

Title: Rapid Determination of pH in Smokeless Tobacco	Control #: ST-TM-410-314	Revision #: 2.1
	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 process to measure the pH of smokeless tobacco aqueous extracts (5 g/100 mL nominal). This test method applies to dry flour, out of cure and finished moist smokeless tobacco products.

B. Definitions

1. pH - Numerical expression of the hydrogen or hydronium ion concentration in an aqueous solution. Mathematically, pH is expressed as the $-\log [H^+]$, where $[H^+]$ is the concentration of hydrogen ions in moles per liter.

C. Responsibilities

1. Laboratory management shall ensure that personnel performing this method have demonstrated the ability to properly perform the method.
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. Mettler Toledo SevenMulti S47 Dual Channel pH/Conductivity Meter, Fisher Scientific Cat. No. 01-913-804 or a meter demonstrated to be equivalent may be used. pH meter equivalency is to meet the following specifications:
 - 1) Resolution: 0.001 pH
 - 2) Relative Accuracy: + 0.005 pH
 - 3) Automatic Temperature Control (ATC) capability and an ATC probe or an electrode with a built-in ATC sensor
 - 4) Optional:
 - a) Automatic buffer recognition is preferred.

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- b) An RS232 output (for connection to pc or LIMS systems).
 - c) Conductivity module is not required for this method.
 - b. pH Electrode, Thermo Orion ROSS Sure-Flow, 8272 BN, Fisher Scientific Cat. No. 13-642-566 or equivalent
 - c. PT 1000 Temperature Probe, Mettler Toledo PT1000, Fisher Scientific Cat. No. 01911506 or equivalent
 - d. Electrode Storage Solution for Ross pH Electrode
 - e. Balance, electronic, top loading; having minimum readability of 0.01g
 - f. Magnetic stirrer(s), capable for maintaining a stir rate of up to ~600 rpm
 - g. Beaker or Erlenmeyer Flask, polypropylene or glass, with the capacity to match sample size and extraction volume. (e.g., For 100 mL extraction volume, preferred capacity is 150 mL) Possible vessel sources include Fisherbrand Polypropylene Disposable Beakers, 150 mL, Fisher Scientific Cat. No. 01-291-11
 - h. Magnetic stir bars, appropriate for the size of container and nature of the sample, Fisher Scientific Cat. No. 14-513-98 or equivalent
 - i. Wipes, lint-free (e.g. Kim Wipes® or equivalent)
 - j. Spatula or laboratory tweezers (for sample weighing)
 - k. Cylinder, graduated, 100 mL capacity, "To deliver"-type, or calibrated bottle-top dispenser capable of achieving equivalent accuracy
 - l. Sample container, 4 ounce capacity with hinged-lid, or equivalent container suitable for use during pH calibration or verification
 - m. Timer
 - n. Optional:
 - 1) Bar code reader
 - 2) Personal computer
 - 3) pH Software WinWedge (Standard Addition) or Mettler Toledo LabX; for capturing pH data
- 2. Instrument Setup
 - a. Setup the pH meter and attachments according to the manufacturer's specifications and operating manual.
 - b. Store buffers and water used for extractions at room temperature.
- 3. Instrument Maintenance
 - a. Refer to instrument manuals.
- E. Chemicals and Reagents
 - 1. Chemicals Required

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- a. NIST Traceable pH Buffers (Calibration Standard Solutions),
 - 1) Thermo Scientific Orion Application Solutions
 - a) pH 4.01 buffer, 500 mL, Fisher Scientific Cat. No. 13-301-133
 - b) pH 7.00 buffer, 500 mL, Fisher Scientific Cat. No. 13-641-857
 - c) pH 10.01 buffer, 500 mL, Fisher Scientific Cat. No. 13-079-319
 - 2) Intermediate Quality Control Buffer Solutions
 - a) Fisher pH 5.00, Fisher Scientific Cat. No. SB102-500
 - b) Fisher pH 8.00, Fisher Scientific Cat. No. SB112-500

NOTE: NIST traceable pH buffers from other manufacturers are acceptable for use.
- b. Milli Q ® Water, or equivalent
2. Reagent Preparation
 - a. N/A
3. Standard Preparation
 - a. N/A
- F. Sample Requirements
 1. Collect samples using containers, techniques and sample volumes specified in this method, applicable sampling methods and/or facility requirements.
 2. Store production samples in an airtight container.
- G. Test Procedure
 1. Environmental Requirements
 - a. Temperature Limits: The system automatically compensates for temperature by using the ATC probe.
 2. Sample Handling
 - a. N/A
 3. Calibration
 - a. Verify balance calibration per test method ST-TM-910-100 Balance Calibration.
 - b. Program the pH meter setup parameters.
 - 1) pH meter resolution - high resolution (for reading to three decimal places)
 - 2) Stability - Normal
 - 3) Endpoint Selection - Auto
 - 4) Calibration Mode - Segmented
 - 5) pH Buffer Mode – Customized Buffer Group, according to the table below

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Customized pH Buffer Settings

Temp (°C)	Buffer 1	Buffer 2	Buffer 3
	Nominal pH 4	Nominal pH 7	Nominal pH 10
10.0	4.000	7.060	10.180
20.0	4.000	7.010	10.060
30.0	4.020	6.980	9.970

- 6) Flush Sure-Flow electrode junction with at least 1-2 mL of filling solution prior to calibration.
 - 7) Fill electrode chamber with filling solution and remove any air bubbles by gently shaking or tapping the electrode.
 - 8) Re-equilibrate electrode in electrode storage solution for at least five minutes before proceeding.
- c. Calibrate pH meter prior to analyzing samples using buffers for pH 4, 7, and 10 by following instructions in the meter's user manual.

