

The results are self-explanatory and show the rather interesting fact that home-grown capsicums are not inferior to imported ones. The fact that Sample 3 shows a greater pungency than Sample 4 would tend to show that the converse was really true. This cannot be accepted as final, however, as we have not proved the botanical source of the samples.

As a whole, however, the experiments prove conclusively that there is a variation in pungency between chillies of different sizes and different botanical sources, and that it is advisable to include only African Chillies in the Pharmacopœia. The Scoville test shows itself readily adaptable to detect this variation in pungency, and due to its simplicity and relatively accurate results, its inclusion in the Capsicum monograph is fully warranted.

HYPOCHLORITE SOLUTIONS.

BY RUTH M. DAVIS AND H. A. LANGENHAN.

(Continued from p. 133, February JOURNAL A. P. H. A.)

(NO. 3) TECHNIQUE OF LABARRAQUE'S SOLUTION.

The method of preparing the solutions of sodium hypochlorite has proved to be important in controlling the strength of the finished product. There are two different methods founded on the materials used, *viz.*, that of passing chlorine gas into alkali solution, and that of mixing a solution of chlorinated lime with a solution of sodium carbonate, sodium bicarbonate, sodium sulphate, or sodium phosphate.

The two methods are basically the same, varying only in small but important details, and these are best noted by reviewing the changes in formula in chronological order.

Durand gives a description of the first method or Labarraque's original process, that of passing chlorine gas into a solution of "subcarbonate of soda." This process consists, first, in the preparation of the "sodium subcarbonate" solution which was to be exactly saturated and must "mark 12 degrees on Baume's aerometer for salts." The chlorine was to be made in the following manner: The sodium chloride and manganese dioxide were placed in a "matrass." "Apply a cork to the mouth of the matrass and place it on a furnace. A hydrostatic funnel for the introduction of the acid is adapted to the cork as well as a safety tube, bent at a right angle, and plunging by its extremity into a Woulfe bottle with two necks, containing water, destined to wash the chlorine. From the second neck of the bottle emerges another safety tube, whose extremity plunges in the bottle containing the solution of carbonate of soda." Introduce the acid into the hydrostatic funnel and place a few "ignited coals under the matrass, and raise the temperature gradually, until the disengagement of chlorine ceases." This should be continued until "one part of the chloride would discolour eighteen parts of sulphate of indigo."

Faraday did not change Labarraque's method, but he passed the chlorine through the wash bottle into the sodium carbonate solution just so that "no carbonic acid was set free." Payen introduced the second type of manipulation. His method consisted in mixing "dry chloride of lime" with twelve parts of water, letting the liquor settle during three hours, in closed vessels, filtering and washing the dregs with two parts more of water, then dissolving the "soda" in the remainder

of the water, with the aid of gentle heat, and allowing the solution to cool. The two solutions were mixed, the mixture well stirred, and the precipitate allowed to settle. The clear liquid was decanted or filtered off and bottled securely. Here he notes an interesting detail that has been forgotten and but recently recalled, *viz.*, that the dregs might be washed to produce a weaker solution.

In all the revisions of the U. S. Pharmacopœia containing *Labarraque's Solution* (from 1840 to 1910), the sodium carbonate is dissolved separately from the chlorinated lime, in water varying in temperature. The revisions of 1840 to 1870 inclusive, directed that the solution should be effected "with the aid of heat." The revision of 1880 prescribed "boiling water." Until 1880 no mention was made as to whether the carbonate solutions should be added cold, warm, or hot. In the 1880 revision the sodium carbonate is dissolved and immediately poured into the chlorinated lime magma. In the 1890 revision the warm solution of the sodium carbonate is poured into the solution of calcium hypochlorite, and the whole mixture heated gently if it gelatinizes. This same method is used throughout the remaining revisions. No notice seems to have been taken by the revision committees of Payen's direction to cool the sodium carbonate solution before adding it to the lime magma. The chlorinated lime magma was treated in several different ways according to the different revisions of the U. S. P.

