pH Analysis of Liquid Fertilizer Samples

1. Scope:

This procedure is to be used for the determination of pH in liquid fertilizers.

2. Principle:

Fertilizer samples are prepared as described in RA-SP-SMPL-PREP.

3. Safety:

The SDS for all chemicals shall be read before performing any part of this method. Gloves, safety glasses, and a lab coat shall be worn when handling hazardous materials and reagents.

When using chemicals and solvents, comply with the instructions of the producer and general lab safety rules.

Always wipe off splashed liquids immediately. The instrument is not waterproof.

4. Equipment, Reagents, and Supplies (equivalents are acceptable):

4.1 Mettler Toledo pH Meter (Model # Seven Multi)
4.2 pH Electrode (WTW SenTix Part # B214216001
4.3 3M Potassium chloride (KCl) solution (Source: NIST)
4.4 pH 1.68, 2.00, 4.00, 7.00, 10.00 standard buffer solutions
4.5 Magnetic stir plate
4.6 Magnetic stir rods
4.7 Mechanical sample shaker
4.8 50mL polypropylene tubes or equivalent
4.9 100mL disposable plastic cups
4.10 DI water
4.11 400mL beaker

5. Calibration:

5.1 Check pH buffer solutions for expiration date. If past the best by date, discard and use new solution that has not expired.
5.2 Pour ~30mL of each buffer solution into its own 50mL polypropylene tubes.
5.3 Remove pH electrode from the reservoir bottle and uncover the hole near the top of the pH electrode.

5.4 Check the amount of potassium chloride solution in pH electrode. If the electrode is not ¾ filled with potassium chloride, refill by using a disposable pipette to add potassium chloride solution through the hole.
5.5 Rinse pH electrode thoroughly with DI water from a squeeze bottle and collect water waste in a beaker. Gently dab off (do not wipe) excess water from pH electrode with Kimwipes until the pH electrode is dry.
5.6 Place pH electrode in the 50mL polypropylene tube containing pH buffer with the starting pH of the curve and press “CAL”.
5.7 When the endpoint stops changing, press “CAL” to save the reading. An “√A” message will appear on the bottom right-hand corner of the display. Note this reading on the pH worksheet and refer to step 5.5 to clean the pH electrode.
5.8 Repeat steps 5.5 to 5.7 for each point on the calibration curve. There needs to be at least three calibration pH points.
    5.8.1 Calibrate at pH 1.68, 4.0, 7.0, and/or 10.0 for fertilizer samples.
5.9 Use the buttons to the right of the display screen to hit “Next” and note the slope and offset on the pH worksheet, then hit “Save” to save the new calibration settings.
    5.9.1 The acceptable slope is 0.95-1.05. Recalibrate with fresh pH buffer solution if it fails.
5.10 Record the slope and Offset value.

6. Check Buffers:

6.1 Two check pH tests should be performed both BEFORE and AFTER the analysis of fertilizer samples. The same ~30mL aliquots of pH buffer solution used for calibration can be used as the check buffer.
    6.1.1 For acidic samples, use check buffers at 2.00 pH and 4.00 pH.
    6.1.2 For basic samples, use check buffers at 4.00 pH and 7.00 pH.
    6.1.3 If using 2.00 pH as a check buffer, pour ~30mL of the 2.00 pH buffer solution into a 50mL polypropylene tube and use this aliquot when obtaining a reading. Check to ensure the buffer solution has not exceeded its best by date.
6.2 Rinse pH electrode thoroughly with DI water from a squeeze bottle and collect water waste in a beaker. Gently dab off (do not wipe) excess water from pH electrode with Kimwipes until the pH electrode is dry.
6.3 Place the pH electrode in the 50mL polyethylene tube containing the first check buffer (refer to 6.1.1 or 6.1.2) and press the green “READ” button. Wait for the endpoint to stop changing and an “√A” message will appear on the bottom right-hand corner of the display. Record the reading on the pH worksheet.
6.4 Refer to step 6.2 to clean the pH electrode and place pH electrode in the 50mL polyethylene tube containing the second check buffer (refer to 6.1.1 or 6.1.2) and press the green “READ” button. Wait for the endpoint to stop changing and an “√A” message will appear on the bottom right-hand corner of the display. Record the reading on the pH worksheet.
6.5 Both check buffers must be within ±0.05 of the assumed pH for the check buffers to be considered passing. If the check buffer is outside this range, prepare a new aliquot of the corresponding buffer solution by pouring ~30mL of the buffer solution into a new 50mL polypropylene tube, and re-check the pH.

6.5.1 If the re-check fails again, open a new bottle of the buffer solution and try again with a new aliquot.
6.5.2 If the check fails after 6.5.1, recalibrate the pH meter and try again.

7. Sample Measurement:

7.1 Homogenize the fertilizer sample bottle(s) by shaking the bottle on the mechanical shaker for 10 minutes.
7.2 Pour 40mL into a 100mL disposable plastic cup immediately after shaking.
7.3 Drop a magnetic stir rod into the 100mL disposable plastic cup containing the fertilizer sample and place the 100mL plastic cup on a stir plate.
7.4 Turn on stir plate and have sample stirred continuously without spilling out.
7.5 Rinse pH electrode thoroughly with DI water from a squeeze bottle and collect water waste in a beaker. Dab off excess water with Kimwipes until the pH electrode is dry.
7.6 Place pH electrode in sample cup, taking care to ensure the stir bar does not come in contact with the electrode.
7.7 Press the green “READ” button and wait for endpoint to stop changing. An “√A” message will appear on the bottom right-hand corner of the display.
7.8 Record the reading on the pH worksheet and clean the pH electrode (refer to step 7.4).
7.9 Repeat steps 7.1-7.7 for each following sample.
7.9.1 Test samples in duplicate.
7.9.2 The difference between duplicates must not exceed +/-0.2 pH unit. If the difference is greater than +/-0.2 pH unit. Repeat 7.1-7.9.

8. Maintenance:

8.1 Turn pH meter off when not in use.
8.2 Cover the hole with parafilm if pH meter is not in use.
8.3 When not in use, the pH electrode will be in the reservoir bottle with enough reservoir solution (3M KCl) in the bottle to completely submerge the pH electrode.

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Revision Log:

Date	What was Revised? Why?