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CONTRIBUTIONS FROM THE LABORATORY OF SANITARY CHEMISTRY.

VIII.

PERMANENT STANDARDS IN WATER ANALYSIS.

BY LILY MILLER KENDALL AND ELLEN H. RICHARDS.

EVERY chemist has noted with regret the hours that are consumed in preparing the various color standards for comparison, now so universally used for the determination of the small amounts of ammonia, nitrites, nitrates, and other substances occurring in water.

Nearly every analyst has tried to minimize this time expended by some mineral solutions of his own or others' devising, which will keep indefinitely and be always ready.

There are two insurmountable obstacles to perfect success; first, that such pure or mixed solutions, frequently strongly acid, have a clearness and brilliancy of tone which the complex sample to be matched never possesses; and second, that the color produced in the solution to be tested depends upon a variety of conditions: temperature, quantity of reagent added, manner of making reagent, variable quantity of accompanying substances, time elapsing after preparation before comparison of color, and a score or two more, practically impossible to control perfectly. The determination of ammonia by the Nessler reagent is a familiar example.

These difficulties may be removed to a greater or less extent by careful preparation of the standards from solutions as nearly as practicable of the same order of variability.

Hence it is that permanent standards, made up to match a given set of conditions, cannot be relied on under *all other circumstances.* Nevertheless they have their uses; and for field work, where comparison within certain limits only is to be exacted, approximately permanent standards for all the common tests serve an admirable purpose.

In preparing a portable field apparatus for the Louisiana Purchase Exposition two or three combinations were devised which may prove

of interest and may suggest other experiments along the same line. The Griess test for nitrites is one of the most valuable for field work, but in the conditions under which tests must be made — lack of pure rinsing water, dusty atmosphere and hasty work — the very unstable nitrite standard is a source of anxiety, and therefore a set of permanent standards is much to be desired. Such a set lessens also the quantity of troublesome reagents to be carried.

A very satisfactory tint of color, which remained permanent for two months in the winter in the even temperature of the laboratory, was given by a solution found among the set of indicators marked "Corallin, Malachite Green." This solution unfortunately was of unknown origin, and attempts to duplicate it exactly have not been wholly successful. The results are here given simply as suggestions for further work. The substance desired is very possibly "corallinephthalin or pseudo-rosolic acid, the chief constituent of commercial aurine." Organic Coloring Matters, Schutze & Julian, translated by Green, 2d edition, p. 198.

A sample of Kaulbaum's aurin found in stock, when made up in about the proportion of 1.5 milligrams per liter matched fairly well the 10 c.c. standard nitrite. A sample of Kaulbaum's rosolic acid of the same strength, to which was added .008 milligram in aqueous solution of Kaulbaum's "malachite green," gave a color very closely approximating to the 20 standard. There are several commercial substances listed under the name malachite green; this seems to be "B," giving a blue-green aqueous solution, which is discharged by excess of potassium hydrate; therefore since aurin or rosolic acid must be in decidedly alkaline as well as alcoholic solution, a somewhat delicate balance of conditions must be formed. The solution once obtained, however, it seems to dilute in proportion and give an excellent match of tint. The blue in the aurin seems to fade out unless the solution is kept very strongly alkaline, leaving a yellower tint than desirable. This is the case with the standards prepared from the usual reagents. If, as now appears probable, the strong solutions carefully protected will keep, and may be diluted as needed, the test may be of some value.

Standards for the Grandval and Lajoux nitrate test were made from the neutral potassium chromate used for indicator in the chlorine test. For the deeper colors and ammonia, a solution made after Tidy's formula was found to be practicable for dilution to the de-

sired tints. $K_2Cr_2O_7$, 0.25 grams; $CoSO_4$ H_2O, 9.05 grams per liter. This solution avoids the strong acidity so unpleasant in portable cases.

In the two long trips[1] made with the portable apparatus described in this journal,[2] it was found that the form of the case, convenient in

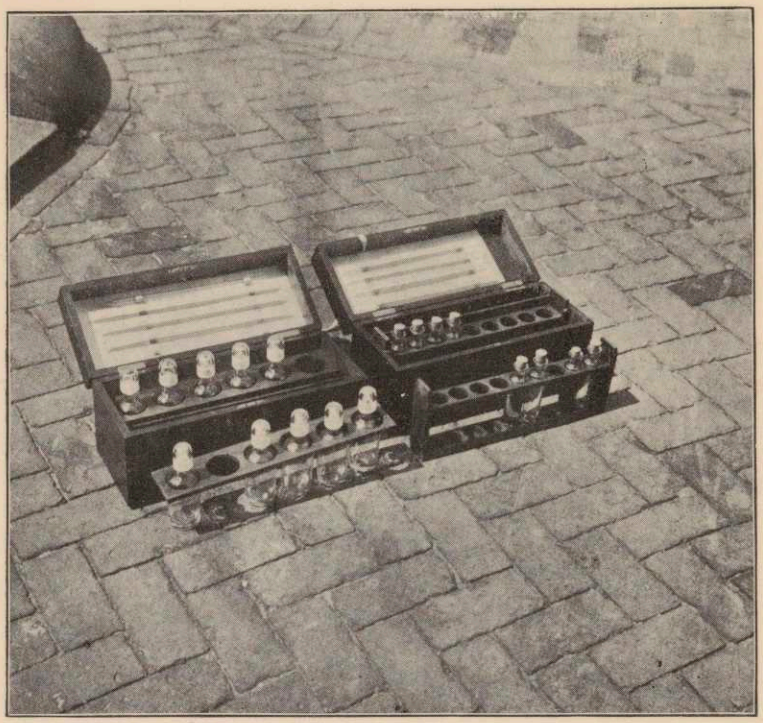

FIG. 1. — PORTABLE CASE FOR FIELD WORK IN WATER ANALYSIS.
Length, 16 inches; depth, 6½ inches; height of each box, 7½ inches.
Designed by Mrs. Kendall.

many respects, was most unsatisfactory to transport in sleeping cars; for, unless watched over like a bag of gold, the porter would inevitably lay it on its side, to the imminent danger of loosened stoppers and

[1] The Water Supplies of Southeastern Alaska. Notes on the Water Supplies in the Black Hills of South Dakota and Vicinity. TECHNOLOGY QUARTERLY, XVI, No. 4, December, 1903.

Notes on the Potable Waters of Mexico. Transactions American Institute of Mining Engineers, XXXII, 1902, p. 335.

[2] A Portable Outfit for Water Analysis. A. G. Woodman, TECHNOLOGY QUARTERLY, XIV, No. 4, December, 1901.

promiscuous mixture of reagents. The same difficulty occurred when driving across country in a buggy. This close, personal care becomes irksome in the course of weeks. The form shown in the figure is designed to obviate these difficulties.

Two boxes fitted up after the manner of instrument boxes are to be strapped together when carried by hand, but when separated may be slipped under the berth or wagon seat and kept right side up.

The removable racks, with the permanent standards and free bottles for comparison, will be found to facilitate observation. The bottles for reagents, with double ground glass stopper joints, may prove too frail for rough carriage; but fastened with the yoke, as shown in the left hand of the illustration, they should stand well.

The padded cover furnishes safe resting place for the pipettes, and the narrow space behind the racks allows for the carriage of the various additions which each chemist would choose for himself. The English Thresh portable case with solid reagents is still more compact but nearly as heavy, and does not permit of all the tests used in this country. Many of the most useful "soloids" can be put up to order in this country and added to the suggested outfit.