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Detergent Residue Test Using a pH Meter, pH Indicator, or Test Kit

The following test procedures are suitable for detecting detergent residues resulting from improper rinsing and can be used to meet laboratory accreditation guidelines and questionnaires such as the College of American Pathologist program of state water lab accreditation programs.

This also establishes compliance with:

- National Environmental Laboratory Accreditation Conference (NELAC) (2003)
- The NELAC Institute (NLI) 2009/2016 Laboratory Standards
- National Environmental Laboratory Accreditation Program (NELAP)

1. pH Meter Method

- a. Rinse a small clean beaker by filling and emptying 3 times with source water.
- b. Fill a 4th time and measure pH using a pH meter.
- c. Record the pH as "source water pH".
- d. Using a piece of recently cleaned glassware, fill to around 10% full of source water (10 ml into 100 ml beaker). Please note that there should be enough water to sufficiently immerse the pH meter electrode in the measuring beaker.
- e. Swish water in glassware to extract residues from all possible surfaces.
- f. Take the pH reading with pH meter.
- g. Record pH reading as "glassware pH".
- h. Significant increases in pH indicates possible alkaline detergent residue. A significant change is 0.2 or more pH units on a pH meter measuring to 0.1 pH units of sensitivity. A result of less than 0.2-pH units change indicates properly rinsed glassware.

Note: If deionized water is used as the sample water, a slight amount (10-20 mg/L) of reagent grade, non-buffering salt (NaCl, CaCl₂) should be added to the sample water to allow the pH meter to function properly. If you want to avoid contaminating clean glassware, dump the glassware testing solution into a triple rinsed beaker and then add the non-buffering salt prior to measuring the pH with meter.



Note regarding pH paper: detergents and surface-active agents can interfere with some pH paper by causing a decrease of several pH units in reading. Please test any pH paper you intend to use with test detergents to determine if there is any interference before adapting this procedure for the use with pH paper.

Testing should be completed on approximately 1% of large frequently washed quantities of glassware and 5% of smaller quantities of less frequently washed glassware. Types of glassware tested should be rotated. Glassware of the narrow-neck variety should be test at an increased frequency. Thorough record keeping should be kept consisting of test date, type of glassware tested, and accompanying results.

2. pH Indicator Method for Alkaline Detergents

Materials required: 0.04% Bromothymol Blue (Aldrich 31,875-2), or prepare in 250 ml reagent grade water with 16 ml of 0.01 ml 0.01 N NaOH to 0.1 g Bromothymol blue (Aldrich cat no. 11,441-3) and reagent grade water (pH 5.5-7.5)

- a. Select glassware to be evaluated for alkaline detergent residue.
- b. Fill glass to 50% capacity with reagent grade water.
- c. Swish water around the glassware to extract any residues from sides.
- d. Add 2-3 drops of 0.04% Bromothymol Blue.
- e. Observe the color change, if any.
- f. If a pale blue color change is observed, the end user performing the test would document a "failed" reading which is indicative of alkaline residue.
- g. If a blue/green color change is observed, the end user performing the test would document a "passing" reading which is indicative of a neutral pH.
- h. If a yellow color change observed, the end user performing the test would document a "passing" reading which is indicative of a water pH of 5.5-6.5.

3. Detergent Test Kit Method for Neutral pH Anionic Detergents

If testing anionic detergent, please adapt the procedure to use a detergent test kit that is sensitive to such detergent residues. These kits are available from the following distributors:

- a. Chemetrics Inc. water testing kit K-9400 or I-2017 for anionic detergents (sensitive to ¼ ppm). Contact 1-800-356-3072.
- b. LaMotte Chemical water testing kit 4507-01 for anionic detergents (sensitive to 1 ppm). Contact 301-778-3100.
- c. Hach Company water testing kit 143203 for anionic detergents (sensitive to 1 ppm). Contact 1-800-227-4224 or 303-669-3050.



4. **Conductivity test for neutral pH ionic detergents**

If testing ionic detergent, please adapt the procedure to use a conductivity meter and deionized water that is sensitive to ionic detergent residue.

- a. Rinse two clean beakers or containers thoroughly with deionized (DI) water that has a conductivity reading of $\leq 1 \mu\text{S/cm}$ at 25°C .
- b. Fill one container sufficiently full of water to measure the conductivity.
- c. Measure and record the conductivity with a properly calibrated conductivity meter (range of at least $0.1 - 100 \mu\text{S/cm}$ at 25°C).
- d. Using recently washed pipettes, transfer sufficient volume of clean container DI water to the second container.
- e. Measure and record the conductivity of the second container.
- f. If an increase of $>0.5 \mu\text{S/cm}$ is observed the presence of detergent residue can be concluded.

Note: All procedures contained within this document were originally crafted by Alconox, Inc. Original documentation can be found at <https://technotes.alconox.com/wp-content/uploads/2013/04/cap.pdf>.