

produce a white loaf of bread, and, to do this, he must have white flour. As has been stated, the flour from the wheat graded No. 4 Northern was not equal in this respect to that from the higher grades. No figures were obtained that would bring out the differences in color, excepting that it was considered in allotting marks for quality in the bread. In fact, it was the chief cause for the lower marks given to the bread from the lower grades of wheat.

In general, the work here reported indicates that the milling, chemical and baking tests fail to bring out any very wide difference in the products of the individual lots of wheat studied. In yield of flour and in color of the bread the products of the lower grades are inferior, but in strength, as shown by the chemical analysis and in yield of bread, there appears to be little or no difference.

When we take into consideration the fact that the spread in price between No. 1 Hard and No. 4 Northern may amount to twenty-five or even forty cents per bushel, it would appear as though, in this case, the lower grades would not receive the price to which they were entitled, and, consequently, the farmer producing them would not receive justice. On the other hand, it is only fair to state that, according to our system of grading, many other samples graded down for other causes than being frosted may be much inferior in quality and thus reduce the standard of these lower grades. The price, however, is regulated by the quality of the grade as a whole and it may thus happen that individual lots are not paid for in their just merits.

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## **DETERMINATION OF MERCURY AND IODINE IN ANTI- SEPTIC SOAPS.**

BY **ATHERTON SEIDELL.**

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THE germicidal character of the halogen salts of mercury has been recognized for a number of years, and comparative experiments, as quoted by Sternberg,<sup>1</sup> have shown that the iodide has

<sup>1</sup> "Text Book of Bacteriology," 1896, p. 188.

more than double the antiseptic value of the bichloride. It has been found by Thomson<sup>1</sup> that mercuric iodide can be introduced into soap without forming an insoluble compound with the fatty acids or losing its antiseptic properties. The mixture is effected by first dissolving the red iodide in a solution of an alkaline iodide and then incorporating it in the melted soap stock. Samples prepared in this manner were found by experiments upon a number of different organisms to be markedly germicidal in their action. More recent experiments along this line appear to have confirmed the observations of Thomson and at present there are a number of mercuric iodide soaps on the market. Rideal, in his book upon "Disinfection and the Preservation of Food," third edition (1903), page 400, states that samples of mercuric iodide soap were found upon analysis to contain 0.37, 0.9 and 3.4 per cents., respectively, of potassium mercuric iodide. Neither the analytical procedure by which these determinations were made nor a reference to it are given by Rideal. Other reference books and a number of journals were examined without success for descriptions of such analyses.

Since no satisfactory method was found by which the mercury and iodine in these soaps could be quantitatively determined, the investigation described in the following pages was undertaken. The plan which first suggested itself was to decompose the fatty material by some suitable means and estimate the mercury and iodine in the residue obtained. Among the methods tried and found unsatisfactory for accomplishing this purpose, were, first, treatment with fuming nitric acid both at the temperature of the steam-bath and in sealed tubes by the method of Carius; second, digestion in a Kjeldahl flask with concentrated sulphuric acid; finally, alternate charring and extraction with acid.

The fatty acids could, of course, be removed from the acidified aqueous solution of the soap by filtration or shaking out with ether, but in either case an unavoidable error would result on account of the solubility of mercuric iodide in the fatty bodies<sup>2</sup>

<sup>1</sup> Thomson: *J. Chem. Ind.* 7, 192 (1888). The *J. Chem. Ind.* reports the following patents obtained by Thomson: "An Improved Antiseptic Soap," English Patent No. 9591, July 24, 1886; "Improvements in the Composition of Mercurial Soaps," No. 7117, May 16, 1887. The *Chemische Centralblatt* reports the German patent, "Preparation of Antiseptic Soaps," D. R. P. No. 49,119, March 22, 1888.

<sup>2</sup> "Solubility of Mercuric Iodide in Fatty Bodies and in Some Other Solvents." Mèhu: *J. Pharm. Chim.* [5] 12, 249 (1885).

on the one hand and its solubility in the immiscible solvents<sup>1</sup> on the other.

After considering all these points experiments were made as follows, using a sample of soap prepared in the laboratory by mixing a weighed amount of white toilet soap with a solution of mercuric iodide in potassium iodide. Portions of this sample were dissolved in warm water, acidified with hydrochloric acid and, after cooling, an immiscible solvent added to take up the separated fatty acids. Hydrogen sulphide was then passed through the two layers of solution until precipitation appeared complete. The two layers of solution were filtered together and in some cases separately, but in spite of the greatest care a portion of the fatty material would separate from solution and remain on the sides of the Gooch crucible. Great difficulty was experienced in endeavoring to remove this deposit and on this account it became necessary to modify the procedure.

