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HASHISH—XIII[1]

ON THE NATURE OF THE BEAM TEST

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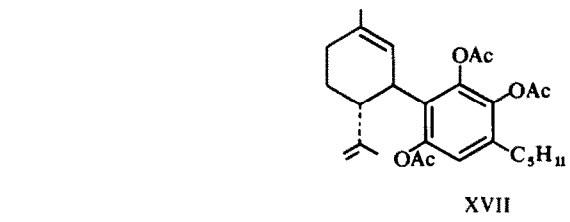
Abstract—The purple colour produced by treatment of cannabidiol (I) with 5% ethanolic potassium hydroxide (Beam test) is due to the anions of the hydroxy-quinone II and its dimer III. Compounds II and III are formed from I by air oxidation during the reaction. The diquinone III is reduced in the mass spectrometer to a $M^+ + 4$ species (probably the dihydroquinone).

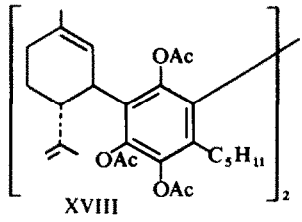
MORE than half a century ago Beam reported[2] that hashish extracts gave a deep purple colour with a 5% ethanolic potassium hydroxide solution. This observation is the basis of the most commonly used colour test for the identification of hashish (marihuana). In view of the considerable importance of cannabis identification for legal purposes this test has been the object of numerous studies as regards its specificity, reliability and sensitivity.[3] From the extensive investigations by Matchett[4] of the effect of variety, maturity, sex and region of growth of hemp on response to the Beam test and from the chemical results of Adams[5] it was concluded that this test is not indicative of a substance with marihuana activity, but is due to cannabidiol (I), a physiologically inactive constituent. We know today[6] that at least two other natural cannabinoids,* namely cannabidiolic acid (IV) and cannabigerol (V), give a positive Beam test, while Δ^1-tetrahydrocannabinol (VI) the major active constituent, $\Delta^{1(6)}$-tetrahydrocannabinol (VII), cannabinol (VIII), cannabichromene (IX) and cannabicyclol (X) give a negative Beam test. Apparently both phenolic groups have to be free for a positive test. As cannabidiolic acid (IV) is decarboxylated under the basic reaction conditions to give cannabidiol (I) and as cannabigerol (V) is a very minor component in hashish it seems that cannabidiol is indeed mainly responsible for the intensity of the Beam test.

For such a simple colour reaction the Beam test is relatively specific.[3,7] Out of 120 plant species (belonging to 28 botanical families) two (*Salvia officinalis* and *Rosmarinus officinalis*) are reported to give a weakly positive reaction, while out of 48 pure substances of vegetable origin (monoterpenes, sesquiterpenes and aromatic compounds) only one, juglone (XI) develops a colour (purple-brown) close to that of the Beam test (strong purple). However, juglone was the only quinone examined.

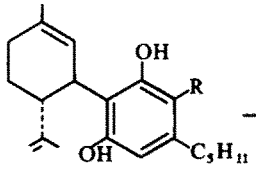
* The term *cannabinoids* has been proposed[6] for the group of C_{21}-compounds typical of, and present in *Cannabis sativa L.*, as well as for their analogs and transformation products.



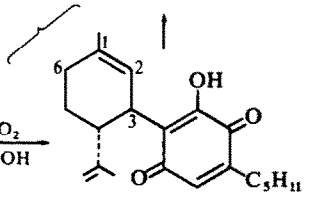
XVII



XVIII



I: R = H
IV: R = COOH



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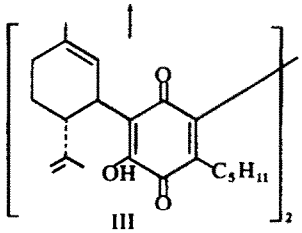

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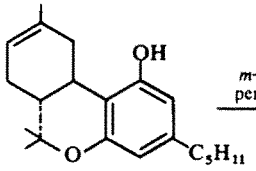

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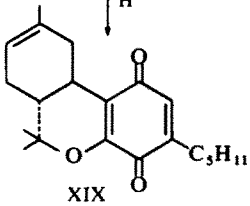

