

Triterpenoids from *Trichilia havanensis* Jacq. Part I. The Acetates of Havanensin and Trichilenone, New Tetracarboxylic Tetranortriterpenes¹

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Four new tetranortriterpenes, havanensin triacetate (1), havanensin 3,7-diacetate (2), havanensin 1,7-diacetate (3), and trichilenone acetate (5), have been isolated from *T. havanensis* Jacq. Their structures have been established by spectral and chemical data and by interrelation with each other and with cedrelone (13). N.m.r. spectra can be used to distinguish between Δ^1 -3-ones and Δ^2 -1-ones in these tetranortriterpenes.

In continuation of our work on tetranortriterpenes (limonoids)² from the Meliaceae, we have examined *Trichilia havanensis* Jacq.; Ehrlich's reagent showed that only the fruit contained furanoid compounds.³

Extraction of ripe fruit with acetone gave an oil which was separated into light petroleum-soluble and -insoluble fractions. Chromatography of both fractions on alumina yielded four new limonoids, havanensin triacetate (1), C₃₂H₄₄O₈, m.p. 188—191°, havanensin 3,7-diacetate (2), C₃₀H₄₂O₇, m.p. 185—187°,† havanensin 1,7-diacetate (3) (non-crystalline), and trichilenone acetate (5) (non-crystalline).‡ Hydrolysis of the triacetate (1) and both diacetates (2) and (3) with base afforded the same triol, havanensin (4), C₂₆H₃₈O₅, m.p. 235—237°, which shows hydroxylic absorption but no carbonyl bands in the i.r.

Reacetylation of havanensin (4) yielded the triacetate (1). Base-catalysed hydrolysis of the acetate (5) gave trichilenone (6), C₂₆H₃₄O₄, m.p. 199—201°, which has u.v. [λ_{max} 222 nm (ϵ 9800)] and i.r. (3450 and 1650 cm⁻¹) bands due to an $\alpha\beta$ -unsaturated ketone and a hydroxy-group.

Havanensin triacetate (1) shows no hydroxylic absorption in the i.r. region but has i.r. and u.v. absorptions characteristic of a furan ring. The n.m.r. spectrum (see Experimental section) confirmed the presence of a β -substituted furan ring⁴ and revealed five tertiary and three acetate methyl groups. A three-proton multiplet at δ 4.71 p.p.m. ($W_{\frac{1}{2}}$ 6 Hz) indicated that the acetates were secondary and axial. Since havanensin (4) has no carbonyl group, the final oxygen must be ethereal, and a singlet at δ 3.42 p.p.m. is consistent⁴ with the presence

† The m.p. given in ref. 1 is incorrect.

‡ For systematic names, see Experimental section.

¹ These results were reported at the Second Natural Products Symposium, Kingston, Jamaica, Jan., 1968, and (in part) as a preliminary communication, W. R. Chan, J. A. Gibbs, and D. R. Taylor, *Chem. Comm.*, 1967, 720.

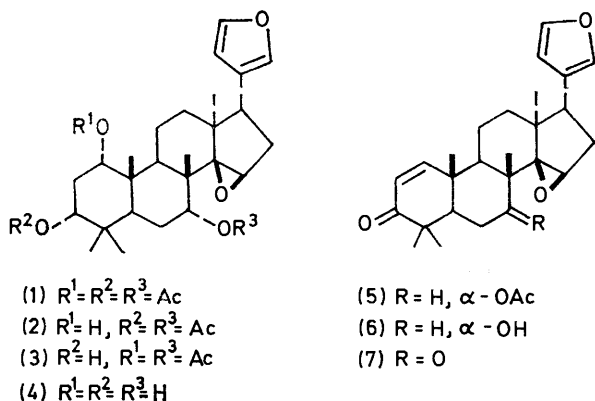
² D. L. Dreyer, *Fortschr. Chem. org. Naturstoffe*, 1968, **26**, 190; J. D. Connolly, K. H. Overton, and J. Polonsky, in 'Progress in Phytochemistry,' eds. L. Reinhold and Y. Liwischitz, Interscience, London, 1970, vol. 2, p. 385.

