

The picric acid method of P. Lemaire was found to be unsuitable for the quantitative determination of sparteine because of the solubility of sparteine picrate in water.

It was found to be impossible to titrate the alkaloid after extraction with chloroform, as methyl red indicator does not give a sharp end-point. This is apparently due to the brown color of the alkaloid and to partial oxidation. If phenolphthalein is used the end-point is fairly distinct but the results are in every case about 10% low.

Another method which consists of shaking the alkaloid out from ether solution with standard acid was found to be impractical because of the difficulty of quantitatively recovering the alkaloid in this way. The method as given recovers about 2.0% of the alkaloid present, two hours' shaking at frequent intervals recovering only 6.1%.

#### BIBLIOGRAPHY.

1. *Philos. Trans.*, 1851 (2), 422.
2. Henry, "The Plant Alkaloids," 1913.
3. Mulliken, "The Identification of Pure Organic Compounds," Vol. II.
4. H. W. Jones, *Am. Druggist*, 62, 369-70, 1914.
5. P. Lemaire, "Repert. Pharm.," (3) 1-4-20.
6. Pictet and Biddle, "The Vegetable Alkaloids," No. 1, 1904.
7. Bamberger, "Liebig's Annalen der Chemie et Pharmacie," 235, 268.
8. Winterstein and G. Trier, "Die Alkaloidie," 1910.
9. Jorrissen, *Jour. phar. chim.*, 8, 1911, 4-25-12.
10. E. H. Grant, *JOUR. A. PH. A.*, 9, 704, 1920.
11. *Compt. rend.*, 1905, 141, 261, 137, 194.
12. "Cohn's Indicators and Test Papers."

---

#### SOLUTION OF CHLORINATED SODA.\*

BY E. F. KELLEY AND JOHN C. KRANTZ, JR.

The well-known Dakin's Solution and the present official Solution of Chlorinated Soda have been the source of extensive research work among the members of the Pharmacopœia Revision Committee. The fact that Labarraque's Solution is seldom used and the increasing demand for Dakin's Solution makes this a question of great importance.

The chief objection to Labarraque's Solution is its strong alkalinity, on account of which it cannot be used as an irrigating solution in antiseptiology. The recent formula submitted by Samuel L. Hilton proposed the use of a mixture of sodium bicarbonate and carbonate to precipitate the lime salt and thus if any alkali salts are carried into the finished product, the acid carbonate ion, furnished by the acid salt, neutralizes the hydroxyl ion, formed by the hydrolysis of the normal carbonate. Dakin's Solution is best suited for clinical purposes when its hydrogen-ion concentration has a value of ( $-10$ ); roughly this degree of alkalinity may be observed when the solution is not colored by solid phenolphthalein but is alkaline to an alcoholic solution of the indicator. The solution prepared by Mr. Hilton's formula meets the above neutrality requirement, but has one objection inasmuch as it is prone to become pink on standing.

---

\* Read before Scientific Section, A. Ph. A., Cleveland meeting, 1922.

It is the purpose of this paper to introduce an alternate formula for Dakin's Solution that will meet the neutrality requirement and overcome the tendency to turn pink.

As the objection to the present Labarraque's Solution is its alkalinity, the first step was to select a sodium salt that was not so alkaline as sodium carbonate, and the acid in combination formed an insoluble calcium salt. For this purpose the official anhydrous disodium hydrogen phosphate was selected, as in dilute solutions it met the neutrality requirement before mentioned.

A series of chlorinated soda solutions were prepared from a sample of chlorinated lime, which assayed 31.24 per cent. available chlorine, using quantities of anhydrous sodium phosphate and the lime salt that would yield theoretically a solution containing 2.5 per cent. of sodium hypochlorite.

Three solutions were made by using 100 Gm. of chlorinated lime, and 105 Gm. of anhydrous sodium phosphate to 1000 Gm. showed 1.6 per cent. of sodium hypochlorite by weight. These solutions, however, were neutral to solid phenolphthalein and alkaline to alcoholic phenolphthalein.

Three solutions were made by using 150 Gm. of chlorinated lime to 1000 Gm. with 105 Gm. of anhydrous sodium phosphate. The results obtained were not much higher in sodium hypochlorite content than in the previously prepared solutions. The average content assayed about 1.79 per cent. sodium hypochlorite.