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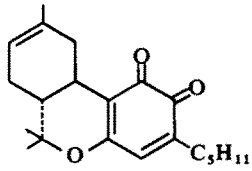

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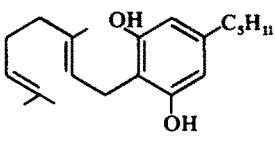

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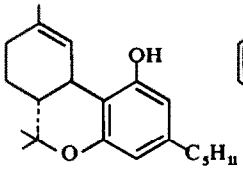

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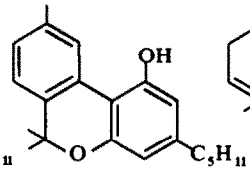

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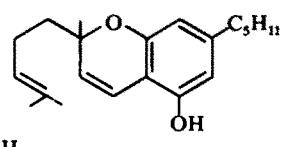

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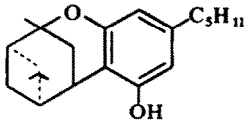
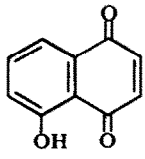
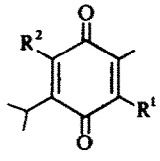


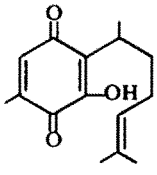
X(?)



XI



XII a: R^1 = OH, R^2 = H
b: R^1 = H, R^2 = OH



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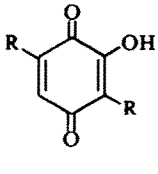
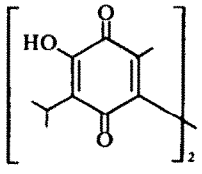
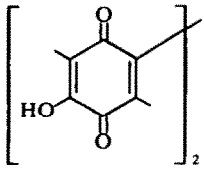


XIV a: R = CH_3
b: R = Cyclohexyl



XV



XVI

We report now our observations on the transformations of cannabidiol (I) under the conditions of the Beam test.* When a solution of I in petroleum ether is mixed with 5% alcoholic potassium hydroxide the typical violet colour appears within a few minutes. When the reaction is performed under nitrogen the starting material can be recovered unchanged on acidification. If, however, the reaction mixture is stirred in an open vessel or if air is bubbled through it, the colour deepens and reaches maximum intensity within 10–15 hr. The colour does not fade for at least 24 hr. On acidification the solution is discoloured. By extraction of the acidified reaction mixture, followed by chromatography, two compounds could be isolated: II, in a variable yield of 5–25%, and III in 50–80% yield. Some of the spectral properties of II and III are given in Table 1. The strong quinonic bands (at 1645 and 1648 cm^{-1}) in the IR of II and III are identical with the corresponding band in monohydroxydialkylquinones such as 3-hydroxythymoquinone (XIIa), perezone (XIII) and 6-hydroxythymoquinone (XIIb).[9] The 890 cm^{-1} band indicates that the terminal methylene group has remained intact. The UV spectrum of II is compatible with those of XIIa, XIIb, 2-hydroxyxyloquinone (XIVa) and 2-hydroxy-3,6-dicyclohexylbenzoquinone (XIVb).[10] The slight differences observed (3–10 mμ) are probably due to solvent effects which are quite pronounced in this series.[11] The UV spectrum of III is comparable to that of di-(6-hydroxythymoquinone) (XV).[11] The NMR spectra of II and III fit the assigned structures. They are very similar, except for the quinonic ring proton in II which is absent in III. This proton appears as a triplet (J = 1·0 c/s) due to splitting by the benzylic protons of the side chain. Such a coupling has been observed in perezone (XIII) and related quinones.[9]

