BRAZILIAN JALAP

PART III. AN EXAMINATION OF THE RESIN AND SOME COMPARISONS WITH THE RESINS FROM VERA CRUZ JALAP AND ORIZABA JALAP

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Received February 4, 1952

INTRODUCTION

IN Parts I and II[1,2] some indication was given of the histological and pharmacognostical features of 4 samples of commercial Brazilian jalap, together with some comparisons with Vera Cruz jalap and Orizaba jalap. Since, however, it is the so called resins of these drugs that are more commonly used in medical and veterinary practice, it was considered desirable to make an examination of Brazilian jalap resin in order to provide a quick and reliable method of identifying it and distinguishing it from the resins of Vera Cruz jalap and Orizaba jalap.

The first reference to work on Brazilian jalap resin appears to be that given by Holmes[3] in 1915, who reported that experiments by Passmore on this particular sample of drug showed "over 20 per cent. of resin answering all the B.P., 1914, and U.S.P. VIII tests for the resin of true or Vera Cruz jalap but only 0·85 per cent. soluble in ether." In 1918 Scoville[4] and Ewing and Clevenger[5] made further examination of the resin obtained from Brazilian jalap, but since that date there is no evidence of further investigation. The resins of Vera Cruz jalap and Orizaba jalap have, on the other hand, both been extensively and critically examined. Much of this work has been concerned with the physical properties and chemical constants of the resins and it is by means of these characters that the resins are at present distinguished.

MATERIALS

Samples of resin were prepared by the method given in the British Pharmaceutical Codex, 1949, from the samples of Brazilian jalap A, B, C and D, Vera Cruz jalap and Orizaba jalap referred to in Parts I and II.

Only small quantities of Brazilian jalap A and B were available so that it was not possible to obtain sufficient resin to enable all the tests to be carried out on these.

EXPERIMENTAL

Loss on Drying at 100° *C.* Determined by the method of the British Pharmacopœia the results obtained were—Brazilian jalap A 3·58, B 4·24, C 3·08, D 2·15; Vera Cruz jalap 3·66; Orizaba jalap 3·38 per cent. All percentages are calculated with reference to the resins dried at 100° C.

Solubility in Organic Solvents. Scoville[4] and Ewing and Clevenger[5] extracted Brazilian jalap resin with various solvents successively in a continuous extraction apparatus, a procedure which had previously been employed by Power and Rogerson[6,7] for the resins of Vera Cruz and

Orizaba jalap. For reference purposes the percentages extracted per solvent as reported by these workers are given in Table I.

TABLE I

EXTRACTION OF JALAP RESIN WITH VARIOUS SOLVENTS

Solvent (in order used)	Brazilian jalap resin (Scoville)	Brazilian jalap resin (Ewing and Clevenger)	Vera Cruz jalap resin (Power and Rogerson)	Orizaba jalap resin (Power and Rogerson)
	per cent.	per cent.	per cent.	per cent.
Light petroleum..	2·09	2·1	1·9	6·2
Ether	2·376	5·4	9·7	64·8
Chloroform ..	2·424	73·4	24·1	0·6
Ethyl acetate ..	(All remainder) 93·1	14·2	22·0	24·8
Ethanol		4·7	38·8	2·3

It will be noted that there is considerable discrepancy between the amounts of Brazilian jalap resin extracted with chloroform as given by Scoville and by Ewing and Clevenger, and it is difficult to attempt an explanation of this. In order to obtain some comparative figures for the resins available, Brazilian jalap resin C and D, Vera Cruz jalap resin and Orizaba jalap resin were successively extracted in a Soxhlet apparatus with the same solvents as those referred to in Table I. In view of previous criticisms of the method, care was taken to ensure that the solvents were dry and in the case of ether and chloroform, that they were free from ethanol.

The percentages of resin extracted by the various solvents are given in Table II.

TABLE II

EXTRACTION OF JALAP RESINS WITH VARIOUS SOLVENTS (AUTHOR'S RESULTS)

Solvent (in order used)	Brazilian jalap resin	Brazilian jalap resin	Vera Cruz jalap resin	Orizaba jalap resin
	Sample C	Sample D		
	per cent.	per cent.	per cent.	per cent.
Light petroleum..	2·86	1·94	2·66	2·36
Ether	2·94	3·61	4·01	73·26
Chloroform ..	48·12	50·11	22·25	1·12
Ethyl acetate ..	42·33	43·06	22·08	19·84
Ethanol		all remainder		

Scoville[4] further determined the solubility of Brazilian jalap resin in various solvents directly and it was decided to make similar determinations of all the resins available. The percentage resin soluble in the solvents are given in Table III, together with Scoville's figures for comparison.

