# Chemical examination of cotton-root bark / by Frederick B. power and Henry Browning, Jun.

### **Contributors**

Power, Frederick B. 1853-1927. Browning, Henry. Wellcome Chemical Research Laboratories.

### **Publication/Creation**

London: Wellcome Chemical Research Laboratories, 1914.]

#### **Persistent URL**

https://wellcomecollection.org/works/jgqhran3

#### License and attribution

This work has been identified as being free of known restrictions under copyright law, including all related and neighbouring rights and is being made available under the Creative Commons, Public Domain Mark.

You can copy, modify, distribute and perform the work, even for commercial purposes, without asking permission.



Wellcome Collection 183 Euston Road London NW1 2BE UK T +44 (0)20 7611 8722 E library@wellcomecollection.org https://wellcomecollection.org

168

# CHEMICAL EXAMINATION OF COTTON-ROOT BARK.\*

BY

FREDERICK B. POWER & HENRY BROWNING, Jun-

Cotton-root bark appears to have been officially recognised only by the United States Pharmacopæia, which describes it as "the dried bark of the root of Gossypium herbaceum, Linné, or of other cultivated species of Gossypium." A decoction and a liquid extract of the bark are, however, included in the Indian and Colonial Addendum to the British Pharmacopæia, the use of these preparations being based upon the reputed value of the bark as an emmenagogue and for arresting hæmorrhage.

The first chemical examination of cotton-root bark was made by E. S. Wayne (Amer. J. Pharm., 1872, 44, 289), who noted the presence of sugar and 3 per cent. of a red, acidic resin, but no definite substance was isolated. Subsequent examinations by Staehle (ibid., 1875, 47, 457) and by Drueding (ibid., 1877, 49, 386) have contributed but little further information respecting its constituents. On account of this deficiency of knowledge, it was deemed desirable to subject the bark to a more complete chemical study.

### EXPERIMENTAL.

The material employed for the principal part of this investigation was kindly supplied by Professor J. U. Lloyd, of Cincinnati, Ohio, to whom our best thanks may here be expressed.

A portion (10 Gms.) of the ground material was subjected to a preliminary test for alkaloid by extraction with Prollius' fluid, but with a negative result.

<sup>\*</sup> Communication from the Wellcome Chemical Research Laboratories, London, E.C., and reprinted from The Pharmaceutical Journal and Pharmacist, September 26, 1914.

Another portion (20 Gms.) of material was extracted successively in a Soxhlet apparatus with various solvents, when the following amounts of extract, dried in a water-oven, were obtained:—

Petroleum (b.p. 35-50°) ext	racted	0.70 gr	amme	-	3.50 p	er cent.
Ether	**	0.35	**		1.75	,,
Chloroform	,,	0.80	,,	=	4.00	***
Ethyl acetate	"	0.60	,,	=	3.00	11
Alcohol	**	1.53	"	===	7 65	,,
Total		3.98 g	ramme	s =	19·9 pe	r cent.

For the purpose of a complete examination of the constituents of the bark 21.66 kilos of the air-dried ground material were completely extracted with hot alcohol. After the removal of the greater portion of the alcohol 3.07 kilos of a viscid, dark-red extract were obtained.

DISTILLATION OF THE EXTRACT WITH STEAM. SEPARATION OF AN ESSENTIAL OIL.

The entire amount of the alcoholic extract was mixed with water and subjected to distillation in a current of steam. On extracting the distillate with ether a very small quantity (2.2 Gms.) of a pale yellow essential oil was obtained, and a further small amount (about 5 Gms.) of the same product was subsequently obtained on working up a somewhat larger quantity of the bark in the above-described manner.

The essential oil, when separately distilled, passed over chiefly between 120 and 135° at the ordinary atmospheric pressure. It gave the colour reaction of furfuraldehyde. When kept for a short time it deposited a few small crystals, which, after recrystallisation from ethyl acetate, were obtained in faintly yellow needles, melting at 112-114°. This substance possessed the characters of acetovanillone, and no depression of the melting-point ensued when mixed with the latter, but the amount was too small to permit of its more complete identification.