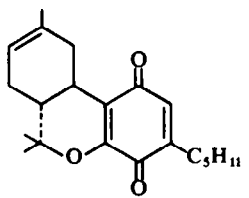
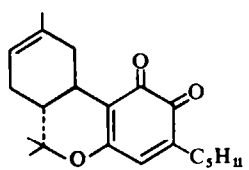
The molecular weights of II and III as determined by the mass spectra are in accord with proposed structures. In III there is an M + 2 peak, whose intensity is slightly stronger than that calculated from the natural abundance and an intense M + 4 peak (160% of the M^+ peak). The formation of hydroquinones from quinones in the mass spectrometer is a frequently observed phenomenon.[12] While in 1,2 quinones the M + 2 peak may be the base peak,[12e] in 1,4 quinones the intensity of the M + 2 peak rarely exceeds one-tenth of that of the M^+ peak.[12c] It seems that the present report is the first one of a reduction of a diquinone in the mass spectrometer to what is probably a dihydroquinone and of a 1,4 quinone showing peaks of a higher intensity than the M^+ peak. The preferential formation of a $M^+ + 4$ species rather than a $M^+ + 2$ species apparently indicates that the intermediate hydroquinone–quinone (or a related intermediate) is reduced faster than the diquinone. On mechanistic grounds this is unexpected as, intuitively, the diquinone should have a higher redox potential than the hydroquinone–quinone.

Folkers[12d] and Lederer[12b] have shown that in polyisoprenoid quinones there is a tendency towards the formation of a pyrilium ion, by ring closure of a double bond with one of the quinonic ketones. We assume that the intense *m/e* 313 ($M^+ - 15$) species in the spectrum of II is similarly formed. The *m/e* 245 ion may be formed by a retro Diels–Alder reaction followed by cyclization.[13]

* We are unaware of any publication dealing with the *chemistry* of the Beam test, though in view of the enormous literature on hashish it is conceivable that we could have missed a chance observation or remark. A recent compilation of references on *Cannabis*[8] lists 1860 articles, most of which we have yet to locate.

TABLE 1

Compound	IR spectrum[a]	UV and visible spectra[b]	NMR spectrum[c]	Mass spectrum[d]
monomeric hydroxy-quinone (II)	1645 (quinone) 890 ($\rangle C{=}CH_2$)	($CHCl_3$) 271 (4·11) 362 (sh) (3·00) 408 (3·03)	0·92 (aliphatic CH_3); 1·60 (olefinic CH_3); 3·68 (1) (d, br) (C_3—H); 4·50 (2) (s) ($\rangle C{=}CH_2$); 5·10 (1) (s, br) (C_2 –H); 6·36 (1) (quinone ring H) (tr, J = 1 c/s); 7·10 (1) (s) (O<u>H</u>) (in CCl_4)	328 (M^+), 313 ($M^+ - 15$) 311 ($M^+ - 17$) 260, 245, 237, 220, 204
dimeric hydroxy-quinone (III)	1648 890 ($\rangle C{=}CH_2$)	($CHCl_3$) 276 (4·42) 367 (sh) (3·37) 411 (3·11)	0·88 (aliphatic CH_3); 1·60 (olefinic CH_3); 3·70 (1) (d, br) (C_3—H); 4·52 (2) (s) ($\rangle C{=}CH_2$); 5·18 (1) (s, br) (C_2—H); 7·10 (1) (s) (O<u>H</u>) (in CCl_4)	658 ($M^+ + 4$) (160%) 654 (M^+) (100%) 642, 626, 600, 584, 675, 507
diacetate of III	1768 (acetate) 1652 (quinone) 900 ($\rangle C{=}CH_2$)	($CHCl_3$) 269 (4·36) 455 (sh) (2·15)	2·25 ($O{\cdot}COCH_3$); 3·75 (2) (br) (C_3—H); 4·62 (2) (s) ($\rangle C{=}CH_2$); 5·16 (1), (s, br) (C_2—H) (in $CDCl_3$)	738 (M^+) 740, 742
acetoxy, cannabidiol diacetate (XVII) $[\alpha]_D^{EtOH} - 76°$ (C = 8·6 g/l)	1780 (acetate) 895 ($\rangle C{=}CH_2$)	(EtOH) 275 (3·03)	2·06, 2·10, 2·16 (acetoxy CH_3); 3·40 (1) (br) (C_3—H); 4·50 (2) (br) ($\rangle C{=}CH_2$); 5·16 (1) (s, br) (C_2—H); 6·72 (1) (s) (aromatic-H) (in CCl_4)	456 (M^+) 441, 413, 388, 373