It must be noted that whereas Scoville obtained his figures by shaking 1 g. of resin with about 4 times its volume of solvent for several hours, filtering, evaporating to dryness and weighing the residue, the method used here was to use 100 ml. of solvent, shake occasionally during 24 hours, decant or filter, recover most of the solvent, evaporate to dryness and weigh.

TABLE III

SOLUBILITY OF JALAP RESIN WITH VARIOUS SOLVENTS

Solvent	Brazilian jalap resin					Vera Cruz jalap resin	Orizaba jalap resin
	Scoville	A	B	C	D		
	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.
Light petroleum..	3·7	—	—	2·84	1·96	2·58	2·62
Ether	5·9	6·26	3·83	3·72	3·94	5·25	74·72
Chloroform ..	19·55	52·31	43·44	53·38	56·55	20·15	71·41
Benzene ..	5·3	—	—	4·25	3·87	5·23	90·64
Acetone	99·4	—	—	98·42	97·92	99·04	98·86

The figures obtained for the percentage soluble in chloroform shows some degree of uniformity for all 4 samples of Brazilian jalap resin and, although the number of samples examined are too few to suggest limits for the solubility of this resin in this solvent, it appears to be between 40 and 60 per cent.

Solubility in Water. Scoville[4] also reported on the solubility of Brazilian jalap resin in water, stating that on shaking 1 g. of resin mixed with washed sand with 100 ml. of distilled water for several hours at room temperature 0·535 g. was found to be dissolved and he commented that 53·5 per cent. was an abnormally high figure for the solubility of a resinous body in water.

It is possible that this figure does not represent the true figure for the amount of resin soluble in water but rather that for the maximum solubility of the resin substance in water at room temperature. In order to ascertain how much of the resin was actually water-soluble 3 quantities each of 1 g. (Sample D) were shaken continuously for 3 hours with 50 ml., 100 ml. and 500 ml. of distilled water respectively. The aqueous extractives were then poured into tared dishes, evaporated to dryness and weighed. Further similar quantities of distilled water were then added and the process repeated until the 3 quantities of resin had been extracted with 200 ml., 500 ml. and 2000 ml. of distilled water respectively. The results obtained are given in Table IV.

TABLE IV

RESIN EXTRACTED BY WATER FROM 1 G. OF BRAZILIAN JALAP RESIN FROM SAMPLE D

	Volume of water used for each extraction	Weight of resin soluble in each volume of water					Total weight of resin extracted
		1	2	3	4	5	
		g.	g.	g.	g.	g.	g.
1	50 ml.	0·1668	0·1640	0·1188	0·0502	—	0·4998
2	100 ml.	0·2340	0·2240	0·0706	0·0208	0·0072	0·5606
3	500 nl.	0·3842	0·3464	0·0702	0·0056	—	0·8064

It will be seen from these results that the amount of resin extracted by water varies with the volume of water used. On all occasions when the resin was shaken with water there was considerable frothing and it was considered that the anomalous results might be due to some hydrolysis of the glycoside constituent of the resins. However, when the aqueous extractive was heated with Fehling's solution no reduction occurred. No further attempt was made to explain the results. The figures do,

however, confirm the work of Scoville that the water-soluble portion of Brazilian jalap resin is abnormally high for a convolvulaceous resin. They also suggest that in order to obtain reproducible results a standardised method must be adopted.

Determination of the water-soluble matter according to the method A of the British Pharmacopœia (using distilled water) gave the following results:—Brazilian jalap resin—A. 20·66; 20·62. B. 32·71; 32·73. C. 22·06; 22·11. D. 21·75; 21·70 per cent.; Vera Cruz jalap resin—0·56; 0·54 per cent.; Orizaba jalap resin—0·32; 0·35 per cent.

There was considerable frothing with the Brazilian jalap resins which persisted for some time, that for Sample B persisting for several hours. The *p*H of the distilled water used was 5·4. The *p*H of the aqueous extractives were:—Brazilian jalap resin—A. 4·35; B. 4·30; C. 4·40; D. 4·35; Vera Cruz jalap resin—4·85; Orizaba jalap resin—5·30.

Specific Rotation.—Although these convolvulaceous resins are not constant in composition, previous workers have given some consideration to the specific rotation of resins which have been decolorised by refluxing ethanolic solutions with animal charcoal. Guigues considered that the optical activity had a better diagnostic value than ether-solubility. Cowie[9] and Power and Rogerson[6,7] have also examined various convolvulaceous resins for optical activity and Ewing and Clevenger[5] have given the only reported specific rotation for Brazilian jalap resin.

