FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
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January 30, 2015

Florida Method of Test
for
pH OF SLURRY

Designation: FM 8-RP13B-4

1. SCOPE

1.1 Field measurement of drilling fluid (or filtrate) pH and adjustments to the pH are fundamental to drilling fluid control. Clay interactions, solubility of various components and contaminants, and effectiveness of additives are all dependent on pH, as is the control of acidic and sulfide corrosion processes.

1.2 The recommended method for pH measurement of drilling fluid is with a glass electrode pH meter. This method is accurate and gives reliable pH values, being free of interference if a high quality electrode system is used with a properly designed instrument. Rugged pH instruments are available that automatically temperature compensate the slope and are preferred over the manually adjusted instruments.

NOTE 1: Color matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. These methods are reliable only in very simple water muds. Mud solids, dissolved salts and chemicals, and dark-colored liquids cause serious errors in pH paper values. Readability is normally about 0.5 pH unit.

1.3 This test method has been adapted from Section 7 of the American Petroleum Institute (API) Recommended Practice 13B-1: Standard Procedure for Field Testing Water-Based Drilling Fluids (RP 13B-1).

2. EQUIPMENT

2.1 pH meter: millivolt range potentiometer calibrated to show pH units for measuring the potential between a glass-membrane electrode and a standard "reference" electrode. The instrument is (preferred) to be water, shock, and corrosion-resistant and portable. Specifications are:

2.1.1 pH range: 0 to 14.

2.1.2 Electronics type: solid state (preferred).

2.1.3 Power Source: batteries (preferred).

2.1.4 Operating temperature range: 0-66°C (32-150°F).

2.1.5 Readout: digital (preferred).

2.1.6 Resolution: 0.1 pH unit.

2.1.7 Accuracy: ±0.1 pH unit.

2.1.8 Repeatability: 0.1 pH unit.

2.1.9 Adjustments.

2.1.9.1 "temperature" compensation of electrode system.

2.1.9.2 "slope" of electrode system (preferred).

2.1.9.3 "calibration" setting of readout. (Instrument with the above internal temperature compensation is preferred.)

2.2 Electrode system: A combination system of a glass electrode for sensing H^+ ions and a standard voltage reference electrode, constructed as a single electrode (preferred). Body of this probe should be constructed of durable material. A flat-end probe is preferred for better protection and easier cleaning of the electrode. Waterproof connection to the meter is recommended. Specifications are:

2.2.1 Glass pH electrode response range: 0 to 14 pH unit

2.2.2 Electrodes: a glass electrode and a silver/silver chloride electrode in combination, having a ceramic or a plastic single or double junction.

NOTE 2: Use double junction electrode for measuring liquids containing sulfide or bromide ion to avoid damaging (silver) reference electrode system.

2.2.3 Electrolyte in reference electrode: KCl gel

2.2.4 Glass composition: suitable for low sodium ion error.

2.2.5 Sodium ion error: at pH = 13 or at 0.1 mole Na^+ ion, an error less than 0.1 ph unit.

2.3 Buffer solutions: three solutions to calibrate and set slope of pH meter prior to sample measurement.

2.3.1 pH = 4.0: potassium hydrogen phthalate at 0.05 molar in water. Gives 4.01 pH at 24°C (75°F).

2.3.2 pH = 7.0: potassium dihydrogen phosphate at 0.02066 molar and disodium hydrogen phosphate at 0.02934 molar in water. Gives 7.00 pH at 24°C (75°F).

2.3.3 pH = 10.0: sodium carbonate at 0.025 molar and sodium bicarbonate at 0.025 molar in water. Gives 10.01 pH at 24°C (75°F).

NOTE 3: Buffers may be obtained from supply houses as pre-made solution, dry-powder packages, or a given formula, but must duplicate National Bureau of Standards primary or secondary buffers. Shelf life of all buffers not to exceed six months before disposal. Date of preparation of buffer should be shown on bottles used in the field. Bottles should be kept tightly stoppered.

