Triterpenoids

J. D. Connolly, R. A. Hill, and B. T. Ngadjui†
Department of Chemistry, Glasgow University, Glasgow G12 8QQ

Reviewing the literature published between January 1990 and December 1991
(Continuing the coverage of literature in *Natural Product Reports*, 1994, Vol. 11, p. 91)

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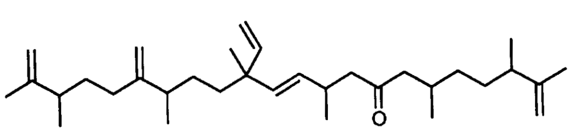
1 Introduction

This article follows the pattern of the previous report. A dictionary of terpenoids has been published containing a section on triterpenoids, in which compounds with the same framework are helpfully grouped together.[1] Reviews have appeared on triterpenoids isolated from *Abies* species[2] and triterpenoid saponins reported from 1987 to 1989.[3]

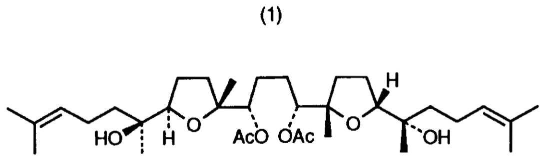
2 The Squalene Group

Botryococcenone (1) has been isolated from *Botryococcus braunii*.[4] Eurylene (2)[5] and longilene peroxide (3)[6] are produced by *Eurycoma longifolia*. The structures of both compounds were established by crystal structure analysis. Full details of the syntheses of teurilene,[7] thyrsiferol,[8] and venustatriol[8] have been published. A new rearrangement reaction involving migration of an allyl group from oxygen to carbon has been applied to the synthesis of squalene.[9]

Several groups have been involved in the purification of oxidosqualene–lanosterol cyclase from yeast[10, 11] and rat liver.[12] The enzyme has been used in further studies on the cyclization of hydroxylated oxidosqualenes[13] and a vinyl analogue of oxidosqualene[14] to the corresponding lanostane derivatives.



(1)



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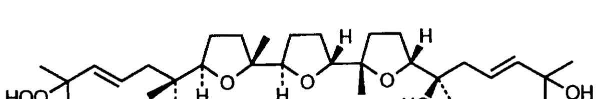
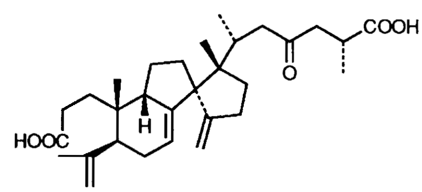


(3)

3 The Fusidane–Lanostane Group

The 3,4-secoabiesane derivative (4) has been obtained from *Abies alba* together with the related 3,4-secolanostane (5).[13] 3-Oxolanosta-7,24-dien-26-oic acid (6) and the corresponding 6β-acetoxy derivative (7) have been isolated from *Santiria trimera*.[16] The structure of (6) was confirmed by *X*-ray analysis, as was that of methyl (24*S*,25*R*)-24,25-epoxy-3α-hydroxylanost-9(11)-en-26-oate (8) from the Siberian larch.[17]



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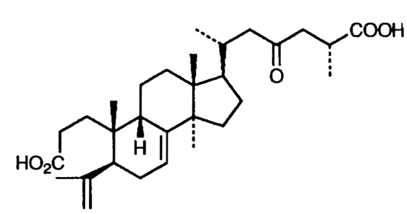

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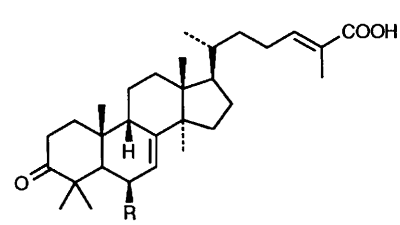
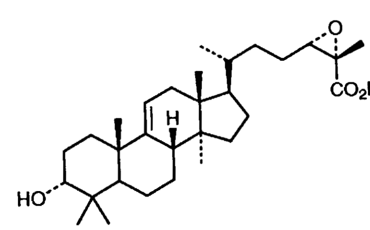


(6) R = H
(7) R = OAc



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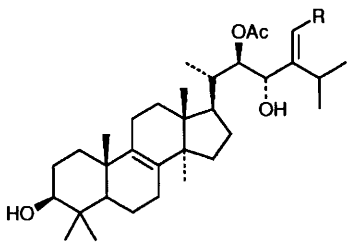
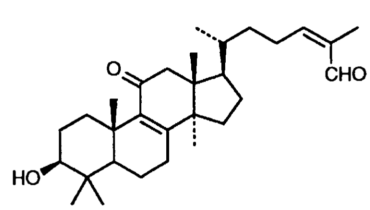
†Present address: Department of Organic Chemistry, University of Yaoundé, BP 812, Yaoundé, Cameroon.



(9) R = H
(10) R = CH3



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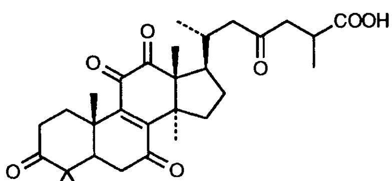

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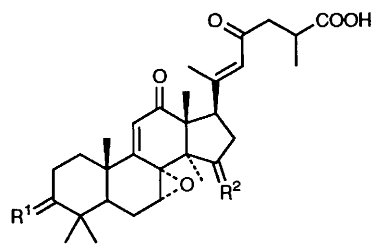
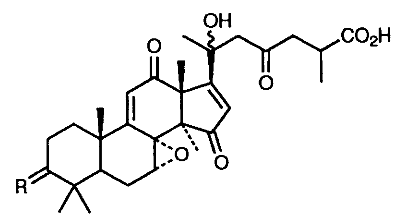
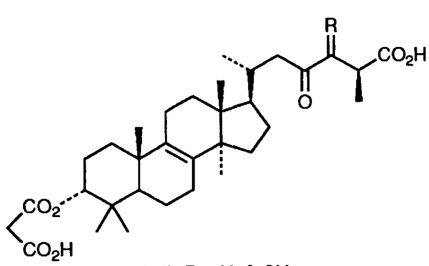


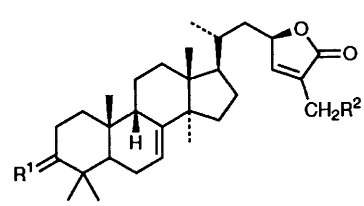
(13) R1 = O; R2 = H, α-OH
(14) R1 = H, β-OH; R2 = O



