

BRAZILIAN JALAP.*

BY WILBUR L. SCOVILLE.

In 1915 a brief article was published in the *Pharmaceutical Journal*¹ of London by E. W. Holmes stating that Brazilian jalap had been for some time imported into Germany and used as a cheap source of resin of jalap. He had therefore procured some of the drug, which was identified as the root of *Piptostegia pisonis* and was examined chemically by Dr. F. W. Passmore. The latter found that the root contained over 20 percent of resin which answered to the U. S. P. & Ph. Br. tests for true resin of jalap, but only 85 percent of it was soluble in ether, and it contained 8.9 percent of ash.

A search of pharmaceutical and chemical literature has failed to disclose any further account of this drug or its chemical composition. Accordingly a small amount was procured from Brazil and examined as follows:

Forty grammes of the ground drug was exhausted with 95 percent alcohol in a Soxhlet apparatus and the tincture diluted to 200 mls. This tincture yielded 7.04 percent of dry solids equivalent to 35.2 percent of extractive in the drug, and on subjecting it to the U. S. P. method of assay for resin of jalap, gave 5.64 percent of resin in the tincture, equivalent to 28.2 percent in the drug. The resin was a light tuff color, slightly hygroscopic, and it melted under boiling water.

Five hundred grammes of the drug was then extracted with alcohol, the tincture was concentrated by distillation to a syrupy liquid measuring about 180 mls, and this was poured slowly into about 7500 mls of cold water containing a little hydrochloric acid. The resin precipitated at first in a fine powder but quickly agglomerated into a sticky mass at the bottom of the jar. It was washed several times with water, then collected and dried, yielding 132.5 Gm. of resin, equivalent to a 26.5 percent yield from the drug.

Portions of this resin were shaken with petroleum benzene, benzol, ether, chloroform, and acetone, and the clear liquids evaporated. By this method it was found that 3.7 percent was soluble in petroleum benzene, 5.3 percent in benzol, 5.9 percent in ether, 19.55 percent in chloroform, and 99.4 percent in acetone. It was nearly all soluble (97.5 percent) in a 1 percent solution of potassium hydroxide, and the addition of acid to this alkaline solution did not reprecipitate the resin.

On shaking 1 Gm. of the resin, mixed with 5 Gm. of washed sand, with 100 mls of distilled water for several hours at room temperature, and filtering, 0.535 Gm. was found to be dissolved by the water. This is a remarkable solubility for a resinous body, and probably accounts for the fact that it could not be precipitated as a powder, even in ice-cold water, but always separated as a soft, sticky mass. On ignition the residue yielded 0.5 percent of ash—considerably less than was obtained by Doctor Passmore.

The acid number, as obtained by the U. S. P. process, was 23.1 and the saponification number was found to be 141.6. An alcoholic solution of the resin gave no color with a solution of ferric chloride and did not reduce Fehling's solution. But on heating the alcoholic solution with sulphuric acid a strong odor of valeric acid appeared and the neutralized liquid reduced Fehling's solution, showing that the resin is of a glucosidal nature.

Two grammes of the resin dissolved slowly in 10 mls of ammonia water, producing a cloudy but limpid solution, and the addition of an excess of hydrochloric acid did not precipitate the resin.

Since the U. S. Pharmacopoeia requires that not more than 12 percent of the resin of jalap shall be soluble in ether, and not more than 30 percent soluble in chloroform, this resin of Brazilian jalap will meet the U. S. P. requirements except as to solubility in water and the acid-resin test, which it slightly exceeds. Even

* Read before Scientific Section, A. Ph. A., Chicago meeting, 1918.

¹ *Pharm. Journal*, 1915, page 671.

the water-solubility test is greatly changed by the presence of a little acid, and the addition of acid to the clear aqueous solution produces a marked precipitate. Probably the temperature also affects the solubility markedly, since hot water dissolves it more readily. To further ascertain its character 100 Gm. of the resin was treated successively by percolation with petroleum ether, ether, chloroform and acetic ether.

The petroleum ether extract yielded 2.09 Gm., and was a reddish, oily, viscid liquid, mostly soluble in alcohol and having a markedly acid reaction. Its acid number was 12.9 and the saponification number 116.

The ether solution yielded 2.376 Gm., was solid and of a buff color, resembling the original resin, had an acid number of 82.6 and a saponification number of 62.5.

The chloroform solution yielded 2.424 Gm., was solid, crystalline and showed an acid number of 19.1 and a saponification number of 58.5.

Acetic ether dissolved all of the remainder of the resin, and this portion gave an acid number of 16.8 and a saponification number of 161.3.