2.4 Distilled or deionized water: in spray bottle.

2.5 Soft tissues: to blot electrodes.

2.6 Thermometer: glass, 0-150°C (32-220°F) .

2.7 Accessory equipment:

2.7.1 Soft-bristle test tube brush: to clean electrode.

2.7.2 Mild liquid detergent: Ivory, or equivalent.

2.7.3 Electrode storage vial: to keep electrode moist.

2.7.4 Sodium Hydroxide: 0.1 molar (approximately); to recondition electrode.

2.7.5 Hydrochloric Acid: 0.1 molar (approximately); to recondition electrode.

2.7.6 Ammonium Bifluoride: 10% solution (approximately); to recondition electrode.
CAUTION: This is a strong and toxic acid.

2.7.7 Hydrofluoric acid: ACS reagent grade.
CAUTION: This is a strong acid.

3. PROCEDURE - pH Measurement

3.1 Obtain sample of fluid to be tested. Allow it to reach 24±3°C (75±5°F).

3.2 Allow buffer solution to also reach the same temperature as the fluid to be tested.

NOTE 4: For accurate pH measurement the test fluid, buffer solution, and reference electrode must all be at the sample temperature. The pH of the buffer solution indicated on the container label is the correct pH only at 24°C (75°F). If attempting to calibrate at another temperature, the actual pH of the buffer at this temperature must be used. Tables of buffer pH values at various temperatures are available from the suppliers and should be used in the calibration procedure.

3.3 Clean electrodes by washing with distilled water and blot dry.

3.4 Place probe into pH 7.0 buffer.

3.5 Turn on meter; wait 60 seconds for reading to stabilize.(See Section 4.1 if meter reading is not stable.)

3.6 Measure temperature of pH 7 buffer solution.

3.7 Set this temperature on "temperature" knob.

3.8 Set meter reading to "7.0" using "calibration" knob.

3.9 Rinse probe with distilled water and blot dry.

3.10 Repeat operations in Sections 3.6 through 3.9 using either pH 4.0 or pH 10.0 buffer. Use pH 4.0 if "acidic" sample, or pH 10.0 if "alkaline" sample is to be tested. Set meter to number "4.0" or "10.0" respectively, using "slope" adjustment knob. (If no "slope" knob exists, use the "temperature" knob to set "4.0" or "10.0" on meter).

3.11 Check the meter with pH 7 buffer again. If it has changed, reset to "7.0" with "calibration" knob. Repeat Sections 3.6 through 3.11. If meter does not calibrate properly, recondition or replace electrodes as given in Sections 4.1 through 4.6.

NOTE 5: Discard and do not reuse the sample of buffer solutions used in calibration. Meter should be fully calibrated every day, Sections 3.2 through 3.11 using two buffers. Check with pH 7 buffer every three hours.

3.12 If meter calibrates properly, rinse electrode with distilled water and blot dry. Place electrode in sample to be tested and stir gently. Allow 60 to 90 seconds for reading to stabilize.

3.13 Record sample pH to nearest 0.1 pH unit and the temperature of sample tested on Drilling Mud Report form.

3.14 Carefully clean the electrode in preparation for next usage. Store in vial of pH 4 buffer. NEVER let the probe tip become dry.

3.15 Turn meter off and close cover to protect instrument. Avoid storing instrument at extreme temperatures (below 0°C (32°F) or above 49°C (120°F)).

4. CARE OF ELECTRODE

4.1 Cleaning the electrode will be necessary periodically, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Clean electrode with a soft-bristle brush and a mild detergent.

4.2 Reconditioning the electrode may be necessary if plugging becomes severe, as indicated by slow response, drifting of readings, or if "slope" and "calibration" cannot be mutually set.

4.3 Recondition by soaking electrode for 10 minutes in 0.1 M HCl followed by rinsing in water and soaking for 10 minutes in 0.1 M NaOH and rinsing again.

4.4 Check electrode for response by performing calibration Sections 3.2 through 3.11.

4.5 If electrode continues to perform poorly, soak electrode for two minutes only in 10% $NH_4F{\cdot}HF$ solution. (CAUTION: This is strong and toxic acid). Repeat Sections 3.2 through 3.11 to check for calibration capability.

4.6 Replace electrode system if above steps fail to recondition it.