³ D. L. Dreyer, *J. Org. Chem.*, 1965, **30**, 749.

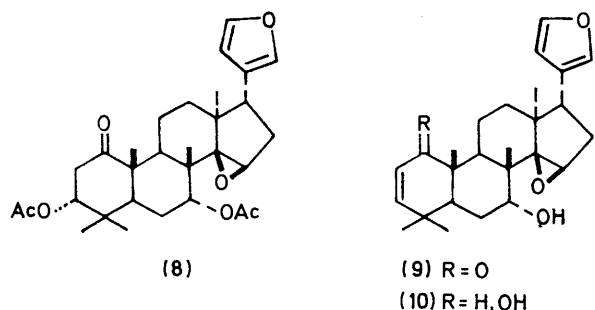
⁴ Cf. J. W. Powell, *J. Chem. Soc. (C)*, 1966, 1794.

of a 14 β ,15 β -epoxide. These data lead to (1) as the biogenetically² most probable structure for havanensin triacetate and confirmation of this assignment is provided below.

The chemical shift of H-15 in both diacetates (2) and (3) is the same as in the triacetate (1). This indicates



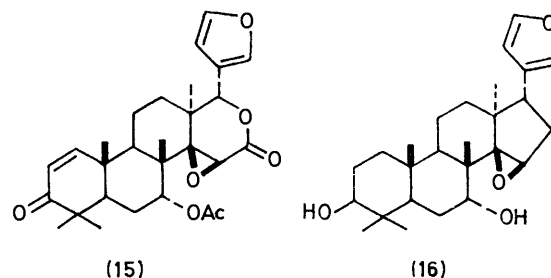
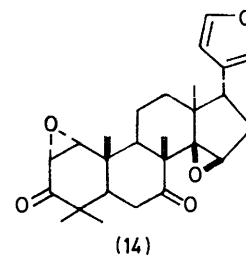
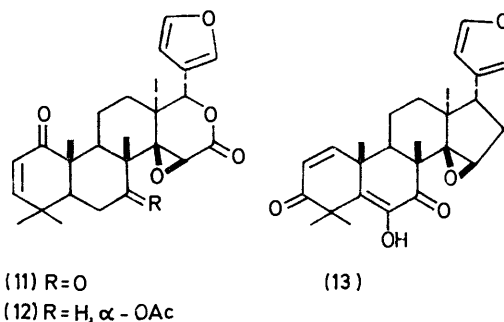
that one of the acetate groups in (2) and (3) is located at C-7 since a change in functionality at C-7 is known to change the chemical shift of H-15.^{4,5} That the diacetates (2) and (3) are isomeric 1,3-hydroxyacetates is shown by the following results. Oxidation of (3) with chromium trioxide in pyridine gave a mixture of two compounds (t.l.c.). The i.r. spectrum of the mixture showed that the main constituent of the mixture was an $\alpha\beta$ -unsaturated ketone. Treatment of the mixture with mild base afforded trichilenone acetate (5), identical with the natural product. This identity was further confirmed by more vigorous treatment with base to give trichilenone (6), whose n.m.r. spectrum shows H-1 and H-2 signals as an AX system (J 10 Hz) at δ 7.18 and 5.82 p.p.m. respectively. This locates the enone system in ring A, and its easy formation shows the diacetate (3) to be a 1,3-hydroxyacetate. A similar oxidation of the isomeric diacetate (2) gave the ketone (8). This did not eliminate



acetic acid under the conditions used above but more vigorous treatment with base caused hydrolysis of the C-7 ester and elimination to yield the enone, isotrichilenone (9), $\text{C}_{26}\text{H}_{34}\text{O}_4$, m.p. 205–207°, λ_{max} 220 nm (ϵ 9700), ν_{max} 3450 and 1680 cm^{-1} . Evidence for the orientation of

⁵ K. W. Gopinath, T. R. Govindachari, P. C. Parthasarathy, N. Viswanathan, D. Arigoni, and W. C. Wildman, *Proc. Chem. Soc.*, 1961, 446; J. D. Connolly, K. L. Handa, R. McCrindle, and K. H. Overton, *J. Chem. Soc. (C)*, 1968, 2227.