As a further comparison between the resins, the specific rotation was determined on samples of resins after purifying as far as possible by refluxing ethanolic solutions with animal charcoal for four hours. The resulting colour in each case was pale straw yellow. The results are given in Table V, together with figures obtained by the previous workers.

TABLE V

SPECIFIC ROTATION

		Guigues[8]	Cowie[9]	Power and Rogerson[6,7]	Ewing and Clevenger[5]
Brazilian jalap	—	—	—	—	−48·5°
Sample A	−19·67°	—	—	—	—
„ B	−20·48°	—	—	—	—
„ C	−20·14°	—	—	—	—
„ D	−20·00°	—	—	—	—
Vera Cruz jalap	−37·6°	−36·0°	−37·3°	−37·0°	—
Orizaba jalap	−25·4°	−24·45°	−27·0°	−23·0°	—

It will be observed that in spite of probable variation in the composition of the resins of each type of jalap, the specific rotation obtained by various workers is fairly constant, except in the case of Brazilian jalap resin, where the value obtained by Ewing and Clevenger is more than double that obtained for each of the 4 samples examined.

Acid Values, Saponification Value, Melting-point. In order to complete the comparative examination of the physical properties and chemical constants of the 3 varieties of resin, acid values, saponification values and melting points were determined on the purified resins. The results

obtained for the acid values and saponification values are given in Table VI, together with values given by previous workers for comparative purposes.

TABLE VI

ACID VALUES AND SAPONIFICATION VALUES

	Acid value		Saponification value	
Brazilian jalap resin—	—	Scoville 23·1	—	Scoville 141·6
Sample C	24·58	—	164·1	—
„ D	23·73	—	158·8	—
Vera Cruz jalap resin ..	17·15	Power and Rogerson 15	142·3	Power and Rogerson 140
Orizaba jalap resin ..	21·65	Power and Rogerson 20	182·4	Power and Rogerson 180

The melting-points of the purified resins obtained by the method described in the B.P. Appendix IV, AI were:—

Brazilian jalap resin *all samples*	94° to 100° C.
Vera Cruz jalap resin	138° to 144° C.
Orizaba jalap resin	121° to 127° C.

Examination under Screened Ultra-violet Light. When the resins were powdered, spread in thin layers and examined in screened ultra-violet light, it was quite easy to distinguish between them by their appearance, which was as follows:—Brazilian jalap resin (all samples), yellowish brown; Vera Cruz jalap resin, pale pinkish violet; Orizaba jalap resin, deep bluish purple.

Elementary Chromatograms. Adsorption chromatograms were prepared from the varieties of resins with heavy magnesium oxide as the adsorbent. Both tube and disc chromatograms were prepared using:—(i) ethanolic solutions of the resins; (ii) ether solutions of ether-soluble portions; (iii) ethanolic solutions of ether-insoluble portions; (iv) chloroform solutions of the chloroform-soluble portions; (v) ethanolic solutions of the chloroform-insoluble portions.

The chromatograms were developed with the same solvent as that used to prepare the solutions. When observed in daylight it was possible to distinguish pale coloured zones in most of the chromatograms but they were not so distinctive as to enable the various resins to be characterised. It was possible to note a difference between the chromatogram of the

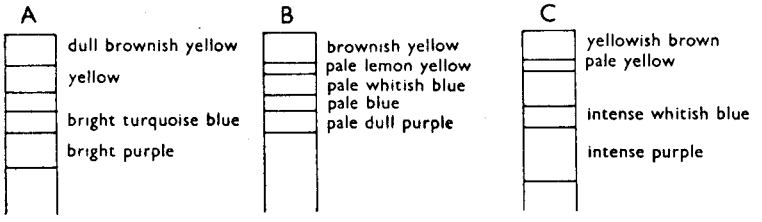

FIG. 1. Appearance of the chromatograms of the resins in screened ultra-violet light. A. Vera Cruz jalap. B. Brazilian jalap, Samples A, C and D. C. Orizaba jalap.

resin of Brazilian jalap B and the other Brazilian jalap resins. The appearance of the chromatograms in screened ultra-violet light was, however, most striking. The results obtained with the tube chromatograms when examined in screened ultra-violet light are given diagrammatically in Figures 1 and 2. It is, however, impossible to describe

