BIBLIOGRAPHY.

1.—"Diethylphthalate," J. A. Handy and L. F. Hoyt, JOUR. A. PH. A., 11, 929–939 (Nov. 1922).

2.—"Detection of Diethylphthalate in Whisky," A. B. Lyons, JOUR. A. PH. A., 11, 262–265, 1922.

3.—"Diethylphthalate," R. E. Andrew, paper read April 6, 1923, at the New Haven meeting of the American Chemical Society; published in *Ind. Eng. Chem.*, 15, 838 (August 1923), under title of "A Modified Test for Phthalates."

4.—"Detection of Diethylphthalate and Phthaleins," R. L. Calvert, *Am. J. Pharm.*, 94, 702–3, 1922.

5.—"Dyes Derived from Saccharin. The Sulphamphthaleins," S. Dutt, *J. Chem. Soc. (London)*, 121, 2389–94, 1922; *Chem. Abstr.*, 17, 554.

6.—"A Practical Method for the Preparation of Phenolsulphonephthalein," E. A. Slagle, *J. Pharm.*, 1, 663–6; *Chem. Abstr.*, 4, 2545, 1910.

7.—"Some Interesting Reactions of Resorcinol," E. Lang, *Bull. Soc. Ind. Mulhouse*, 89, 24–30, 1923; *Chem. Abstr.*, 17, 2052–3.

8.—"Condensation of Diphenic Anhydride with Resorcinol," F. Bischoff and H. Adkins, *J. Am. Chem. Soc.*, 45, 1030–33, 1923.

9.—"Diethylphthalate," Steinkopff, *Pharmazeutische Zeitung*, 68, 86 (Jan. 1923).

10.—Address by J. M. Doran, *American Perfumer*, 18, 63 (Apr. 1923).

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ZINC HYPOCHLORITE.

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The preparation of solid zinc hypochlorite was attempted by Lunge and Landolt.[1] Their procedure consisted in passing dried chlorine gas over freshly prepared and dried zinc hydroxide. They also attempted to prepare magnesium hypochlorite in a similar manner. However, their results were negative in both cases.

In the preparation of calcium hypochlorite it has been found that the presence of water is a necessary condition, the degree of chlorination varying with the original water content of the calcium hydroxide. Calcium hypochlorite, having an available chlorine content of 76.62%, has been described by Hofmann and Ritter.[2] This material was furnished them by the Griesheim-Elektron Company and was prepared according to the specifications of a German patent.[3] The manufacture of pure calcium hypochlorite $Ca(OCl)_2$ is described in a later patent.[4] The specifications of this patent state that chlorine gas is passed through milk of lime, the suspended calcium hydroxide dissolves and a precipitate forms which redissolves after continued passage of chlorine gas through the solution. Upon the addition of a saturated solution of calcium chloride, pure calcium hypochlorite crystallizes from this solution.

Solutions of zinc hypochlorite are described in various books.[5] These solutions were prepared by passing chlorine gas through suspensions of zinc hydroxide or zinc oxide in water; the suspended material dissolves forming zinc hypochlorite, chloride and chlorate. In some of these experiments a solid residue remained after the chlorine gas ceased to be absorbed. It seems that none of these investigators examined this residue.

While analyzing a number of zinc hypochlorite solutions, the writer found that solutions containing some of this suspended residue had a larger available chlorine content than clear solutions. This led to an examination of the solid material.

It was found that the residues contain chlorinated compounds, the available chlorine content varying with the manner of chlorinating. Therefore, it seems probable that the presence of water is a necessary condition for the preparation of solid zinc hypochlorite.

To test this, chlorine gas was passed through suspensions of large amounts of zinc oxide in water. As the chlorination progresses a change is noticeable in the solid material; it becomes granular in structure, and if the material is permitted to stand in contact with the solution of zinc hypochlorite it forms a hard cake. Evolution of oxygen takes place at the surface of contact.

The solid residue when treated with acids liberates chlorine gas; dissolved in ammonia water, nitrogen is evolved. It chlorinates phenol and it liberates iodine from iodides in the presence of acids. The moist mass gradually loses its available chlorine, and also when the residue is dried. The loss upon drying is larger the higher the temperature.

When the chlorination proceeds in the presence of sodium hydroxide, it was found that the solid residue contains a larger amount of available chlorine.