After the above-described treatment there remained in the distillation flask a dark-brown aqueous liquid (A) and a considerable quantity of a deep purplish-coloured resin (B). The resin was granular in character, and after being washed and dried amounted to 2300 Gms., thus corresponding to 10.6 per cent. of the weight of bark employed.

# Examination of the Aqueous Liquid (A).

The aqueous liquid, after concentration under diminished pressure, was extracted many times with ether, the ethereal extract being then successively shaken with aqueous ammonium carbonate, sodium carbonate, and sodium hydroxide. For the more complete examination of the products thus obtained, a larger quantity of bark was subsequently employed. The acid material removed by the ammonium

carbonate was esterified with methyl alcohol, and the product distilled in a current of steam. The volatile esters were extracted from the distillate by ether, and then separated into phenolic and non-phenolic portions by shaking the ethereal solution with aqueous 1 per cent. potassium hydroxide. From the non-phenolic product a small amount of ester crystallised in colourless plates, melting at 97-98°, but the amount was not sufficient for further examination. The ester from which these crystals had been separated yielded a very small amount of a liquid acid.

The phenolic portion of the volatile esters was treated with petroleum of high boiling-point, when a small amount of a sparingly soluble, crystalline substance separated. This was found to be an acid, since it remained unchanged on heating with an alcoholic solution of potassium hydroxide, and its ester must, therefore, have become hydrolysed by the abovementioned extraction with aqueous alkali. The acid sublimed in minute, colourless crystals, melting at 265-270°, and gave no coloration with ferric chloride, but the amount obtained was only 0.04 Gm.

The petroleum mother-liquors from the acid melting at 265-270° were evaporated to dryness, and the residue hydrolysed. The acid so obtained was partly crystalline and of a dark-It was dissolved in a mixture of alcohol and brown colour. benzene, from which it crystallised on evaporating the alcohol. After several such treatments it was obtained in clusters of nearly colourless needles, melting constantly at 196-199°, with The acid was very sparingly soluble in evolution of gas. chloroform, benzene, or high-boiling petroleum, but dissolved readily in water, methyl or ethyl alcohol. Its aqueous solution gave with ferric chloride an intense blue colour, changing rather rapidly to an olive-green or black. With lead acetate a white precipitate was produced. The characters of this phenolic acid render it probable that it was 2:3dihydroxybenzoic acid, which is stated to melt at 204°, but the amount obtained was not sufficient to permit of analysis or further direct confirmation.

The benzene mother-liquors remaining after the separation of the above-described acid were evaporated, and the residue crystallised twice from dilute hydrochloric acid, when a very small amount (0.01 Gm.) of an acid was obtained in the form of small, colourless needles. This acid gave a violet coloration with ferric chloride, and when warmed with methyl alcohol and a drop of concentrated sulphuric acid it developed the odour of methyl salicylate. Although melting somewhat lower than salicylic acid, as no depression of the melting-point ensued when mixed with the latter, no doubt could be entertained of its identity.

The phenolic portion of the esters which had not been volatilised by the above-described distillation with steam yielded a further small amount of the acid melting at 196199°, whereas nothing could be isolated from the non-phenolic product.

ISOLATION OF A NEW, PHENOLIC SUBSTANCE, C9H8O(OH)2.

The ethereal extract of the aqueous liquid (A), which had been shaken with aqueous ammonium carbonate, was next extracted with successive small amounts of aqueous sodium carbonate, the strength of the latter being gradually increased from 1 to 10 per cent. The first four extractions yielded a product which, after fractional crystallisation from dilute alcohol, separated in nearly colourless needles, melting at 258-260°, and amounting to 0.34 Gm. The substance was not readily soluble in cold alcohol, acetone, chloroform, or methyl alcohol, but dissolved readily in pyridine. It dissolves in aqueous potassium hydroxide, forming a bright yellow liquid, and its solution in aqueous alcohol gives with ferric chloride a purple colour.

