

THE LEAD CONTENT OF BUROW'S SOLUTION.*

BY JOSEPH L. MAYER.

Burow's Solution, official in the NATIONAL FORMULARY as Liquor Alumini Acetatis, is prepared by dissolving 150 grams of lead acetate in 500 cc of water, and pouring this in cold solution in a thin stream, with constant stirring, into a cold solution of 85 grams of aluminum sulphate in 500 cc of water. The mixture is then set aside in a cold place (about 10° C.) for 24 hours, stirring it occasionally and, finally, making the filtered liquid measure 1000 cc.

The quantities of lead acetate and aluminum sulphate employed are the result of stoichiometric calculations, the object being to produce a solution containing 5% of aluminum acetate, with practically no lead in the finished preparation.

The directions to employ cold solutions and set the mixture aside at a temperature of about 10° C. have for their object the precipitation of lead sulphate, which is formed as a result of the chemical interaction of the lead acetate and aluminum sulphate employed in the preparation of the solution. This procedure is based upon the solubility of lead sulphate in pure water which, according to Seidel, is 0.035 gram in 1000 cc water at 10° C. ("Solubility of Inorganic and Organic Compounds," 2nd edition, page 363.)

Being interested in the question of how much lead Burow's Solution, made according to the NATIONAL FORMULARY, should contain, and, having consulted various reference books without finding any other information than that there should be a trace only, I conducted the following experiments.

A sample of uneffloresced lead acetate, taken from a cork-stoppered bottle, was assayed by the U. S. P. method; it showed the presence of 87.099% of anhydrous lead acetate, which meets the official requirement—"It contains not less than 85.31 percent nor more than 89.57 percent of anhydrous lead acetate."

A sample of aluminum sulphate, taken from a cork-stoppered bottle, when assayed by the method official in the NATIONAL FORMULARY, showed the presence of 16.458% aluminum oxide; this fully meets the requirement of the N. F.—"The weight of aluminum oxide is not less than 16.1 percent of the weight of aluminum sulphate."

Employing these chemicals, making all weighings on an analytical balance with analytical weights, and the final measurements in a glass-stoppered volumetric flask, 200 cc of Burow's Solution were made in strict accordance with the directions of the N. F. The finished product giving heavy precipitates with all of the commonly employed reagents for lead, thus indicating the presence of more than a trace of the metal, I quantitatively determined the amount by the following methods:

A. Five cc of the solution—acidified with slight excess of nitric acid, heated to boiling and electrolyzed for about 3 hours, 3 volts, 1½ amperes—showed the presence of 0.070 gram of lead as metallic lead per 100 cc.

B. Ten cc of the solution, to which a slight excess of sulphuric acid was added, were evaporated down to white fumes; 75 cc of cold distilled water were cautiously added, and the solution stirred thoroughly, 25 cc of alcohol were then added, and the solution allowed to stand over night. The material was then filtered through asbestos on a Gooch crucible; the precipitate was washed with alcohol until free from acid, the lead sulphate was dissolved in ammonium acetate solution.

* Read before New York State Pharmaceutical Association, 1922.

and the crucible and its contents thoroughly washed. This filtrate and wash water were made slightly acid with acetic acid and heated to boiling; the lead was precipitated by means of boiling 10% potassium chromate solution, slowly added from a pipette, the material being stirred vigorously during the precipitation. After the lead chromate had settled the mixture was filtered, while hot, through asbestos on a Gooch crucible and washed with hot distilled water until the filtrate did not show the slightest tinge of yellow, after which the precipitate was dried at about 150° to constant weight.

This method showed the presence of 0.068 gram of lead as metallic lead per 100 cc.

C. Twenty-five cc of Burow's Solution were diluted to about 300 cc with distilled water, and 5 cc glacial acetic acid added; the mixture was heated to boiling; the lead was precipitated by means of boiling potassium chromate solution and the analysis completed as under B.

The analysis showed the presence of 0.0666 gram of lead as metallic lead per 100 cc. This method requires less time for completing the assay than the above in which the lead is precipitated as sulphate and dissolved in ammonium acetate before adding the potassium chromate, and yields results which are practically identical.

Having determined the quantity of lead present, it occurred to me that it would be a good plan to quantitatively estimate the amount of aluminum present in this same sample of Burow's Solution, before and after the removal of the lead, therefore I made these analyses with results as follows:

D. The original solution, assayed by the method official in the NATIONAL FORMULARY, showed the presence of 0.01326 gram of aluminum as Al_2O_3 per cc.

