by manufacturers of volatile oil, especially one, but years afterwards the sample in question was picked out of the list presented and the distiller admitted it contained two percent. ot water. The records of the Association gave the data but I am not able to recall the time it was published.

MR. STOCKBERGER: I know it has been Mr. Rabak's custom in working with these oils to take all precautions, so far as possible, to remove moisture from the oil. Whether it was absolutely removed or not, of course, it is impossible for me to say, but I know that is my impression, that they were moisture free samples.

NOTES ON A NEW ALKALOID FOUND IN NUX VOMICA.*

HUGO H. SCHAEFER.

We find from time to time in pharmaceutical literature, reports of the discovery of new alkaloids in nux vomica beans, that is, alkaloids other than strychnine or brucine. In the Jour. Chem. Soc. (39-457), Shenstone reports the finding of an alkaloid freely soluble in warm water. After describing the substance, the author states that it is probably brucine with some persistent impurity. In the Pharm. Jour. (III, XIV, 1025) Dunston reports the discovery of a glucoside in nux vomica, which he terms loganin. Desnoir (Jahresbericht 50-54, p. 48) describes a new nux vomica alkaloid, which he calls igasurine. This, he claims, is an intensely bitter substance, giving tests entirely different from those of strychnine or brucine. Again, in Gmelin (XVII—589), we find that igasurine is a very bitter substance resembling brucine, but more soluble in water. Schutzenberg (Am. Jour. Pharm., 1858-535) reports that igasurine is really composed of nine different and distinct alkaloids. He separates these by fractional crystallizations and gives a brief description of each one. Finally we find, in Am. Jour. Pharm. (1872-256, Jörgenston), evidence that there is no such alkaloid as igasurine, or at least, that the substance so-called, can be readily converted into brucine, by treating with potassium iodide, filtering off and regenerating the alkaloid, which will be found to be brucine.

We therefore see, from the above contradicting statements, that it is an open question as to whether such an alkaloid as igasurine really exists. However, the author now wishes to report the finding of an alkaloid in nux vomica, which is totally different from strychnine, brucine, or any of the igasurines, as described in literature, and which alkaloid he proposes to call *struxine.*

This alkaloid makes its appearance during the process of manufacturing strychnine. In neutralizing the acid-solution of the raw alkaloidal sulphates of nux vomica, the new alkaloid separates out as a base, when the liquors become just neutral, or are still slightly acid, while strychnine and brucine remain in solution as sulphates. It was found that only few lots of nux vomica beans contained this substance, and the quantities, in those which did contain it, differed greatly with different lots. Careful record was made of the beans which yielded this new alkaloid, and this proved that all such beans were from shipments made from Cochin-China. Those lots which consisted mostly of small beans, insect-eaten

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* Read before Scientific Section at Detroit.

and partly decomposed by prolonged exposure in wet fields, contained the largest percentage of the alkaloid. The average amount found in these beans was 0.1%.

Struxine, which separates from the solution of raw alkaloids as above stated, was filtered off, dissolved in an excess of dilute sulphuric acid, made alkaline, and the precipitated alkaloid again filtered off. This was now purified by repeated recrystallizations from alcohol.

The final pure product, when freshly made, consists of colorless crystals containing no water of crystallization. Upon exposure to air and light, however, the crystals gradually develop a faint yellowish color. No melting point could be determined, as the substance begins to char at about 250° C. A solution of it gives typical alkaloidal reaction with gold chloride, platinum chloride, mercuric chloride, Bouchardat's, Marme's, Mayer's, Dragendorff's, Sonnenschein's, and Scheibler's reagents, and with tannic acid and picric acid. The alkaloid is only very slightly bitter, which lack of taste is probably due to its extreme insolubility in water, since the more soluble salts of *struxine* are distinctly bitter, but have not the intensity of either strychnine or brucine, or any of the igasurines, as reported in literature.

The following solubility determinations were made:—

In water 1-5000 cc.
In alcohol 1-190 cc.
In methyl 1-210 cc.
In chloroform 1-1 2/3 cc.
In ether 1-450 cc.
In benzol 1-35 cc.

Upon microscopical examination, it was found that the crystals are all of well-defined rhombic form. This, together with the fact that various attempts at fractional crystallization proved failures, shows that the substance is not a mixture of alkaloids, but is a uniform product.

The ultimate analysis of the alkaloid gave the following composition:—

	A.	B.	Average
C.	67.31%	67.11	67.21
H.	7.89	8.13	8.01
N.	7.62	7.44	7.53
O.	17.18	17.32	17.25

A careful determination by means of the freezing-point method, indicated that the alkaloid had a molecular weight of 371. From this data, it was calculated that the empiric formula of *struxine* is $C_{21}H_{30}N_2O_4$, which would give it a molecular weight of 374.53. As will be noted below, this molecular weight is between that of strychnine, and of brucine, and its formula in certain respects resembles that of strychnine and brucine.