the enone systems in trichilenone (6) and isotrichilenone (9) comes from the n.m.r. data for H-1 and H-2 in these compounds. The chemical shifts for H-1 and H-2 in trichilenone (6) agree well with those in other Δ^1 -3-ones.⁴ However, the chemical shifts for H-2 and H-3 in a Δ^2 -1-one with a *gem*-dimethyl group at C-4 had not been previously reported. In isotrichilenone (9), H-2 and H-3 occur as doublets (J 10 Hz) at δ 5.68 and 6.34 p.p.m. respectively, thus providing a clear distinction between Δ^1 -3-ones and Δ^2 -1-ones in these degraded triterpenes. Similar chemical shifts (δ 5.74 and 6.43 p.p.m.) are shown by H-2 and H-3 in deacetoxy-7-oxoisogedunin (11)⁶ whose n.m.r. spectrum had not been previously reported.



Narayanan *et al.*⁷ have subsequently shown that n.m.r. spectroscopy similarly allows the differentiation of Δ^1 -3-ones and Δ^2 -1-ones in pentacyclic triterpenes.

The downfield shift of H-15 in (6) and (9) (δ 3.59 and 3.57 p.p.m.) compared with its position in the triacetate (1) (δ 3.42 p.p.m.) supports the presence of a 7 α -acetate in (1).^{4,5} Confirmation of these structural assignments was obtained by correlation with cedrelone

⁶ A. Akisanya, E. O. Arene, C. W. L. Bevan, D. E. U. Ekong, M. N. Nwaji, J. I. Okogun, J. W. Powell, and D. A. H. Taylor, *J. Chem. Soc. (C)*, 1966, 506.

⁷ C. R. Narayanan, R. V. Pachapurkar, B. M. Sawant, and M. S. Wadia, *Indian J. Chem.*, 1969, 7, 187.

(14,15 β :21,23-diepoxy-6-hydroxy-4,4,8-trimethyl-24-nor-13 α ,14 β ,17 α -chola-1,5,20,22-tetraene-3,7-dione) (13)⁸ whose structure has been unequivocally defined by X-ray analysis.⁹ Oxidation of trichilenone (6) with chromium trioxide in pyridine gave the 7-ketone (7). Oxygenation of (7) in the presence of potassium t-butoxide¹⁰ yielded a gum which could not be characterised. Cedrelone (13) is unstable to the same reaction conditions and gave a gum with the same spectral properties. However, the 1,2-epoxide (14) obtained by treatment of (7) with alkaline hydrogen peroxide afforded the known 1,2-epoxycedrelone⁸ in high yield on potassium t-butoxide-catalysed autoxidation.

That trichilenone (6) possesses the expected 5 α -configuration is shown by the similarity of its c.d. curve [$\Delta\epsilon_{400}$ 0, $\Delta\epsilon_{377}$ -1.00(i), $\Delta\epsilon_{357}$ -2.40(i), $\Delta\epsilon_{345}$ -2.61, $\Delta\epsilon_{330}$ -1.81(i), and $\Delta\epsilon_{282}$ 0] to that of gedunin (15)^{11,12} [$\Delta\epsilon_{400}$ 0, $\Delta\epsilon_{380}$ -0.79(i), $\Delta\epsilon_{360}$ -2.07(i), $\Delta\epsilon_{347}$ -2.34, and $\Delta\epsilon_{284}$ 0].¹³ The c.d. curves of isotrichilenone (9) ($\Delta\epsilon_{340}$ -2.52) and deacetyl-7-oxoisogedunin (11) ($\Delta\epsilon_{332}$ -2.85) also show negative Cotton effects for the enone but lack the fine structure shown by trichilenone (6) and gedunin (15). Thus c.d. data also differentiate Δ^1 -3-ones from Δ^2 -1-ones.

