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A simple method for making p-arsanilic acid.

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The preparation of what is now known as primary arsanilic acid, the starting point and the basis of Ehrlich's synthesis of salvarsan, was first mentioned by Béchamp[1] in 1863. In spite of Béchamp's published work, and that of many others, the fact remains that the literature does not contain definite directions for making the primary arsanilic acid. Many laboratories have spent much effort, money and time in trying to evolve the synthesis from the general directions given in the literature, but without much success. The process in its highest form is generally understood to be a trade secret.

Without going into details, it can be shown that the directions either call for too much aniline or too high a temperature, or both. By controlling one or both of these factors one can obtain the primary free from secondary in large yields, practically pure, with ease.

One thousand c.c. of crude arsenic acid (75 per cent.) are heated in an open beaker or vessel, at 120° to 140° C. for 12 to 15 hours by means of an oil or "Crisco" bath. This should concentrate the acid to practically 100 per cent.

[1] *Compt. rend.*, LVI., 1172, 1863.

Fourteen hundred c.c. of dry aniline oil are cooled with an ice mixture to 0° C. or lower, and the cooled arsenic acid is added slowly with vigorous stirring. The mixture soon becomes thick and then granular, after which it is finely ground and thoroughly mixed. This powder has roughly the composition of $(C_6H_5NH_2)_3 - (H_3AsO_4)_2$.

Two hundred grams of this powder are heated in an Erlenmeyer flask, by means of a "Crisco" bath to 160° C., when the powder begins to melt. The substance is stirred continually and when it has all melted, a reflux condenser is then attached; for one and a half hour it is heated from 160°–170°, and then one hour from 180° to 183° C. After allowing the mixture to cool somewhat 225 c.c. of 6N sodium hydroxide and 225 c.c. of water are added, which causes the substance to dissolve and separate out the aniline oil left uncombined.

When the mixture is cool, the aqueous layer is drawn off with the aid of a separatory funnel, and after shaking with 15 to 20 grams of infusorial earth or kaolin, it is filtered with suction.

To the clear filtrate is added 100 c.c. of 6 N hydrochloric acid.

By means of 25 c.c. portions and further additions of 0.5 c.c., 1.00 c.c., 2.00 c.c., etc., of 6 N hydrochloric acid, one finds out if further addition of acid to the whole filtrate will give a larger yield or not. When after waiting a few minutes for the crystallization to take place, and when the best medium has been determined on the small aliquot portion, an equivalent amount of acid is then added to the whole filtrate. Usually the crystals fill the entire solution so that it has the appearance of being solid.

After standing for an hour or longer the precipitate, which is white and crystalline, is filtered with suction. It is then washed by suspending the precipitate in 200 c.c. of water and filtering with suction. When all the wash liquid has drained from the precipitate, it is dried by fanning, or in any other suitable way. The yield is about 30 per cent. of the theory, whereas 10 per cent. seems to be the average heretofore.

The products obtained by the above directions are almost pure enough for further use, but if necessary can be reprecipitated with alkali and acid, or by dissolving it in boiling water and allowing the solution to cool.

The analysis of a crude product once purified by hot water crystallization and dried in a vacuum desiccator gave:

	As, %	N, %
Product	34.20	6.02
Product	34.50	6.13
Theory for primary	34.60	6.45
Theory for secondary	25.65	9.65

9 (1384)

Vitamines in green leaves.

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In view of the very scanty data recorded respecting the relative content of vitamine in green foods compared with other plant and animal products which have been studied more in detail, we have fed albino rats on diets containing, as the source of water-soluble vitamine, spinach, cabbage, clover, timothy and alfalfa, dried in their immature state. Far less dried spinach supplies sufficient water-soluble vitamine to promote normal growth than do whole wheat, soy beans, dried egg, meat, milk or potatoes. Spinach leaves are much richer in the fat-soluble vitamine than are most of the products used in our ordinary rations. Thus rats fed for over 160 days, during which they consumed only 25 to 34 grams of spinach, have grown from 60 to 250 grams at a nearly normal rate and have as yet shown no evidence of deficient nutrition. Such quantities are not much larger than have heretofore been considered to be necessary when butter fat supplied the fat-soluble vitamine. A somewhat larger quantity of cabbage than of spinach leaves is needed to promote normal growth. It probably also contains the fat-soluble vitamine. Timothy, clover and alfalfa contain both vitamines, but further experiments are needed to establish the relative amounts of these. Tests of a large variety of tubers, stems, leaves and fruits are now in progress.

If one may draw conclusions from the limited data at present available, it seems that the green vegetables and fodders are richer