

## Scientific Section

Papers Presented at the Sixty-Second Annual Convention

### THIRD ALKALOID FROM GELSEMIUM.

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The Proceedings of the American Pharmaceutical Association for a number of years past have recorded the progressive work of the laboratory of the University of Kansas in regard to the alkaloids of Gelsemium.

Last year a report was made on the further study of Gelsemium, giving, in the first place, the percentage of various alkaloidal and acid products from fifty pounds of the crude drug; secondly, a very complete statement as to the physiological action of the alkaloids, Gelsemine and Gelseminine. This latter work was contributed by Doctor F. P. Chillingsworth. It may be said in passing that during the past year, Doctor S. A. Mathews, pharmacologist, has gone over the physiological action of Gelseminine and his results confirm those of Doctor Chillingsworth.

During the past year, two separate lots of Gelsemium, from different sources, have been investigated for alkaloidal constituents. The results of this work, being somewhat noteworthy, we desire to record them in the present Proceedings of this Association.

The first lot of Gelsemium consisted of one pound of the concentrated fluid extract, which assayed .4% of total alkaloids. The second lot consisted of five pounds of powdered gelsemium root, ground especially for investigation.

By a change of a method of procedure, suggested by one of us (Stevenson) we have secured, as a result, a purification of what seems to promise a third alkaloid, which furnishes definite forms of crystals in the alkaloidal condition as well as in two of its salts, namely, the hydrochloride and the nitrate, the only ones thus far studied.

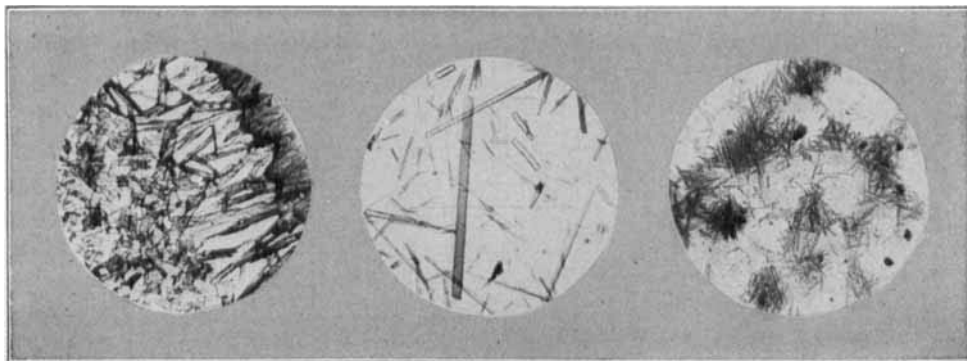
The method of procedure for the isolation of this alkaloid was practically the same in the two cases and, as results were the same, we shall give only the details for the isolation from the powdered drug, which was as follows:—

Five pounds of the finely-powdered drug were first treated with 70% alcohol until exhausted. The tincture thus obtained was evaporated to a viscous liquid. This concentrated liquid was then extracted with five litres of chloroform, divided into five one-litre portions. The chloroformic washings were then mixed and the chloroform distilled off until the liquid was concentrated to 100 cc. This second concentrate was then treated with .5% hydrochloric acid, washing the concentrate in a separatory funnel with five portions of 100 cc. each of the above acid; then six times with 100 cc. each of .25% hydrochloric acid. The combined acid solutions now were shaken out with 900 cc. of benzol in three portions of 300 cc. each. This was to remove the gelsemic acid. Then the residue was shaken out with

chloroform, 400 cc., 300 cc., 300 cc., and 300 cc.—in all, 1300 cc. in order to remove any trace of gelsemic acid.

The chloroformic solution was distilled until a highly concentrated chloroformic solution was left behind. This concentrate was washed with water. This aqueous solution, which was found to be strongly alkaloidal, was evaporated to a solid. This was completely soluble in alcohol. The alcoholic solution was evaporated until highly concentrated and then mixed with sand and dried in current of warm air. The powder which resulted was extracted first with acetone; the residue, left behind from the acetone treatment, was then extracted with alcohol. On evaporation of the alcohol, the hydrochloride of the alkaloid in question crystallized out. Photographs of the crystals of the alkaloid, the nitrate and hydrochloride, are herewith appended.

