FACTS REGARDING THE MANUFACTURE, PHYSICAL CONSTANTS AND KEEPING QUALITIES OF SOAP, CHLOROFORM AND CAMPHOR LINIMENTS OF THE U. S. P. X.*

BY SAMUEL SHKOLNIK.

Last year, at the Philadelphia meeting of the A. PH. A., L. E. Warren of the Drug Research Unit of the Bureau of Chemistry of the U. S. Department of Agriculture intimated that he would like to gather some definite data regarding such physical constants as optical rotation, and specific gravity and the per cent of solids of soap and chloroform liniments. It was the request of Mr. Warren, therefore, supported by the advice of Prof. Clyde M. Snow of the University of Illinois School of Pharmacy that prompted my undertaking of the compilation of the data on the two above-named liniments. Before presenting the compiled data, however, I shall briefly discuss the ingredients and manipulations in the manufacture of Soap Liniment.

The formula for Soap Liniment as given in the U. S. P. X (and as had been given in the U. S. P. IX) and the directions for its manufacture are to dissolve 45% of camphor and 1% of oil of rosemary in 70% of alcohol and then add 5% of soap and enough water to make 100%. This is to be agitated until the soap dissolves, after which time the solution is to be set aside in a cool place for twenty-four hours and then filtered. We understand, of course, that the soap used in this liniment is the official "castile soap" made by the reaction between olive oil and sodium hydroxide. Since palmitin is a constituent of olive oil there is in the finished soap more or less sodium palmitate which is less soluble in cold alcohol than in warm and, to avoid precipitation in cold weather, the preparation should be kept cold for twenty-four hours and then filtered; otherwise the sodium palmitate, being in a finely divided condition, would pass through the filter paper and consequently would later precipitate on standing. Both the duration of time and the proper temperature must be observed if satisfactory results are to be obtained in determining the per cent of solids and specific gravity.

It may be pointed out at this time that the setting aside of the liniment for twenty-four hours in a "cool place" must not be interpreted as meaning a place like an ice box or any other refrigerator, for in such cases the preparation will congeal into a jelly-like, somewhat opaque mass necessitating a subsequent "thawing out" or warming before filtering. Such procedure would only cause unnecessary delay; for on warming, the sodium palmitate dissolves and a subsequent cooling is required before it could be removed.

Comparing the ingredients and the manner of preparation as outlined in the U. S. P. with those outlined in the British Pharmacopœia we find that the British formula calls for soft soap which is made from potassium hydroxide and olive oil (unlike the soft soap of the U. S. P. X, which is made from linseed oil and sodium and potassium hydroxide). Since the soft soap according to the British formula contains no sodium palmitate separation will not occur, even in cold weather, for potassium soap is soluble in alcohol, cold or warm, hence the British Pharmacopœia does not require cooling the liniment before filtering.

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* Section on Practical Pharmacy and Dispensing, St. Louis meeting, 1927.

Another peculiarity must be pointed out at this time which may affect the specific gravity and per cent of solids in the case of "Chloroform Liniment" which is prepared by agitating 30% of chloroform with 70% of soap liniment. The peculiarity in this case is that while soap liniment, as such, will show precipitation on standing in the cold, if the sodium palmitate had not been removed by twenty-four hours of cooling followed by filtration, my observations show that "Chloroform Liniment" will not precipitate on standing in the cold even though it had been prepared from soap liniment, the sodium palmitate of which had not been removed by cooling and filtering. This indicates then that the "would be precipitated" sodium palmitate is dissolved by the chloroform used in the manufacture of chloroform liniment, hence, a perfectly clear chloroform liniment might contain some of the "would be precipitated" sodium palmitate if it were prepared from a soap liniment which had not sufficiently cooled for twenty-four hours before filtering and in such case the per cent of solids is appreciably higher although the difference in specific gravity is negligible.

Having thus discussed the various factors which may affect the results I shall now outline the methods employed and results obtained.

METHODS.

The methods employed as suggested by Mr. Warren are as follows:

(*1*) *Specific Gravity.*—This was determined at 25/25° C. by means of a pycnometer.

(*2*) *Optical Rotatory Power.*—This was determined at 20° C. by the use of both 100-mm. and 200-mm. tubes and the findings reported in angular degrees. If the material be too dark for direct reading it will clear up considerably by diluting it with one of three volumes of alcohol at 20° C. and report findings accordingly.

(*3*) *Per Cent of Solids.*—This was determined by placing 1 Gm. of sand and a stirring rod in a beaker of about 150-cc. capacity and drying at 100° C. to constant weight. To this 10 cc. of the liniment in question was added by means of a pipette. The mixture was then evaporated on a steam-bath with occasional stirring until the odor of camphor had disappeared. The residue was then dried in an oven at 110° to constant weight.

RESULTS.

Linimentum Saponis—U. S. P. X.

1. Specific Gravity.—0.876 at 25° C. (average of 3 trials).

2. Optical Rotatory Power.—(*a*) Using a 100-mm. tube (average of 7 trials) +1.51° Dextrorotatory. (*b*) Using a 200-mm. tube (average of 5 trials) +3.34° Dextrorotatory.

3. Per Cent of Solids.—5.955 Gm. in 100 cc. (average of 3 trials).

Linimentum Chloroformi—U. S. P. X.