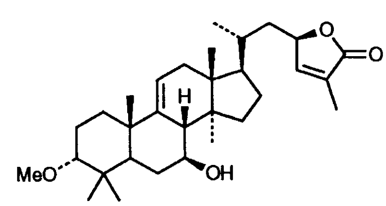
(15) R = O
(16) R = H, β-OH



(17) R = H, β-CH3
(18) R = CH2
(19) R = H, β-CH2OH



(20) R1 = O; R2 = H
(21) R1 = O; R2 = OH
(22) R1 = H, β-OH; R2 = H
(23) R1 = H, α-OH; R2 = H
(24) R1 = H, α-OMe; R2 = H; Δ9(11)



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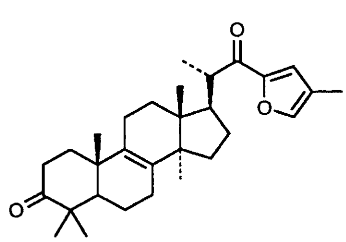

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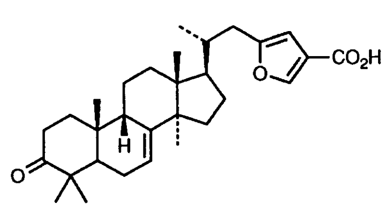

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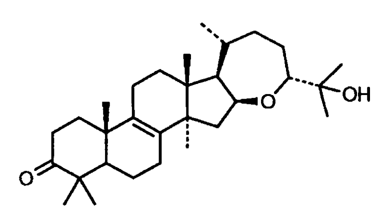


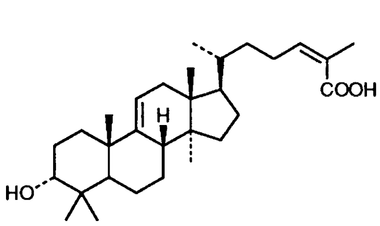
(28)

The C-22 and C-23 configurations of the lanostanes (9) and (10), from *Pisolithus tinctorius*, have been revised.[18]

The mushroom *Ganoderma lucidum* appears to have dried up as a source of new lanostanes and confusing names! Only ganoderic aldehyde A (11)[19] and ganosporenic acid A (12)[20] have been reported. The related species *G. applanatum* contains applanoxidic acids A (13), B (14), C (15), and D (16),[21] while an Indonesian *Ganoderma* species yielded the malonate esters (17)–(19),[22] of which the first is the known carboxyacetylquercinic acid; its structure was confirmed by *X*-ray analysis. Biosynthetic studies of the triterpenoids of *G. lucidum* using [1,2-$^{13}C_2$]acetate have shown that the fungal pathway also uses squalene 2,3-epoxide.[23]

(23*R*)-3-Oxolanosta-7,24-dien-26,23-olide (20) has been isolated from *Abies firma*[24] together with the closely related 27-hydroxy derivative (21) and the 3β-hydroxy derivative (22).[25] The stereochemistry of (20) was established by *X*-ray analysis. Another *Abies* species, *A. veitchii*, yielded the corresponding 3α-hydroxy derivative (23), the 3α-methoxy-7,9(11),24-trienolide (24),[26] and 7β-hydroxy-3α-methoxylanosta-9(11),24-dienolide (25), veitchiolide.[27] Pomacerone (26) from *Phellinus pomaceus*[28] and pseudolarifuroic acid (27) from *Pseudolarix kaempferi*[29] have furan-containing side chains. Isoargentatin B (28)[30] and isoanwuweizic acid (29)[31] have been obtained from *Parthenium argentatum* and *Kadsura heteroclita*, respectively. Other new lanostanes include poricoic acids A (30) and B (31) from *Poria cocos*,[32] (24*S*)-24-methyllanosta-9(11),25-dien-3-one (32) from *Bridelia tomentosa*,[33] antiquol A (33) from *Euphorbia antiquorum*,[34] schisanlactone F (34) from *Kadsura longipedunculata*,[35] and the acetonide of (24*S*)-3β-methoxylanost-9(11)-ene-24-25-diol (35) from *Pinus armandii*.[36]

Papers have appeared on the synthesis of various C-30 (C-32) substituted lanostanes[37–39] and on the isolation of the enzyme which catalyses 14-demethylation of lanosterol.[40] Investigations of lanostane saponins include sarasinosides A_1, A_2, A_3 B_1, B_2, B_3, C_1, C_2, and C_3 from *Asteropus sarasinosum*;[41] cucumarosides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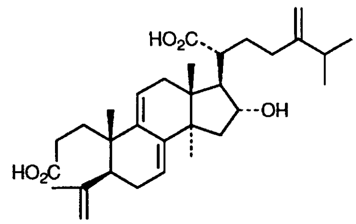
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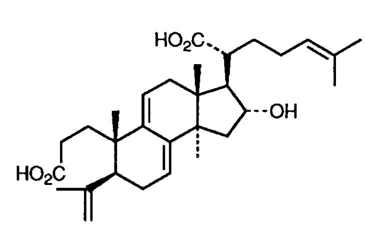
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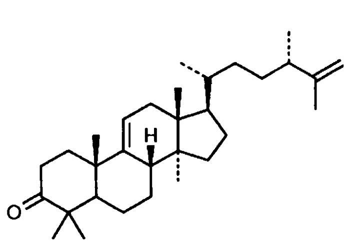
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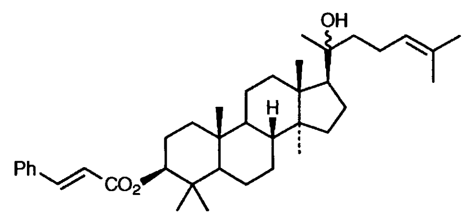
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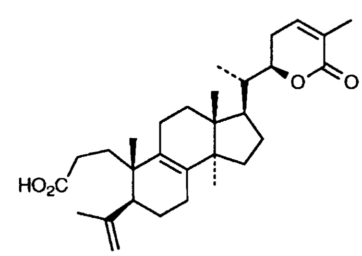
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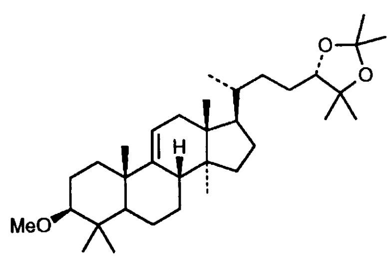
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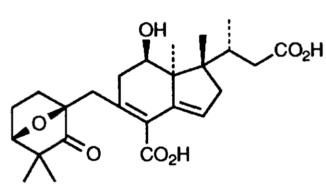
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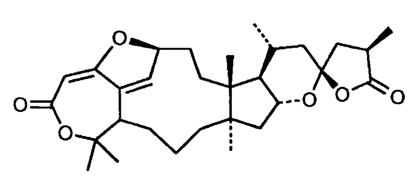
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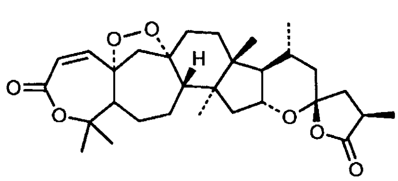
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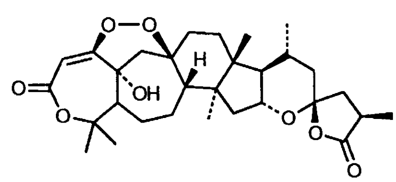
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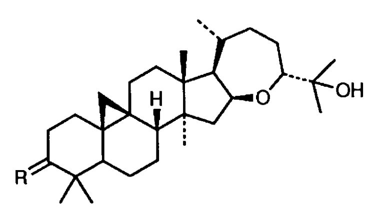
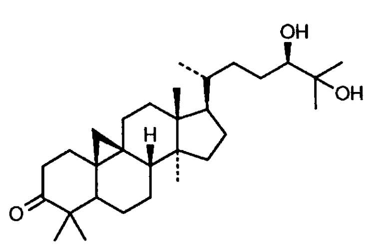