E. Another portion of the original solution which had been acidified with hydrochloric acid and from which the lead was precipitated by means of H_2S , showed the presence of 0.01298 gram of aluminum as Al_2O_3 per cc.

SUMMARY.

1. Burow's Solution made strictly according to the NATIONAL FORMULARY shows the presence of 0.0682 gram of lead as metallic lead per 100 cc.

2. The results of the three methods submitted for the quantitative determination of lead are as follows:

(a) Electrolytic	0.0700 Gm. lead as metallic lead per 100 cc
(b) Precipitation as $PbSO_4$, solution in $NH_4C_2H_3O_2$, precipitation with K_2CrO_4	0.0680 Gm. lead as metallic lead per 100 cc
(c) Precipitation with K_2CrO_4 in presence of $HC_2H_3O_2$	0.0666 Gm. lead as metallic lead per 100 cc

	0.2046
Mean,	0.0682

Method C, which is a simple and rapid one, yields results practically the same as the others, which are more involved and time-consuming. The object of employing the glacial acetic acid in this method, is to keep the aluminum chromate in solution without affecting the insolubility of the lead chromate and, therefore, the lead need not be separated from the aluminum as it is in Method B.

These results confirm what I expected—that while the solubility of lead sulphate at 10° C. is 0.035 gram in 1000 cc of water, it would be very much greater in the presence of the aluminum acetate and acetic acid contained in the Burow's Solution and, therefore, there would be more than a trace of lead, even in a preparation made from uneffloresced lead acetate.

3. The original solution shows the presence of 0.01326 gram of aluminum as Al_2O_3 per cc.

The original solution, freed from lead by means of H_2S , shows the presence of 0.01296 gram aluminum as Al_2O_3 per cc, thus indicating that the results are practically identical whether the aluminum is determined in the presence or absence of lead.

4. The amount of aluminum as Al_2O_3 found—0.01326 gram per cc in the presence of lead and 0.01298 gram per cc in the solution freed from lead by H_2S —proves that the NATIONAL FORMULARY standard, "each milliliter of solution of aluminum acetate corresponds to not less than 0.01126 Gm. nor more than 0.01376 Gm. of aluminum oxide (Al_2O_3)," is correct.

RESEARCH AND ANALYTICAL LABORATORIES
OF THE LOUIS K. LIGGETT COMPANY, NEW YORK.

THE DETERMINATION OF TOTAL IRON IN PHARMACEUTICAL PREPARATIONS.

BY J. FORBES WILLIAMS.

There are numerous official syrups, elixirs, tinctures and pills which contain iron in various forms. In most of these preparations no assay is given for the iron determination. Besides the official preparations there are hundreds of tonics on the market, the majority of them containing iron in some form. An effort has been made to establish an assay which gives accurate results in preparations of this class, but which can be performed in a reasonable length of time.

A short résumé of the methods tried and in some degree found unsatisfactory, might be given:—Those depending upon the quantitative liberation of I from KI in the presence of HCl, can be used only when the iron is in the ferric state. Those depending upon the precipitation as $\text{Fe}(\text{OH})_3$ are not quantitative when sugar or certain organic acids are present. Those by igniting the sample, taking up in acid and precipitating as $\text{Fe}(\text{OH})_3$; the residue after ignition, in the cases where sugar and sodium hypophosphite were present, was only dissolved with difficulty in acids. That, by precipitating as the sulphide, dissolving in HCl, oxidizing with HNO_3 and precipitating as $\text{Fe}(\text{OH})_3$, igniting and finally weighing as Fe_2O_3 , in nearly all determinations gave accurate results, but required about three hours to run, and when manganese was present in considerable quantity, gave high results.

The results in the last-mentioned method having been for the most part satisfactory, some modification whereby it could be used in the presence of manganese and by which the time required would be reduced, was sought.

By dissolving the FeS in dilute H_2SO_4 and titrating with $N/10$ KMnO_4 , it was found that Mn did not interfere, besides eliminating a drying, ignition and weighing.

An outline of the assay finally decided upon is given.

To 10 cc of the sample in a 125-cc beaker, add 10 cc. H_2O , 5 cc HNO_3 , and boil for 5 minutes. Add 5 cc of 10% NH_4Cl solution, then add NH_4OH until neutral or only slightly acid. Cool and run in H_2S for 1 minute; add excess of NH_4OH and continue to pass in H_2S until com-