The 1840 and 1850 revisions, after Payen's formula, were not so particular as to details as was Payen's in his procedure. Essentially the technique was the same but the U. S. P. process omitted the washing of the lime precipitate. The chlorinated lime was added to the water "in small portions." Here arises a question as to the possibility of obtaining as smooth a mixture as when the chlorinated lime is triturated with water added gradually. In the 1860 revision this idea was incorporated into the text for along with the change of terms was a change in manipulations. The chlorinated lime was triturated with small portions of water, and then set aside for twenty-four hours. (Payen called for three hours of maceration and the 1840 revision suggests "several.") This revision allowed the lime residue to drain, then washed it with water sufficient to collect eight pints. The mixture of sodium carbonate and chlorinated lime was also allowed to drain and was washed until eleven and one-half pints of the finished product were obtained. Thus the main modification was the requirement of definite volumes of chlorinated lime filtrate and of the finished product. This undoubtedly resulted in the preparation of a more uniform product as compared with older revisions. The 1870 revision introduced no change but a modification appears in that of 1880. This revision destroyed in part the attempts of the former revisions to control the volume of the finished product. It directed that the chlorinated lime be mixed with water, in a tared, covered dish, the solution of sodium carbonate added and, when cold enough, water to make the entire mixture weigh 1000 parts. After standing the mixture was strained through muslin, and further separation effected by siphoning off the clear liquid from any sediment that might appear. It was quite evident that less than 1000 parts of the finished product would be obtained, due to the fact that the insoluble portion was included in original 1000 parts. Even though this revision eliminated the attempts of previous revisions to standardize the finished products, by controlling the amounts obtained, it was generally accepted as an improvement over former revisions since it was an evidence of a better understanding of the

properties of the material used. The great ease with which chlorine may be lost and the action of atmospheric carbon dioxide in precipitating the soluble lime salts are guarded against by the covered dish and the addition of the sodium carbonate solution to the lime magma. The method appeared to be feasible, especially since the requirement of available chlorine was only 2 per cent. but even this requirement was not attained in the finished product, so in 1890 a complete change was made. Payen's process was again adopted. The chlorinated lime was triturated with two portions of water, transferred to a filter paper, and finally washed. The filtrate was mixed with the sodium carbonate solution, the mixture strained, and the residue washed until the filtrate obtained, weighed 1000 parts. Though the care in manipulation, as directed by the previous revision, was ignored, the mixing of the two solutions and the subsequent washing of the precipitate gave a finished product of definite weight with little loss of hypochlorite in the residue. But this process gave no better results than the process of the 1880 revision. There, little chlorine had been lost by evaporation, but much loss held by the magma; here, little chlorine had been lost in the magma due to the washings, but much lost by evaporation due to prolonged handling.

In the 1890 and 1900 revisions this process was still maintained, but in 1910 a new technique was instituted, a simplification as to detail of preparation. Half of the water is added gradually to the chlorinated lime, the calcium hypochlorite solution is filtered off and mixed with the sodium carbonate solution, the mixture is then filtered, and the residue washed with sufficient water to make the filtrate weigh 1000 grams. But the chlorinated lime residue is not washed and as usual the loss of chlorine during the preparation makes it impossible to prepare a solution of the required strength, even though the purity rubric of the finished product had been lowered.

There can be little doubt, after having reviewed the changes in the methods, that most of the important details in manipulation have at one time or another been detected but at no time have they all been included in the one perfect formula.

Credit should be given to Army and Dawson, who have combined the good points of these methods into one which seems to invariably give solutions of standard and uniform strength. According to their procedure, the chlorinated lime is placed in a wide-mouthed bottle with the water and allowed to stand twenty-four hours with occasional shaking. The sodium carbonate solution is then added, the whole shaken for several minutes, and the mixture filtered. The lime residue is carefully washed to produce a product measuring 900 mils.

Several other methods may be here noted. Graebe followed Labarraque's procedure, that of passing chlorine into sodium hydroxide solution of known strength, thus preparing a stronger solution than that of the U. S. P. requirement. Dakin was the first to dissolve the sodium carbonate and macerate the chlorinated lime in the same water. He placed the entire amount of sodium carbonate and chlorinated lime in a flask, added the water, and shook the whole occasionally for an hour, allowed the precipitate to settle, and siphoned off the supernatant fluid. Dausfresne adopted Army and Dawson's technique to the extent of macerating the chlorinated lime for six hours before adding the sodium carbonate solution.

