

40 percent to 60 percent in value as compared with the original shipments from which the seeds were obtained. One strain has been continued under cultivation in the same locality and upon the same soil for four consecutive years, and its value as indicated by physiological tests has fluctuated between 40 percent and 65 percent. This fluctuation has been intermittent, and not in the nature of a regular annual increase or decrease. During this time, however, a marked improvement has resulted in the size and character of the inflorescence. By selection, this has become heavier, more compact, larger and less leafy. A dwarf form has also resulted which would greatly simplify the process of collection.

Figures No. I, II, III and IV show some of the experimental plots, and convey some idea of the scale upon which the work is being done. Large numbers of plants are being used, and these are observed throughout the entire growing season before any selections are made. In this manner the entire life history of the plants, from earliest seedling stage to maturity, is made to serve as a record from which intelligent selections can be made.

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METHODS FOR THE ANALYSIS OF CASTILE SOAP.*

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Aside from the fact that the sale of a spurious castile soap may subject the seller to legal prosecution, its use causes the difficulty so frequently encountered in preparing soap liniment, and as many soaps sold as castile are not what they are labeled it is necessary to subject samples to analysis in order to determine whether they are properly made olive oil soaps.

In my own work I have employed the following methods with excellent results:

Sampling. Select a sample which is representative of the whole lot or bar. If in the latter form, shavings should be taken from different parts, such as the outer and inner surfaces, and after being thoroughly mixed kept in a tightly corked bottle from which samples are taken for analysis.

Water. The method of U. S. P., that is taking 0.500 gram of sample, placing in a previously tared beaker containing 1 gram of sand, adding 10 cc. of alcohol and evaporating to dryness and then drying at 110° C. to constant weight is entirely satisfactory. Care must be exercised in heating to conduct the evaporation on a water bath and to employ a small flame, otherwise the sand may be very forcibly ejected from the beaker and the determination ruined.

The quantity of water allowed by the Pharmacopœia, 36 percent, is excessive and should be very much reduced.

Tests for Animal Fats. The Pharmacopœia states that if a four percent alcoholic solution of soap be allowed to cool it should not gelatinize, indicating

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the absence of animal fats. The most satisfactory method of carrying out this test is to place the alcohol and soap in an Erlenmeyer flask and heat on a water bath, employing a reflux condenser to prevent evaporation. When solution is complete the material is allowed to cool to room temperature (not below 20° C.).

This test is not very satisfactory as the Pharmacopœia allows 36 percent of water and as most samples do not contain that much, it is easy to see that instead of a four percent solution one may have almost any strength, depending upon the quantity of moisture, the result being that the solution gelatinizes and indicates animal fat, where none was used.

Therefore the Pharmacopœia should provide tests to determine the origin of the fat employed in making the soap, such as determining the iodine number of the fatty acids and their melting points.

Separation of the Fatty Acids. To a portion of the soap dissolved in water add an excess of diluted sulphuric acid and heat on a water bath until the fatty acids rise to the top in a clear layer, then cool in ice water and when the fatty acids have solidified pour off the water. Repeat this heating and cooling process twice, then filter through paper wetted with water; this will retain the fatty acids which after drying are ready for the tests.

Iodine Number of Fatty Acids. Determine the iodine number of fatty acids as directed by the Pharmacopœia for fats and oils. The writer employs the Hanus method, and as suggested by him (Drug. Circ., 1910, page 106) this should be adopted as the official method because of the keeping qualities of the solution and the shorter time required to make a determination. Having determined the iodine number of the fatty acid a reference to Allen's Organic Analysis, will indicate the fat or oil which was employed in the preparation of the soap.

Melting Point of Fatty Acids. Take some of the fatty acids prepared as above, gently melt and immerse the bulb of a thermometer in the liquid; in a few seconds it will have congealed and all that remains is to put the thermometer through a cork in an ounce wide-mouth bottle and then suspend the bottle and thermometer in a beaker of water and heat the water slowly. The melting point is regarded as the time when the material forms a clear drop on the tip of the thermometer. Allen's Organic Analysis gives the melting points of various fatty acids.

Tests for Silica and Other Insoluble Matter. The Pharmacopœia determines these by dissolving 20 grams of soap in hot alcohol, washing with hot alcohol, then with hot water and weighing the insoluble residue as silica. A better plan is to take 5 grams of soap, dissolve in about 150 cc. hot water and collect the insoluble matter on a tared ashless filter paper and after washing with hot water drying at 105° C. and weighing. The increase in weight of the filter paper indicates the total insoluble matter. After igniting the residue represents insoluble mineral matter.

Sodium Carbonate. The Pharmacopœia directs 20 grams of soap to be dissolved in hot alcohol and poured on a tared filter paper; the increase in weight of the paper after washing with hot alcohol is regarded as sodium carbonate,

silica, etc. After pouring water on this the residue is silica and other insoluble matter; the difference between the two being regarded as sodium carbonate.

Free Alkali. If upon adding a few drops of alcoholic solution of phenolphthalein to the freshly cut surface of the soap a pink color is not developed, the absence of free caustic alkali is indicated.

The Pharmacopœial method of determining alkalinity is inexact and indefinite.

The method of determining sodium carbonate as above directed can with slight modification be employed for the quantitative determination of free alkali.

In place of 20 grams, take 2 grams of soap, dissolve in about 150 cc. of hot neutral alcohol and filter. After washing the filter thoroughly with hot neutral alcohol add phenolphthalein to filtrate and titrate with N/10 H_2SO_4 . The alkalinity found is calculated as free alkali due to sodium hydroxide. The material insoluble in alcohol is then dissolved in water and titrated with N/10 H_2SO_4 , using methyl orange as indicator. This alkalinity is calculated as free alkali due to sodium carbonate.

If the soap contains both free alkali and free fat the heating with alcohol will influence the result and for that reason it is often advantageous to follow Devine's method of determining free alkali (Journal American Chem. Soc., 1900, page 693), which is carried out as follows:

Weigh 2 grams of soap into a 300 cc. flask, add 50 cc. of alcohol, and excess of N/10 stearic acid in alcohol, a few drops of phenolphthalein solution, connect with a reflux condenser and place the whole on a water bath for half an hour. The stearic acid should constantly be in excess, indicated by the solution remaining colorless. The excess of acid is determined by means of N/10 alcoholic KOH, the difference is the amount required to combine with the total free alkali in the 2 grams of soap taken.

One cc. N/10 acid is the equivalent of .00397 gram caustic soda or .00526 gram sodium carbonate.

Should it be necessary to determine what quantity of the above is free caustic alkali and what quantity is carbonated, Devine's method provides that a second determination similar to the first be started and having calculated the total alkali from the first determination as sodium carbonate add barium chloride to precipitate the alkali, heat a few minutes and after adding phenolphthalein titrate with N/10 stearic acid. This figure represents the cc. required to neutralize the caustic alkali in the soap and the difference between this and the total alkali will correspond to the carbonate.

Refractive Index of Fatty Acids. The determination of the refractive index of the fatty acid often gives valuable information with reference to the origin of the fat employed in making the soap. This determination is easily made if a refractometer is at hand.

More tests could be applied by the pharmacist to enable him to differentiate genuine and spurious olive oil soaps; the above in addition to being sufficient are simple, accurate and easily performed and should therefore be considered for inclusion in the Pharmacopœia.