(40) R = O
(42) R = H, β-OH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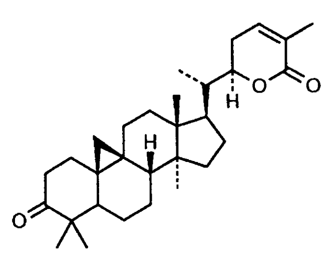
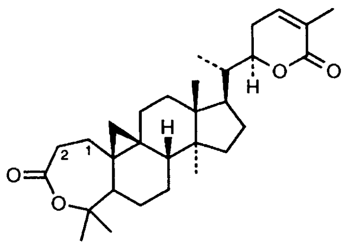
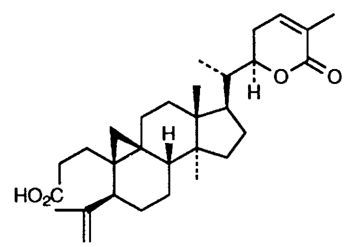

(43)

A_1-2, A_2-3, A_2-4, and A_4-2 from *Cucumaria japonica*;[42] two saponins from an *Erylus* sponge;[43] and chikusetsusaponin VI from *Panax pseudo-ginseng*.[44] Studies of the metabolism of ginseng saponins in rats have been reported.[45-47]

Glycinoeclepin A (36), the natural hatching stimulus for the soybean cyst nematode, has been synthesized.[48] Three unusual dilactones, pseudolarolides E (37),[49] H (38),[50] and I (39),[51] have been found in the root of *Pseudolarix kaempferi*. Pseudolarolide E (37) appears to be a 3,4:8,9:9,10-cleaved cycloartane derivative while the other two probably arise from a cycloartane skeleton by 3,4:9,10-cleavage. The structures were all established by *X*-ray analyses. The structure of argentatin B (40) has been revised on the basis of an *X*-ray analysis.[52] Details of its spectroscopic properties have also been published.[53] *X*-ray analyses have been performed on argentatins C (41) and D (42), which co-occur with argentatin B (40) in *Parthenium argentatum*.[30] A series of cycloartanes has been isolated from *Kadsura* species: kadsulactone (43) from *K. longipedunculata*



(44)
(47) 1,2-Didehydro



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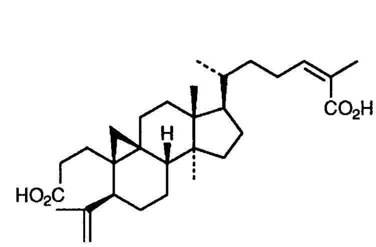

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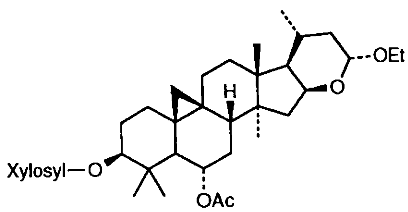

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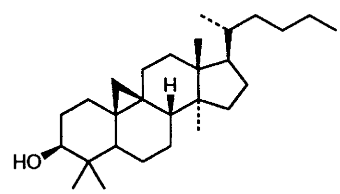

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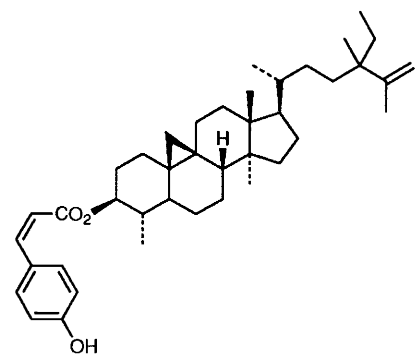

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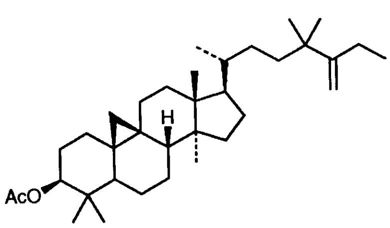

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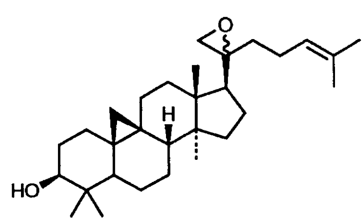

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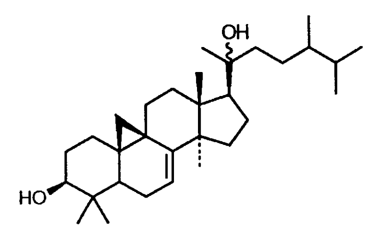

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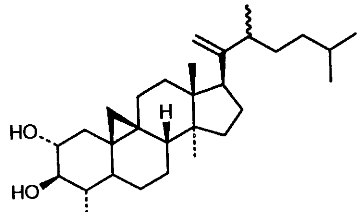

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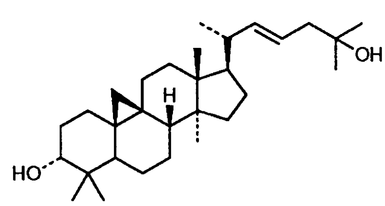

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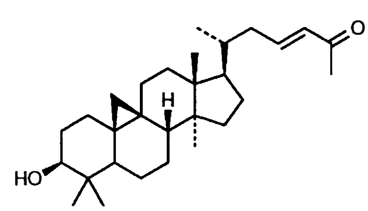

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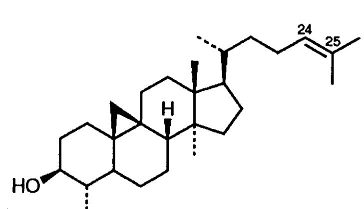
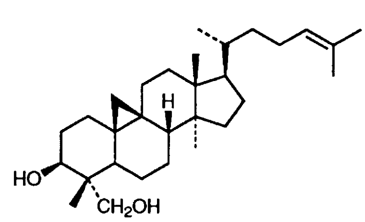
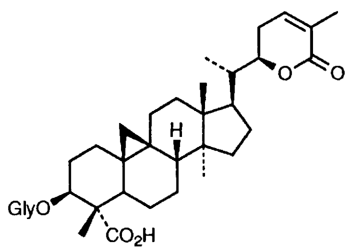


(57)
(58) 24ξ,25-Epoxide



(59)

and the ring-A-cleaved lactone kadsudilactone (44) from *K. coccinea*;[54] schisanlactone E (45) and changnanic acid (46) from *K. longipedunculata*;[55] and kadsulactone A (47) from *K. heteroclita*.[56] The ethanol extract of *Astragalus tomentosus* is the source of the tetranorcycloartane ethyl acetal tomentoside I (48).[57] Side-chain variations are apparent in 26,27-dinorcycloartan-3β-ol (49) from opium marc,[58] uniflorin (50) from *Coelogyne uniflora*,[59] and cyclopodmenyl acetate (51) from *Polypodium* species.[60] Other cycloartanes include cyclonivuliaol (52) from *Euphorbia nivulia*;[61] curculigol (53) from *Curculigo ochioides*;[62] 22ξ-methyl-29-norcycloart-20-ene-2α,3β-ol (54) from *Swietenia mahagoni*;[63] cycloart-22-ene-3α,25-diol (55) from *Pentatropis spiralis*;[64] 3β-hydroxy-27-norcycloart-23-en-25-one (56) from *Garcinia mangostana*;[65] 29-norcycloartenol (57), 24,25-epoxy-29-norcycloartenol (58) (mixture of 24-epimers), and 28-hydroxycycloartenol (59) from *Garcinia*



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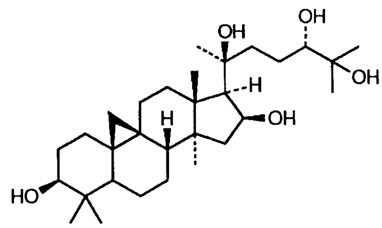

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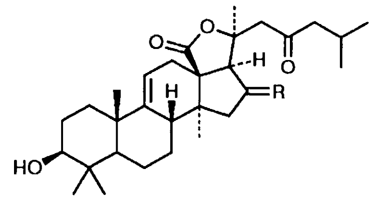
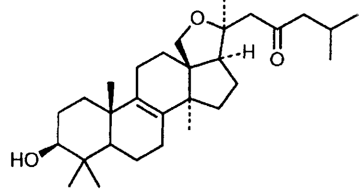


(62) R = O
(63) R = H,H



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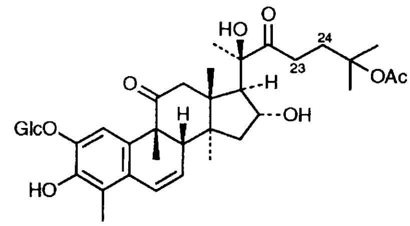
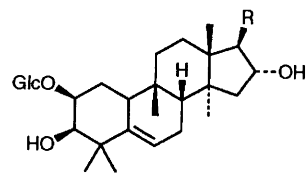
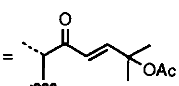
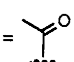
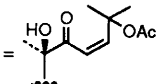


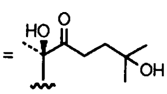
(65)
(66) 23,24-Didehydro

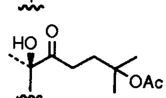


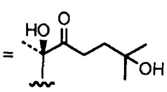
(67) R = 

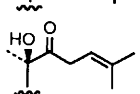
(71) R = 

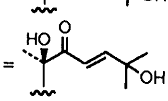
(68) R = 

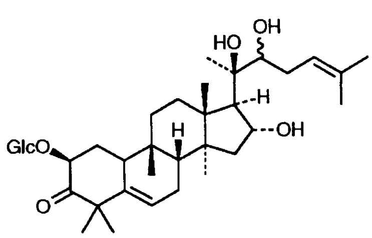
(72) R =  ; aglycone

(69) R = 

(73) R =  ; 6′-cinnammyl

(70) R = 

(74) R = 



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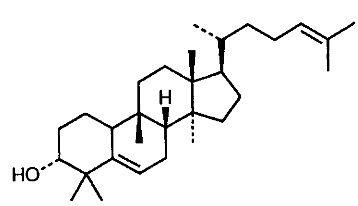


(76)

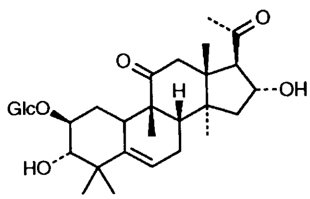
lucida;[66] the *p*-hydroxycinnamate of cycloartenol (arundinol) from *Arundina bambusifolia*;[67] and the 24,25-acetonide of (24*S*)-cycloartane-3*β*,24,25-triol from *Notholaena rigida*.[68]

The alkaloid cycloprotobuxine has been synthesized from lanosterol.[69] The value of 1D ^{1}H–^{13}C NMR correlations in structure elucidation has been demonstrated using a cycloartane glycoside, abrusoside D (60).[70] On exposure to a growing culture of *Mycobacterium* species, cycloartenol, 24-methylene-cycloartanol, and lanosterol were metabolized to androsta-4,8(14)-diene-3,17-dione.[71]

(20*S*, 24*S*)-Cycloartane-3*β*,16*β*,20,24,25-pentaol (61) occurs as a glycoside in *Oxytropis bicolor*.[72] Other cycloartane saponins include cycloaralosides C[73] and E[74] from *Astragalus amarus*, cyclocarposide from *A. coluteocarpus*,[75] new glycosides from *A. membranaceus*[76] and *Oxytropis bicolor*,[77] cycloorbicoside B from *A. orbiculatus*,[78] astailienin A from *A. iliensis*,[79] astrachrysoside A from *A. chrysopterus*,[80] beesioside IV from *Beesia calthaefolia*,[81] and mussaendoside M from *Mussaenda pubescens*.[82]