The substance, dried at 105°, was analysed, and its molecular weight determined by the microscopic method, with the following results:—

 $C_9H_{10}O_3$  requires C = 65.1; H = 6.0 per cent. M.W. = 166.

An acetyl derivative of the substance was prepared by dissolving 0.09 Gm. in a little pyridine, and gently boiling for an hour with acetic anhydride. The liquid was then concentrated and shaken with water, the substance which thus separated being collected, dissolved in ether, and the solution washed with aqueous sodium carbonate. After removing the ether the acetylated compound was crystallised from alcohol, when 0.05 Gm. of small, tabular crystals were obtained, which melted at 152°.

0.0491 gave 0.1120 CO<sub>2</sub> and 0.0274 H<sub>2</sub>O. C = 62.2; H = 6.2.  $C_9H_8O(0.CO.CH_3)_2$  requires C = 62.4; H = 5.6 per cent.

A further small amount (0.06 Gm.) of the substance was converted into a benzoyl derivative by boiling for half an hour with benzoyl chloride and a little pyridine. The product was cautiously treated with hot water, and then a current of steam passed through the liquid, when, after crystallisation from alcohol, colourless microscopic crystals were obtained, melting at about 155°.

A consideration of these results indicates the above-described substance to be a new dihydric alcohol, possessing the formula  $C_9H_{10}O_3$ , but the amount available was not sufficient for its further investigation.

Isolation of a New, Yellow, Phenolic Substance, C<sub>13</sub>H<sub>16</sub>O<sub>3</sub>(OH)<sub>2</sub>.

The later washings of the ethereal extract of the aqueous liquid (A) with sodium carbonate, and also with aqueous sodium hydroxide, were of a deep purple colour. The intensity of colour was but slightly diminished after many extractions with the first-mentioned alkali, but the substance producing the colour was rapidly removed by sodium hydroxide. These two alkaline liquids, when acidified and extracted with ether, yielded small amounts of crystalline yellow products, which were similar in appearance. After numerous crystallisations from dilute alcohol and dilute acetone a substance was obtained in slender, orange-yellow needles, melting at 210-212°. The mother-liquors from the latter yielded a further small amount of substance in the form of brown hexagonal plates, melting at 214-217°. These crystals, although different in appearance from the yellow needles, were found to be identical with the latter. This was indicated by a determination of their molecular weight by the microscopic method (mean value in acetone solution = 263; compare result below) and by the fact that when subsequently removed from the solution the substance was obtained in the yellow, needle-shaped form.

The substance in the form of yellow needles was dried at 115° without any loss in weight. Its analysis and a determination of the molecular weight by the microscopic method gave the following results:—

0.0648 gave 0.1464  $CO_2$  and 0.0447  $H_2O$ . C = 61.6; H = 7.7 0.0364 , 0.0813  $CO_2$  , 0.0248  $H_2O$ . C = 60.9; H = 7.6 0.0534, in 7 Cc. of acetone, was between 0.0275 and 0.03 mol., using benzil as the standard. Mean M.W. = 266.

 $C_{13}H_{18}O_5$  requires C = 61.4; H = 7.1 per cent. M.W. = 254.

A small portion of the substance was converted into an acetyl derivative by heating with acetic anhydride in the presence of a little pyridine. This derivative separated from alcohol in small, nearly colourless tabular crystals, melting at 147-149°. It was dried at 110° and analysed.

0°0732 gave 0°1621  $CO_2$  and 0°0410  $H_2O$ . C = 60°4; H = 6°2  $C_{15}H_{16}O_3(O.CO.CH_3)_2$  requires C = 60°4; H = 6°5 per cent.

A further small amount of the yellow substance of slightly lower melting-point (207°) was heated with benzoyl chloride in the presence of a little pyridine. The product, when crystallised from alcohol, separated in small nodules, melting at 153-157°, but the amount was not sufficient for analysis.

