

Title: Determination of Oven Volatiles in Smokeless Tobacco Products by Microwave Moisture Analyzer	Control #: ST-TM-440-264	Revision #: 3.1
	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 for rapid microwave moisture determination of finished smokeless tobacco products, intermediate process stage and other smokeless tobacco matrices. Results are reported as percent.

B. Definitions

1. Oven Volatiles (OV) - Compounds in tobacco that are evolved by exposure in a mechanical convection oven at 100°C for 3 hours.

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. CEM SMART System Microwave Moisture/Solids Analyzer, CEM Corporation, Matthews, NC.
 - b. CEM Square Sample Pads, Part # 200150, CEM Corporation, Matthews, NC.
 - c. Intelli-Temp Calibrator, CEM Corporation.
 - d. Petri Dishes, Pyrex 100mm x 10mm, Fisher Scientific, Cat # 08-747B, or equivalent, not to exceed 40g
 - e. Disposable Pipettes
2. Instrument Setup
 - a. Refer to [Attachment II](#) for parameter settings.

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3. Instrument Maintenance
 - a. Refer to instrument manual.
- E. Chemicals and Reagents
 1. Chemicals Required
 - a. Deionized Water
 - b. Sodium Chloride 20% (w/v) aqueous solution, Fisher Scientific, Cat. # 72186-16.
 2. Reagent Preparation
 - a. N/A
 3. Standard Preparation
 - a. N/A
- F. Sample Requirements
 1. This method requires approximately 7 g/sample for each % volatile determination.
 - a. Turns, Out-of-Cure, Finish, and Finish Loose Pouch materials: Weigh 7.25g (\pm 0.25g) per sample. Refer to [Attachment I](#) for suggested loading pattern.
 - b. Dry Pouch – (Cope Pouch and Skoal Pouch): Place 7 pouches per sample. Refer to [Attachment I](#) for suggested loading pattern.
 - c. Dry Pouch – (Bandits): Place 9 pouches per sample. Refer to [Attachment I](#) for suggested loading pattern.
 - d. Flotop and Protop Solutions: Weigh 2.5g (\pm 0.25g) per sample.
 2. Obtaining Samples:
 - a. Collect samples using containers, techniques and sample volumes specified in this method, applicable sampling methods and/or facility requirements or requests.
- G. Test Procedure
 1. Sample Handling
 - a. Samples must be transported to and stored in the lab in approved containers.
 - b. Sample containers should not be opened until the sample is ready to be prepared.
 2. Calibration
 - a. Internal Balance Calibration

NOTE: Balance calibration will be performed at the beginning of each production day.

 - 1) Open the SMART System cover using the gray CEM latch.

NOTE: DO NOT open or close the system without using the gray latch.

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- 2) Verify that the balance is clean; clean if required. Expect an audible click.
- 3) From the Main Menu, Select "Set Up" by pressing "4" on the control pad.
- 4) Select "Calibrate Balance" by pressing "7" on the control pad.
- 5) Select "Tare".
- 6) Open the cover and add 10g weight onto the balance. Close cover and press "Ready".
- 7) The instrument screen will display "calibration complete".
- 8) Open the cover and remove the weight.
- 9) The "Exit" button will return to the previous screen. Press Exit Button twice and return to CEM Main Menu.
- 10) Record appropriate data in calibration log book or LIMS database.

3. Analysis

a. Load Method

- 1) Using the "CEM SMART System Methods to Use for Smokeless Products" list in [Attachment III](#), select the appropriate SMART System method.
- 2) From the Main Menu, Select "Load Method" by pressing "3" on the control pad.
- 3) Select the desired method by pressing the number beside the chosen method on the control pad.
- 4) Once selected, the method appears at the bottom of the main menu.
NOTE: If the product to be tested calls for the use of the SMART System method "H2O FOUR" set the Bias Adjustment parameter to the value for that product as listed in [Attachment III](#).
- 5) Press the "Ready" button to move to the testing screen.

b. Sample Analysis

- 1) Open the cover. Place two (2) sample pads and petri dish on the balance pan and close the cover with the gray latch. Expect an audible click.
- 2) Press the "Tare" button.
- 3) Open the cover and add sample to the petri dish.
- 4) Close the cover and press the "Start" button. The instrument will beep and print a result when the analysis is complete.
- 5) Open the cover and remove the petri dish. The same sample pads may be used for each petri dish.

4. Calculations and Reporting

a. N/A

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5. Quality Control and Acceptance Criteria

a. System Verification with 20% Salt Solution

NOTE: Verification will be performed at the beginning of each production day for error measurement determination.

