
 UNIVERSITY OF KWAZULU-NATAL	Standard Operating Procedure  PRG pollution research group	Effective Date: 20 June 2013	Version: 003
		Reviewed: 1 Nov 2017	
SOP_Chem_012 Chemical Analysis_ pH Analysis of Faecal Sludge			Page # 1 of 5

Standard Operation Procedure – pH of Faecal Sludge

1. Scope and Application

- This method is an electrometric procedure for measuring pH in soils and waste samples.
- Wastes may be solids, sludge, or non-aqueous liquids.
- If water is present, it must constitute less than 20% of the total volume of the sample.

2. Apparatus and Glassware

- pH meter with means for temperature compensation.
- Glass electrode.
- Reference electrode- A silver-silver chloride or other reference electrode of constant potential may be used.
- 50 ml beaker.
- Thermometer and/ or temperature sensor for automatic compensation.
- Analytical balance- capable of weighing 0.1 g.

3. Safety Precautions

- Always use safety goggles, gloves and laboratory coat while working in laboratory.
- After the analysis, clean bottles and beakers with clear water keep it for drying.
- Dispose the used gloves after completion of analysis.
- Clean hands using antiseptic soap.
- Disinfect hands after washing with soap.
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

4. Interferences

- Samples with very low or very high pH may give incorrect readings on the meter.
- For samples with a true pH of >10, the measured pH may be incorrectly low.
- This error can be minimized by using a low-sodium-error electrode.
- Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.
- Errors will occur when the electrodes become coated.
- If an electrode becomes coated with an oily material that will not rinse free, the electrode can:

- be cleaned with an ultrasonic bath, or,
- be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or,
- be cleaned per the manufacturer's instructions.

5. Procedure

Sample Preparation

- To 20 g of waste sample in a 50 ml beaker, add 20 ml of distilled water, cover, and continuously stir the suspension for 5 min.
- Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.
- Let the waste suspension stand for about 15 min to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.
- **NOTE:** If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 ml of reagent water.
- **NOTE:** If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

Measurement of pH

- Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground glass joint or the fibre-capillary hole.
- Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.
- If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

6. Results

- Report the results as "waste pH" measured in water at ___°C" where "___°C" is the temperature at which the test was conducted.

Tips for new probes in extended dry storage

- Stir the probe in pH buffer 7 solution to dislodge air bubbles.
- Before use, soak new probes or probes that are stored for an extended time in pH 7 buffer for at least 5 minutes.
- Remove reference gel from the probe sensor and the inner surface of the rubber dust cap.
- Calibrate the probe. First with pH 7, then with a second buffer (usually pH 4 or pH 10) and then a third buffer, if necessary.
- Check the calibration. Put the probe into the pH 7 buffer. If the reading is incorrect, the probe is not hydrated. Soak the probe for 5 minutes in pH 7 and repeat the re-calibrate.
- Store the probe dry with the sensor cap on.

- For semi-solids use, gently twist the probe to make sufficient contact with the sample to the sensor.

Clean the pH probe

Before the probe is cleaned:

- For use in dairy, cheese or meat applications, soak the probe in Pepsin Cleaning Solution for 15 minutes.
- Do not use sharp metal objects (needle, pin, etc.) to clean the sensor surface. Scratching the surface of the sensor may cause permanent damage to the probe.
 1. Use a soft-bristle toothbrush to clean the sensor with soap and water.
 2. Make sure all the debris is removed from the sensor surface.
 3. Rinse the probe fully and re-calibrate.

Maximize probe life

- Rinse the probe fully after any calibration, measurement and cleaning.
- Add sensor cap to the end of the probe when not in operation.
- Cool samples to room temperature.
- When semi-solids are tested, make sure solid objects (i.e., bone or gristle) do not scratch the sensor.
- Use new buffers and rinse solution.
- Do not use the probe in environments that will damage the epoxy materials used in the probe tip.
- Keep the probe away from acetone, toluene, methylene chloride, xylene and other strong organic solvents.
- Avoid environments with static electricity. Electrostatic discharge (ESD) can permanently destroy the probe.
- Avoid use at temperatures more than 60°C. Thermal cycling can decrease the life of the probe.
- Do not let oil, fat, food particles, starch, protein or other materials stay on the probe after use.
- Prevent damage to the silicon chip sensor.
- Do not use the probe in hydrofluoric acid or abrasive samples or other environments that can cause damage to the silicon chip sensor.

7. References

<http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9045d.pdf>

APPROVAL OF STANDARD OPERATING PROCEDURE

PRG Head: Prof C.A. Buckley

Signature:

Date:

Author: Merlien Reddy

Signature:

Date: