111. *Sumatrol. Part I.*

By Alexander Robertson and George L. Rusby.

In the course of an examination of a resin obtained from a species of *Derris* (probably *Derris malaccenis*, var. *Sarawakenis*; compare Henderson, *Malayan Agric. J.*, 1934, **22**, 125) rich in toxicarol, Cahn and Boam (*J. Soc. Chem. Ind.*, 1935, **54**, 42T) isolated in small amount (0·55% of crude resin) a new colourless crystalline compound which appeared to be dimorphic, m. p. 189° and m. p. 192—194°, and which they considered to have the empirical formula $C_{23}H_{22}O_7$, although their analytical results did not exclude the formula $C_{20}H_{20}O_6$. This substance, which, on the basis of the former formula, is isomeric with tephrosin and toxicarol, gave a strong ferric reaction, could not be dehydrated by means of a mixture of acetic acid and sulphuric acid, and gave a liquid product on treatment with acetylating agents. Preliminary tests indicated that, like other derris constituents, the new compound had insecticidal properties. Through the kindness of Dr. R. S. Cahn and of Messrs. Cooper, McDougall, and Robertson, Ltd., of London, in placing a supply of the resin at our disposal we have been able to make a detailed examination of the new compound, which has now been named *sumatrol* (private communication from Dr. Cahn).

Sumatrol was isolated from the resin according to the procedure employed by Cahn and Boam (*loc. cit.*), but the melting point phenomena observed by us are not quite identical with those previously reported (see experimental section). Until a detailed crystallographic examination of the forms, m. p. 189° and m. p. 194° (Cahn and Boam, *loc. cit.*, and present work), is available it is impossible to say whether the results obtained are due to solvents of crystallisation or to the existence of dimorphic forms, but it may be noted that similar properties are exhibited by a surprisingly large number of members of the rotenone group, where in some cases the existence of dimorphic forms has been clearly established (compare

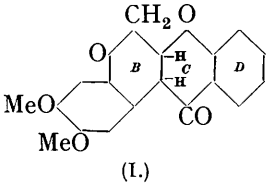
La Forge and Keenan, *J. Amer. Chem. Soc.*, 1931, **53**, 4450; Gooden and Smith, *ibid.*, 1935, **57**, 2616). The analytical results obtained for sumatrol, together with those of certain derivatives subsequently discussed, clearly show that the compound has the formula $C_{21}H_{16}O_5(OMe)_2$ and is thus isomeric with tephrosin and toxicarol as Cahn and Boam suggested (*loc. cit.*). We have also confirmed the observations of these workers that, unlike tephrosin, sumatrol cannot be dehydrated. The formation of an *oxime* establishes the presence of an active carbonyl group, and this fact, in conjunction with the strong ferric reaction, clearly indicates that there is a phenolic hydroxyl group in the *o*-position to the carbonyl group; in agreement with this conclusion it has been observed that, like toxicarol, sumatrol is almost insoluble in aqueous sodium hydroxide. Acetylation of sumatrol gave rise to an amorphous product, which has not yet been obtained crystalline and has a negative ferric reaction; this product may be the monoacetate, the diacetate derived from an enolic form, or, more probably, a mixture of both.

Whilst the oxidation of sumatrol with potassium ferricyanide did not appear to proceed smoothly, treatment of the compound with iodine and sodium acetate and elimination of iodine from the product by means of zinc dust and acetic acid according to the standard procedure gave rise to *dehydrosumatrol* which, like the parent compound, is optically active, is almost insoluble in dilute aqueous sodium hydroxide, and gives a strong ferric reaction. As in the case of sumatrol itself, the last two properties indicate that the dehydro-derivative contains a phenolic group in the *o*-position to a carbonyl group, and the presence of this hydroxyl group in the dehydro-compound and hence in sumatrol itself is clearly established by the formation of a crystalline O-*acetyldehydrosumatrol* having a negative ferric reaction.