Three new aglycones, cucumechinols A (62), B (63), and C (64), have been isolated from the extract of the sea cucumber *Cucumaria echinata*.[83] Cucumechinosides A–F have been isolated from the same species,[84] whereas *C. frondosa* yields frondoside A.[85] Other saponins in this area include 24-dehydroechinoside B from *Actinopyga mauritiana*,[86] holothurinosides A–D and desholothurin A from *Holothuria forskalii*,[87] and neothyonidioside C from *Neothyonidium magnum*.[88]

Two ring-A-aromatic norcucurbitacins [(65) and (66)] have been isolated from a *Wilbrandia* species.[89] *Picrorhiza kurrooa* is a rich source of cucurbitacin derivatives. It contains the glycosides (67)–(71), the aglycone (72),[90] 6′-cinnamate (73),[90] and the glycosides (74) and (75).[91] A simple cucurbitacin alcohol, antiquol B (76), has been obtained from *Euphorbia*



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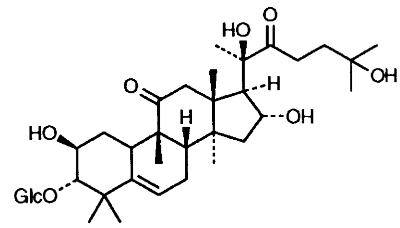

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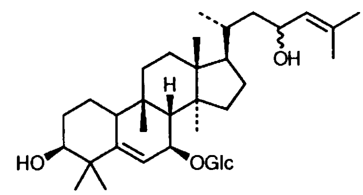

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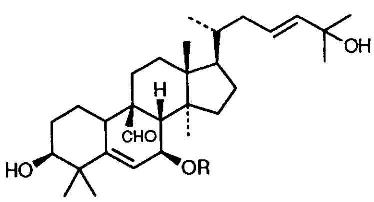
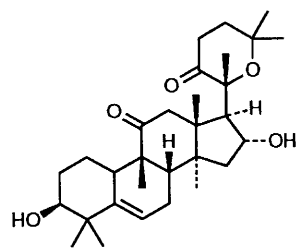


(80) R = Glc
(81) R = H



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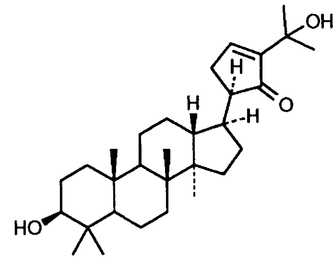

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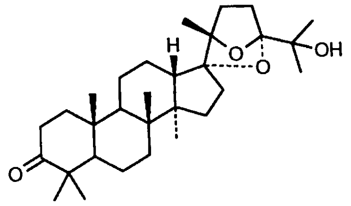
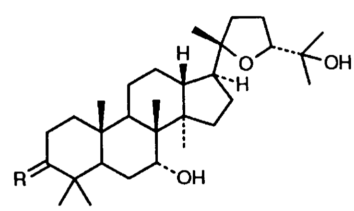
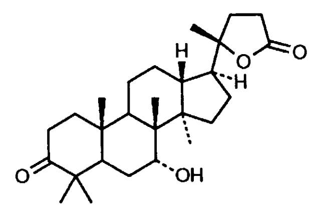


(84)



(85) R = H, β-OH
(86) R = O



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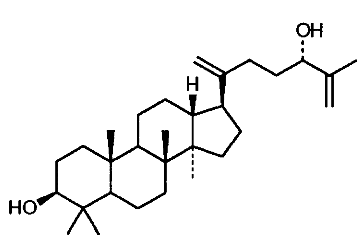
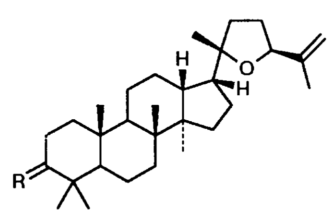
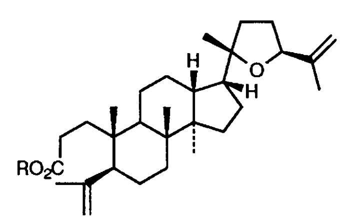


(88)



(89) R = H, β-OH
(90) R = O



(91) R = H
(92) R = Me

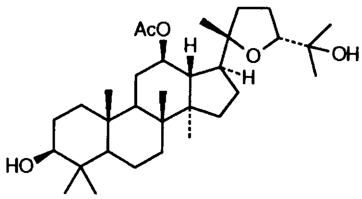
antiquorum.[34] Several other glycosides have been published, including perseapicroside A (77) from *Persea mexicana*;[92] the 3-*O*-glucoside (78) from *Hintonia latiflora*;[93] and the 7-*O*-glucosides (79) and (80), together with the aglycone (81), from *Momordica charantia*.[94]

The mass-spectral fragmentation of cordifolin (82) and other cucurbitacins has been studied.[95] Hebevinosides XII, XIII, and XIV, cucurbitacin saponins from the mushroom *Hebeloma vinosophyllum*, have received attention.[96] Mogroside III is a tasteless cucurbitacin saponin from *Sivaitia grosvenori*. The structure–taste relationship of a range of cucurbitacin glycosides has been described.[97]

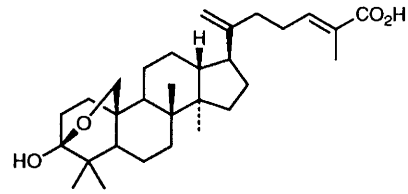
4 The Dammarane–Euphane Group

Several dammarane structures have been solved by *X*-ray analysis. Gymnogenin (83), from *Gymnostemma pentaphyllum*, has a novel side chain containing a cyclopentenone[98] and cleocarpone (84), from *Cleome brachycarpa*, has a side-chain acetal.[99] Salvilymitol (85) and the corresponding 3-ketone, salvilymitone (86), have been isolated from *Salvia hierosolymitana*.[100] An *X*-ray analysis was performed on the trisnor-γ-lactone (87). A natural ant repellant from *Abuta racemosa* has been revealed as (24*S*)-dammara-20,25-diene-3β,24-diol (88) by *X*-ray analysis.[101]