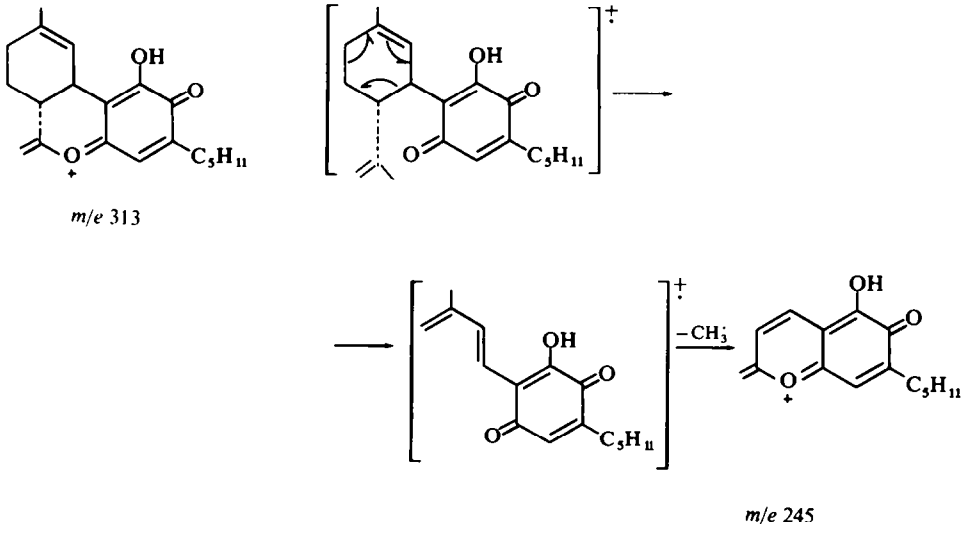
hexacetate XVIII $[\alpha]_D^{EtOH}$ −131° (C = 5·2 g/l)	1775 (acetate) 892 (>C=CH$_2$)	(EtOH) 278 (sh) (3·61) 285 (3·62)	2·08, 2·20 (acetoxy groups, the second with double intensity); 3·30 (1) (br) (C_3—H); 4·52 (2) (br) (>C=CH$_2$); 5·26 (1) (d, br) (C_2—H) (in CCl_4)	910 (M^+) 868, 826, 784, 742
 XIX	1660, 1650 (quinone) 1600	(EtOH) 269 (4·12) 362 (sh) (2·84) 400 (2·9)	0·92 (aliphatic CH_3); 1·14, 1·42 ($CH_3\alpha$ to 0); 1·68 (olefinic CH_3); 3·02 (1) (br) (C_3—H); 5·32 (1) (br) (C_6—H); 6·22 (1) (tr, J = 1·2 c/s) (quinonic ring H)	328 (M^+) 313 (M^+ − 15) 311, 309, 285, 272, 260 257, 245, 243
 XX	1645, 1580	(EtOH) 279 (3·72) 450 (3·14)	0·90 (aliphatic CH_3); 1·20, 1·42 ($CH_3\alpha$ to 0); 1·70 (olefinic CH_3); 3·10 (1) (br) 5·30 (1) (br) (C_6—H); 6·40 (1) (quinonic ring H)	328 (M^+) 313, 300, 257 258

[a] In CCl_4, values given in cm^{-1}.

[b] Solvent as indicated. Values given in mμ. Figure in parenthesis is log ε. (sh) indicates "shoulder".

[c] Values given in ppm relative to tetramethylsilane as internal standard (frequency zero). Number in parentheses denote number of protons, determined by integration of areas. Letters in parentheses denote singlet (s); doublet (d); triplet (t); quartet (q); broad (br) and multiplet (m).

[d] Direct inlet system (TO-4 ion source). The ionizing current was kept at 30 μA and the ionizing energy at 70 eV.