	Empiric Formula	
Struxine	$C_{21}H_{30}N_2O_4$	374.53
Strychnine	$C_{21}H_{22}N_2O_2$	334.466
Brucine	$C_{23}H_{26}N_2O_4$	394.518

Because of these resemblances, it is thought that, possibly, the new alkaloid is a product of decomposition, by fermentation or oxidation of either strychnine or brucine. The fact that those beans which are insect-eaten and partly decomposed, contain more of the alkaloid than good beans, would also indicate this.

Various salts of *struxine* were made, and it was found that it forms normal and acid salts. The latter, in most cases, however, only from a solution containing a very large excess of acid. All the salts react acid to litmus, and when these acid solutions were neutralized with ammonia water, the free alkaloid separated out, even while the solution showed still a faint acid reaction. No melting points could be obtained, since all these salts charred at 240°-250° C.

Among the salts made and examined, were the following:—

Sulphate—$(C_{21}H_{30}N_2O_4)_2H_2SO_4.8H_2O$.

This is a yellowish, crystalline salt, possessing a bitter taste, soluble in water 1-120, in boiling water 1-12, and in alcohol 1-100.

Bisulphate—$C_{21}H_{30}N_2O_4.H_2SO_4.1H_2O$.

This is a white crystalline salt, possessing a bitter taste, soluble in water 1-75 and in alcohol 1-210.

Hydrochloride—$C_{21}H_{80}N_2O_4.H\,Cl.3H_2O$.

This is a yellow crystalline salt, which with water, gives a golden yellow-colored solution, having a bitter taste. It is soluble in water 1-85 and in alcohol 1-75. The alkaloid does not combine with two molecules of hydrochloric acid, since even from a solution of a large excess of acid, the neutral salt is obtained.

Hydrobromide—$C_{21}H_{80}N_2O_4.H\,Br.2H_2O$.

This is a yellowish, bitter crystalline salt, soluble in water 1-115 and in alcohol 1-100. A di-hydrobromide could not be obtained even from a solution containing a large excess of hydrobromic acid.

Bitartrate—$C_{21}H_{30}N_2O_4.H_2C_4H_4O_6.2H_2O$.

This salt consists of white, glistening, flaky crystals possessing a bitter taste. Only the acid salt could be obtained. In attempting to make the normal salt, the theoretical quantities of acid and alkaloid in hot water, formed a product which was found to be a mixture of the alkaloid and acid tartrate. Upon using double the amount of tartaric acid, however, all the alkaloid went into solution, from which upon cooling, the acid tartrate crystallized out in beautiful flaky crystals, sparingly soluble in water or alcohol. These crystals, when dissolved in hot water, form a solution of strong acid character. If this solution is neutralized with ammonia water until neutral to litmus, no normal tartrate is obtained, but free alkaloid crystallizes out.

An attempt was made to obtain the acetate of the alkaloid. It was found, however, that no such salt could be obtained in crystallized form.

Many tests were made with the new alkaloid, in order to find characteristic reactions and possible resemblances to strychnine or brucine.

Among these reactions were the following:—

A. A small quantity of the alkaloid will give no color when a drop of concentrated sulphuric acid is added. If to this mixture, however, a crystal of potassium dichromate be added, a yellow color will be obtained, which slowly changes to green.

B. If to a small quantity of the alkaloid, a drop of concentrated sulphuric acid be added, and then a small particle of sodium nitrite, a dark brown color is obtained.

C. If to a small quantity of the alkaloid, a drop of concentrated sulphuric acid be added, and then a small particle of lead peroxide, a dark brown color is obtained, which slowly changes to purple, and finally to violet.

D. If to a small quantity of the alkaloid, a drop of concentrated sulphuric acid be added, and then a small crystal of potassium ferricyanide, an orange color is obtained which changes to brown.

E. If to a small quantity of the alkaloid, a drop or two of concentrated sulphuric acid be added, and then a few particles of manganese dioxide, a brown color is obtained which slowly changes to purple.

F. A small quantity of the alkaloid will give a brown color, upon addition of a drop of concentrated nitric acid, which color does not change upon addition of a small quantity of zinc chloride.

G. If a small quantity of the alkaloid be dissolved in a few drops of nitric acid and the mixture evaporated to dryness, a light yellow residue remains behind. If this residue be dissolved in ammonia water, a reddish-colored solution is obtained. If alcoholic potassium hydroxide solution now be added, a red to brown precipitate is obtained.