The original yellow substance gave with ferric chloride solution a brown tint. With the alkali carbonates and hydroxides, with lead acetate and barium acetate, and also with concentrated sulphuric acid, a deep purple colour was produced. These characters suggested that the substance

might be an anthraquinone derivative, but the analytical

figures indicated that this could not be the case.

With consideration of the above results it would appear probable that the yellow crystalline substance is a new dihydric phenol, possessing the empirical formula  $C_{13}H_{18}O_s$ . The exceedingly small proportion of this substance contained in the cotton-root bark precluded its further investigation.

After the above-described extraction of the ethereal liquid with alkali carbonates and hydroxide, the ether was evaporated, but nothing definite could be isolated from the

residue.

The original aqueous liquid (A) which had been extracted with ether, as above described, was subsequently shaken a number of times with warm amyl alcohol. These extracts were united, washed with a little water, and concentrated under diminished pressure. On keeping, some material separated, and fractions of varying solubility were obtained on further concentration. Nothing crystalline could be obtained from these products, either directly or after acetylation. They were consequently brought together and boiled for three hours with 5 per cent. hydrochloric acid, when a considerable amount of indefinite resinous material separated. No sugar was produced by this treatment, and there was, therefore, no evidence of the presence of a glucosidic substance in the amyl-alcohol extract.

After extracting the aqueous liquid with ether and amyl alcohol, as above described, it was next treated with a slight excess of a solution of basic lead acetate. An abundant light-brown precipitate was thus produced, which was collected, thoroughly washed with water, then suspended in water, and decomposed by hydrogen sulphide. The filtered liquid gave no evidence of the presence of tannin, nor did it yield anything definite when boiled with an amount of sulphuric acid representing 5 per cent. of the solution. On subsequently rendering the liquid strongly alkaline, and again heating, only a trace of substance was obtained, which gave a green coloration with ferric chloride, and appeared to be 3: 4-dihydroxycinnamic acid.

The filtrate from the above-mentioned basic lead acetate precipitate was treated with hydrogen sulphide for the removal of the excess of lead, and the filtered liquid concentrated under diminished pressure to a thin syrup, which amounted to 818 Gms. This syrup gave no reaction with the usual alkaloid reagents, and no precipitate with mercuric nitrate, but when heated with potassium hydroxide much ammonia was evolved. It contained an abundance of sugar, since it readily

yielded d-phenylglucosazone, melting at 212-214°.

A quantity of the above-mentioned syrup was deprived as completely as possible of water, and then acetylated by means of acetic anhydride. The product, after keeping for some

time, yielded a small amount of colourless crystals, which after recrystallisation from dilute alcohol separated in needles, melting at 125-127°. This substance was evidently penta-acetyldextrose, since no depression of the melting-point ensued when mixed with a known specimen of the latter.

# Isolation of Betaine, C5H11O2N.

A portion (50 Gms.) of the above-mentioned syrupy liquid was acidified with sulphuric acid, and a solution of phosphotungstic acid added. The resulting precipitate was collected, washed with water, decomposed with cold aqueous barium hydroxide, and the mixture filtered, after which the excess of baryta was removed from the filtrate by means of carbon dioxide. The liquid being then rendered exactly neutral with hydrochloric acid, it was evaporated to dryness under diminished pressure, and the residue extracted with alcohol. To this alcoholic liquid a saturated alcoholic solution of mercuric chloride was added, and the mixture kept for several hours, after which the resulting precipitate was collected and dissolved as completely as possible in hot water. The aqueous solution was then treated with hydrogen sulphide for the removal of the mercury, filtered, and the filtrate evaporated to dryness under diminished pressure. A crystalline product was thus obtained which showed no sign of deliquescence, nor was anything appreciable removed by treatment with absolute alcohol, thus indicating the absence of choline. separated from alcohol containing a little water in colourless crystals, melting at 244-246°. The identity of this substance with betaine hydrochloride was established by the following results of its analysis, after drying at 105° :-

0.0863 gave 0.1228 CO<sub>2</sub> and 0.0638 H<sub>2</sub>O. C = 38.8; H = 8.2 0.1120 gave 8.8 Cc. N<sub>2</sub> at 15.5° and 761 Mm. N = 9.2 0.0679 gave 0.0643 AgCl. Cl = 23.4.

