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A COMPARISON OF NEOARSPHENAMINE AND SULPHARSPHENAMINE WHEN THEY ARE DIALYZED.*

BY A. E. JURIST AND W. G. CHRISTIANSEN.

It is well known that both neoarsphenamine and sulpharsphenamine are colloids, at least in part. Several investigators have subjected neoarsphenamine to dialysis and thereby demonstrated this fact. Extensive investigations have been made on neoarsphenamine by Freundlich, Stern and Zocker (1), Hirschfelder and Wright (2), and Raiziss and Gavron (3), but none of these investigators have compared neoarsphenamine and sulpharsphenamine. In this paper we shall describe a method for carrying out the dialysis of neoarsphenamine or sulphars-

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phenamine under anærobic conditions and the results obtained when this method is used.

These results show a very distinct difference in the behavior of these two compounds upon dialysis. When neoarsphenamine is dialyzed for twenty-four hours with running water 61.4% of the arsenic and 17.5% of the sulphur remain in the dialysis bag; these are averages from a number of experiments with a single brand of neoarsphenamine in which the undialyzable arsenic varied between 51.7 and 75.5% and the undialyzable sulphur between 13.6 and 21.7%. During the dialysis a precipitate forms in the originally clear solution; this precipitate dissolves upon addition of sodium hydroxide and re-forms when CO_2 is passed into the alkaline solution. On the other hand sulpharsphenamine shows only 39.7% of the arsenic and 28.9% of the sulphur still undialyzed; these are averages of several experiments with a single brand of sulpharsphenamine in which the undialyzable arsenic varied between 28.0 and 55.18% and the undialyzable sulphur between 16.7 and 35.77%. Sulpharsphenamine solutions remain clear throughout the dialysis.

Since the per cent arsenic lost by dialysis of sulpharsphenamine is greater than with neoarsphenamine and since the reverse is true for the sulphur content, there is strong evidence that a distinct difference exists in the structures of neoarsphenamine and sulpharsphenamine. This fact was first pointed out by Jurist and Christiansen (4). However, in addition to the differences in the chemical structure of these compounds a distinct dissimilarity in colloidal character is indicated. This can be ascribed either to the fact that the colloidal particles of neoarsphenamine are much larger than those of sulpharsphenamine or to the fact that a much larger portion of the latter is in the true solute form. The latter is the more probable explanation since the dialyzing membrane used here is of a type which usually retains colloids even when highly dispersed.

In addition to these experiments on a single brand of neoarsphenamine and a single brand of sulpharsphenamine a series of experiments were carried out on single lots of the different market brands of neoarsphenamine. The results of these experiments are briefly summarized in the following tabulation.

Brand of Neoarsphenamine.	% of Original Arsenic Undialyzed.	% of Original Sulphur Undialyzed.	Condition of Undialyzed Solution.
A	76.8	16.4	Precipitated
B	44.9	11.2	Clear
C	73.9	10.4	Precipitated
D	63.0	19.0	Precipitated
E	57.7	9.8	Precipitated
F	29.3	21.5	Clear
G	54.6	14.8	Precipitated

These results show wide variations in the undialyzable arsenic ranging from 29.3% to 76.3% and definite, but smaller differences in the undialyzable sulphur ranging from 9.8 to 21.5%. The results obtained with brands "C" and "G" are very similar to those obtained with sulpharsphenamine and further these two brands show one of the other characteristics of sulpharsphenamine, namely, that the undialyzed solution is clear and not precipitated. This series of experiments further emphasizes the fact that there are wide differences between different brands of neoarsphenamine, especially since two brands show characteristics more like those of sulpharsphenamine than neoarsphenamine. The differences in the chemi-

cal composition of market brands of neoarsphenamine pointed out by Elvove (5) and by Jurist and Christiansen (6) appear to extend also to differences in behavior on dialysis.

EXPERIMENTAL.

