A SINGLE REAGENT FIELD TEST FOR QUATERNARY AMMONIUM SOLUTIONS

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A test method is described for the verification of proper use concentrations of solutions of straight quaternary ammonium rinses or formulated detergent-quaternary preparations. The simple test kit and direct procedure afford an economical and easily used test method for the sanitarian and the food and beverage worker. The estimation of the quaternary concentration is accomplished by the reaction of the quaternary with a definite amount of bromphenol blue accurately buffered on the acid side. The blue color of the dye-quaternary complex is dominant if 200 ppm. or more of quaternray is present in the solution. If less than 200 ppm. quaternary is present various shades of green are produced by the partial conversion of the dye to its blue complex plus the acidified yellow excess. The test reagent may be used with many proprietary preparations after the dye demand of the specific quaternary at the recommended use concentration is established.

The test described is simpler than related methods in eliminating use of an organic solvent and in its wide adaptability to service conditions. It is simple and accurate and economical enough to encourage greater use of controlled solutions.

INTRODUCTION

Numerous procedures for the assay of quaternary ammonium sanitizing solutions have been developed in the past few years for use in the laboratory or in the field. Methods showing a wide range of complication in use and accuracy of results are available but none have combined the elements of accuracy, economy, speed, and simplicity to a degree leading to a desirable widespread acceptance and application by sanitarians and other users of quaternary ammonium compounds.

The reaction occurring between certain quaternary ammonium compounds and tetrabromphenolsulfonphthalein (bromphenol blue) leading to an intensely blue reaction product has been widely used in principle by workers in the past ([1, 2, 3, 4]). The unique feature of the present method is that no volatile organic solvent is required since the dye-quaternary salt is used directly as formed in the aqueous phase.

The prime objective of this work was to develop a single test reagent suitable for use in the field merely to differentiate minimum use concentrations of certain quaternary ammonium type sanitizing agents from marginal or improperly dilute solutions. Such a reagent was to be furnished in a simple kit for use in evaluating either a straight quaternary rinse or a formulated, alkaline detergent-sanitizer. Since it was intended that the kit be suitable for use by both control enforcement agents and by the non-skilled food and dairy worker as well, simplicity of use was an important requirement. The composition range of water supplies and the variation in pH and titratable alkalinity of the solutions to be tested were additional considerations. Further, it was desired that the same reagent volume addition be used in all tests for any particular quaternary.

MATERIALS AND METHOD

In addition to bromphenol blue, the reagent contains buffering chemicals to bring the final solution to a pH very close to 3, regardless of water supply or initial alkalinity of the solution. Sodium acetate and acetic acid at a ratio of 1 to 5 were found to be suitable.

The composition of the reagent is as follows:

	Percent by weight
Bromphenol blue	0.08
Sodium acetate C. P. ($NaC^2H^3O^2$-$3H^2O$)	12.50
Glacial acetic acid	62.50
Distilled water	24.92

The solution is quite easily prepared by dissolving the bromphenol blue in the acid and water mixture and then adding the acetate salt. In preparing large volumes, the solution of the dye may be hastened by first dissolving it in a minimal amount of 95 percent ethanol.

A photograph of a complete and practical testing kit is shown in the illustration.

For two typical products, Spartec* and Tri-Bac,* the test is conducted by adding 0.40 ml of the reagent to 6.0 ml of the quaternary solution in a small specimen vial graduated at the 6.0 ml level. The volume of reagent may be added from a dropping pipette standardized to deliver the required volume in a definite number of drops. Then, simply by counting the number of drops, the operator can easily add the proper amount of reagent. Upon mild agitation of the vial a clear blue color appears if the quaternary content of the solution under test is of a magnitude of 200 ppm or more. This is a commonly recommended sanitizing strength. A blue-green, green or yellowish color indicates less than 200 ppm, 100 ppm or less, and 50 ppm or less, respectively.

* Trade marks registered by Wyandotte Chemicals Corporation. Spartec is a sanitizing agent based on methyldodecylbenzyltrimethyl ammonium chloride and Tri-Bac is a mildly alkaline detergent-sanitizer containing di-isobutylphenoxyethoxyethyldimethylbenzyl ammonium chloride.

DISCUSSION

The quantitative color effect results from the high affinity of bromphenol blue for quaternary ammonium compounds and their relative concentrations in the final solution. The complex formed is essentially deep blue in color in the amounts in which it is present in these tests. It is formed from the dye as long as there is quaternary and dye available in the solution. Assuming that 200 ppm quaternary is the desired dilution for most purposes, then the concentration of the bromphenol blue in the final solution is adjusted to yield a definite blue color at this level. Four tenths ml of the reagent furnishes this amount of dye plus sufficient buffer to give the solution a pH of about 3. The lower quaternary concentrations are detected when the formation of the blue complex is limited in amount by the quaternary and the excess bromphenol blue retains the yellow color of its acidified solution. When the mixture is viewed by transmitted light the two colors blend into various greens indicative of the concentration of the quaternary.

The need for careful control of pH in such a system is obvious since bromphenol blue is itself an indicator dye showing color transformation in the pH range 3.0–4.6. Unless the solution is heavily buffered at the proper pH as described, minor differences in dissolved salts may interfere with the color. With the use of the acetic acid-sodium acetate system, successful tests are obtained in synthetic hard waters containing up to 30 grains per gallon total hardness. This buffer system also allows the application of the test to moderately alkaline quaternary formulations now widely used where washing and sanitizing are to be accomplished in one operation. Interference of many contaminating agents such as beer, milk, synthetic detergents and even soaps is similarly overcome up to a point where the concentration of such materials begins to inhibit the sanitizing action of the quaternary.

The bromphenol blue-quaternary complex suspended in water exhibits a certain amount of wine red fluorescence when the vial is held in such a way that the solution is seen in reflected light. If the test is viewed in transmitted white light, that is, with the vial between the light source and the eye of the observer, the true colors are seen. The reflected light emission must be ignored, or the vial may be placed in a viewing block that would allow only transmitted light to be used. Some fading of the test colors occurs if the vial is allowed to stand very long after completion of a test but since the test can be interpreted immediately this has no significance.

The test reagent has proven to be quite stable in storage at ordinary conditions. Accelerated storage tests have shown that the solution retains its original characteristics for a period equivalent to at least six months at room temperature when kept in tightly capped bottles.

The method has been found to work satisfactorily with many quaternary preparations. However the dye requirement may vary from one quaternary to another and in each case the volume of reagent necessary to produce differentiating colors should be determined from known dilutions.

A great advantage of the described test is its low cost. The reagent and accompanying kit can be prepared and distributed economically enough to allow its frequent use by all interested in determining the strength of quaternary ammonium sanitizing solutions. Increased vigil resulting from the use of this practical test should assist in the movement toward better utensil sanitation in the food and beverage industries.

CONCLUSIONS

A simple method has been devised for the colorimetric differentiation of the 200 ppm range quaternary ammonium sanitizing solutions from those of lesser strengths. The addition of a measured volume of a single reagent to an aliquot of the quaternary use-solution allows the operator to determine immediately if the solution has the proper sanitizing strength. Simplicity, low cost and adaptability to a variety of quaternary products and water conditions promises wide acceptance of this method.

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