 $C_5H_{12}O_2NC1$  requires C=39.1; H=7.8; N=9.1; Cl=23.1 per cent.

The amount of betaine  $C_5H_{11}O_2N$  obtained, as calculated from the hydrochloride, was equivalent to 0.18 per cent. of the weight of bark employed.

# EXAMINATION OF THE RESIN (B).

A portion (575 Gms.) of the resinous material, which had been separated from the aqueous liquid (A), as previously described, was distributed by means of alcohol over purified sawdust, and the thoroughly dried mixture then extracted in a large Soxhlet apparatus with various solvents. The weights of the products were as follows:—

				Grammes.
Petroleum (b.p.	35-50°)	extracted		61.0
Ether				
Chloroform				5.5
Ethyl acetate .				25.0
Alcohol				
			To	otal 204.0

It will be observed that in the course of the above-described operations more than half of the resin had been rendered insoluble.

# PETROLEUM EXTRACT OF THE RESIN.

This extract, after the removal of the solvent, was heated with an alcoholic solution of potassium hydroxide, the greater portion of the alcohol then removed, water added, and the The amount of alkaline mixture extracted with ether. material thus obtained was 32.7 Gms. For the complete examination of this material the remaining portion (1725 Gms.) of the resin was extracted with light petroleum, and the extract treated as above described. The whole of the neutral material was then heated with phthalic anhydride in the presence of a little pyridine, the mixture being maintained at a temperature of 140° for four hours. The product was dissolved in a mixture of ether and chloroform, the solution being first washed with water, and then shaken with aqueous sodium carbonate During the latter operation an emulsion was formed, owing to the separation of a sodium salt of an acid phthalic ester.

# ISOLATION OF A FATTY ALCOHOL, C20H42O.

The above-mentioned emulsion was washed several times with chloroform and ether, these liquids being added to the main portion of the chloroform-ether mixture, after which the sodium salt of the acid phthalic ester was heated with an alcoholic solution of potassium hydroxide. By this means a small amount of a colourless, crystalline substance was obtained, which evidently was an alcohol, and melted at 77.5°-78.5°. This melting-point remained unchanged after several further crystallisations from both alcohol and high-boiling petroleum. It was dried at 110° and analysed.

0.0868 gave 0.2561 CO<sub>2</sub> and 0.1104 H<sub>2</sub>O. C = 80.5; H = 14.1 0.0805 , 0.2373 CO<sub>2</sub> , 0.1027 H<sub>2</sub>O. C = 80.4; H = 14.2  $C_{20}H_{42}O$  requires C = 80.5; H = 14.1 per cent.

An alcohol of the composition  $C_{20}H_{42}O$  appears to have first been obtained by Étard (Compt. rend., 1892, 114, 365) from the leaves of Medicago sativa, Linné, and was termed medicagol. It was described as forming colourless, microscopic crystals, melting at 80°. Subsequently Haller (Compt. rend., 1907, 144, 595) obtained from the wax of a Madagascar palm, Raphia Ruffia, an alcohol melting at 80°, and agreeing most nearly in composition with the formula  $C_{20}H_{42}O$ , but which was considered not to be identical with the medicagol of Étard. The very small amount of substance obtained in the present investigation did not permit of its further characterisation.

# ISOLATION OF A PHYTOSTEROL, C27H46O.

The above-mentioned chloroform-ether mixture, from which the fatty alcohol had been removed by means of aqueous sodium carbonate, was heated with an alcoholic solution of potassium hydroxide for the removal of the excess of phthalic anhydride, after which a product was obtained which consisted chiefly of a mixture of a phytosterol and a hydrocarbon. After repeated fractional crystallisation from alcohol the phytosterol separated in colourless, glistening plates, melting at 130°, and amounted to about 0.6 Gm.