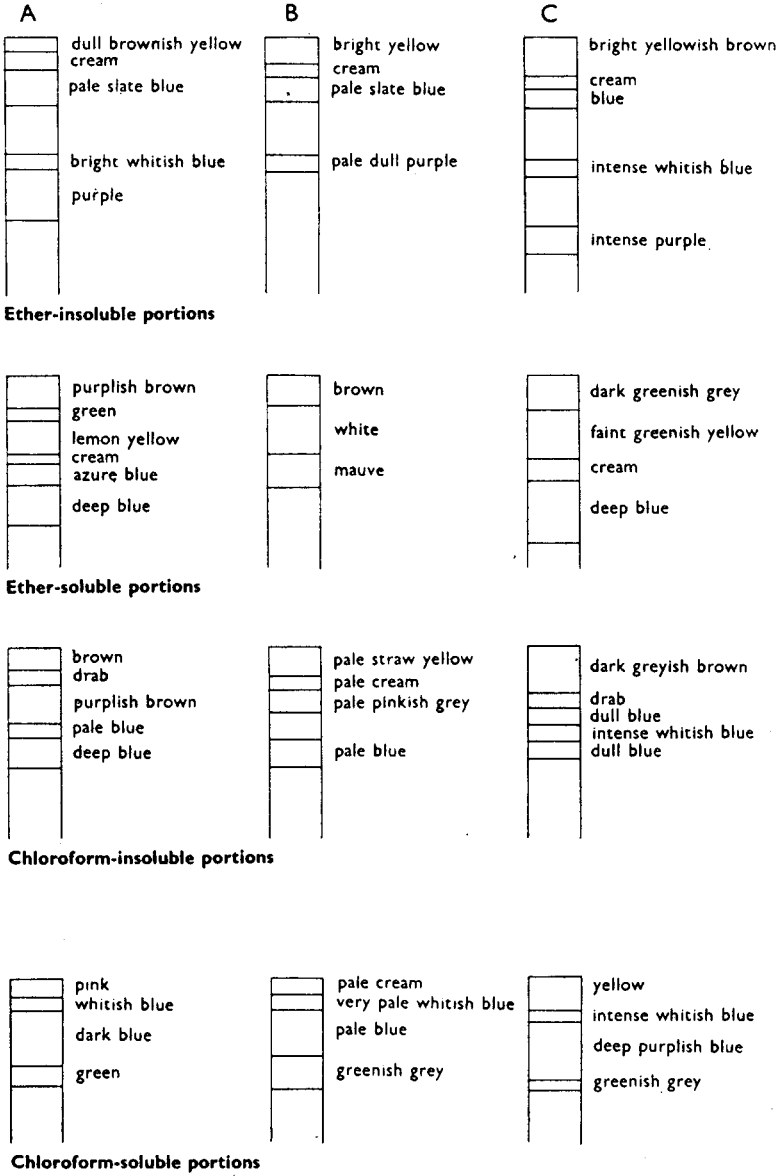

FIG. 2. Appearance of chromatograms in screened ultra-violet light. A. Vera Cruz jalap resin. B. Brazilian jalap resin. C. Orizaba jalap resin.

adequately the actual colours observed, and it is necessary therefore to make or emphasise the following points:—(1) the chromatogram of the resin of Brazilian jalap B differed from the resins of the other samples of Brazilian jalap by having a rather yellower first zone and a second narrow zone which was cream in colour rather than pale lemon yellow. Further, the following whitish blue zone was far less intense; (2) in all the chromatograms prepared from Brazilian jalap resin the blue and purple zones were pale and dull, while in the resins from Vera Cruz and Orizaba jalap the blue and purple zones were bright and often intense; (3) when adsorbed on magnesium oxide, β-methylæsculetin gives a bright pale yellow fluorescence.

Summary and Conclusions

The resins obtained from the 4 samples of Brazilian jalap have been examined and compared with the resins of Vera Cruz jalap and Orizaba jalap. The appearance of the chromatograms under ultra-violet light indicated that the resin of Brazilian sample B differed slightly from those of the other three samples which appeared to be identical.

Brazilian jalap resin can be characterised by:—

(i) Slight solubility in ether.
(ii) Considerable solubility in water, the aqueous solutions giving copious persistent frothing when agitated.
(iii) A yellowish brown colour when observed in screened ultra-violet light.
(iv) The almost complete absence of blue and purple zones from the chromatograms when viewed in screened ultra-violet light.
(v) Specific rotation (of the purified resin) about $-20°$.
(vi) Melting-point (of the purified resin) of 94 to 100° C.

Brazilian jalap resin may be distinguished from Vera Cruz jalap resin by the following tests:—

(i) Appearance in screened ultra-violet light.
(ii) Appearance of chromatograms in screened ultra-violet light.
(iii) Solubility in water.
(iv) Specific rotation (of purified resins).
(v) Melting-point (of purified resin).

It may be distinguished from Orizaba jalap resin by the above tests and, in addition, by—

(vi) Solubility in ether.

References

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