Barton *et al.*^{14,15} found that in several pairs of Δ^1 -3-ones and Δ^2 -1-ones derived from lanostane and oleanane, the Δ^2 -1-ones always had a much more positive specific rotation. This difference is, however, not shown by the isomeric pairs trichilenone (6), $[\alpha]_D +17^\circ$, and isotrichilenone (9), $[\alpha]_D +19^\circ$, and gedunin (15), $[\alpha]_D +44^\circ$,¹¹ and isogedunin (12), $[\alpha]_D +46^\circ$.⁶

The enone systems in trichilenone (6) and isotrichilenone (9) have different reactivities towards nucleophilic reagents. Whereas 7-dehydrotrichilenone (7) underwent easy epoxidation with alkaline hydrogen peroxide (see above), isotrichilenone (9) was recovered unchanged when treated under the same conditions. This difference in reactivity of ring-A-isomeric enones towards peroxide ion has also been observed¹⁵ in the oleanane series. However, Ekong and his co-workers¹⁶ have recently shown that the Δ^2 -1-one system in 7-deacetylisogedunin can be epoxidised with hydrogen peroxide and alkali. This latter substrate is tricyclic under the reaction conditions since the lactone would be opened, and the difference in electrophilicity of the $\alpha\beta$ -unsaturated carbonyl system is presumably a reflection of conformational changes. Also, reduction of trichilenone (6) with sodium borohydride in methanol gave the saturated diol (16), while similar treatment of isotrichilenone (9) afforded the allylic alcohol (10). Adesogan *et al.*¹⁷ obtained only 1,2-dihydrotrichilenone from reduction of trichilenone (6) with sodium borohydride in a mixture of methanol and chloroform.

⁸ R. Hodges, S. G. McGeachin, and R. A. Raphael, *J. Chem. Soc.*, 1963, 2515.

⁹ I. J. Grant, J. A. Hamilton, T. A. Hamor, J. M. Robertson, and G. A. Sim, *J. Chem. Soc.*, 1963, 2506.

¹⁰ D. H. R. Barton, S. R. Pradhan, S. Sternhell, and J. F. Templeton, *J. Chem. Soc.*, 1961, 255.

¹¹ A. Akisanya, C. W. L. Bevan, T. G. Halsall, J. W. Powell, and D. A. H. Taylor, *J. Chem. Soc.*, 1961, 3705.

The havanensin acetates (1), (2), and (3) have also been obtained from *Khaya anthotheca* Welw. C.D.C.¹⁷

EXPERIMENTAL

Optical rotations were determined for chloroform solutions; c.d. data for dioxan solutions (i = inflection); u.v. spectra were obtained for ethanolic solutions; i.r. spectra for Nujol mulls; and n.m.r. spectra were recorded on a Varian A-60 spectrometer for deuteriochloroform solutions (tetramethylsilane as internal reference) unless stated otherwise.

Extraction.—The ripe fruit (3.0 kg) were blended with acetone (2 l). Evaporation of the acetone left an oily residue which was separated into light petroleum (b.p. 60–80°)-soluble (1.2 kg) and -insoluble (750 g) fractions. Chromatography of the insoluble gum (34 g) on alumina (700 g) and elution with benzene-ethyl acetate (8 : 2) gave a gum (3.4 g) which (by n.m.r.) was mostly *trichilenone acetate* (5), ν_{\max} (CHCl₃) 1727, 1650, 1501, 1248, and 877 cm⁻¹, δ (CCl₄) 7.31 (H-23), 7.19 and 5.77 (AX system, J 10 Hz, H-1 and H-2), 7.05 (H-21), 6.13 (H-22), 4.65 (H-7), 3.33 (s, H-15), 2.10 (OAc), 1.14, 1.05, 1.01 (6H), and 0.81 (5 \times CMe). Elution with benzene-ethyl acetate (7 : 3) gave impure *havanensin 1,7-diacetate* (3) (13 g), ν_{\max} (CHCl₃) 3333 and 1742 cm⁻¹, δ 7.40 (H-23), 7.14 (H-21), 6.19 (H-22), 4.95 (H-1), 4.78 (H-7), 3.42 (s, H-15), 3.41 (H-3), 2.12 and 2.09 (OAc), 1.06, 0.99 (6H), 0.94, and 0.86 (5 \times CMe). The soluble oil (39 g) was chromatographed on alumina (750 g). The light petroleum-benzene (8 : 2) eluate was crystallised from methanol to yield *havanensin 3,7-diacetate* (2) (5.4 g), m.p. 185–187°, $[\alpha]_D -39^\circ$, λ_{\max} 212 nm (ϵ 3800), ν_{\max} 3425, 1724, 1504, and 877 cm⁻¹, δ 7.35 (H-23), 7.10 (H-21), 6.17 (H-22), 4.92 (H-3), 4.70 (H-7), 3.54 (H-1), 3.42 (s, H-15), 2.15 (2 \times OAc), 1.08, 1.05, 0.94, 0.91, and 0.79 (5 \times CMe) (Found: C, 70.5; H, 8.25. C₃₀H₄₂O₇ requires C, 70.0; H, 8.2%). Light petroleum-benzene (1 : 1) eluted *havanensin triacetate* (1), prisms (3.4 g) (from methanol), m.p. 188–191°, $[\alpha]_D -76^\circ$, λ_{\max} 212 nm (ϵ 3700), ν_{\max} 1739, 1504, and 877 cm⁻¹, δ 7.37 (H-23), 7.11 (H-21), 6.16 (H-22), 4.71 (3H, H-1, H-3, and H-7), 3.42 (s, H-15), 2.12 (6H) and 2.03 (3 \times OAc), 1.16, 1.01 (6H), 0.92, and 0.81 (5 \times CMe) (Found: C, 68.9; H, 8.1. C₃₂H₄₄O₈ requires C, 69.0; H, 8.0%).