0.0868, on heating at 120°, lost 0.0035  $H_2O$ .  $H_2O = 4.0$  0.0833\* gave 0.2567  $CO_2$  and 0.0900  $H_2O$ . C = 84.0; H = 12.0

 $\mathrm{C}_{27}\mathrm{H}_{46}\mathrm{O},\mathrm{H}_2\mathrm{O}$  requires  $\mathrm{H}_2\mathrm{O}=4.5$  per cent.

 $C_{27}H_{46}O$  requires C = 83.9; H = 11.9 per cent.

A portion of the phytosterol was converted into an acetyl derivative, which melted at 122°.

The above-mentioned hydrocarbon, from which the phytosterol had been separated by fractional crystallisation, was found still to contain some fatty alcohol, which could only be removed by again submitting it to treatment with phthalic anhydride. A very small amount of a hydrocarbon was thus finally obtained, which melted at 60-61°.

0.0409 gave 0.1272  $CO_2$  and 0.0555  $H_2O$ . C=84.8; H=15.1  $C_{30}H_{62}$  requires C=85.3; H=14.7 per cent.

It appears probable that this substance was triacontane, but the amount was not sufficient to ascertain the constancy of its somewhat low melting-point.

## EXAMINATION OF THE FATTY ACIDS.

The aqueous alkaline liquid resulting from the hydrolysis of the petroleum extract of the resin, which had been extracted with ether for the removal of unsaponifiable material, was next acidified, and again extracted with ether. After the removal of the solvent the fatty acids were converted into their methyl esters, which distilled for the most part between 220 and 225°, 22 Mm., and amounted to 27.5 Gms. This mixture of esters was then hydrolysed, and the fatty acids separated into liquid and solid portions by conversion into their lead salts and treatment of the latter with ether.

The Liquid Acid.—The portion of lead salt which was soluble in ether, when decomposed by hydrochloric acid, yielded 13.5 Gms. of liquid acid. The latter, after being distilled at 17 Mm. pressure, gave on analysis C = 76.5; H = 11.8 per cent., had an iodine value of 98.5, and a neutralisation value of 199.1.

 $C_{18}H_{34}O_2$  requires C = 76.6; H = 12.1 per cent. I.V. = 90.1; N.V. = 198.9.

<sup>\*</sup> Anhydrous substance.

The liquid acid was thus found to consist of nearly pure oleic acid.

The Solid Acid.—The portion of lead salt which was insoluble in ether, when decomposed by hydrochloric acid, yielded a comparatively small amount of solid acid, which was crystallised three times from alcohol. A trace of an acid melting at 72° was thus obtained, while the greater part melted at 58-60°. This gave on analysis C = 75·1; H = 12·5 per cent., and had a neutralisation value of 215·2.

 $C_{16}H_{32}O_2$  requires C = 75/0; H = 12.5 per cent. N.V. = 219.1.

It is seen from the above results that the solid acid consisted almost wholly of palmitic acid.

# ETHER EXTRACT OF THE RESIN.

This extract was of a beautiful dark-red colour. Its ethereal solution was shaken successively with aqueous ammonium carbonate, sodium carbonate, and sodium hydroxide, when the first-mentioned alkali was found to remove material possessing a red colour. On acidifying the alkaline liquid, however, and extracting again with ether, the greater portion of the material remained undissolved, and nothing definite could be obtained from it. The sodium carbonate extract, on acidification, yielded 32 Gms. of a black resin. The sodium hydroxide extract, when acidified, gave a product from which nothing definite could directly be obtained. It was, therefore, acetylated, and the resulting product hydrolysed, when a small amount of substance was obtained, which melted at 72°. After repeated separation from alcohol, and, finally, from high-boiling petroleum, the substance was obtained in a definitely crystalline form, melting at 76-77.5°, and evidently consisted of ceryl alcohol.