No compound of definite structure was isolated. The highest available chlorine content of the chlorinated material was found to be 29.98%. The compound $ZnOCl_2$ contains 46.57% available chlorine. If the chlorine content of the above material is calculated as $ZnOCl_2$, it is found that this corresponds to a conversion of 72.96% of the residue into $ZnOCl_2$.

EXPERIMENTAL.

A. 25 Gm. of zinc oxide were triturated in a mortar with 300 cc. of water and the resulting suspension was strained through a fine mesh screen to separate the coarser particles. Through this suspension a stream of chlorine gas was passed until no further absorption of chlorine occurred. The container was cooled by a stream of water maintaining the temperature between 18° and 20° C. The material was stirred occasionally. The liquid was separated from the undissolved portion by means of a suction filter. The filtrate was removed and the residue on the filter was washed until the odor of chlorine was faintly perceptible. Part of this material was dried in a water jacketed drying oven for 48 hours at a temperature varying between 40° and 60° C. The analysis of the moist and dry materials yielded the following results.

	Percentage.
Moisture	78.83
Available Cl_2	3.85
Available Cl_2 calculated as dry mass	18.2
Available Cl_2 dry mass found	16.4
Available Cl_2 loss on drying	1.8

B. 25 Gm. of zinc oxide were mixed with 300 cc. of distilled water containing 50 cc. of 4 *N* sodium hydroxide solution. The experiment was conducted as described under A, excepting that during the chlorination the temperature rose to 48° C. for a short time, and that for the last 8 hours of the drying period the temperature of the drying oven was maintained at 80° C. The following results were obtained by analyzing the materials.

	Percentage.
Zinc Oxide	11.
Moisture	82.14

Available Cl_2	5.35
Available Cl_2 calculated as dry mass	29.98
Available Cl_2 dry mass found	17.96
Available Cl_2 loss on drying	12.02

The moist material was kept in a tightly closed vessel. After a lapse of two weeks the contents were analyzed for the available chlorine.

	Percentage.
Available Cl_2 at the beginning	5.35
Available Cl_2 after two weeks	3.67
Available Cl_2 loss on standing	1.68

SUMMARY.

Dry chlorinated zinc compounds have been prepared with an available chlorine content of 16.4% and 17.96%, respectively.

Moist chlorinated zinc compounds lose their available chlorine content at an appreciable rate.

In the presence of OH^-, the chlorination produces a mixture of larger available chlorine content.

Pure zinc hypochlorite should be an important therapeutic agent, that may be of service in the treatment of certain diseases of the eyes. The advantages of this chemical are manifold, since it possesses the combined oxidizing and antiseptic properties of the OCl^- and the astringent property of the Zn^{++}.

At a later date, work on this investigation will be resumed.

REFERENCES.

1. *Z. Ch. Ind.*, v. 8, 1885, p. 337. C. 1886, p. 57.
2. B. 47, p. 2233.
3. D. R. P. 188,521. C. 1907, II, p. 1950.
4. D. R. P. 195,896. C. 1908, I, p. 1109.
5. Gmelin-Kraut, "Handbuch der Chemie;" Moissan, "Traite de chemie."

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THE ANALYSIS OF TINCTURE OF SWEET ORANGE PEEL.

BY FRANK A. LEE.

Recently we had occasion to examine samples of so-called "Tincture of Sweet Orange Peel U. S. P.," to determine whether or not these were as labeled. The State Drug Inspector suspected that they were made from dried orange peel, rather than the peel grated from the fresh fruit, as directed by the U. S. P., and were intended primarily to be consumed as beverages. In the drying of the orange peel, practically all of the oil is lost. The samples of the tincture gave little or no precipitation on the addition of water; the taste was not bitter as would be expected. In view of the fact that the alcohol used as a menstruum for such purposes runs about 5% water, and considering the moisture content of the grated rinds of fresh oranges, the alcohol present should presumably be about 70 or 75%. Our results as tabulated below tend to confirm this assumption. The labels of the tinctures submitted stated alcohol content 85%. The most important index of purity should be the amount of orange oil present and, secondly, the amount of alcohol.

These results were obtained from samples prepared according to U. S. P. IX,[1]

[1] U. S. P. IX, p. 447.