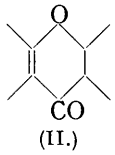
Hydrogenation of sumatrol was readily accomplished with the aid of a platinum catalyst and gave rise to approximately equal amounts of two optically active products, *dihydro-* and *tetrahydro-sumatrol*, which were readily separated by taking advantage of the fact that the latter derivative is easily soluble in 2% aqueous sodium hydroxide. Dehydrogenation of these compounds by the iodine method yielded *dehydrodihydro-* and *dehydrotetrahydro-sumatrol* respectively. Of these, only the former is optically active. Dehydrodihydro-sumatrol was also obtained by the hydrogenation of dehydrosumatrol, but in this case the alkali-soluble fraction of the product, which in all probability consisted largely of dehydrotetrahydrosumatrol, could not be purified.

On being boiled with aqueous alcoholic potassium hydroxide, dehydrosumatrol was converted by the addition of the elements of two molecules of water into a monobasic acid, *sumatrolic acid*. Similarly, dehydrodihydrosumatrol furnished a product which, though it has not yet been obtained pure, is undoubtedly dihydrosumatrolic acid.

The isomerism of sumatrol with tephrosin and toxicarol, its insecticidal activity, and its co-existence with rotenone and toxicarol (or a toxicarol precursor) in the same plant, which we believe in the circumstances argues a close biogenetic relationship, clearly appear to justify the assumption that sumatrol is closely related structurally to other members of the rotenone series. Although we have not yet been able to complete our studies on the degradation of the compound, nevertheless, starting with this hypothesis, we are able to allocate rational structural formulæ to sumatrol and its derivatives.



(I.)



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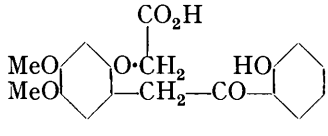


(III.)

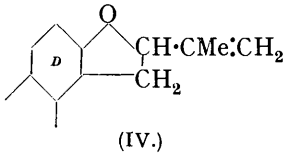
Apart from the phenolic hydroxyl group, sumatrol, like all other members of the rotenone series, possesses two methoxyl groups, an active carbonyl group, and three indifferent oxygen atoms. Further, sumatrol and dihydrosumatrol can be almost quantitatively dehydrogenated to give dehydro-derivatives which furnish acids of the derrisic acid type by standard procedures. It therefore appears practically certain that sumatrol possesses

the chromanochromanone residue (I) which gives the chromenochromone type (II) capable of forming the derrisic acid type (III) (J., **1933**, 489, **1163**; **1935**, 993).

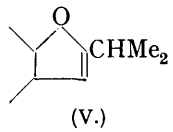
Since it has been clearly established that, like its isomeride toxicarol, sumatrol has a phenolic hydroxyl group in the *o*-position to the carbonyl group, this hydroxyl group must be at the 5-position of the chromanochromanone (I) (*i.e.*, ring *D*). Apart from the ferric reaction, the possibility that the hydroxyl group is at the 2- or 3-position is excluded, because, unlike tephrosin and the rotenolones-I and -II (Takei and co-workers, *Ber.*, **1933**, **66**, 479), sumatrol and its hydrogenation products cannot be dehydrated and give dehydro-products by oxidation only. Since in all members of the rotenone series ring *D* carries two ethereal oxygen atoms, consequently in sumatrol this ring is either a hydroxyquinol or a phloroglucinol residue. Comparison of the ferric reactions of sumatrol and toxicarol and of their respective derivatives, as set out below, indicates that in sumatrol ring *D* of (I) is in all probability a phloroglucinol residue (compare J., **1935**, 681).

	Sumatrol.	Toxicarol.
Parent compound	Brown, tinged green.	Deep green.
Dehydro-compound	Deep green.	,, ,,
Dihydro-compound	Brown.	Brown.
Tetrahydro-compound	Olive-green.	—
Dehydrodihydro-compound	Deep green.	Deep green.
Dehydrotetrahydro-compound	,, ,,	—

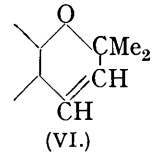
All the known members of the rotenone series may be considered to be built up from the chromanochromanone type (I) by addition of an isoprene residue forming a fifth ring system fused to ring *D* which is of one of the following types—(IV), present in rotenone, (V) in *iso*rotenone, and (VI) in deguelin, tephrosin, and toxicarol.



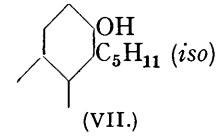
(IV.)



(V.)



(VI.)



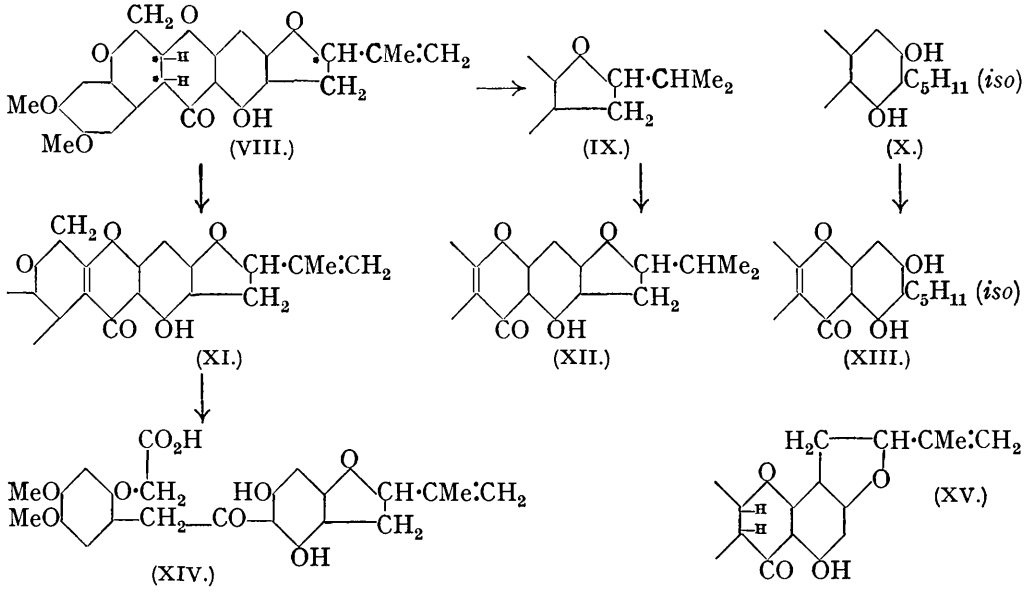
(VII.)

On hydrogenation, types (V) and (VI) behave normally, giving saturated dihydro-derivatives, but the *iso*propenyl type (IV) gives in addition to an *iso*propyldihydrofuran an alkali-soluble tetrahydro-product, *e.g.*, tetrahydrorotenone (or dihydrorotenonic acid of older nomenclature) type (VII). The behaviour of sumatrol in forming an alkali-insoluble saturated dihydro- and an alkali-soluble tetrahydro-derivative on hydrogenation is identical with that of rotenone and therefore we consider that the sumatrol molecule contains the system (IV). Consequently it would appear that sumatrol is a 5-hydroxyrotenone and may be represented by formula type (VIII) and hence dihydro-, tetrahydro-, dehydro-, dehydrodihydro- and dehydrotetrahydro-sumatrol have formula types (IX), (X), (XI), (XII) and (XIII) respectively.

The structure type (VIII) suggested for sumatrol possesses three asymmetric carbon atoms (marked*). In the formation of dihydrosumatrol (IX) none of these centres is affected, but in the conversion of both sumatrol and dihydrosumatrol into the respective dehydro-derivatives (XI) and (XII) the two centres at C_2 and C_3 in the chromanochromanone residue are destroyed and, since these compounds are both optically active, the asymmetric C-atom in the dihydrofuran system is a centre of optical activity. In the formation of tetrahydrosumatrol, which is optically active, the latter centre of activity is destroyed and therefore the C-atoms at positions 2 and 3 of the chromanochromanone residue are also centres of optical activity, a conclusion in agreement with the fact that the optically active centres C_2 and C_3 are destroyed in the dehydrogenation of tetrahydrosumatrol, thus giving an optically inactive dehydro-derivative.