Havanensin (4).—A solution of *havanensin triacetate* (1) (50 mg) in 5% methanolic potassium hydroxide (5 ml) was heated under reflux for 6 h. Most of the solvent was evaporated off *in vacuo* and the product was precipitated with cold water (25 ml). Crystallisation from methanol gave *havanensin* (14,15 β :21,23-diepoxy-4,4,8-trimethyl-24-nor-5 α ,13 α ,14 β ,17 α -chola-20,22-diene-1 α ,3 α ,7 α -triol) (4) as prisms (37 mg), m.p. 235–237°, ν_{\max} 3350, 1502, and 877 cm⁻¹ (Found: C, 72.6; H, 8.9. C₂₆H₃₈O₅ requires C, 72.5; H, 8.9%).

Similar hydrolysis of the diacetates (2) and (3) also gave *havanensin* (4), which on acetylation with acetic anhydride

¹² S. A. Sutherland, G. A. Sim, and J. M. Robertson, *Proc. Chem. Soc.*, 1962, 222.

¹³ G. Sztatke, *Tetrahedron*, 1965, 21, 421.

¹⁴ D. H. R. Barton, P. J. L. Daniels, J. F. McGhie, and P. J. Palmer, *J. Chem. Soc.*, 1963, 3675.

¹⁵ D. H. R. Barton, E. F. Lier, and J. F. McGhie, *J. Chem. Soc. (C)*, 1968, 1031.

¹⁶ M. E. Obase, J. I. Okogun, and D. E. U. Ekong, *J.C.S. Perkin I*, 1972, 1943.

¹⁷ E. K. Adesogan, D. A. Okorie, and D. A. H. Taylor, *J. Chem. Soc. (C)*, 1970, 205.

and pyridine at room temperature for 3 days yielded havanensin triacetate (1).

Trichilenone (6).—(a) *From trichilenone acetate* (5). Trichilenone acetate (650 mg) was hydrolysed by heating in 5% methanolic potassium hydroxide for 6 h to give *trichilenone* (14,15 β :21,23-diepoxy-7 α -hydroxy-4,4,8-trimethyl-24-nor-5 α ,13 α ,14 β ,17 α -chola-1,20,22-trien-3-one) (6) (320 mg) as prisms (from methanol) m.p. 199–201°, $[\alpha]_D^{20} +17^\circ$, λ_{\max} 222 nm (ϵ 9800), ν_{\max} 3450, 1650, 1502, and 877 cm^{-1} , δ 7.34 (H-23), 7.18 and 5.82 (AX system, J 10 Hz, H-1 and H-2), 7.09 (H-21), 6.16 (H-22), 3.59 (2H, H-7 and H-15), 1.18, 1.14, 1.09, 1.07, and 0.97 (5 \times CMe) (Found: C, 75.7; H, 8.2. $\text{C}_{26}\text{H}_{34}\text{O}_4$ requires C, 76.1; H, 8.3%).