- 1) From the Main Menu, Select "Load Method" by pressing "3" on the control pad.
- 2) Load "H2OVER".
- 3) Open the cover and add two sample pads to the balance. Close cover and press "Ready". Select Tare.
- 4) Shake the 20% salt solution thoroughly.
- 5) Open the cover and add 1.00 g (\pm 0.25 g) of solution onto pads.
- 6) Close the cover.
- 7) Select "Start". The instrument will beep and print a result when the analysis is complete.
- 8) Record result in appropriate calibration logbook or LIMS database.
- 9) Perform "Recheck" if value has shifted more than 1% from previous calibrations. Rechecks may be performed at technician's discretion for shifts less than 1%. Record rechecks in appropriate calibration logbook or LIMS database.
- 10) Open the cover, and remove and discard the pads.
- 11) Repeat at the end of each shift.

H. Related Documents

1. Validation Report Rev 01. 2010. Determination of Oven Volatiles by CEM Smart5 Microwave System.
2. ST-TM-440-204 Determination of Volatiles by Mechanical Convection Oven
3. ST-TM-440-254 Determination of Volatiles in Smokeless Tobacco Products by Mechanical Convection Oven
4. CEM SMART System Products List
5. SMART System Operation Manual

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Attachments

Attachment I

Example Petri Dish Sample Loading Patterns

Long Cut Tobacco



Fine Cut Tobacco

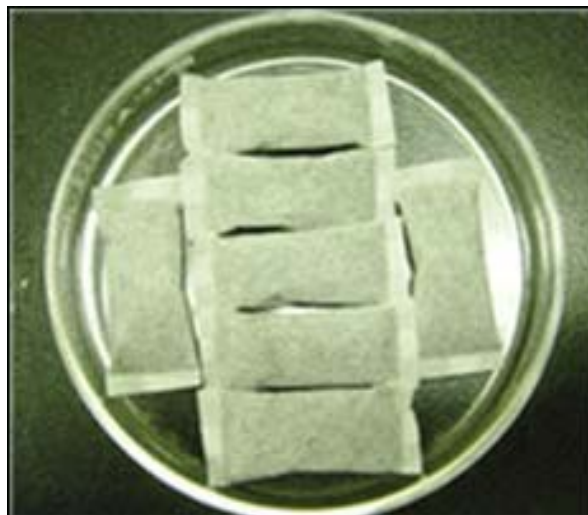


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Finish Loose Pouch Tobacco

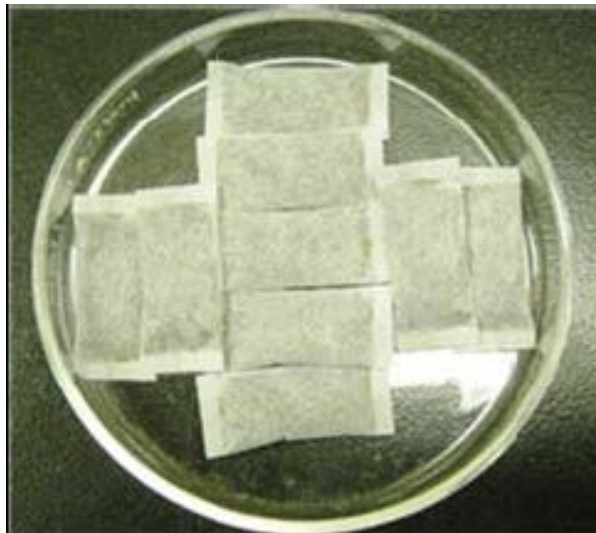


Cope Pouch and Skoal Pouch



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Bandits



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Attachment II

CEM SMART System Parameter Settings

SMART System Method	H2OONE	H2OTWO	H2OTHREE
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	100	100	100
Delta Wt (mg)	0.5	0.5	0.5
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	0.00%	0.00%	0.00%
Max Temp C	125°C	135°C	140°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	10.00
Wt Comp	OFF	OFF	OFF

SMART System Method	H2ODFONE	H2ODFTWO	H2ODFTHREE
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	100	100	100
Delta Wt (mg)	0.3	0.3	0.3
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	+ 1.45%	+ 1.00%	+ 0.50%
Max Temp C	120°C	120°C	120°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	10.00
Wt Comp	ON	ON	ON

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SMART System Method	H2OVER	H2O TURNS	MONITOR
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	100	95	100
Delta Wt (mg)	0.2	0.5	0.2
Delta Time (sec)	8	8	10
Max Time (mins)	10	10	10
Bias (%)	0.00%	0.00%	0.00%
Max Temp C	120°C	126°C	110°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	4.00
Wt Comp	OFF	OFF	ON

SMART System Method	H2OAPPCH	H2OBAPPPCH	H2OBBCHYPCH
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	85	85	85
Delta Wt (mg)	0.3	0.3	0.3
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	+ 0.30%	+ 0.30%	+ 0.40%
Max Temp C	105°C	105°C	105°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	10.00
Wt Comp	OFF	OFF	OFF