NOTE: Calibrations should be performed once per shift, or prior to use or, if necessary, after failed Quality Control Buffer Samples (Refer to "[Quality Control and Acceptance Criteria](#)" section in this document").

- 1) Add each buffer to a clean, dry beaker or sample container containing a stir bar that will provide a slight vortex while mixing. Do not allow the stir bar to contact the electrode or damage will result.
- 2) Rinse pH electrode thoroughly and blot dry with a clean wipe. Do not wipe electrode to reduce the chance of error due to polarization.
- 3) Lower electrode into the cup so that the Sure-Flow Junction (and temperature probe) is covered by the solution, allowing 30 seconds for the electrode to equilibrate.
- 4) While measuring the buffer, confirm that the internal filling solution is at least one inch above the solution to be measured and the Sure-Flow Junction (reference is covered by the solution).
- 5) Read pH.
- 6) Verify quality of the calibration using the following criteria:
 - a) Calibration slope: 92 –102%
 - b) Offset: ± 30 mV

NOTE: If either the slope or Offset is outside of these ranges, do not proceed with sample analysis and consult the meter's User Manual and/or departmental technical leadership for troubleshooting.

- c) pH 7 buffer offset: 0 ± 30 mV
- 7) Record calibration slope, offset and pH buffer lots.

4. Analysis

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- a. Analyze the Quality Control (QC) buffers.
 - 1) Pour appropriate amount of QC buffer into a sample container (or equivalent).
 - 2) Add stir bar and stir at a rate to achieve a slight vortex while measuring.
 - 3) Rinse pH electrode thoroughly and blot dry.
 - 4) Lower electrode into the cup so the Sure-Flow Junction (and temperature probe) is covered by the solution, allowing 30 seconds for the electrode to equilibrate.
 - 5) Read pH.
 - 6) Acceptance criterion: ± 0.04 pH units from the certified pH at the prescribed temperature. If the QC buffer value is not within the acceptance limits, then recalibrate.
 - 7) If the QC buffer value is not within the acceptance limits after performing an additional calibration, do not proceed with sample analysis and begin troubleshooting. Do not resume sample analysis until the problem is resolved.
 - 8) Measure pH of the appropriate QC buffers after every tenth sample and after last sample of the sample set.
 - 9) The same aliquot of the QC buffer may be used throughout the same day as long as it is covered between uses and continues to meet the acceptance criteria.
- b. Analyze the test samples.
 - 1) Weigh 5.00 ± 0.05 g tobacco sample into a clean plastic extraction cup.
 - 2) Rinse electrode thoroughly and blot dry.
 - 3) Add stir bar to the sample cup.
 - 4) Using a 100 mL graduated cylinder or bottle-top dispenser, add 100 ml Milli Q water and begin timing.
 - 5) Place sample on a magnetic stir plate and stir mixture, forming a slight vortex.
 - 6) Mix sample for a minimum of 30 seconds for out of cure and finished products. Stir dry flour samples ~7-8 minutes, with an additional swirl to fully wet the tobacco.
 - 7) Lower electrode into tobacco mixture so the bulb and temperature probe are covered by the solution.
 - 8) Read solution pH at 5 minutes for out of cure and finished products. Read solution pH at 10 minutes for dry flour. Record results.
 - a) pH result to the nearest 0.01 pH unit
 - b) Solution temperature to the nearest 0.1°C

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- 9) Rinse pH electrode and temperature probe thoroughly between all successive measurements and before reading the QC buffer to assure proper cleaning and avoid any potential contamination affecting pH determination.
 - 10) Upon completion of testing, store electrode as specified by manufacturer
5. Calculations and Reporting
- a. The pH value is read directly from the pH meter display. If the optional computer and software are used, the pH value is transferred to an Excel spreadsheet.
6. Quality Control and Acceptance Criteria
- a. Meter and Electrode Performance
 - 1) Follow the manufacturer's instructions regarding the care and use of the equipment.
 - 2) Inspect, clean, and maintain the electrode according to the electrode manual. Electrode should be cleaned weekly per the manufacturer's recommendation and documented.
 - b. Sample Set Acceptance Criteria
 - 1) Sample readings are acceptable if they are preceded by a passing calibration and bracketed by the appropriate Quality Control Buffer meeting the acceptance criteria. Refer to the "Analysis" section of this document for additional information.
 - 2) Repeat analysis of any samples not meeting these criteria.
 - 3) If a quality control sample fails in the middle of a sample set without an assignable cause, recalibrate the meter and do not resume analyzing samples until the meter is confirmed to be working properly.
- H. Related Documents
1. Validation Report Rev 02. 2011. Rapid Determination of pH in Smokeless Tobacco. - Supplement for (b) (4)
 2. Validation Plan Rev 02. 2010. Rapid Determination of pH in Smokeless Tobacco – Supplement for (b) (4)
 3. Validation Report Rev 01. 2010. Rapid Determination of pH in smokeless Tobacco.
 4. Validation Plan Rev 01. 2010. Rapid Determination of pH in Smokeless Tobacco.
 5. ST-TM-910-100 Balance Calibration.
- I. Attachments
1. N/A