The foregoing optical rotation phenomena observed for sumatrol and its hydrogenation and dehydrogenation products are identical with those exhibited by rotenone and its corresponding derivatives, thus affording strong evidence in support of the structural formulæ proposed for sumatrol and its degradation products. Further, these structures are also supported by the fact that the dehydro-compounds (XI) and (XII), like the members

of the dehydrorotenone series, are converted into acids of the derrisic acid type, of which that obtained from (XI) may be represented by formula type (XIV).



The formulæ (VIII), (IX), (XI) and (XII) proposed for sumatrol and its derivatives are of the linear type with respect to rings *C*, *D*, and *E*, but, as is obvious, the angular type (XV) present in rotenone may well obtain. The facts at our disposal do not permit us to make a choice between these two types, but the existence of a number of linear furanocoumarins (bergapten type) having a phloroglucinol nucleus (*iso*bergapten appears to be the only angular type known) might possibly be considered to favour the same structure for sumatrol.

By analogy with rotenone it should be possible according to formulæ (VIII) and (XI) to isomerise sumatrol and dehydrosumatrol by conversion of the *iso*propenyldihydrofuran system into an *iso*propylfuran residue, *i.e.*, conversion of (IV) into (V), and, although all attempts to effect this change have been unsuccessful (see experimental section), it should be emphasised that in no case was the starting material recovered unchanged (compare Cahn and Boam, *loc. cit.*).

Experimental.

Sumatrol.—The compound was isolated from the resin by the method described by Cahn and Boam (*loc. cit.*), but the yield (40 g.) of crude material separating from the carbon tetrachloride solution of the resin (500 g.) was increased somewhat by keeping this solution for 4 weeks. Repeated extraction of the crude product with boiling alcohol (first portion of 150 c.c.; succeeding portions, 50 c.c.) gave a series of twelve to thirteen fractions, having m. p. from 168—172° to 194—195°, of which the final fraction was almost pure sumatrol. Repeated crystallisation from alcohol (less wasteful than acetone) finally gave the compound in slender needles which, on being collected immediately and pressed on porous tile, had m. p. 192—194°. After having been kept in air for 2—3 days (on one occasion for only 6 hours), the dried material had m. p. 195—196°, $[\alpha]_D$ — 184° (*c*, 1·335 in benzene) [Found : C, 67·2, 67·2; H, 5·4, 5·4; OMe, 16·5; *M*, 396, 383. Calc. for $C_{21}H_{16}O_5(OMe)_2$: C, 67·3; H, 5·4; OMe, 15·1%; *M*, 410]. In view of the melting point phenomena of sumatrol observed by Cahn and Boam the following observations are recorded : Recrystallisation of the material, m. p. 195—196°, from acetone gave colourless needles, m. p. 194°, unaltered after the compound had been kept for several days. On one occasion pure sumatrol was twice recrystallised from acetone and each time had a constant m. p. of 194°, but on being recrystallised for a third time the compound had m. p. 183°, which after 3 days changed to 192—194° and then remained constant. A small specimen of sumatrol kindly presented to us by Dr. Cahn and labelled m. p. 189° was found to have m. p. 187—188° and after being recrystallised from alcohol and dried for 3 days had m. p. 194—195°.

Sumatrol is sparingly soluble in methyl alcohol, cold acetic acid, and in 8% aqueous sodium hydroxide, moderately soluble in ethyl acetate and benzene, and readily soluble in chloroform. With alcoholic ferric chloride it gives a deep brown coloration tinged with green, which is unaltered on dilution with water. A mixture of sumatrol (0·5 g.), hydroxylamine hydrochloride (0·5 g.), and dry pyridine (5 c.c.) was heated on the steam-bath for 20 hours, cooled, and poured on ice. After being kept for 1 hour, the resulting *oxime* was collected, washed, dried, and crystallised from alcohol and then from 50% alcohol, forming colourless slender needles, m. p. 245—247° (Found : C, 65·1, 65·1; H, 5·6, 5·6; N, 3·5. $C_{23}H_{23}O_7N$ requires C, 64·9; H, 5·5; N, 3·3%). When the reaction mixture was heated for shorter periods, oximation appeared to be incomplete and a mixed product resulted.