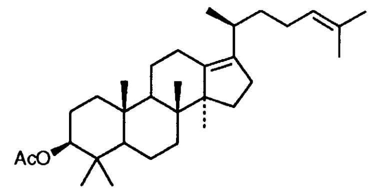
Dysoxylum richii contains richenol (89) and richenone (90) together with the ring-A cleaved derivatives richenoic acid (91) and its methyl ester (92).[102] (20*S*,24*R*)-20,24-Epoxydammarane-3β,12β,25-triol 12-acetate (93) has been isolated from *Notholaena rigida*.[68] Other new dammaranes include semialatic acid (94) from *Rhus semialata*,[103] 3β-acetoxy-(20*S*)-dammara-13(17),24-diene (95) from *Salvia salicifolia*,[104] dammara-



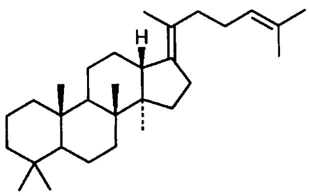
(93)



(94)



(95)



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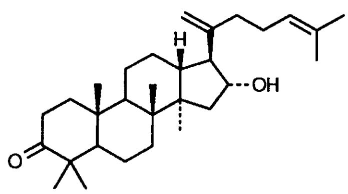

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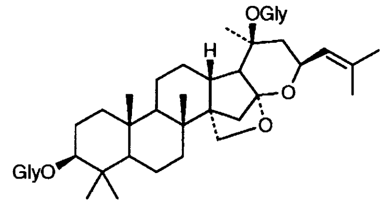
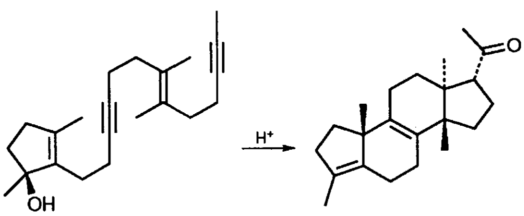
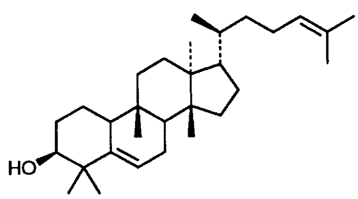


(98)



(99) (100)



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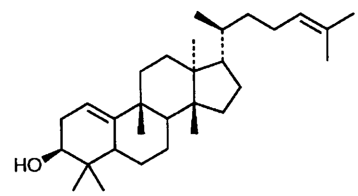

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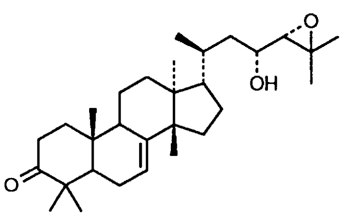
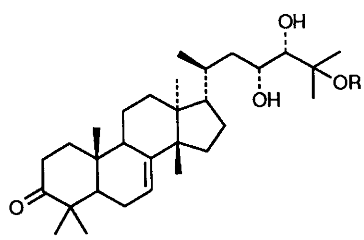
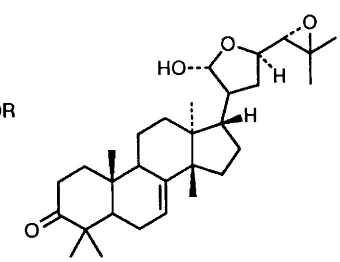


(103)



(104) R = Me
(106) R = H



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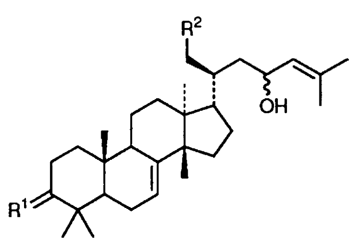
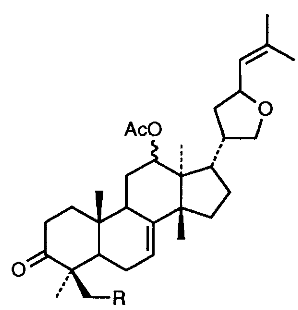


(107) R^1 = H, β-OH; R^2 = H
(108) R^1 = H, β-OH; R^2 = OH
(109) R^1 = O; R^2 = H
(110) R^1 = O; R^2 = OH



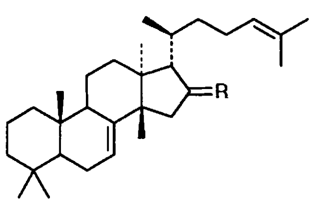
(111) R = H
(112) R = OH

17(20),24-diene (96) from *Polypodium* species,[60] and 16α-hydroxydammara-20,24-dien-3-one (97) from *Elaeodendron buchananii*.[105]

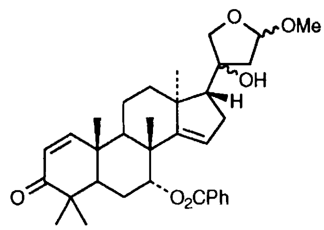
New saponins have been obtained from *Panax pseudo-ginseng*[106] and *P. ginseng*,[107] including ginsenoside La.[108] The structure of ziziphin (98), one of the antisweet jujubosaponins from *Ziziphus jujuba*, has been revised.[109] Capsugenin 25,30-diglucopyranoside has been reported from *Corchorus capsularis*[110] and two dammarane saponins have been found in *Gymnostemma longipes*.[111]

Euphol and tirucallol have been synthesized.[112] Acid-catalysed cyclization of the monocyclic precursor (99) afforded (100) as the major product. Subsequent modification of the latter gave euphol and tirucallol.

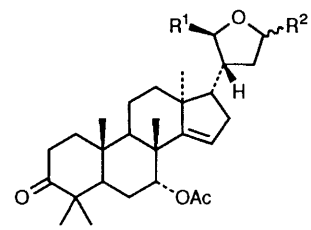
Two rearranged tirucallanes, euferol (101) and melliferol (102), have been isolated from *Euphorbia mellifera* and their structures resolved by *X*-ray analysis.[113] The structure of niloticin (103) has also been established by *X*-ray analysis. It occurs in the fruits of *Phellodendron chinense* together with phellochin (104) and melianone (105).[114] Phellochin (104) is the 25-*O*-methyl derivative of 23,24,25-trihydroxytirucall-7-en-3-one (106) from *Cedrela odorata*.[115] Other new tirucallanes include tirucalla-7,24-diene-3β,23-diol (107), tirucalla-7,24-diene-3β,21,23-triol (108), the corresponding 3-ketones [(109 and (110)] from *Paramignya monophylla*,[116] and prieurone (111) and 29-hydroxyprieurone (112) from *Trichilia*