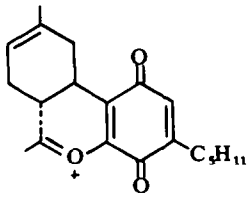
m/e 313

m/e 245

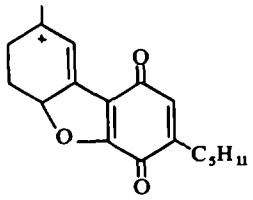
As mentioned above the intensity of the purple colour from cannabidiol (I) increases for a few hours reaching a plateau. The exact spectrum at this point (ca. 520–535 mμ) depends on the proportion of II and III formed, which varies considerably. Pure II or III, under the basic conditions of the test give *at once* the typical purple colour (II: 525 mμ, log ε, 3·28; III: 538 mμ, log ε, 3·42, both in ethanol). These data parallel those reported for XIVa and XVI in alkaline solution.[14,15]

On acetylation the dimer III gives a diacetate. On reductive actylation with zink and acetic anhydride II gives the triacetate XVII, while III is reduced to the hexa-acetate XVIII. The spectra of these compounds (see Table) fit the assigned structures. Thus the mass spectrum of XVII possesses most of the prominent peaks of cannabidiol (I),[13] increased by 142 mass units (two acetyl and one acetoxy group). In XVIII the corresponding cannabidiol peaks (calculated for a dimeric molecule) also appear but the main ones are those formed by the successive loss of 42 mass units (ketene) of the phenolic acetates. In the diacetate of III there is a M + 4 peak (30% of M^+), as well as a M + 2 peak which is stronger than that calculated from the natural abundance.

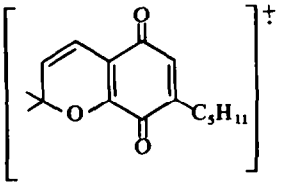
We have been unable to obtain any *o*-quinones through Beam oxidations. An entry into this series, albeit in very poor yield, can be achieved through oxidation of VII by *m*-chloroperbenzoic acid. In this reaction two compounds were isolated: the *p*-quinone XIX (in 10% yield) and the *o*-quinone XX (in 3% yield). The *p*-quinone XIX was also obtained by acid catalyzed cyclization of II. In this reaction the Δ^1 double bond migrated to the $\Delta^{1(6)}$ position. This isomerization is well known in the cannabinoids.[6] The mass spectrum of XIX resembles that of $\Delta^{1(6)}$-tetrahydrocannabinol (VII). Most of the prominent peaks of the latter[15] appear in XIX (adjusted by 14 mass units). The following species can be rationalized:



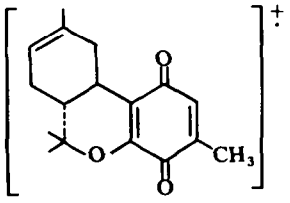
m/e 313



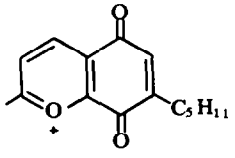
m/e 285



m/e 260



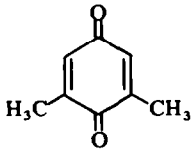
m/e 272



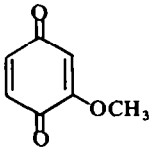
m/e 245

The *m/e* 245 peak in XIX, however, is amongst the smaller ones (ca. 5% of M^+ which is also the base peak), while the corresponding one in VII is the base peak.[13]

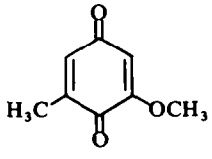
ortho and *para* Quinones can be differentiated by their UV spectra.[16] A pronounced bathochromic shift is generally observed in the *ortho*-series. As an example, three pairs of quinones[18] are described below (the log ε is given in brackets).