The following attempts to isomerise sumatrol proved unsuccessful : Sumatrol (0·5 g.), suspended in acetic acid (3·5 c.c.), was treated with concentrated sulphuric acid (1·5 c.c.), and the mixture kept at 60—65° for 12 minutes, gradually heated to 95° in the course of 10 minutes, and then maintained at this temperature for 8 minutes. The product obtained by addition of much ice to the reaction mixture separated from hot alcohol in tiny yellow spherical masses, which gave a deep green ferric reaction. Addition of methyl alcohol (3 vols.) to a warm solution of this product in hot chloroform (1 vol.) caused the separation of a similar solid, but all attempts to obtain crystalline material were unsuccessful. The products melted over a range which varied, according to the solvent used, from 190° to 210°. Amorphous material was also obtained when sumatrol (0·25 g.) was dissolved in concentrated sulphuric acid (4 c.c.) in the course of 4 minutes, and the product precipitated with ice-water. In another experiment a mixture of acetic acid (3 c.c.) and sulphuric acid (1 c.c.) containing sumatrol (0·5 g.) was gently refluxed for 2 minutes, cooled, and poured on ice ; only a small amount of a dark amorphous solid separated from a cooled alcoholic solution of the product.

When a solution of sumatrol (0·5 g.) in alcohol (80 c.c.) containing concentrated sulphuric acid (8 g.) was refluxed for 4 hours, the crystalline material (0·4 g.) which separated from the cooled solution consisted of unchanged sumatrol, m. p. and mixed m. p. 195—196°.

Dehydrosumatrol.—Iodine (1·2 g.), dissolved in a little alcohol, was added in the course of 5—10 minutes to a solution of sumatrol (1 g.) in boiling alcohol (140 c.c.), containing sodium acetate (3 g.), and the mixture then boiled for 1¾ hours ; after the addition of the greater part of the iodine (about 1 g.) the solution retained a permanent brown colour. Next day the resulting crystalline iodo-derivative (0·45 g.) was collected, washed, and dried ; a further quantity (0·9 g.) of crude iodo-compound was obtained when the alcoholic filtrate was reduced to a volume of 20 c.c. and the residue treated with water (200 c.c.). The crystalline product gave a deep green ferric reaction and on being heated decomposed at about 200°.

A mixture of the iodo-derivative (0·45 g.), acetic acid (10 c.c.), and zinc dust (0·8 g.) was refluxed for 2 hours ; after 1½ hours more zinc (0·4 g.) was added. The hot solution was filtered, and the zinc washed with boiling acetic acid (5 c.c.). On cooling, the combined solutions deposited the *dehydro*-compound (0·3 g.), which separated from chloroform–methyl alcohol in tiny, pale yellow prisms, $[\alpha]_D$ — 55° (*c*, 2·500 in chloroform), m. p. 190—192°, containing an indefinite amount of solvent of crystallisation [Found in material dried at 80° in a high vacuum : C, 67·4, 67·4, 67·6; H, 4·9, 4·9, 5·0; OMe, 15·1. $C_{21}H_{14}O_5(OMe)_2$ requires C, 67·6; H, 4·9; OMe, 15·2%]. This compound is readily soluble in chloroform, sparingly soluble in hot methyl alcohol, and slightly soluble in warm benzene, acetone, or ethyl acetate. With alcoholic ferric chloride it gives a deep green coloration.

Acetylation of dehydrosumatrol (0·35 g.) with acetic anhydride (5 c.c.) and pyridine (2·5 c.c.) at 100° for 1 hour and then at room temperature for 18 hours gave rise to the *acetate* (0·38 g.), which separated from chloroform–methyl alcohol in clusters of pale yellow needles, m. p. 256—259° after darkening at 250°, having a negative ferric reaction [Found : C, 66·3; H, 5·0; CH_3·CO, 11·7. $C_{23}H_{19}O_7(CH_3{\cdot}CO)$ requires C, 66·7; H, 4·9; CH_3·CO, 9·6%].