1. Specific Gravity.—1.060 at 25° C. (average of 3 trials).

2. Optical Rotatory Power.—(*a*) Using 100-mm. tube (average of 7 trials) +1.23° Dextrorotatory. (*b*) Using 200-mm. tube (average of 5 trials) +2.30° Dextrorotatory.

3. *Solids on Drying.*—4.155 Gm. in 100 cc. (average of 3 trials).

The above determinations were made in November 1926, and a copy of same was forwarded to Mr. Warren; since then, on his suggestion, I kept divided portions under different conditions of storage (such as flint and amber-colored bottles, filled and partially filled bottles, exposure to sunlight and in a dark closet) until August 1927, when I undertook to determine what effect, if any, the different conditions of storage had on the two liniments. The results were so much similar to those obtained in November that to outline them would be repeating the first results with slight variations which are negligible. It is interesting to note that even in the case of chloroform liniment—a preparation with such a highly volatile constituent—where a variation in the results was expected no appreciable difference was observed. The paper of J. W. E. Harrisson from the laboratory of Charles H. LaWall of Philadelphia, read before the Section on Practical Pharmacy and Dispensing at the Cleveland meeting of the A. PH. A. in 1922 or 1923, contains similar results in regards to the stability of chloroform liniment kept for three months in glass or cork-stoppered containers. I may say that it was this information that led me to investigate the effects of keeping under different conditions of storage on **Linimentum Camphoræ** which will be next considered.

Early in the fall of 1926 an article appeared in a "health column" of one of Chicago's daily newspapers in which it was stated that "Camphorated Oil" will lose its strength, decompose or deteriorate, even if kept under the most suitable conditions of storage. Prof. Wm. B. Day, dean of the University of Illinois School of Pharmacy called my attention to this article which, in view of the report of Mr. Harrisson (to the effect that "Chloroform Liniment" was fairly stable under different conditions of storage) sounded very unlikely. I, therefore, undertook to determine whether the article in the "health column" is correct and, in December 1926, I prepared a sample of camphorated oil in accordance with the U. S. P. X and determined the specific gravity at 25/25° C. by the use of a pycnometer and the per cent of camphor following the assay process as outlined in the U. S. P. X, which consists of heating a definite weight of camphorated oil in a tared porcelain dish at about 110° C. for 90 minutes (or until the odor of camphor has disappeared) and cooling and weighing; thus ascertaining the loss of camphor. I also kept portions of this liniment until August 1927, under different conditions of storage and tested again.

RESULTS.

Linimentum Camphoræ—U. S. P. X.

December 1926.

1. *Specific Gravity.*—0.939 at 25° C. (average of 2 trials).
   Per Cent of Camphor.—19.964% (average of 3 trials).

August 1927.

2. *Specific Gravity.*—0.938 at 25° C. (average of 2trials).
   Per Cent of Camphor.—19.959% (average of 3 trials).

CONCLUSION.

It follows that, contrary to expectations and the health column report, the above liniments discussed are fairly stable at ordinary conditions of storage,

the difference in stability under different forms of storage being negligible. I, therefore, recommend that the above standards be included under the respective preparations in the next Pharmacopœia—provided, of course, that the Revision Committee find it advisable and decide to make "official" data of this kind. I am of the opinion that the expression "Cool Place" in the U. S. P. X used in the directions for making soap liniment is too indefinite and conveys the idea that as long as it is kept for twenty-four hours away from the radiator or stove and not exposed to the direct sunlight it is sufficient compliance, but, for reasons pointed out before, it is not sufficient. Neither can satisfactory results be obtained by keeping it at or about 0° C. I should, therefore, suggest that a definite temperature or a range of temperatures, at least, be given in the next Pharmacopœia and my opinion is that about 10° C. would be cool enough to cause all the sodium palmitate to settle out and yet not so cold as to cause the preparation to congeal.

I take the opportunity at this time to acknowledge my indebtedness to Professors Clyde M. Snow and Albert H. Clark and S. W. Morrison of the University of Illinois School of Pharmacy and L. E. Warren of the Drug Research Unit for the valuable aid they have rendered in connection with this work.

BIBLIOGRAPHY.

J. W. E. Harrisson, JOUR. A. PH. A., 12 (1923), 333.

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ANALYSIS OF TINCTURE OF LEMON.

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Some time ago, one of us[1] reported the results of an investigation on Tincture of Sweet Orange Peel, showing the amounts of oil of orange and of alcohol that the official preparation should contain. The results of the work demonstrated that a tincture prepared according to the U. S. P. IX should contain not less than 1 per cent of oil of orange, and not more than 77 per cent of alcohol. In the present work, Tincture of Lemon was studied to determine the amount of oil of lemon present in the tincture prepared as directed by the U. S. P. X. The quantity of alcohol was also determined, although the importance of this assay is not so great as in the previous investigation, because the present pharmacopœia has set a standard for the amount of alcohol that should be present in the official tincture.

The lemons used included four different grades from California, and a mixed grade from the same state; the *Villa Franca* and the *Ponderosa* from Florida; and lemons from Sicily. The usual maceration period of three days was allowed in all cases, accompanied by the customary agitation. Alcohol of the U. S. P. X was used as menstruum, and was shown by analysis to meet the official requirements. The completed tinctures prepared according to the directions of the U. S. P. X, were yellow in color, and were possessed of an odor decidedly more aromatic and suggestive of lemon, than a solution of citral in water. They pro-

[1] F. A. Lee, "Analysis of Tincture of Sweet Orange Peel," THIS JOURNAL (Aug. 1924), 712.