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SMART System Method	H2OBMTPOCH	H2OBRYPOCH	H2OBWGPCH
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	85	85	85
Delta Wt (mg)	0.3	0.3	0.3
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	+ 0.90%	+ 0.40%	+ 1.40%
Max Temp C	105°C	105°C	110°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	10.00
Wt Comp	OFF	OFF	OFF

SMART System Method	H2O FOUR	STD SOLUTION
Calculation Mode	Moisture/Solids	Moisture/Solids
Time Parameter	Set Time	Set Time
Test Mode	Standard	Standard
Power (%)	100	100
Run Time (mins)	10	3.5
Bias (%)	Refer to Attachment III	0.00
Max Temp C	140°C	110°C
Result Range	MOISTURE	MOISTURE
Min Result (%)	0.00%	0.00%
Max Result (%)	100.00	100.00
Min Wt Range (g)	2.00	2.00
Max Wt Range (g)	10.00	4.00
Wt Comp	OFF	ON

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SMART System Method	H2OCITPCH	H2OCPCH	H2OMTPCH
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	85	85	85
Delta Wt (mg)	0.3	0.3	0.3
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	+ 0.40%	+ 0.25%	+ 0.60%
Max Temp C	105°C	105°C	105°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	10.00
Wt Comp	OFF	OFF	OFF

SMART System Method	H2OSTPCH	H2OSKWGPCH	H2OWETPCHONE
Calculation Mode	Moisture/Solids	Moisture/Solids	Moisture/Solids
Time Parameter	Constant Weight	Constant Weight	Constant Weight
Test Mode	Standard	Standard	Standard
Power (%)	85	85	100
Delta Wt (mg)	0.3	0.3	0.3
Delta Time (sec)	8	8	8
Max Time (mins)	10	10	10
Bias (%)	+ 0.30%	+ 0.30%	-1.00%
Max Temp C	107°C	105°C	125°C
Result Range	MOISTURE	MOISTURE	MOISTURE
Min Result (%)	0.00	0.00	0.00
Max Result (%)	100.00	100.00	100.00
Min Wt Range (g)	2.00	2.00	2.00
Max Wt Range (g)	10.00	10.00	15.00
Wt Comp	OFF	OFF	ON

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Attachment III

CEM SMART System Methods to Use for Smokeless Products

(b) (4)

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Attachment IV

Validation of New Sample Matrices

Development of new SMART System OV methods may be needed for new or modified finished products, for intermediate process stages or for other matrices where OV measurements are required. The following is the process to be followed to assure that any new or modified SMART System OV methods are properly validated and considered suitable for use in the laboratory.

1. The facility responsible for the new or modified product or intermediate process should validate SMART System OV method for the new matrix.
 - a. Obtain sufficient sample for mechanical convection oven ("Oven method") and SMART System testing at the designated facility.
 - b. Perform three (3) replicates of Oven Volatiles by an Oven test method (ST-TM-440-204 Determination of Volatiles by Mechanical Convection Oven or ST-TM-440-254 Determination of Volatiles in Smokeless Tobacco Products by Mechanical Convection Oven). Determine the average of the three replicates and use as the reference OV value.
2. Begin the SMART System validation process by performing a series of "scoping runs" using one or more of the existing SMART System methods that may be appropriate for the matrix to be tested. The purpose of the scoping runs is to find the SMART System method that gives OV results closest to the Oven method reference OV value.
 - a. If the results of the scoping runs using the existing SMART System methods give OV results within ± 0.25 OV units of the Oven method reference OV value, complete the validation as follows:
 - 1) Based on the results of the scoping runs, select the most appropriate SMART System method for the new matrix validation.
 - 2) Use the method selected to perform six (6) replicates of OV for the sample.
 - 3) Calculate the average of the six replicates and use as the SMART System OV value.
 - 4) Determine the difference between the Oven method reference OV value and the SMART System OV value.
 - 5) If the SMART System OV value is within ± 0.25 OV units of the Oven method reference OV value the methods are considered to deliver equivalent results.
 - b. If the results of the scoping runs using the existing SMART System methods give OV results greater than ± 0.25 OV units as compared to the Oven method reference OV value, it may be necessary to develop a SMART System method that uses a Bias Adjustment.
 - 1) Based on the results of the scoping runs, select the most appropriate SMART System method for the new matrix validation.
 - 2) Use the method selected to perform six (6) replicates of OV for the sample.

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- 3) Calculate the average of the six replicates and use as the SMART System OV value.
 - 4) Determine the difference between the Oven method reference OV value and the SMART System OV value. The difference between the two methods will be used as the Bias Adjustment parameter that will be entered in the SMART System method designated for that matrix.
 - 5) If the SMART System OV value (including the Bias Adjustment) is within ± 0.25 OV units of the Oven method reference OV value the methods are considered to deliver equivalent results.
3. Upon completion of testing for a new matrix validation, all analytical data and supporting documentation will be maintained by the facility that developed the method.