After a number of trials it was finally ascertained that advantage could be taken of the solubility of the fatty acids in strong alcohol, to obtain a homogeneous solution of both the organic and inorganic constituents of the sample. The solution is best obtained by adding to the weighed sample of soap contained in an Erlenmeyer flask about 150 cc. of 95 per cent. alcohol, and 3 to 5 cc. of concentrated hydrochloric acid. The solution is warmed and successive small portions of water added until a perfectly clear solution is obtained on shaking. If suspended particles of impurities are present, the solution should, of course, be filtered. A slow stream of hydrogen sulphide gas is then passed through the liquid for about an hour, after which time the precipitated mercuric sulphide is filtered on a carefully prepared Gooch crucible. The fatty material present in the solution appears to have a tendency to cause the precipitate to pass through the filter, but no trouble on this account need be experienced, if care is taken to use the least amount of suction possible until the sulphide has been washed several times with 95 per cent. alcohol. Although considerable time is required for this filtration, no great delay need be encountered, if a number of determinations are made

<sup>1</sup> "Notice Concerning the Solubility of Mercury Haloid Salts, Especially of Mercuric Iodide in Organic Solvents," *Sulc: Z. anorg. Chem.* **25**, 399 (1900). "On the Effect of Various Solvents on the Allotropic Change of Mercuric Iodide," Kastle and Clarke: *Am. Ch. J.* **22**, 473 (1899).

simultaneously. The weight of mercuric sulphide obtained multiplied by 1.955 gives the amount of mercuric iodide in the weight of sample used.

The filtrate and washings from the mercuric sulphide are transferred to a beaker and placed on the steam-bath. When the volume of the solution has been reduced to about one-half or one-third of the original volume, water is added to replace the alcohol, the solution cooled and the separated fats removed by filtration. The clear solution obtained is placed in a glass stoppered separatory funnel together with 25 to 50 cc. of chloroform. The combined iodine is then liberated by the addition of a few drops of nitrous acid. The iodine thus set free is quickly dissolved by the chloroform on shaking, and by repeating the operation two or three times the whole of the iodine is found in the chloroform solution. This latter is then washed by being shaken several times with distilled water. The washings are treated with fresh chloroform to recover any iodine which may have been taken up by them. Finally, the chloroform solution of the iodine is titrated to loss of color with standard sodium thiosulphate solution. The number of cubic centimeters required multiplied by the factor for the standard solution gives the grams of iodine in the sample used.

The nitrous acid solution is prepared by adding to a mixture of about 10 grams of starch and 10 grams of arsenious acid contained in a 700 cc. Erlenmeyer flask about 150 cc. of nitric acid of 1.3 specific gravity. The solution is warmed gently and the reddish  $\text{NO} + \text{NO}_2$  fumes conducted by means of a suitably bent glass tube into a bottle containing about 100 cc. of concentrated sulphuric acid. The solution keeps well and only a few drops are required at a time to liberate the iodine present in the solution under examination.

The method as described above was used for the analysis of several samples of mercuric iodide soap prepared in the laboratory and purchased samples of soap claimed to contain certain amounts of mercuric iodide. The results obtained are shown in the accompanying table.

## RESULTS OF THE DETERMINATIONS OF THE MERCURY AND IODINE IN SOAPS CONTAINING MERCURIC IODIDE.

Sample.	Soap taken. Grams.	HgI <sub>2</sub> added or claimed as present. Per cent.	HgI <sub>2</sub> found. Per cent.	I (total) found. Per cent.	I as alkali iodide (calculated). Per cent.
A .....	10.0	0.83	0.92	1.00	0.48
A .....	10.0	0.83	0.72	0.94	0.54
A .....	10.0	0.83	0.85	1.18	0.71
A .....	15.0	0.83	0.96	1.07	0.54
B .....	3.14	1.0	1.13	1.63	1.00
C .....	3.69	1.0	0.70	0.97	0.58
C .....	2.87	1.0	0.78	0.90	0.46
D .....	6.39	1.0	1.21	1.39	0.72
D .....	8.38	1.0	0.94	1.30	0.78
E .....	4.33	0.1	0.11	0.17	0.11
E .....	11.80	0.1	0.10	0.22	0.16
E .....	3.15	0.1	0.10	0.12	0.06

Sample A prepared in the laboratory.

B, C and D, different samples of the same brand of soap.

Sample E, a purchased preparation.

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## A METHOD FOR THE DETERMINATION OF BLACK ALKALI IN IRRIGATING WATERS AND SOIL EXTRACTS.<sup>1</sup>

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SUCCESSFUL irrigation in an arid region depends to a large extent upon a knowledge of the soluble salts, the so-called alkali, in both soils and the water to be applied to the soils. This is particularly true of black alkali, which is the term applied to certain salts that have a corrosive action upon plants and also have the power of dissolving the humus content of soils. It is from this last characteristic that the prefix black originated, for the humus dissolved by the alkali gives the solution a dark brown or black color, which, when collected in depressions of the soil and evaporated to dryness, produces the very characteristic black alkali spots. Black alkali may therefore be the normal carbonate or bicarbonate of any of the alkalies. As a matter of fact, however, the term has been restricted to mean the carbonate and bicarbonate of sodium. The carbonates of potas-

<sup>1</sup> By permission of the Secretary of Agriculture.