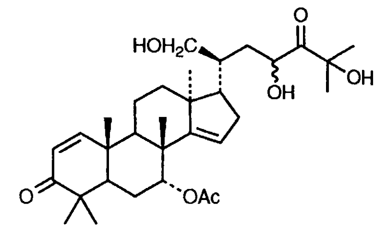
(113) R = H, β-OH
(114) R = O, Δ^8



(115)



(116) R^1 OMe; R^2 = H
(117) R^1 = H; R^2 = OMe



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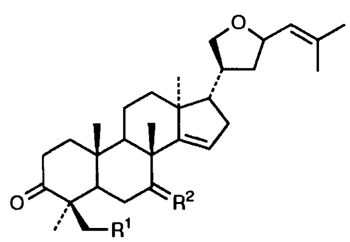
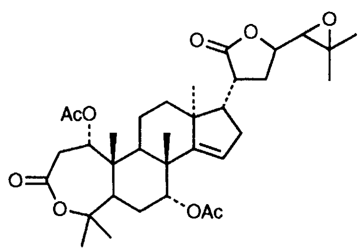


(119) R^1 = OH; R^2 = O
(120) R^1 = OH; R^2 = H, α-OH
(121) R^1 = H; R^2 = O; 1,2-didehydro
(122) R^1 = H; R^2 = α-OH; 1,2-didehydro
(123) R^1 = OH; R^2 = O; 1.2-didehydro



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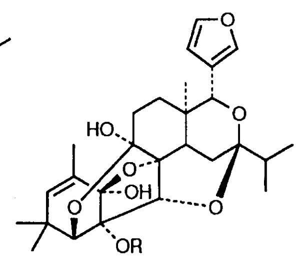
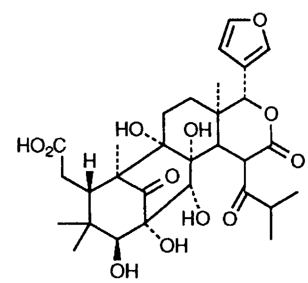


(125) R = H
(126) R = Ac



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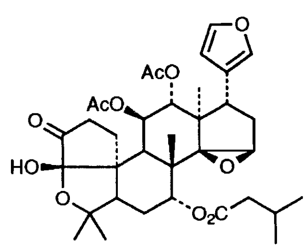

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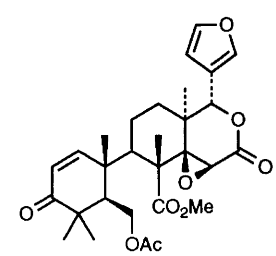

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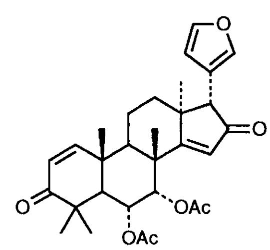

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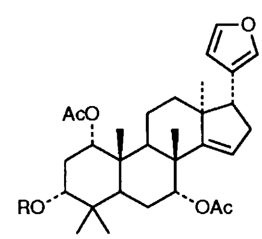
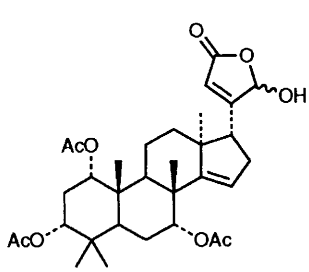


(131) R = H
(132) R = Ac



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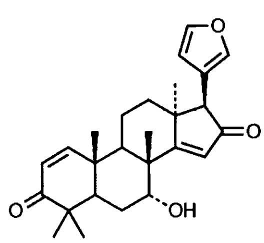
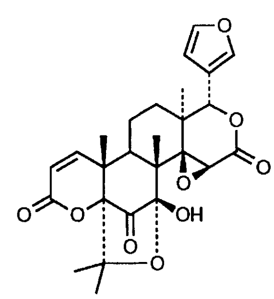
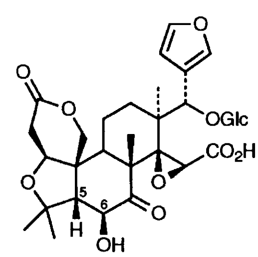


(134)



(135)



(136)
(137) 5,6-Didehydro

prieuriana.[117] Euphol cinnamate has been found in *Euphorbia antiquorum*.[34]

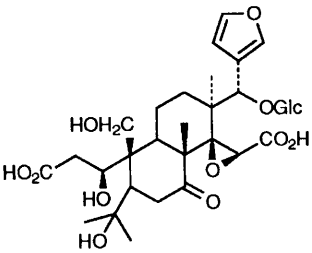
A further group of tirucallanes and apotirucallanes has been isolated from *Azadirachta indica*. The tirucallanes limocinol (113) and limocinone (114) are accompanied by the apo-tirucallanes limocinin (115), limocins A (116) and B (117),[118] and azadiol (118).[119] Five new apo-compounds, dysonones A (119), B (120), C (121), D (122), and E (123), have been found in *Dysoxylum roseum*.[120] Phebaloparvilactone (124) is a ring-A-cleaved derivative from *Phebalium squamulosum*.[121]

4.1 Tetranortriterpenoids

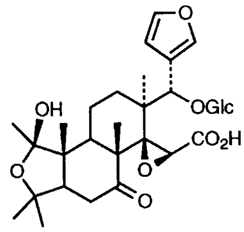
Entilins A (125) and B (126) are highly cleaved heptanor-triterpenoids from the stem bark of *Entandrophragma utile*.[122] The compounds could arise from a 15-acyl derivative (127) by a sequence of retro-aldol, retro-Michael, and decarboxylation processes. Dumsin (128), an insect antifeedant from *Croton jatrophoides*,[123] has an unusual ring-A arrangement. Its structure was solved by *X*-ray analysis. Further tetranortriterpenoids from the seeds of *Swietenia mahogani* include the first 6,7-seco derivative, secomahoganin (129), and 6α-acetoxyazadiradione (130) (mahonin).[124]

14,15-Deoxyhavanesin diacetate (131), the corresponding triacetate (132), and the modified furan derivative (133) have been isolated from *Trichilia havanensis*.[125] 17-Epinimbocinol (134) has been reported from neem oil.[126] An *X*-ray analysis of perforatin from *Harrisonia perforata* revealed the structure (135).[127]

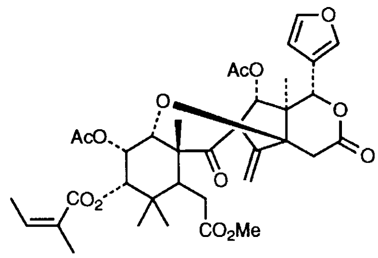
Many limonoid glucosides have been reported including 6-hydroxy-5-epilimonin 17-*O*-β-D-glucopyranoside (136) and limonin diosphenol 17-*O*-β-D-glucopyranoside (137) from