“SEMPERVIRENE” SALTS AND ALKALOID.



HYDROCHLORIDE.  
(Crystals on left are the more characteristic.)

NITRATE.

FREE ALKALOID.

We would suggest as a name for this alkaloid, “Sempervirene.” This alkaloid exists in gelsemium only in minute quantities (the exact amount obtained was not determined), and it was only through the insolubility of its salts that it was discovered. On adding hydrochloric acid, sulphuric acid, or nitric acid to a neutral aqueous solution of the hydrochloride, the hydrochloride, sulphate and nitrate, respectively, of the alkaloid are precipitated. Phosphoric acid does not cause a precipitate. The alkaloid is so completely precipitated by the addition of nitric acid to the aqueous solution of the hydrochloride, that only a faint opalescence is produced by the addition of Mayer’s reagent to the filtrate. In the insolubility of its nitrate the alkaloid shows a striking similarity to cinchonamine.

The nitrate is slightly soluble in alcohol from which it may readily be crystallized, separating in the form of well-defined yellow needles. The nitrate begins to darken when heated to 210°; at 280° C., it formed a black, partially fused mass.

The free alkaloid was prepared from the re-crystallized nitrate. Its chloroformic solution was yellow. The alkaloid formed pale yellow crystals, the characteristic form from alcoholic solution seeming to be short and rather thick needles.

The hydrochloride formed pale yellow crystals easily soluble in alcohol, which

crystallized on evaporation of the aqueous solution in the form shown in the photograph above.

We are convinced that this principle differs from any one that has heretofore been reported and, since we have obtained the same alkaloid from both the fluid extract and from the drug, we cannot believe that it is due to any foreign admixture in the drug. But, in order to assure ourselves that this is not the case, we are having ground, at the present time, a specially selected gelsemium for the purpose and will go over the same work repeatedly in order to reassure ourselves that no error could have crept in from such a source.

Referring to a letter from J. U. Lloyd, with whom one of us (Sayre) has had some correspondence in connection with this subject, he says: "I take it you have surely taken the pains to see that your new alkaloid is not a chemical product, in which some reaction on another of the alkaloids has produced the new one as a split off?" He further states,—“Please bear in mind the fact that drugs at different seasons of the year contain different constituents or varying amounts of the usual constituents, and that possibly you will find variations in gelsemium, in more directions than one.”

It would be of interest to us to continue this investigation to see whether there are gelsemiums of different collections in which this alkaloid is absent.

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## THE STRUCTURAL VARIATION OF ALLSPICE.

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Allspice, *Pimenta officinalis*, is a New World spice. It was not known in Europe until after the discovery of America. The exact history of its discovery, and its subsequent introduction into commerce, is obscured in the haze of passing years. It is definitely known, though, that in the seventeenth century it was imported into England, where it was known by a variety of names. About the close of the seventeenth century, it was in wide use in England as a condiment, and was sold as Jamaica Pepper or allspice. The latter name is in common use to-day, but we do not hyphenate the word as they did formerly. At the beginning of the eighteenth century, allspice was used in medicine. It was not until 1721, however, that allspice was made official in the British Pharmacopœia; and not until 1816, was it made official in the United States Pharmacopœia. In the 1816, U. S. P., allspice was recognized under the name of Pimento; the common name was given as Jamaica Pepper, and its botanical origin as *Myrtus Pimento*. In the 1820 and 1828, Pharmacopœia the oil was official under the title of *Oleum Pimentæ*. In the 1830, U. S. P., its title was Oil of Pimento, from Pimento. In the 1831, U. S. P., both the fruit and the oil were official. The title of the fruit was *Pimenta—Myrtus pimenta*, the berries. The title of the oil was *Oleum Pimentæ*. In the 1842, U. S. P., the title of the fruit was *Pimenta*, Pimento. The unripe berries of *Myrtus Pimenta*. The title of the oil was *Oleum Pimentæ*, Oil of Pimento, from Pimento. In the 1857, U. S. P., the title of the fruit was *Pimenta*, Pimento,—the unripe berries of *Myrtus Pimenta*. The title of the oil