In the preceding series of tests on ampuls, very slight differences in free alkali were observed. The results would indicate that regardless of the initial p_H, the final results are practically identical. We are dealing, in the case of ampuls, with very slight amounts of alkali.

An analysis of the series of tests on bottles reveals that regardless of the initial p_H, up to 6.0, the gain in p_H is similar. When the original p_H of water is adjusted to 7.0, the increase in p_H upon applying the heat test is but 60% of that obtained with original water at 5.5 p_H.

The length of time of the heat test used in our procedure is too severe, as will be shown by the following observation:

Bottles of the same stock were completely filled with water of known p_H. The sealed bottles were permitted to stand at room temperature for one year. The gain in p_H in one year at room temperature was but 0.5. Ordinary glass bottles increase the p_H of water 3.0 after 16 hours at 80–90° C.

SUMMARY.

This paper was written with the object in mind of drawing attention to the necessity of the establishment of a standard method of determining the free alkali in glass.

In our work we attempted to obtain purely relative results in order to make fair comparisons of glass.

A standardized method should be established only for glass bottles and ampuls that are used for finely adjusted pharmaceutical and chemical solutions.

ANALYTICAL DEPARTMENT,
PARKE, DAVIS & CO.

A NOTE ON THE U. S. P. MONOGRAPH ON CHRYSAROBIN.*,[1]

BY JOHN H. GARDNER.

For several years past, there has been an investigation in progress in this laboratory on the constituents of the anthracene drugs. In the early part of this work, it was shown that the acetate of chrysophanic acid-9-anthranol can be isolated from demethylated and acetylated chrysarobin (1), indicating that chrysophanic acid-9-anthrone is a constituent of the drug. On repeating this work with several samples of chrysarobin, it was found that the yields were extremely variable, ranging from nearly zero to about fifty per cent of the weight of the drug taken. In attempting to trace the cause of this variation, all of the samples were subjected to the tests for identity given in the U. S. P. monograph on chrysarobin. For comparison, similar tests were made on pure chrysophanic acid and on pure, synthetic chrysophanic acid-9-anthrone. The results of these tests are given in Table I.

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* From the Chemical Laboratory of Washington University, St. Louis. This investigation was made possible by a grant from the fund given by the Rockefeller Foundation to Washington University for research in science.

[1] Scientific Section, A. PH. A., Washington meeting, 1934.

TABLE I.

Reagent.	U. S. P.	Chrysarobin.	Chrysophanic Acid.	9-Anthrone.
5% NaOH	Deep red	Red	Red	Pink
Concd. H_2SO_4	Deep red	Brownish red	Cherry red	Yellow
Fuming HNO_3	Red brown	Red brown	Yellow	Light brown
$+NH_4OH$	Violet red	Brownish red + precipitate	Violet	Brown

It will be noted that in two of the four cases the results with chrysarobin differ decidedly with the tests given by the U. S. P., that is, with concentrated sulphuric acid and on treating the fuming nitric acid solution with ammonia. In the latter case, the monograph also states that chrysophanic acid gives a yellow color with those reagents. The production of a violet color was checked using chrysophanic acid from three sources, one sample from chrysarobin and two synthetic. The colors obtained with five samples of chrysarobin from as many different dealers gave the same colors, with the exception that a few samples showed some bluish green fluorescence in sodium hydroxide solution.

These results can only lead to one conclusion, that the tests for identity for chrysarobin given in the U. S. P. are incorrect and in need of revision. No data are as yet at hand to justify any positive suggestions.

REFERENCE.

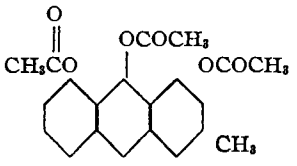
(1) Naylor and Gardner, *J. Am. Chem. Soc.*, 53 (1931), 4114.

THE PREPARATION OF CHRYSOPHANIC ACID FROM CHRYSAROBIN.*,[1]

BY JOHN H. GARDNER.

In the course of our studies on the chemistry of the natural purgatives it has become necessary to find a source from which chrysophanic acid can be readily obtained. Since chrysarobin is made up almost entirely of derivatives of chrysophanic acid and of emodin monomethyl ether, it seemed a logical material to investigate.

Several years ago, the author and Naylor (1) found that by demethylating chrysarobin with hydrobromic acid and acetylating the product, a mixture was formed from which chrysophanic acid-9-anthranol triacetate (Formula I) could be readily separated by fractional crystallization. Chrysophanic acid can be readily



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* From the Chemical Laboratory of Washington University, St. Louis. This investigation was made possible by a grant from the fund given by the Rockefeller Foundation to Washington University for research in science.

[1] Scientific Section, A. PH. A., Washington meeting, 1934.