Title: Determination of pH in Smokeless Tobacco	Control #: ST-TM-410-334	Revision #: 1.1
	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 a smokeless tobacco aqueous extract (5g /100mL). It applies to tobacco samples generated throughout the USSTC manufacturing process.

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 #. 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-I 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, 8172 BN or 8272 BN, Fisher Scientific # 13-642-566.

c. Thermo Orion 917007 ATC probe, modified by end-user with RCA connector, Fisher Scientific # 136411119 or equivalent.

d. Electrode Storage Solution

e. Balance, electronic, top loading; having minimum readability of 0.01g

f. Magnetic stirrer(s), capable for maintaining a stir rate of up to ~300-400 rpm.

g. Griffin-type 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 # 01-291-11.

h. Magnetic stir bars of appropriate size.

i. Cylinder, graduated, 100 mL capacity, Class B, “To deliver”-type, or calibrated bottle-top dispenser capable of achieving equivalent accuracy.

j. Optional:

1) Bar code reader

2) Personal computer

3) Mettler Toledo LabX software for capturing pH data.

2. Instrument Setup

a. Install the pH meter and attachments according to the manufacturer’s specifications and the operating manual.

b. Buffers and water used for extraction should be stored at room temperature.

3. Instrument Maintenance

a. Refer to manufacturer’s instructions.

E. Chemicals and Reagents

1. Chemicals Required

a. NIST Traceable pH Buffers (Calibration Standard Solutions),

1) Thermo Scientific Orion Application Solutions

a) pH 4.01 buffer, 500 mL, Fisher Scientific # 13-301-133

b) pH 7.00 buffer, 500 mL, Fisher Scientific # 13-641-857

c) pH 10.01 buffer, 500 mL, Fisher Scientific # 13-079-319

2) Intermediate Quality Control Buffer Solutions

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a) pH 5.00 buffer, 500 mL, Fisher Scientific # 13-640-8

b) pH 8.00 buffer, 500 mL, Fisher Scientific # SB112-1

NOTE: NIST traceable pH buffers from other manufacturers are acceptable.

b. Milli-Q Water.

2. Reagent Preparation

a. N/A

3. Standard Preparation

a. N/A

F. Sample Requirements

1. Samples should be stored in well-sealed containers.

G. Test Procedure

1. Sample Handling

a. N/A

2. Calibration

a. Verify balance calibration per test method ST-TM-910-100 Balance Calibration or other site-specific procedure as required.

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) Alarm Limits function – On; maximum offset 30mV, minimum offset -30mV, maximum slope 102%, minimum slope 92%.

5) Calibration Mode - Segmented.

6) pH Buffer Mode – standard buffer group 1: pH 1.68, 4.01, 7.00 and 10.01 at 25 °C

c. Calibrate the meter prior to analyzing samples using buffers for pH 4, 7, and 10 by following the instructions in the meter's user manual.

NOTE: Calibrations should be performed once per shift as needed, or, if necessary, after failed Quality Control Buffer Samples (see section "Quality Control and Acceptance Criteria").

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) Flush the Sure-Flow electrode junction with at least 1-2 mL of filling solution prior to calibration.

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3) Fill the electrode chamber with filling solution and remove any air bubbles by gently shaking or tapping the electrode.

4) Rinse the pH electrode thoroughly and blot dry with a clean wipe.

5) Lower the electrode into the cup so that the Sure-Flow Junction (and temperature probe) is covered by the solution

6) 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 junction) is covered by the solution.

7) Read the pH.

8) Repeat for remaining buffers.

9) Verify the quality of the calibration using the following criteria:

   a) Calibration slope: 92 –102%.

   b) Offset = ± 30mV

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

10) Record the calibration slope, offset, instrument, and operator initials.

3. Analysis

   a. Analyze the Quality Control (QC) buffers.

      1) Choose the QC buffer which best matches the samples to be analyzed.

      2) Pour the appropriate amount of QC buffer into a sample container (or equivalent).

      3) Add a stir bar and stir at a rate to achieve a slight vortex while measuring.

      4) Rinse the pH electrode thoroughly and blot dry with a clean wipe.

      5) Lower the 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.

      6) Read the pH.

      7) 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, the recalibrate.

      8) 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.

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9) Measure the pH of the QC buffers after every tenth sample and after the last sample of the sample set.

10) 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 ± 0.05g of tobacco sample into a clean plastic extraction cup.

2) Rinse the electrode thoroughly and blot with a clean dry wipe.

3) Add a magnetic stir bar to the sample cup.

4) Using a 100 mL graduated cylinder or bottle-top dispenser, add 100 ml of Milli Q water.

5) Place the sample on a magnetic stir plate and stir the mixture vigorously (approximately 300-400 rpm recommended).

6) Mix the sample for 60 minutes and analyze for pH as quickly as possible.

7) Lower the electrode into tobacco mixture so that the bulb and temperature probe are covered by the solution.

8) While stirring as in step 5, measure the pH of the solution. Record the Results.

a) pH result to at least two decimal places

b) Solution temperature to the nearest 0.1°C.

9) Rinse the pH electrode and temperature probe thoroughly between all successive measurements and before reading the QC solution to assure proper cleaning and avoid any potential contamination affecting pH determination.

10) Upon completion of testing, store the electrode as specified in the electrode manual.

NOTE: Periodically inspect, clean, and maintain the electrode according to the electrode manual.

4. Calculations and Reporting

a. N/A

5. Quality Control and Acceptance Criteria

a. Once per week, clean the electrode.

1) Soak for at least 15 minutes in Electrode Cleaning solution

2) Open the electrode junction and flush electrode filling solution

3) Rinse the electrode junction with Milli-Q water to remove any remaining cleaning solution

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4) Refill the electrode filling solution and rewet junction as described above.

5) Allow the electrode to soak in electrode storage solution for at least 60 minutes before use.

b. Sample Set Acceptance Criteria

1) Sample readings are acceptable if they are preceded by a passing calibration and bracketed by Quality Control Buffers meeting the acceptance criteria described previously.

2) Repeat the 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 01. 2010. Determination of pH for Tobacco Samples – USSTC.
2. Validation Plan Rev 01. 2010. Determination of pH for Tobacco Samples – USSTC.
3. ST-TM-410-304 Determination of pH in Smokeless Tobacco – language from ST-TM-410-304 has been both directly transcribed and / or adapted for this test method.

I. Attachments

1. N/A