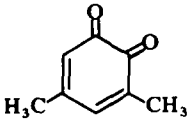
319 mμ (2·54)
in $CHCl_3$



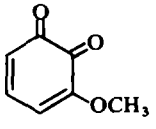
343 (3·04)
in hexane



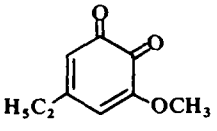
355 (2·97)
in CCl_4



410 mμ (3·20)
in $CHCl_3$



370 (3·31)
in C_2H_5OH

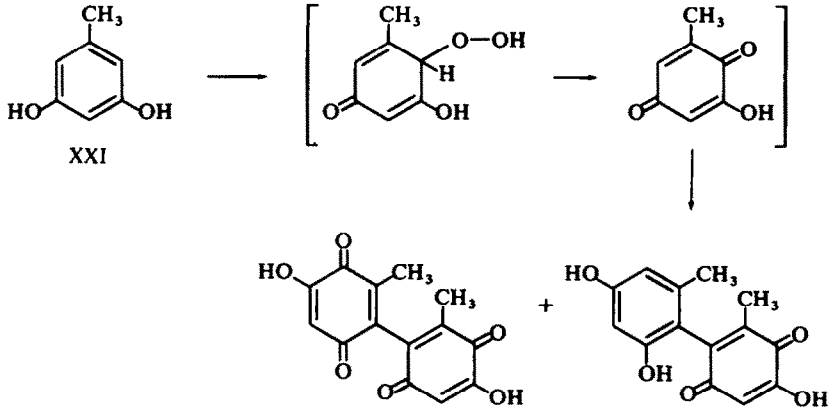


470 (3·19)
in $CHCl_3$

In our case there is a red shift of 50 mμ in XX as compared to XIX. The rest of the new quinones described in this paper have UV spectra which are very similar to those of related known *para*-quinones (*vide supra*). This represents, therefore, a proof of their *para* structure, which is according to expectation as, in general, on oxidation *p*-quinones are formed in preference to *o*-quinones.

Cannabidiolic acid (IV) reacts similarly to cannabidiol (I) in the Beam test, though at a somewhat lower rate. When a petroleum ether solution of IV was mixed with ethanolic potassium hydroxide for 20 hr and worked up, as described for I, only II and III could be isolated.

The oxidation of resorcinol derivatives has been studied mainly in connection with the formation of orceine and litmus dyes. In recent years, Musso has investigated numerous compounds of this series.[17] He has shown that orcinol (XXI) in basic medium is autoxydized through the peroxy derivative to a mixture of the dimeric hydroquinone–quinone and diquinone. With 5-t-butyl resorcinol,[18] which is sterically hindered, the monomeric hydroxyquinone was also isolated, but in this case it was accompanied by numerous side products.



XXI

EXPERIMENTAL

IR spectra were recorded on a Perkin–Elmer Infracord 137-B(I), UV spectra were measured on a Ultrascan, Hilger and Watts spectrophotometer, NMR spectra were determined on a Varian A-60 and mass spectra were measured on a Atlas CH4. TLC plates made of Kieselgel G, Merck, Darmstadt, were eluted with pet. ether (b.p. 40–60)–ether (80:20) and sprayed with a $KMnO_4$ soln. Column chromatographs were done on Florisil (compound-absorbent ratio 1:50). Microanalyses were performed by the microanalytical departments of the Hebrew University and the Weizmann Institute.

Oxidation of cannabidiol (I) *to the hydroxyquinones* II *and* III. Cannabidiol (1 g) dissolved in pet. ether (b.p. 40–60°; 90 ml) was stirred for 24 hr in an open beaker with 5% KOHaq in EtOH (10 ml). The reaction mixture was cooled to 0° and 5% HClaq (20 ml) was poured into it. The purple colour of the soln disappeared at once. The soln was extracted thrice with ether (50 ml each time). The organic layer was washed with $NaHCO_3$aq and with water, and then dried ($MgSO_4$). Removal of the solvent under reduced press yielded a glassy oil (1·1 g) which showed two spots on TLC. On column chromatography the less polar monomeric hydroxyquinone (II; 115 mg) was eluted with pet. ether (b.p. 40–60°)–ether (95:5) while the dimeric III (800 mg) was eluted with pet. ether–ether (90:10). Both II and III are labile and deteriorate within a few days giving very polar materials (polymers?). For analysis II and III were purified by preparative TLC. (Compound II. Found: C, 76·58; H, 9·02. $C_{21}H_{28}O_3$ requires: C, 76·79; H, 8·59; Compound III. Found: C, 77·50; H, 8·82. $C_{42}H_{54}O_6$ requires: C, 77·03; H, 8·31%). *Bis-3,5-dinitrobenzoate*, m.p. 119–120° (ether–pentane) (Found: C, 64·43; H, 5·48; N, 5·32. $C_{56}H_{58}N_4O_{16}$ requires: C, 64·48; H, 5·60; N, 5·37%). The *diacetate*, uncrystallizable, was purified by preparative TLC. (Found: C, 74·32; H, 8·38. $C_{46}H_{58}O_8$ requires: C, 74·76; H, 7·91%).