All these extracts therefore contained free acids, the ether solution being richest in them, and also fatty esters.

Comparing these with an investigation of resin of jalap by Frederick B. Power and Harold Rogerson (International Congress of Applied Chemistry, 1909), an investigation which was very complete, and which established the very complex character of resin of jalap, they found the following:

Soluble in petroleum ether.....	1.9 percent
Soluble in ether.....	9.7 percent
Soluble in chloroform.....	24.1 percent
Soluble in ethyl acetate.....	22.0 percent
Soluble in alcohol.....	38.8 percent

The above were obtained by successive treatments, and represent the percentages which were dissolved after the preceding solvent had been employed.

Solubility results are not directly comparable unless obtained under identical conditions and with the same amounts of solvents. Presumably the above solubilities were obtained by extracting the resin, previously mixed with purified sawdust, in a Soxhlet apparatus, using the solvents successively. This would result in a thorough extraction of all substances which are soluble in the particular solvent used. In my work the powdered resin was simply shaken for several hours with about four times its volume of the solvent, the latter decanted, and the resin rinsed with fresh portions of solvent. In this method only the more readily soluble constituents would be extracted.

It is evident, however, that the Brazilian resin is less soluble in ether and much more soluble in ethyl acetate.

On analysis of the various fractions of the resin, Power and Rogerson found formic, acetic, butyric, valeric and other higher volatile acids, also palmitic, stearic and convolvulic acids, and by hydrolysis azelaic and sebamic acids, and a new acid which they had previously found in the root of *Ipomoea purpurea*, and which they called ipurolic acid.

The neutral bodies found were cetyl and ipurganol alcohols, a physosterol, beta-methylaesculetin and glucose, the latter showing the glucosidal nature of the resin.

I did not attempt so complete a separation of the constituents of Brazilian jalap resin, but formic and valeric acids were recognized by their odor and the

former was identified by chemical tests, glucose was produced by hydrolysis with sulphuric acid and the ready saponification of some of the fractions strongly suggested stearic or similar acids. If ipurolic acid could be separated and identified, the chemical similarity of the two resins would be established.

In their work on *Ipomoea purpurea* Power and Rogerson separated this acid as follows:

The resin was hydrolyzed by heating in alcoholic solution, containing enough sulphuric acid to make a 5 percent acid solution, for four hours under a reflux condenser. The alcohol was then removed, and the hydrolyzed resin distilled with steam. When the liquid in the still had cooled, the acid separated in fine, interlaced crystals from the aqueous fluid and was collected by filtration. It was found to be readily soluble in a weak solution of sodium hydroxide, and after rendering this acid with acetic acid the crystalline body was extracted with chloroform. To further purify it, it was again crystallized from hot water, in which it is slightly soluble. The melting point was then found to be 101° C.

On applying this process to the resin of Brazilian jalap, an acid body separated in the still and was purified by dissolving in weak sodium hydroxide solution and extracting with chloroform after acidulating. On recrystallizing from hot water it was obtained in colorless condition and had a melting point of 83.5° C. Further purification showed no change in the melting point.

It combined readily with weak alkalies, and had a saponification value of 174, as found by direct titration in alcoholic solution with decinormal alcoholic potassium hydroxide. This is a lower saponification number as well as a lower melting point than that of ipurolic acid. It is therefore a different body, but it is obtained in the same way, and does not correspond to any of the commonly known organic acids.

The resin of Brazilian jalap is thus shown to be similar in chemical character to that of *Exogonium Purga*. It is a complex body of a glucosidal nature, and contains constituents of like character. This resin meets the U. S. P. requirements for resin of jalap except that of solubility in water and the acid number. The yield is three to four times as great, and the physiological action is similar.

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KARAYA GUM, A SUBSTITUTE FOR TRAGACANTH.*

BY CLARE OLIN EWING.

The commercial value of a gum depends largely upon the purpose for which it is best suited, the most valuable generally being those varieties suitable for sizing silks and for use in confectionery and pharmacy. For pharmaceutical use the desirable qualities are a fairly high viscosity, suitable adhesive power, and freedom from appreciable odor, color and acidity. One of the gums which admirably fulfills these requirements is gum tragacanth, which is highly prized by pharmacists and is official in most pharmacopoeias. It is defined by the United States Pharmacopoeia IX (1916) as "The spontaneously dried gummy exudation from the stems of *Astragalus gummifer* Labillardiere, or from other Asiatic species of *Astragalus* (fam. *Leguminosae*.)" Gum tragacanth belongs to the class of gums

* Contribution from the Pharmacognosy Laboratory, Bureau of Chemistry, Department of Agriculture, Washington, D. C.