H. If to a small quantity of the alkaloid, a few drops of aqueous solution of potassium hydroxide be added, and the mixture evaporated to dryness, a brown residue remains behind.

I. If to a small quantity of the alkaloid, a drop of a solution of stannous chloride be added, a yellow color is obtained.

J. If to a small quantity of the alkaloid, a drop of concentrated hydrochloric acid be added, a yellow color is obtained.

K. If to a small quantity of the alkaloid, a drop of alcoholic solution of potassium hydroxide be added, an orange color is obtained, which slowly changes to red and then to brown, and finally to black. This test can also be performed by treating the alkaloid with a drop of aqueous solution of potassium hydroxide, which produces no change in color. However, as soon as a drop of alcohol is now added, to the mixture, the above color-changes occur.

All the foregoing tests were made on watch-glasses, or crucible covers with the dry alkaloid. The following tests were made in test tubes, using a saturated aqueous solution of the sulphate:—

L. A small quantity of the saturated solution when treated with an equal portion of concentrated sulphuric acid gives a pink color. If now a particle of sodium nitrite be added, a red-colored solution is slowly formed.

M. The solution of the sulphate gives a yellow-colored precipitate, upon addition of a solution of picric acid.

N. Upon addition of an equal portion of concentrated hydrochloric acid to the saturated solution, no color is obtained even on heating.

O. Upon addition of chlorine water to the saturated solution of the sulphate, a yellow-colored mixture is obtained with no precipitate.

P. A heavy yellow precipitate is obtained from the solution of the sulphate upon addition of as little as one drop of bromine water.

Q. If a small quantity of the saturated solution be diluted with an equal portion of alcohol, and two or three drops of bromine water now be added, no precipitate appears and the solution remains colorless. However, if a large excess of bromine water be added, a yellow precipitate is formed.

R. If an excess of sulphuric acid be added to the solution of ***struxine sulphate***, and sodium carbonate or bicarbonate be added in excess, a heavy white precipitate is formed, showing that the alkaloid is not soluble in a solution of carbon dioxide.

S. A solution of potassium chlorate, when added to the solution of sulphate, will cause no color-change or precipitation.

T. If to the saturated solution, a solution of potassium ferri-cyanide be added, a dense white precipitate is obtained.

U. If to the solution of the sulphate a solution of potassium ferro-cyanide be added, an amorphous yellow precipitate is obtained. If an equal portion of alcohol now be added, the precipitate immediately dissolves.

V. Tincture of iodine, when added to the solution of the sulphate, gives a brown precipitate.

W. If to the solution of the sulphate, some alcoholic potassium hydroxide solution be added, the mixture slowly takes on a pink color, gradually changing into cherry, then red, and, finally, purple.

This test is characteristic of this alkaloid only, and therefore many modifications were tried, until the following was finally determined as being the sharpest and most definite:—

Y. If to the saturated alcoholic solution of the sulphate (1-100) an equal part of alcoholic solution of potassium hydroxide 10% be added, within one minute, the mixture will take on a cherry-red color, which turns darker upon standing. If to this colored mixture, four drops of alcoholic solution of resorcinol (1-100) be added, the color immediately changes to intense dark green. If alcoholic solutions of either hydrochinone, or anthra-quinone (1-100) be used in place of the resorcinol, the same green color will be obtained.

These experiments, reactions and descriptions prove conclusively that there is a third alkaloid in nux vomica, unlike any described in literature under the name igasurine. While studying the alkaloid, much more research work suggested itself to the author, and the results of some of it will be published in a later paper. Experiments will also be made to obtain the physiological reaction of *struxine*.

Laboratory N. Y. Quinine and *Chem. Works.*

LABORATORY NOTES.

GEORGE E. E'WE AND CHARLES E. VANDERKLEED.

In every laboratory where a large variety of work is being done, many observations are made from time to time, which are not of enough importance in themselves to be dignified by making them the subjects for scientific papers; yet they may be of interest and importance to workers in other laboratories. Under the title of "Laboratory Notes," therefore, we have collected a number of such observations of miscellaneous character, hoping that they may prove to be of interest to members of the Scientific Section, and that they may prove to be a step in the direction of encouraging, at our meetings, the mutual exchange of laboratory ideas.

Criticism of U. S. P. Directions for Making Decinormal Iodine Solution:— The U. S. Pharmacopœia directs, in making N/10 Iodine solution, to

> "Dissolve 12.59 gm. pure iodine in a solution of 18 gm. of potassium iodide in 300 cc. water."

This procedure of making the solution, requires from one to three hours. A great saving in time may be effected by cutting down the amount of water to about 40 cc. By this modified method, the solution may be prepared in about 10