Comments on Dr. Cranston's Method

The method employing dithizone extraction is an improvement over Dr. Cranston's previous technic. The use of dithizone is an obvious development that, however, is useful only in certain instances. The method is entirely satisfactory when sufficient lead is extracted so that the concentration of lead in the solution to be polarized is at least of the order of 1 microgram of lead per ml. This means that large aliquots must be extracted and one can readily see that when small samples are handled, the lead extracted must be concentrated in some fashion. This increased manipulation will offer opportunities for losses and contamination, and, in addition, when dealing with normal lead, many instances will arise where it will not be feasible to concentrate far enough to pick up the lead.

In our own polarographic method we also include a dithizone extraction, but further concentrate the lead electrolytically whenever necessary. In this fashion we are able to plate out as little as 1 microgram of lead, which can be stripped into 1 ml of solution. The acid washings of the dithizone extracts in our procedure are polarized directly only when relatively large amounts are present. Incidentally, the above technic is only a modification of our electrolytic technic, and has been gotten ready for publication along with our purely electrolytic technic. The latter method plates out lead directly from the prepared solution of ashed material,

and the extraction modification has been included only to decrease the time required to analyze feces and food. These materials require at least 1 hour to plate out by the direct method, and this time may be greatly reduced, if a dithizone extraction is first employed. The paper has been written up and we are working on our last table, which we hope to complete this week. Our tables give comparative results with the dithizone and spectrographic methods and prove the complete adequacy of our polarographic method although we do not claim accuracies of 1%.

Dr. Cranston's request for samples can easily be complied with if he is willing to take solutions of ashed samples. We can easily withdraw aliquots corresponding to 100 ml of original urine, and place the aliquots in containers supplied by Dr. Cranston. If his method is adequate he should be able to employ aliquots no larger than that above in which he should be able to pick up 1 - 3 micrograms of lead corresponding to normal urine. We would expect to include some very low urine samples and I doubt very much that he will pick up the lead, unless some contamination occurs.

I do not approve of his method of ashing in which H_2SO_4 is employed. This introduces an additional amount of lead which he may not be able to pick up as a blank, but which in a sample boosts the amount sufficiently to compensate for other losses.

I believe that a 1% accuracy is not possible by Dr. Cranston's method, because the effects of even slight

changes in composition, drop rate, or temperature greatly affect the curves. Our method corrects for these variations by employing an internal standard; the latter development while not revolutionary, is the first successful application to polarographic analysis of biological material. It was first suggested by a German, but the technic does not seem to have been used at all by others.

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