A solution of 6 Gm. of Parlodion (DuPont) in 50 cc. of ether and 50 cc. of ethyl alcohol is prepared according to the directions of Eggerth (7). This solution is poured into a clean, dry 500-cc. Erlenmeyer flask, and by rotating the flask as the solution is poured out slowly the entire inside of the flask is coated with the solution. The flask is then allowed to drain, inverted for 15 minutes. Then the membrane in the upper part of the neck of the flask is loosened by means of a knife blade. The inside of the flask containing the membrane is filled with water and then emptied to insure wetting the entire inner surface of the membrane, then water is poured between the membrane and the wall of the flask, loosening the membrane from the side of the flask as the flask fills. When the membrane has been loosened from the flask it can be easily pulled out; it must not be allowed to dry out. Immediately cut off that portion of the membrane which was inside the neck of the flask. Carefully open the membrane at the top so that a glass tube $^1/_2$ inch in diameter can be inserted for a distance of $^3/_4$ of an inch. Then the membrane is attached to this tube by wrapping around the tube a $^1/_2$ inch wide strip of adhesive tape. This should fit as tightly as possible. Then the membrane in the collapsed state is put in the dialysis bath and suspended in it by means of a clamp on the glass tube. The membrane is filled, through the glass tube, with water and lifted slightly so that 1.5 inches of the membrane are out of the water and exposed to the air. Then the exposed portion of the membrane as well as the strip of adhesive tape and the glass tube for a short distance are painted with a complete but thin coat of shellac. Any excess of shellac lying on the surface of the water of the dialysis bath is immediately skimmed off before it can harden. When the shellac is dried (about one hour is required for this), the exposed portions of the membrane, etc., are painted with a coat of the same Parlodion solution which was used in preparing the membrane. When this has dried the membrane is carefully emptied by removing it from the clamp and inverting it. This also serves to sweep the air out of the membrane as it collapses. It is then put back in the bath and the neoarsphenamine solution put in it. In our experiments 0.9 Gm. of neoarsphenamine was dissolved in 20 cc. of water. The atmosphere above the neoarsphenamine solution is cleared of air with a nitrogen stream using care to avoid breaking the membrane. Then a slight positive pressure of nitrogen is obtained by closing the glass tube with a one-hole stopper which is connected to a small gasometer. Before the membrane is expanded by the gas pressure it should be lifted by means of the clamp and glass tube to such a position that the surface of the neoarsphenamine solution inside the membrane is just below the surface of the water in the dialysis bath. The membrane containing the solution must then be lowered from time to time as the dialysis proceeds and causes it to increase in volume. Such an apparatus as this has been found to be entirely leak-proof and can be maintained under anærobic conditions. A sufficiently extensive dialysis of neoarsphenamine has been obtained by dialyzing in running water for 24 hours. The temperature of the water was maintained at 20° C. throughout.

When the dialysis has been completed the solution is transferred anærobically to a glass-stoppered cylinder. Any precipitate present is dissolved by means of sodium hydroxide. Then aliquot portions of this solution are used for arsenic and sulphur assays. The arsenic was determined by the Newbery (8) method and the sulphur by the method described by Elvove (5). The per cent of the total sulphur and arsenic remaining after dialysis can then be readily obtained by the following:

$$\frac{\text{Gm. Arsenic Undialyzed} \times 100}{\text{Gm. Total Arsenic Present}} = \% \text{ Arsenic Undialyzed}$$

$$\frac{\text{Gm. Sulphur Undialyzed} \times 100}{\text{Gm. Total Sulphur Present}} = \% \text{ Sulphur Undialyzed}$$

This method has been applied successfully to both neoarsphenamine and sulpharsphenamine and typical results are given in the table below. It is interesting

to note that at the end of 24 hours' dialysis there was a precipitate in the solution remaining in the dialysis bag in the case of neoarsphenamine, but none in the case of sulpharsphenamine. This precipitate was flocculent in character and was readily soluble in sodium hydroxide.

TABLE I.

Compound.	Total Arsenic Present—Mg. before Dialysis.	Total Sulphur Present—Mg. before Dialysis.	Undialyzed Material. Arsenic. Mg.	Arsenic. %	Sulphur. Mg.	Sulphur. %
Neoarsphenamine	178.2	56.4	111.0	66.3	10.4	18.5
Sulpharsphenamine	197.5	95.4	72.4	38.4	29.7	31.1

CONCLUSIONS.

1. These results show that neoarsphenamine has a larger undialyzable arsenic content than sulpharsphenamine.

2. It is also shown that the sulphur content of sulpharsphenamine is less readily removed by dialysis than that of neoarsphenamine.

3. In the course of the dialysis of neoarsphenamine a portion of the material remaining undialyzed precipitates in the dialysis bag. This is not true in the case of sulpharsphenamine.

4. The previous conclusion of a fundamental structural difference between neoarsphenamine and sulpharsphenamine is confirmed.

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THE POTASSIUM MERCURIC IODIDE REAGENTS FOR ALKALOIDS.

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A number of potassium mercuric iodide solutions have been recommended as precipitating reagents for alkaloids. Of these, Mayer's reagent is the most widely used and is regarded as an exceedingly sensitive qualitative solution for alkaloids in general. It was found, however, that this reagent would not detect codeine unless present in a concentration of at least 1 in 5000 parts, but that modification of the reagent rendered the reaction with codeine and other alkaloids much more delicate. These experiments show that Mayer's reagent, which is advocated as a qualitative test solution by the United States Pharmacopœia X and by textbooks generally, is probably the least sensitive of the potassium mercuric iodide reagents which have been described. It seemed worth while, therefore,