Section on Scientific Papers

Papers Presented at the Sixtieth Annual Convention

THE ELECTROLYTIC DETERMINATION OF SOME OF THE ZINC SALTS OF THE PHARMACOPOEIA.

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The literature on the estimation of zinc is very extensive and more seems to have been written about the determination of zinc than that of any other of the technically important metals. Yet a rapid and accurate method for its estimation, gravimetrically or volumetrically, is still a problem to be solved. The report of the Committee on Quantitative Methods of the Pharmaceutical Section of the American Chemical Society[1] shows that widely disagreeing results were obtained by different workers using the same method.

The gravimetric methods are time consuming, and even with the exercise of the greatest care, present several sources of error. Of the volumetric processes, the sodium sulphide and the ferrocyanide are reliable and are extensively used in technical laboratories. These two methods are, however, too much influenced by experimental conditions, and while satisfactory for the analysis of zinc ores and alloys, they are not readily applicable to the determination of the pharmacopoeial zinc salts. With a requirement of 99 to 99.5 per cent. purity for zinc salts, but little allowance remains for error in the determination, because of the high molecular weights of most of the zinc salts of the pharmacopoeia, the least variation in the result makes a considerable difference in the percentage of the salt. For the determination of zinc salts a method is therefore needed which is first of all the most accurate, and second, if possible, rapid. The electrolytic method meets both these requirements.

With the aid of the electric current zinc salts are rapidly and most accurately determined. The procedure is very simple, hence its accuracy—and requires no extraordinary skill or care. A determination can easily be made in the course of less than one hour, the analyst's time consumed amounting only to 15-20 minutes. There being practically no source of error, unless ordinary care be grossly neglected, the method is such as to inspire one with confidence in the results—a point of the utmost value in any method, in any line of work.

Several electrolytes can be used[2], all yielding equally good results. Sodium hydroxide is the simplest and is well adopted for the determination of all the pharmacopœial zinc salts. A weighed quantity of the salt corresponding to 0.1-

[1] J. Ind. Eng. Chems., p. 467. 1912.

[2] E. F. Smith's Electrochemistry.

0.2 grams of metal contained in the previously weighed electrolyzing dish is dissolved in a little water or dilute sulphuric, or hydrochloric acid, 75 cc. of 10 per cent sodium hydroxide solution added, diluted with water to about 120-130 cc., the solution heated almost to boiling and electrolyzed for 20-30 minutes with a current of 4-5 amperes and 5-6 volts, the anode making about 600 revolutions per minute. Without interrupting the current, the deposit is then washed with distilled water with the aid of a siphon, until the current drops to nearly zero, then remove the dish and wash the deposited zinc with a little alcohol and ether, dry in the disiccator for a few minutes and weigh.

For the analysis of zinc metal about 1.5 gm. of it is dissolved in dilute hydrochloric acid, filtered if necessary, diluted with water to 100 cc. and 10 cc. taken for the determination. Zinc stearate dissolves but very slowly in caustic soda. To determine the zinc in it, it is best to boil about one gram with 10 cc. of dilute sulphuric acid and 25 cc. of water, filter while hot into the electrolyzing dish, wash with hot water until the volume of the filtrate amounts to about 120 cc. then add 8 gm. Sodium Hydroxide and electrolyze.

The following figures will illustrate the results obtained by the electrolytic method. It will be noted that the percentages of the salts containing water of crystallization are over 100. This is due to a deficiency in the water of crystallization.

Zinc Metal

	Per cent. zinc found
1	99.07
2	99.04
3	99.09

Zinc Acetate

	Per cent. zinc found	Per cent. zinc acetate
1	30.40	102.01
2	30.34	101.82
3	30.37	101.94

Zinc Oxide

	Per cent. zinc found	Per cent. ZnO
1	80.00	99.58
2	80.10	99.70
3	80.10	99.70
4	80.06	99.64

Zinc Phenolsulphonate

	Per cent. zinc found	Per cent. zinc phenolsulphonate
1	11.80	100.28
2	11.79	100.19
3	11.78	100.11
4	11.79	100.19

Zinc Stearate

	Per cent. zinc found	Per cent. ZnO
1	11.70	14.56
2	11.72	14.59
3	11.69	14.55

For all these determinations a nickel dish was used. Nickel is admirably well adopted for the depositing of zinc by electrolysis. It has the advantage over platinum because the deposit dissolves very readily in dilute sulphuric acid and the cost of nickel as compared with platinum is nominal. If platinum is used it should be first coated with a layer of silver (see Smith's Electrochemistry).

The results obtained by the electrolytic method leave nothing to be desired. The installation of an electrolytic apparatus in an analytical laboratory is an investment which rapidly pays for itself.

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