(b) *From havanensin 1,7-diacetate* (3). A solution of havanensin 1,7-diacetate (8.6 g) in cold pyridine (20 ml) was added dropwise to a cold solution of chromium trioxide (8 g) in pyridine (10 ml). The mixture was set aside at room temperature overnight and then diluted with benzene (200 ml). The benzene solution was filtered through a short column of alumina and the solvent evaporated off *in vacuo* to afford a gum which was a mixture of mainly two compounds (t.l.c.). Treatment of the mixture with methanolic 5% potassium hydroxide at room temperature for 20 min. left only the major component, trichilenone acetate (5) (6.4 g), identical with the natural product (t.l.c., n.m.r. and i.r. spectra), which yielded trichilenone (6) (m.p., mixed m.p., and i.r.) on hydrolysis at elevated temperature as described above.

1-Dehydrohavanensin 3,7-Diacetate (8).—Havanensin 3,7-diacetate (100 mg) was oxidised with chromium trioxide in pyridine as described above to give *1-dehydrohavanensin 3,7-diacetate* (8) (78 mg), m.p. 189–191° (from methanol), ν_{\max} 1733 and 1695 cm^{-1} , δ 7.38 (H-23), 7.13 (H-21), 6.22 (H-22), 5.07 (t, J 4 Hz, H-3), 4.71 (H-7), 3.42 (s, H-15), 2.06 and 2.04 (OAc), 1.36, 1.15, 1.09 (6H), and 0.83 (5 \times CMe) (Found: C, 70.0; H, 8.0. $\text{C}_{30}\text{H}_{40}\text{O}_7$ requires C, 70.3; H, 7.9%). The diacetate (8) was recovered unchanged after treatment with 5% methanolic potassium hydroxide at room temperature for 30 min.

Isotrichilenone (9).—A solution of 1-dehydrohavanensin 3,7-diacetate (8) (100 mg) in 5% methanolic potassium hydroxide was heated under reflux for 6 h and the product crystallised from methanol to yield *isotrichilenone* (9) (77 mg), m.p. 205–207°, $[\alpha]_D^{20} +19^\circ$, λ_{\max} 220 nm (ϵ 9700), ν_{\max} 3450 and 1680 cm^{-1} , δ 7.35 (H-23), 7.17 (H-21), 6.34 and 5.68 (AB system, J 10 Hz, H-3 and H-2), 6.20 (H-22), 3.57 (2H, H-7 and H-15), 1.27, 1.09, 1.06 (6H), and 1.03 (5 \times CMe) (Found: C, 75.5; H, 8.2. $\text{C}_{26}\text{H}_{34}\text{O}_4$ requires C, 76.1; H, 8.3%).

Deacetoxy-7-oxoisogedunin (11).—Gedunin (500 mg) was converted into deacetyl-1,2-epoxygedunin (350 mg), m.p. 289–292° (lit.,⁶ 285–291°) with alkaline hydrogen peroxide as described.⁶ Treatment of the epoxide with hydrazine⁶ afforded deacetylisedgedunin (131 mg), m.p. 244–246° (lit.,⁶ 241–244°), which was oxidised with Jones reagent to give deacetoxy-7-oxoisogedunin (11) (31 mg), m.p. 253–254° (lit.,⁶ 252–254°), ν_{\max} 1727 and 1685 cm^{-1} , δ 7.41 (2H, H-23 and H-21), 6.38 (H-22), 6.43 and 5.74 (AB system, J 10 Hz, H-3 and H-2), 5.47 (s, H-17), 3.69 (s, H-15), 1.43, 1.21,

1.17, 1.14 and 1.10 (5 \times CMe) (Found: C, 68.5; H, 6.6. Calc. for $\text{C}_{26}\text{H}_{30}\text{O}_6, \text{H}_2\text{O}$: C, 68.4; H, 7.1%).