Attempts to isomerise dehydrosumatrol (0·3 g.) with warm acetic acid (5 c.c.) and sulphuric acid (1·7 c.c.) gave rise to a product which separated as an amorphous solid, m. p. 170—178°, from chloroform–methyl alcohol and gave a deep green ferric reaction.

Hydrogenation of Sumatrol.—Absorption of hydrogen (approx. 1·5 mols.) by sumatrol (1 g.), dissolved in ethyl acetate (200 c.c.) containing a platinum catalyst (0·15 g., prepared according to directions of Adams, Voorhees and Shriner, " Org. Synth.," Coll. Vol. I, 1932, p. 452), ceased in about 45 minutes. After the removal of the catalyst, the volume of the solution was reduced to 75 c.c., an equal volume of ether added, and the mixture extracted several times with 2% aqueous sodium hydroxide. Acidification of the combined extracts with dilute hydrochloric acid gave *tetrahydrosumatrol* (0·5 g.), which separated from chloroform–methyl alcohol in colourless, stout,

well-formed prisms, m. p. 222—223°, containing solvent of crystallisation, $[\alpha]_D$ + 122° in chloroform (*c*, 1·0827) [Found in material dried in a high vacuum at 100° : C, 66·7, 66·7; H, 6·3, 6·3; OMe, 14·9, 14·6. $C_{21}H_{20}O_5(OMe)_2$ requires C, 66·6; H, 6·3; OMe, 15·0%]. This compound is more soluble in alcohol than the parent compound. Addition of one drop of ferric chloride to an alcoholic solution of tetrahydrosumatrol gives an almost black coloration, which becomes violet-brown on addition of water. With more ferric chloride the black solution becomes olive-green.

The ether–ethyl acetate solution left on removal of the tetrahydro-derivative was washed with water, dried, and evaporated. Crystallisation of the residue (0·45 g.) from chloroform–methyl alcohol gave *dihydrosumatrol* in colourless slender rods, m. p. 184—185°, $[\alpha]_D$ − 32° (*c*, 0·2667 in chloroform) [Found : C, 66·8, 66·8; H, 6·0, 6·0; OMe, 15·6. $C_{21}H_{18}O_5(OMe)_2$ requires C, 67·0; H, 5·9; OMe, 15·1%], which gave a deep brown coloration with alcoholic ferric chloride, unchanged on addition of water.

Dehydrodihydrosumatrol.—Iodine (0·7 g.) was added to a solution of dihydrosumatrol (0·7 g.) in boiling alcohol (70 c.c.) containing sodium acetate (1·8 g.) in the course of 5 minutes, and the mixture refluxed for 1¾ hours. 20 Minutes later, pale yellow crystals of the iodo-compound began to separate and next day this product (0·26 g.), m. p. 191°, was collected; a further quantity of crude material (0·7 g.) was obtained by concentration of the alcoholic liquors and the subsequent addition of water. Acetic acid (13 c.c.), containing the iodo-compound (0·4 g.) and zinc dust (0·8 g.), was refluxed for 1¼ hours; after ¾ hour, more zinc (0·4 g.) was added. After the removal of the zinc (wash with 5 c.c. of boiling acetic acid) the cooled solution deposited *dehydrodihydrosumatrol* (0·25 g.), which crystallised from chloroform–methyl alcohol and then chloroform–acetone as a matte of slender yellow needles, m. p. 235°, $[\alpha]_D$ −· 63° (*c*, 0·2700 in chloroform), sparingly soluble in hot alcohol or acetone, readily soluble in hot benzene, and giving a deep green coloration with alcoholic ferric chloride (Found : C, 67·5, 67·4; H, 5·6, 5·5. $C_{23}H_{22}O_7$ requires C, 67·3; H, 5·4%). The yield of the dehydro-compound was approx. 70% of the theoretical.

