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No. III

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TUBEROUS ROOT OF *Ipomœa Horsfalliæ*, HOOKER

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A Contribution from the Wellcome Chemical Research Laboratories, London.

In the Spring of 1909 one of us was kindly presented by Mr. E. M. Holmes, F.L.S., Curator of the Museum of the Pharmaceutical Society of Great Britain, with a large tuberous root which had been received from the West Indies, and was evidently the product of a species of *Ipomœa.* It had been sent to Mr. Holmes by Messrs. Westmacott & Son, of Manchester, England, who had likewise favored him with the following information concerning it. "It grows wild, and is not cultivated for any purpose. This specimen was gathered in the woods of Maypen, Clarendon District, Jamaica, by our client, Col. Barlow, of Bury, who states that it is used for starch making, although it produces a yellow product, and that it is also used for food in some instances."

The root in question was a very large one, and, as it could not conveniently be preserved in its entire, fresh state, it was thought that it might be utilized for a chemical examination, so far at least as the amount of material would permit. Some additional interest was imparted to the subject by the fact that the present authors had recently made a complete chemical study of the stems of *Ipomœa purpurea,* Roth,[1] and also of the official jalap,[2] from *Ipomœa Purga,* Hayne (*Exogonium Purga,* Benth).

In order to ascertain the botanical source of the root referred

[1] This Journal, 1908, 80, pp. 251–286.

[2] *Journ. Amer. Chem. Soc.,* 1910, 32, pp. 80–113.

to, Mr. Holmes had obtained from Jamaica, through the kindness of Col. Barlow, specimens of the flowering plant which produced it, and these were found to agree completely with the *Ipomœa Horsfalliæ,* as described by Sir W. J. Hooker, who established the species (compare *Botanical Magazine,* N. S., Vol. viii, *tab.* 3315. *Ind.* or.). Although the *Ipomœa Horsfalliæ* is described as an East Indian species, there would appear to be no doubt respecting the botanical identity of the plant received from Jamaica, where it must have been introduced. The remarks of Sir W. J. Hooker (*loc. cit.*) concerning the plant to which he had given the above-mentioned name may be deemed of sufficient interest to record in this connection.

In so extensive a genus as the present, and where many of the species are necessarily very imperfectly described, it behooves us to constitute new ones with great caution; and it is not until a careful comparison of the present individual, unquestionably one of the most beautiful, with all the descriptions to which I have had access, and with a most extensive collection of the genus in my herbarium, that I have considered it to be new, and have given it the name of the lady to whose kindness I am indebted for the drawing. The seeds were received by Charles Horsfall, Esq., either from Africa or from the East Indies, and raised by his very skilful gardener, Mr. Henry Evans, at Everton, where the plants produced their lovely blossoms in great profusion during the months of December and January (1833–4), a season when so gay a visitor is particularly welcome to the stove. Mr. Evans informs me that he has it under the name of *I. pentaphylla;* but the species so called by Jaquin has hairy leaves, and is in other respects quite a different plant, while the *I. pentaphylla* of Cavanilles (*I. Cavanillesii,* Roemer et Schultes) is still more at variance with our species. *I. Horsfalliæ,* in its inflorescence and blossoms, bears the closest affinity with *I. paniculata,* Br. (*Convolvulus,* L.), but their foliage is so different that the two plants can never be confounded: the former having compound and quinate leaves, while those of the latter are simply lobed.

EXPERIMENTAL.

The tuberous root of *Ipomœa Horsfalliæ,* Hooker, which formed the subject of the brief investigation here described, is represented in the plate accompanying this paper. It was light brown in color, with darker colored spots, and showed in places an exudation of black resin. The length of the root was 0.38 metre, its circumference at the largest part 0.37 metre, and it weighed 2395 grammes. The presence of an abundance of starch was evident when a section of the root, moistened with iodine, was observed under the microscope.

The root was sliced, and then dried in a water-oven, when it weighed 408 grammes, the loss of weight in drying having thus been 1987 grammes, or nearly 83 per cent. The dried material was ground to a fine powder, when it amounted to 385 grammes. It was then brought into a large Soxhlet apparatus, and thoroughly extracted with hot alcohol, the greater portion of the alcohol being subsequently removed, and the resulting extract distilled in a current of steam. The distillate was found to contain only traces of formic and butyric acids.

After the above-described treatment, the contents of the distillation flask consisted of a dark red, aqueous liquid, together with a small amount of resinous material. The liquid was filtered, and the resin repeatedly washed with hot water, after which the resin was dissolved in a little alcohol, the solution evaporated to dryness, and the residue dried at 100° C. The amount of resin obtained was 9.6 grammes, thus corresponding to 2.5 per cent. of the weight of dried, or 0.4 per cent. of the weight of the entire fresh root.

The resin formed a dark brown, spongy mass, which became somewhat sticky on exposure to the air. It was found to be almost completely soluble in ether.

Optical Rotation of the Crude Resin.