One solution was made using 200 Gm. of the lime salt with 150 Gm. of anhydrous sodium phosphate. This solution yielded 2.75 per cent. of sodium hypochlorite, but was alkaline to solid phenolphthalein. This excess alkalinity was possibly due to the increased quantities of both ingredients used in the preparation.

Following these results, another solution was prepared using 170 Gm. of chlorinated lime and 150 Gm. of sodium dihydrogen phosphate crystallizing with one molecule of water of hydration. Due to the acidity of this phosphate chlorine was immediately liberated from the bleaching powder and the solution turned pink while filtering. The pink coloration is likely due to the oxidation of some manganese salt in the chlorinated lime to the permanganic acid state by the action of the liberated chlorine on the water. This solution yielded only 1.38 per cent. of sodium hypochlorite.

Two other solutions were prepared using 170 Gm. of chlorinated lime and the alkaline and acid sodium phosphates in different proportions. In each solution the chlorine content assayed between 1.4 and 1.5 per cent. and they did not react with an alcoholic solution of phenolphthalein showing a hyperacidity.

In searching for the cause of the low sodium hypochlorite content of the previously made solutions, it was noticed that the precipitate of calcium phosphate was quite voluminous compared with the precipitate of calcium carbonate, when sodium carbonate was used for the precipitation. After enough water had been washed through the precipitate to make 1000 Gm. of finished solution, the precipitate was again washed, and the washings were found to contain a considerable quantity of sodium hypochlorite, showing that the shortage in hypochlorite content was due to the incomplete washing of the calcium phosphate. Many attempts were made, by changing the temperature and concentrations of the reacting solutions, to reduce the bulk of the precipitate, but little decrease in volume was noticed.

In view of the fact that the 2.5% solution is used only after proper dilution to 0.5 per cent., *i. e.*, the original Dakin's Solution, experiments were carried out to prepare the latter. In the making of the first solution 30 Gm. of chlorinated lime and 26 Gm. of anhydrous sodium phosphate were used to make a liter. This solution met the neutrality requirement and assayed 0.83 per cent. of sodium hypochlorite.

Using the last-mentioned quantities and 0.83 per cent. as a basis, the following quantities of chlorinated lime and anhydrous sodium phosphate were used for a liter of a solution that should assay between 0.45 per cent. and 0.50 per cent. of sodium hypochlorite.

Chlorinated lime	20 Gm.
Sodium phosphate, anhydrous	20 Gm.
Water, sufficient to make	1000 cc

Solution (1) prepared from this formula assayed 0.514 per cent. of sodium hypochlorite and solution (2) assayed 0.49 per cent. These solutions were neutral to solid phenolphthalein and alkaline to an alcoholic solution of phenolphthalein.

The directions for preparing the solution are as follows:

Mix the chlorinated lime intimately with 400 cc of water. Dissolve the sodium phosphate in 400 cc of water heated to 50° C. and pour the sodium salt solution in the lime-salt mixture; shake well and allow to stand for 15 minutes; transfer the precipitate to a filter and after all of the liquid has drained from the precipitate, wash the filter and contents with sufficient water to make the finished product measure one liter.

The method of assay is the same as any other standard chlorine assay using 0.003723 as the sodium hypochlorite factor, *i. e.*, the equivalent for one cubic centimeter of tenth normal sodium thiosulphate V. S. used.

The stability of this solution is comparable to other sodium hypochlorite solutions of a corresponding strength. The percentage of decomposition one week after preparation is approximately 8 per cent. Therefore, any solution assaying 0.5 per cent. when prepared can be safely used one week later, since it would not fall below the lower limit of hypochlorite content, that is, 0.45 per cent. In two weeks the average decomposition was 14 per cent., showing that the solution should be made for periods of 7 to 10 days.

From the experiments described above the writers draw the following conclusions:

1. A 2.5% solution of sodium hypochlorite cannot be conveniently prepared by the above method.
2. The 0.5% solution of sodium hypochlorite can be conveniently prepared, which meets the neutrality requirement and does not become pink on standing.
3. The solution prepared by the phosphate method is as stable as other solutions of sodium hypochlorite of corresponding strengths.