Dehydrotetrahydrosumatrol.—A solution of iodine (2·3 g.) in alcohol (25 c.c.) was gradually added to a boiling solution of tetrahydrosumatrol (2·2 g.) in the same solvent (150 c.c.), containing sodium acetate (6·5 g.), the mixture refluxed for 1¾ hours, the greater part of the alcohol distilled, and the residual liquid (25 c.c.) mixed with water (200 c.c.). 3 Hours later, the solid (2·4 g.) was collected, washed, dried, and heated with boiling acetic acid (30 c.c.) and zinc dust (5 g., added in two portions of 4 g. and 1 g.) for 1½ hours. After removal of the zinc dust by filtration (wash with 5 c.c. of hot acetic acid) the combined solution and washings were diluted with water (200 c.c.). Repeated crystallisation of the product (2 g.) from alcohol (charcoal) gave *dehydrotetrahydrosumatrol* in pale yellow plates (1·1 g.), m. p. 218°, giving a deep green coloration with alcoholic ferric chloride and showing a zero rotation (Found in material dried in a high vacuum at 100° : C, 67·0, 67·2; H, 5·9, 6·0. $C_{23}H_{24}O_7$ requires C, 67·0; H, 5·9%).

Acetylation of this compound (0·14 g.) with acetic anhydride (2 c.c.) and pyridine (1 c.c.) at 100° for 1 hour and then at room temperature for 48 hours gave the *diacetate*, which separated from aqueous acetone in tiny, pale straw-coloured needles, m. p. 197°, having a negative ferric reaction and insoluble in 8% aqueous sodium hydroxide (Found : C, 65·5; H, 5·7. $C_{27}H_{28}O_9$ requires C, 65·3; H, 5·7%).

Sumatrolic Acid.—A mixture of dehydrosumatrol (0·7 g.), potassium hydroxide (3·5 g.), zinc dust (0·7 g.), alcohol (65 c.c.), and water (5 c.c.) was refluxed for 6 hours, filtered, and diluted with water (65 c.c.), the greater part of the alcohol was removed by distillation, and the cooled residue was acidified with hydrochloric acid. 24 Hours later, the semi-solid was collected, washed, and dissolved in aqueous sodium bicarbonate. After having been filtered to remove traces of insoluble material, the solution was acidified with hydrochloric acid, the pale pink solid (0·5 g.) thus precipitated was dried and dissolved in a little hot acetone, and the solution was treated with charcoal, filtered, diluted with benzene (3 vols.), and then evaporated until crystalline material began to separate. Purification of the product which separated from the cooled liquor was repeated several times from hot benzene containing a little chloroform, finally giving *sumatrolic acid* in spherical aggregates of rather ill-defined colourless crystals, m. p. 150°, readily soluble in alcohol, chloroform, acetone, or ethyl acetate and sparingly soluble in hot benzene or water (Found : C, 62·2; H, 5·9. $C_{23}H_{24}O_9$ requires C, 62·1; H, 5·5%). With alcoholic ferric chloride this acid gives a violet coloration.

Hydrolysis of dehydrodihydrosumatrol (0·3 g.) with boiling aqueous-alcoholic potassium hydroxide (1·6 g. of hydroxide in 5 c.c. of water and 30 c.c. of alcohol) in the presence of zinc dust (0·4 g.) for 3½ hours and purification of the crude product with aqueous sodium bicarbonate gave rise to a solid which separated from benzene in short rods, m. p. 130—132°; this un-

doubtedly consisted of almost pure dihydrosumatrolic acid, but was not obtained analytically pure owing to lack of material.

Hydrogenation of Dehydrosumatrol.—Dehydrosumatrol (1 g.), dissolved in ethyl acetate (400 c.c.), was treated with a platinum oxide catalyst (0·15 g.) and hydrogen; absorption (approx. 1·5 mols.) was complete in about 20 minutes. After the removal of the catalyst the solvent was evaporated, leaving a bright yellow solid, which, by means of 2% aqueous sodium hydroxide, was resolved into an alkali-soluble and an alkali-insoluble fraction. After being extracted three times with boiling acetone (5 c.c.) to remove traces of impurities, the latter consisted of dehydrodihydrosumatrol, m. p. 235°, unaltered by recrystallisation from chloroform–acetone, and was identified by comparison with an authentic specimen, m. p. and mixed m. p. 235°. The alkali-soluble fraction separated readily from warm alcohol in pale yellow crystals, which, however, could not be obtained homogeneous but appeared to consist mainly of dehydrotetrahydrosumatrol.

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