The optical rotatory power of the convolvulaceous resins has been considered to afford some indication of their identity or purity.[1] As this factor has previously been determined for several such resins, including those of *Ipomœa purpurea,* Roth, and jalap by the present authors,[2] it was deemed of interest to make this determination with the resin under examination. For this purpose 1.0 gramme of the crude resin was dissolved in absolute alcohol, and the solution treated with successive small quantities of animal charcoal until it was nearly deprived of color. The rotation of this liquid in a 2 dcm. tube was —0°54′, and the amount of substance contained in 10 c.c. of the liquid, after drying at 105–10° C., was 0.1584 gramme, whence $[a]_D$ —28.4°. It may be noted that this degree of optical

[1] Compare Guigues, *Journ. de Pharm. et de Chim.,* [6], 22, 241, and *Chem. Centralblatt,* 1907, Bd. I, p. 309. Also *Bull soc. chim.* [4], 3, 872, (1908).

[2] This JOURNAL, 1908, 80, p. 253, and *Journ. Amer. Chem. Soc.,* 1910, 32, p. 85.

activity is considerably lower than that of either of the above-mentioned resins, and appears to approximate most nearly to that of the resin from *Ipomœa orizabensis,* Ledanois, which has been recorded by Guigues (*loc. cit.*) as varying from — 23.30′ to — 25°.

Extraction of the Resin with Various Solvents.

A small portion of the resin (1.0 gramme) was reserved for a physiological test, and the remainder (7.6 grammes) dissolved in alcohol, the solution being brought onto prepared sawdust, and the mixture thoroughly dried. It was then successively extracted with light petroleum, ether, and alcohol, when the following amounts of extract, dried at 100° C., were obtained:

Petroleum (b. p. 40–60° C.)	extracted 4.92 grammes, or	64.7 per cent.
Ether	extracted 1.90 grammes, or	25.0 per cent.
Alcohol	extracted 0.60 grammes, or	8.0 per cent.
Total	7.42 grammes, or	97.7 per cent.

Petroleum Extract.—This was a soft, brown resin. It was heated in a reflux apparatus with an alcoholic solution of potassium hydroxide for several hours, after which the alcohol was removed, water added, and the alkaline mixture extracted with ether. The ethereal liquid was dried, and the solvent evaporated, when a small quantity of solid material was obtained. This yielded a little of a crystalline substance, which melted at 132–133° C., and gave the color reactions of the phytosterols.

The alkaline liquid which had been extracted with ether was acidified, and again extracted with this solvent, the ethereal liquid being dried and evaporated. A small amount of acid was thus obtained, which was distilled under diminished pressure, when it partially solidified. The oily portion was unsaturated, since it absorbed bromine in chloroform solution. The solid portion was recrystallized from dilute acetic acid, when it melted at 56–58° C. It was thus evident that the original product consisted of a mixture of acids, but the amount was too small to permit of their further separation.

Ether Extract.—This extract, like the preceding one, was a soft, brown resin. On redissolving it in ether a very small amount of a sparingly soluble substance separated, which was collected. This was found to give a color reaction similar to that yielded by

the phytosterols, and it probably consisted of one of the dihydric alcohols, such as ipuranol and ipurganol, which have previously been isolated by us from convolvulaceous resins (*loc. cit.*) and other sources. The exceedingly small amount of this substance rendered it impossible to identify it.

The ethereal liquid was subsequently shaken successively with solutions of sodium carbonate and sodium hydroxide, but only resinous material was thus removed, and on finally evaporating the ether only a trace of yellow resin remained.

Alcohol Extract.—This extract, which was very small in amount, resembled the two preceding ones, and even after prolonged drying it could not be reduced to a powder. Its alcoholic solution was treated with baryta, and allowed to stand in a warm place for twelve hours, after which the alcohol was removed, water added, and the barium precipitated by means of sulphuric acid. The filtered aqueous liquid did not reduce Fehling's solution until after heating with a drop of concentrated sulphuric acid, thus indicating that at least a portion of the alcoholic extract of the resin was glucosidic.

Examination of the Aqueous Liquid.

The aqueous liquid, as above indicated, represented that portion of the alcoholic extract of the root which was soluble in water, and from which the small amount of resinous material had been removed. It was first shaken with ether, and on adding to the ethereal liquid a little aqueous alkali the latter solution showed a blue fluorescence. This was probably due to the presence of traces of β-methylæsculetin, $C_9H_5(CH_3)O_4$, a substance which we have previously shown (*loc. cit.*) to be a constituent of jalap resin.

After extracting the aqueous liquid with ether, it was treated with a solution of basic lead acetate, which produced a light brown precipitate. This was collected, washed with water, and then suspended in water and decomposed by hydrogen sulphide. On filtering the mixture a liquid was obtained which gave a slight brown coloration with ferric chloride, but it yielded nothing definite. The filtrate from the lead subacetate precipitate was treated with hydrogen sulphide for the removal of the excess of lead, and again filtered. On concentrating this liquid a dark colored syrup was obtained, which contained a quantity of sugar, since it readily yielded *d*-phenylglucosazone, melting at 213–214° C.

In order to ascertain whether the resin obtained from the root of *Ipomœa Horsfalliæ,* Hooker, possesses any physiological activity, a test was kindly conducted for us by Dr. H. H. Dale, Director of the Wellcome Physiological Research Laboratories. One gramme of the resin was administered to a small dog, but no purgation was produced, nor could any other effect be observed.

It is evident from the results of the preceding investigation that the root of *Ipomœa Horsfalliæ,* Hooker, does not possess any constituent which would render it of medicinal value, for even the very small proportion of resin which it contains appears to be devoid of any marked physiological action.

In conclusion we desire to express our thanks to Mr. E. M. Holmes, F.L.S., for the great pains he has taken to secure the botanical identification of the material supplied to us.