

exception of the seeds of *Rhynchosia pyramidalis* and *Clitoria arborescens*. These were found to have some curare-like action which, compared to that of *Erythrina* seeds, was extremely weak and probably represented only traces of active alkaloids.

The curare-like action of the extracts of all species of *Erythrina* tested in frogs was of short duration when compared to the duration of paralysis of extracts of curare or curare plants.

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THE DETERMINATION OF IRON IN IRON SALTS OF ORGANIC ACIDS CONTAINING PHOSPHORUS.*

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The gravimetric estimation of iron is complicated by the presence of phosphorus. There are several methods mentioned in the literature which may be used to assay iron salts of organic acids containing phosphorus. One method is based upon the titration of the iron, previously reduced to the ferrous state, with an oxidizing agent as described by Kolthoff and Furman (1). Another, in which the iron is extracted as ferric chloride from a strong hydrochloric acid solution with ether, is described by Scott (2). Kolthoff and Furman (3) also mention a third one based on the reduction of ferric iron to ferrous iron by iodide and subsequent titration of the liberated iodine with thiosulfate. Also, modifications of these basic methods appear in the literature on this subject.

In the case of organic compounds interference due to organic matter must be considered. The methods mentioned above are adaptable only after destruction of the organic matter. We have used one which, in addition to being applicable after destroying organic matter, can apparently in some instances at least be employed without doing that. This method comprises the precipitation of the iron as sulfide (4, 5) in ammoniacal solution, separating it in this way from the phosphorus and other interfering constituents; the sulfide is then converted to the hydroxide, for weighing as ferric oxide. The method has been applied to samples of iron adenylate with satisfactory results.

Iron adenylate has the empirical formula $C_{10}H_{12}N_5O_7PFe$, containing 13.93% of iron. Values obtained after oxidation of the samples range from 13.36% to 14.42%. When the destruction of organic matter was omitted and the method was applied directly, the findings ranged from 13.72% to 14.05%.

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EXPERIMENTAL.

The following results were obtained when iron adenylate was assayed by the two methods described herein.

Sample.	After Oxidation.	Iron Content.	Direct.
A	13.45%		...
B	13.36%		...
C	14.42%		...
D	13.42%		...
E	14.37%		14.05%
F	13.77%		13.72%
G	...		13.88%

The methods used to obtain the above results are described as follows:

Iron Assay with the Destruction of Organic Matter.—Place a 0.5-Gm. sample in a 250-cc. Erlenmeyer flask and add 5 cc. of concentrated sulfuric acid. Warm gently on a hot plate for several minutes. Cool thoroughly. Add 5 cc. of 30% hydrogen peroxide and warm gently on a hot plate until white fumes appear. Again cool thoroughly. Repeat the treatment with 5 cc. of 30% hydrogen peroxide once more and then repeat using 2-cc. portions of the hydrogen peroxide until organic matter has been destroyed, taking care to cool thoroughly between each treatment with hydrogen peroxide. After thorough cooling, add 25 cc. of water and 10 cc. of concentrated hydrochloric acid. Warm gently until all the material has dissolved and a clear solution exists. Dilute to 100 cc. with water and heat to incipient boiling. Cool. Pass into the solution a slow current of hydrogen sulfide gas for several minutes, and then add slowly diluted (1 + 1) ammonium hydroxide until an excess has been added. Continue the slow current of hydrogen sulfide for 15 minutes. Set aside on a steam-bath for 30 minutes. Collect the ferrous sulfide on a filter paper and drain completely. Then, wash the precipitate twice with ammonium sulfide water by filling the filter completely and allowing it to empty completely before adding the second portion. The original flask in which the iron sulfide was precipitated should be rinsed with ammonium sulfide water, passing the rinsings through the filter. Dissolve the ferrous sulfide on the filter by pouring several 25-cc. portions of hot diluted (1 + 20) nitric acid through the filter, collecting the filtrate in a 600-cc. beaker. Finally, transfer the filter paper to the original flask, and warm with a 50-cc. portion of the diluted nitric acid. Decant the liquid through a fresh filter paper into the main filtrate in the 600-cc. beaker. Repeat with an additional 50-cc. portion of hot diluted nitric acid. Heat the filtrate gently until the solution is clear. Cool, and stir in a small amount of filter paper pulp. Add slowly, while stirring, diluted (1 + 1) ammonium hydroxide until an excess has been added. Collect the precipitate on a rapid filter (Whatman #41) and wash twice with hot water. In order to transfer all the iron from the beaker to the filter, add some filter paper pulp to the beaker and some diluted (1 + 1) hydrochloric acid. Rotate the beaker until all the iron hydroxide on the sides of the beaker has dissolved; rinse the sides of the beaker with hot water, neutralize with an excess of diluted (1 + 1) ammonium hydroxide, using methyl red indicator solution and transfer the iron hydroxide to the main precipitate. Wash copiously with warm water. Transfer the precipitate and filter to a platinum dish, dry, and ignite slowly and carefully. Cool and weigh. Calculate the iron from the weight of ferric oxide thus obtained.

Iron Assay without the Destruction of Organic Matter.—Weigh 0.5-Gm. of the sample into a 250-cc. Erlenmeyer flask and add 20 cc. of diluted (1 + 1) hydrochloric acid. Warm gently until complete solution results. Dilute to 100 cc. with water, and proceed with the precipitation of iron sulfide, etc., as in the iron assay following the destruction of the organic matter.

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