0.0822 gave 0.2454 CO<sub>2</sub> and 0.1075  $H_2O$ . C = 81.4; H = 14.5.  $C_{27}H_{56}O$  requires C = 81.8; H = 14.1 per cent.

After the extraction of the ethereal liquid with aqueous alkalis the ether was evaporated, but from the small amount of material thus obtained only a trace of phytosterol could be isolated.

As all the attempts to obtain anything further of a definite character from the ether extract of the resin were futile, a portion of it was mixed with zinc dust and heated in a current of hydrogen. A trace of a brown oil was thus obtained which possessed the odour of pyrrol, and gave the reaction of the latter with a pine chip moistened with hydrochloric acid. The amount of nitrogenous substance in the resin must, however, have been extremely small.

CHLOROFORM, ETHYL ACETATE, AND ALCOHOL EXTRACTS OF THE RESIN.

The chloroform and alcohol extracts were very small in amount, and nothing of interest could be obtained from them. The ethyl acetate extract had a dark-red colour, and was similar in appearance to the portion of the ether extract which was soluble in aqueous ammonium carbonate. The ethyl acetate and alcohol extracts were separately heated with dilute acid and with caustic alkali, but they yielded nothing definite, and a similar negative result was obtained on fusing a quantity of the original crude resin with potassium hydroxide.

#### SUMMARY.

The results of the present investigation of cotton-root bark may be summarised briefly as follows:—

On distilling an alcoholic extract of the bark in a current of steam a very small amount of a pale yellow essential oil was obtained. This product gave the colour reaction of furfuraldehyde, and on keeping it deposited a few crystals melting at 112-114°, which appeared to consist of acetovanillone.

The other constituents of the bark were found to comprise: (1) A phenolic acid (melting-point 196-199°), which gives an intense blue colour with ferric chloride, and is probably 2:3dihydroxybenzoic acid; (2) salicylic acid; (3) a new, colourless phenolic substance (melting-point 258-260°), to which the formula C<sub>9</sub>H<sub>10</sub>O<sub>3</sub> has been assigned, and which yielded an acetyl derivative melting at 152°. It dissolves in aqueous alkalis, forming bright yellow liquids, and with ferric chloride a purple colour is produced. (4) A new, yellow phenolic substance (melting-point 210-212°), which appears to possess the formula C<sub>13</sub>H<sub>18</sub>O<sub>5</sub>, and which yielded an acetyl derivative melting at 147-149°. With aqueous alkalis and with concentrated sulphuric acid it gives a deep purple colour, and with ferric chloride a brown coloration is produced. (5) Betaine, C<sub>5</sub>H<sub>11</sub>O<sub>2</sub>N; (6) a fatty alcohol, C<sub>20</sub>H<sub>42</sub>O, melting at 77.5°-78.5°; (7) a phytosterol, C<sub>27</sub>H<sub>46</sub>O (melting-point 130°); (8) a small amount of a hydrocarbon (melting-point 60-61°), which apparently is triacontane,  $C_{30}H_{62}$ ; (9) ceryl alcohol,  $C_{27}H_{56}O$ ; (10) a mixture of fatty acids, consisting chiefly of oleic and palmitic acids. The bark contains, furthermore, a considerable proportion of sugar, from which d-phenylglucosazone (melting-point 212-214°) was prepared, and by the acetylation of the sugar a small amount of penta-acetyl-dextrose was obtained.

No alkaloid is contained in the bark, and no evidence could be obtained of the presence of tannin. The resinous material, from which some of the above-mentioned substances were isolated, was of a deep purplish colour, and amounted to 10.6 per cent. of the weight of air-dried bark employed. PRINTED BY
ST. CLEMENTS PRESS, LTD.,
PORTUGAL STREET,
KINGSWAY, LONDON, W.C.

the first state and a sound of him simple or behavior