven was loaded to the nearest minute on the Oven Log sheet. Attach a label to the oven door that reads "Oven In-Use, Do Not Open Door."

b. Unloading the Oven

CAUTION: The following steps require the operator to touch and work near very hot surfaces. Always wear protective gloves (insulating type) when transferring dishes. Portable sleeves are available for operators who desire to use them.

1) Before unloading the oven, ensure that the stopper on the side of the desiccator lid is in place and secure. Check that the O-ring is in good condition and has an applied layer of silicone grease.

2) Remove samples from the oven after 3 hours ± 5 minutes.

3) Just prior to opening the door of the oven to remove the samples, record the time and the oven temperature on the Oven Log Sheet. Unload the bottom shelf first to minimize sample loss in the event of spillage. Replace the lid on the dish as it is removed from the oven, and place the dish in the desiccator. Close the desiccator after loading the dishes (a maximum of 20 dishes per desiccator).

4) Allow the OV dishes to cool to room temperature. This takes approximately thirty to forty-five minutes. After the cooling period, touch the surface of the dish to ensure adequate cooling has occurred prior to weigh-back. Samples are to be removed and weighed as described below as soon as is practical after they have cooled. Dishes must not be cooled overnight.

c. Reweigh Samples:

NOTE: All dishes in the desiccator may be removed at the same time providing all weighing is completed within five minutes from the time the dishes are removed. Keep desiccator covered at all times except to remove dishes for weighing.

1) Record the OV monitor values in the control chart as described in the Quality Control section and determine if there are any out of control conditions.

5. Quality Control

a. Prior to loading and removing dishes, check the oven temperature and initiate corrective action if temperature is outside acceptable range.

b. An Oven Check is required in the following cases:

1) following maintenance before the oven can be used for sample testing;

2) when the method is out of out of statistical control, as determined by monitor control chart evaluation, without a known root cause.

c. The Oven Check is accomplished by weighing 10 dishes of a freshly opened monitor sample. Place the dishes in the oven with 10 empty dishes. If the monitor results are acceptable as determined by monitor control chart evaluation, release the oven into service.

d. Following the sample analysis, plot the average of the two monitors on the QI Macros Control Chart and evaluate against the limits determined for the monitor. The rules and information on how to address out-of-control conditions are documented on the control charts.

   1) If the monitor indicates that the method is out of statistical control, reanalyze the monitor once to identify if the root cause is the monitor measurement.

   2) If the monitor value exceeds the limits:

      a) Do not authorize sample batch data. Add comments to the control chart to indicate the sample batch is rejected.

      b) Obtain reserve samples and process as described in Section H. Reweigh and retest samples in a different oven, using a new can of monitor. NOTE: In the event of an insufficient supply of samples or other unusual circumstances, contact laboratory management.

      c) Record out-of-control situations and corrective actions in the Control Chart.

   3) If the monitor results are acceptable, close and authorize the batch.

   4) Discard the contents of the OV dishes after authorizing data.

e. Precision ovens give results that are somewhat position dependent between the top shelf and the bottom shelf. The OV monitor difference, with shelf position, is approximately 0.1% OV which is not considered significant.

f. Typical practice is to load an oven the same work day. But on occasion, situations may arise due to time constraints where weighed OV dishes with monitors are allowed to remain in conditioned areas/ambient conditions up to 72 hours prior to actual oven loading.

6. Calculations

The percent OV for each dish is calculated as:

$$\text{OV (\%)} = \frac{\text{Sample Wt. Original - Sample Wt. After Drying}}{\text{Sample Wt. Original}} \times 100$$

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I. REFERENCES

1. Validation of Oven Volatiles (OV), Precision Model Ovens. August 27, 2010. In support of test method ST-TM-440-204.
2. ST-TM-440-204, “Oven Volatiles (OV), Precision Model Ovens.”
3. ST-TM-440-201, “Installing and Calibrating OV Ovens.”
4. SOP 095-0061Generalized Procedure for Determining Uncertainty.
5. SOP 095-0029 Sample Handling and Conditioning
6. WI 097-1108 Sample Preparation
7. SOP 095-0037 Results Verification

J. FORMS

1. 099-3106 Dish Maintenance Form
2. 099-3107 OV Manual Calculation Sheet
3. 099-3114 CDC Oven Log Worksheet