Oxidation of cannabidiolic acid (IV) *to* II *and* III. This oxidation was performed exactly as the one described above for cannabidiol (I). While with cannabidiol, however, the purple colour of the Beam test appears within 10–15 sec with cannabidiolic acid after ca. 5 min a brownish colour is observed which within

30 min turns into purple. The final products, II and III are identical with those, obtained from cannabidiol as judged by identical TLC and IR, UV and NMR spectra.

Reductive acetylation of II *to acetoxycannabidiol diacetate* (XVII). The hydroxyquinone (II; 160 mg) was dissolved in a soln of pyridine (1 ml) and Ac_2O (0·5 ml) and left at room temp for 16 hr. After work-up an oil (152 mg) was obtained, which without further purification was dissolved in Ac_2O (5 ml) and AcOH (5 ml). Zn (0·5 g) was added and the mixture was boiled under reflux for 30 min. The residue was filtered off, pyridine (20 ml) was added to the filtrate and the soln was left at room temp overnight when it was poured into ice–water and extracted with ether (twice 100 ml). The etheric soln was washed with 5% HCl aq, 5% $NaHCO_3$ aq and water, then dried over Na_2SO_4. The oil (170 mg) obtained after removal of the solvent gave a single spot on TLC and a single peak on VPC (2% SE-30 on Chromosorb W at 240°, flow rate of 150 ml/min on a Packard Gas Chromatograph using N_2 as carrier gas). The analytical sample was purified on preparative TLC. (Found: C, 70·83; H, 8·32. $C_{27}H_{36}O_6$ requires: C, 71·03; H, 7·95%).

Reductive acetylation of III *to the hexaacetate* XVIII. This reaction was performed exactly as described above for the preparation of XVII. The dihydroxydiquinone III (150 mg) gave XVIII (175 mg) which could not be induced to crystallize. It gave a single spot on TLC and the analytical sample was purified in this fashion. (Found: C, 70·88; H, 7·52. $C_{54}H_{70}O_{12}$ requires: C, 71·18; H, 7·74%).

Cyclization of the hydroxyquinone II *to quinone* XIX. The hydroxyquinone II (50 mg) was boiled with a soln of *p*-toluenesulphonic acid (10 mg) in dry benzene (25 ml) for 2 hr. The cooled soln was then washed with 5% $NaHCO_3$ aq and water, dried over $MgSO_4$ and evaporated to dryness. The material obtained was purified on preparative TLC. An uncrystallizable oil (17 mg) was obtained. (Found: C, 76·40; H, 8·23. $C_{21}H_{28}O_3$ requires: C, 76·79; H, 8·59%).

Oxidation of $\Delta^{1(6)}$*-tetrahydrocannabinol* (VII) *with* m*-chloroperbenzoic acid.* A soln of VII (2·13 g) and *m*-chloroperbenzoic acid (1·37 g) in chloroform (50 ml) was left in the dark for 16 hr at room temp. The precipitated *m*-chloroperbenzoic acid was filtered and the filtrate was diluted with water (25 ml) and ether (200 ml). The organic layer was washed with 5% NaOH aq and water, dried over $MgSO_4$ and evaporated under reduced press. The residue was chromatographed. The *p*-quinone XIX (150 mg) was eluted with ether–pet. ether (ratio 5:95), with the same solvent system (ratio 15:85) the *o*-quinone XX (48 mg) was eluted. Compound XIX was identical with the product obtained in the cyclization of II (comparison by TLC and by IR, UV and NMR spectra). The *o*-quinone XX was purified by preparative TLC, giving an oil. (Found: C, 76·48; H, 8·38. $C_{21}H_{28}O_3$ requires: C, 76·79; H, 8·59%).

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