7-Dehydrotrichilenone (7).—Trichilenone (500 mg) was oxidised by chromium trioxide in pyridine as usual to give 7-dehydrotrichilenone (7) (390 mg), m.p. 183–184° (from methanol) (lit.,¹⁷ 179–181°) ν_{\max} 1715 and 1650 cm^{-1} , δ 7.39 (H-23), 7.26 and 5.95 (AX system, J 10 Hz, H-1 and H-2), 7.17 (H-21), 6.21 (H-22), 3.66 (s, H-15), 1.37, 1.22, 1.17 (6H), and 0.80 (5 \times CMe) (Found: C, 76.4; H, 8.0. Calc. for $\text{C}_{26}\text{H}_{32}\text{O}_4$: C, 76.4; H, 7.9%).

1,2-Epoxy-7-dehydrotrichilenone (14).—A solution of 7-dehydrotrichilenone (200 mg) in acetone (20 ml) was stirred at room temperature with 30% hydrogen peroxide (5 ml) and 8% sodium hydroxide (5 ml) for 8 h. Isolation of the product with ethyl acetate and crystallisation from methanol gave *1,2-epoxy-7-dehydrotrichilenone* (14) (135 mg), m.p. 205–208°, ν_{\max} 1719 cm^{-1} , δ 7.38 (H-23), 7.17 (H-21), 6.20 (H-22), 3.68 and 3.47 (AB system, J 4.7 Hz, H-2 and H-1), 3.65 (s, H-15), 1.21 (6H), 1.12, 1.04, and 0.85 (5 \times CMe) (Found: C, 73.5; H, 7.6. $\text{C}_{26}\text{H}_{32}\text{O}_5$ requires C, 73.6; H, 7.6%).

1,2-Epoxycedrelone.—(a) *From cedrelone*. Cedrelone (200 mg) was epoxidised with alkaline hydrogen peroxide as described⁸ to yield 1,2-epoxycedrelone (146 mg), m.p. 228–230° (lit.,⁸ 222–228°), ν_{\max} 3425, 1715, 1684, and 1631 cm^{-1} .

(b) *From 1,2-epoxy-7-dehydrotrichilenone*. 1,2-Epoxy-7-dehydrotrichilenone (50 mg) and *n*-potassium *t*-butoxide in *t*-butyl alcohol (10 ml) were shaken with oxygen for 7 h. The solution was diluted with water (20 ml) and the product extracted into ethyl acetate. Crystallisation from chloroform-ethanol gave 1,2-epoxycedrelone (27 mg) identical with the authentic material described above (m.p., mixed m.p., and i.r.).

Sodium Borohydride Reduction of Trichilenone.—A solution of trichilenone (200 mg) in methanol (20 ml) was treated with sodium borohydride (200 mg). After 1 h at room temperature, most of the solvent was removed *in vacuo* and the product was precipitated by the addition of water (20 ml). Crystallisation from methanol afforded *1,2-dihydrotrichilenol* (16) (122 mg), m.p. 234–235°, ν_{\max} 3427 cm^{-1} , δ 7.37 (H-23), 7.13 (H-21), 6.19 (H-22), 3.57 (2H, H-7 and H-15), 3.45br (H-3), 0.99, 0.97 (6H), 0.92, and 0.78 (5 \times CMe) (Found: C, 75.5; H, 9.3. $\text{C}_{26}\text{H}_{38}\text{O}_4$ requires C, 75.3; H, 9.2%).

Sodium Borohydride Reduction of Isotrichilenone.—Isotrichilenone (140 mg) in methanol (15 ml) was reduced with sodium borohydride as for trichilenone, yielding *isotrichilenol* (10) (97 mg), m.p. 164–165° (from methanol), ν_{\max} 3623 and 3448 cm^{-1} , δ 7.37 (H-23), 7.13 (H-21), 6.20 (H-22), 5.25–5.90 (2H, complex, H-3 and H-2), 3.60 and 3.57 (2H and 1H, H-1, H-7, and H-15), 1.05, 1.01, 0.99, and 0.90 (6H) (5 \times CMe) (Found: C, 75.9; H, 9.0. $\text{C}_{26}\text{H}_{36}\text{O}_4$ requires C, 75.7; H, 8.8%).

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