In conclusion it may be said that two factors influence the strength of the finished product, *viz.*, first, the available chlorine content of the chlorinated lime

used, and second, the complete extraction of this chlorinated lime by the amount of water prescribed.

REFERENCES (NOS. 2 AND 3).

- , *Jour. de Chimie Medicale de Pharm. et de Toxicologie*, 2 (pt. 1), p. 167, 1826.
 Durand, *Am. Jour. Pharm.*, 1, p. 272, 1830.
 Cohen, *Am. Jour. Pharm.*, 4, p. 205, 1833.
 Arny and Dawson, *Proc. Am. Pharm. Assoc.*, 56, p. 841, 1908.
 Elvoe, *Am. Jour. Pharm.*, 82, p. 161, 1910.
 Dakin, *Brit. Med. Jour.*, 11, p. 318, 1915.
 ———, *Jour. A. M. A.*, 65, p. 880, 1915.
 ———, *Jour. A. M. A.*, 65, p. 1221, 1915.
 Carrel, *Jour. A. M. A.*, 67 p. 1777, 1916.
 ———, *Jour. A. M. A.*, 67, p. 1798, 1916.
 ———, *Jour. A. M. A.*, 67, p. 1108, 1916.
 ———, *Jour. A. M. A.*, 67, p. 1795, 1916.
 Thum, *Jour. A. Ph. A.*, 6, p. 558, 1917.
 Griffith, *Am. Jour. Pharm.*, 89, p. 497, 1917.
 Wischo and Freiberger, *Jour. Chem. Soc. Lond.*, 114 (2), p. 193, 1918.
 Sells, *Jour. A. Ph. A.*, 9, p. 881, 1920.
 ———, Editorial, *Am. Jour. Pharm.*, 95, p. 67, 1923.
 Ebert, *Year Book, A. Ph. A.*, 8, p. 97, 1919.
 Cowley, *Year Book, A. Ph. A.*, 8, p. 92, 1919.
 ———, *Chemical News*, 11, p. 132, 1865.
 ———, *Jour. A. Ph. A.*, 9, p. 1087, 1920.
 Éwe, *Jour. A. Ph. A.*, 9, p. 46, 1920.
 Thomas, *Jour. A. Ph. A.*, 11, p. 940, 1922.
 Kelly and Krantz, *Jour. A. Ph. A.*, 12, p. 112, 1923.
 Dakin and Carlisle, *Year Book, A. Ph. A.*, 6, p. 89, 1917.
 Stimson, *Jour. Am. Med.*, 67, p. 1687, 1917.

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(To be continued.)

CHEMISTRY AND PREPARATION OF DECOLORIZED TINCTURE OF IODINE.

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A half-century ago, the colorless tincture was highly recommended for its external application on account of its discutient propensities, and the supposed repulsive tendencies further ascribed to the tincture placed it in position as an indicated remedy by internal administration in general inflammatory affections.¹

The preparation is practically devoid of the therapeutic virtues ordinarily attributed to elementary iodine, nevertheless constitutes a galenical of staple demand in pharmaceutical practice.

The method of preparation, as prescribed in the N. F. IV,² represents a totally inadequate procedure for production of the commodity upon a more or less extensive scale.

Numerous conditions and difficulties, encountered in the course of digestion in particular, contribute to the uncertainty of quantity in the final yield of finished product. The application of heat to facilitate solution and accelerate decolorization, presents a degree of danger due to the liability of precipitation of varying amounts of nitrogen iodide occasioned by the contact of ammonia and iodine. Nitrogen iodide is characterized by a violently explosive tendency, and when purified and dried is susceptible to detonation by the mere contact of a feather.

The precipitate, which is subsequently dissipated on completion of decoloriza-

* Cincinnati, Ohio.

¹ *Am. J. M. Sc.*, 9, 398, 1865.

² "N. F. IV," p. 228.