(138)



(139)



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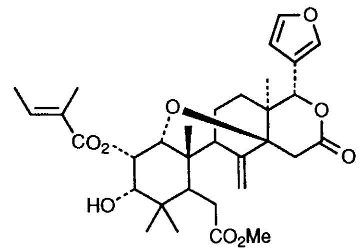
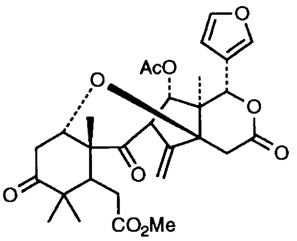
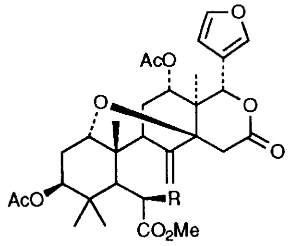


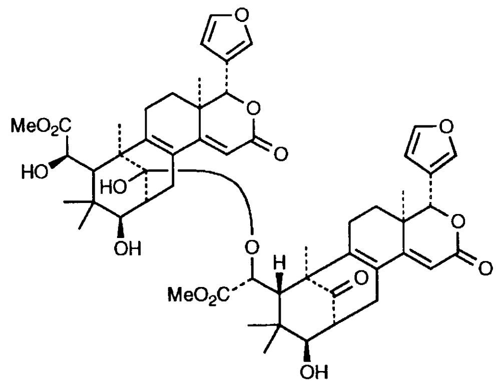
(141)



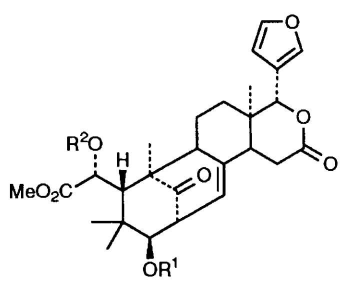
(142)



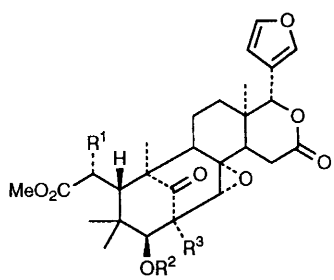
(143) R = H
(144) R = OH



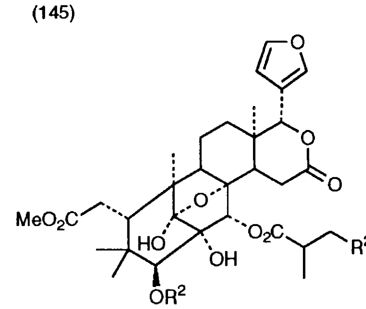
(145)



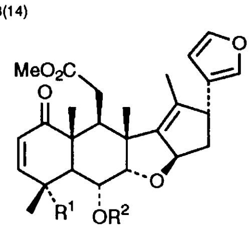
(146) R^1 = $COCH_2CH_3$; R^2 = H
(147) R^1 = $COCH(CH_3)_2$; R^2 = H
(148) R^1 = $COC(CH_3)=CH_2$; R^2 = H
(149) R^1 = $COCH(CH_3)CH_2CH_3$; R^2 = H
(150) R^1 = COPh; R^2 = H
(151) R^1 = Ac; R^2 = H; $\Delta^{8(14)}$
(152) R^1 = H; R^2 = Ac; $\Delta^{8(14)}$
(153) R^1 = Tigloyl; R^2 = Ac; $\Delta^{8(14)}$



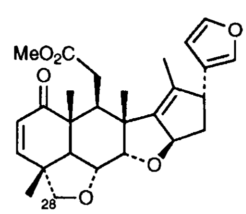
(154) R^1 = OH; R^2 = $COCH_2CH_3$; R^3 = H
(155) R^1 = OAc; R^2 = $COCH_2CH_3$; R^3 = H
(156) R^1 = OAc; R^2 = $COCH(CH_3)_2$; R^3 = H
(157) R^1 = OH; R^2 = Ac; R^3 = H
(158) R^1 = OH; R^2 = tigloyl; R^3 = H
(159) R^1 = OAc; R^2 = tigloyl; R^3 = H
(160) R^1 = OH; R^2 = tigloyl; R^3 = OH
(161) R^1 = H; R^2 = tigloyl; R^3 = H



(162) R^1 = Ac; R^2 = CH_3
(163) R^1 = Ac; R^2 = H
(164) R^1 = R^2 = OC



(165) R^1 = CHO; R^2 = H
(166) R^1 = CH_2OH; R^2 = Ac

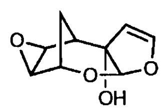


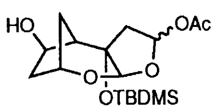
(167)

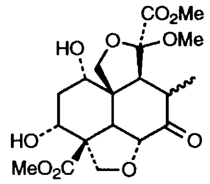
Tetradium rutaecarpa;[128] the 17-*O*-β-D-glucopyranosides of isolimonic acid, ichangic acid, and 19-hydroxydeacetylnomilic acid (138) from *Citrus aurantium*;[129] and of ichangesin (139) from *C. junos*, *C. sudachi*, and *C. sphaerocarpa*.[130] The 4-*O*-β-D-glucopyranosides of ichangin and nomilic acid have been found in *C. limon*.[131] A crystal structure analysis of limonin has appeared.[132]

The methyl angolensate derivative (140) from *Ekebergia pterophylla* has been shown by *X*-ray analysis to have the contracted ring-C skeleton similar to that of trijugins A and B (see *Nat. Prod. Rep.*, 1989, **6**, 483). It is interesting to note that while the crystal structure showed the presence of a dimethylacrylate ester attached to C-3, the ^{1}H and ^{13}C NMR spectra of (140) clearly indicated an angelate. The normal methyl angolensate derivative (141) was also isolated.[133] Trijugin B acetate (142) has been identified in *Heynea trijuga*.[134] *X*-Ray structures have been published for sandoricin (143) and 6-hydroxysandoricin (144) from *Sandoricum koetjape*.[135] Mahagonin (145) is a stable hemiacetal dimer from *Swietenia mahagoni*.[136] It is accompanied by a host of esters of swietenine [B (146), C (147), D (148), E (149), and F (150)], swietenolide [(151), (152), and (153)],[137] and swietemahonin [A (154), B (155), C (156), D (157), E (158), and F (159)].[138,139] The 2-hydroxyderivative swietemahonin G (160) and the 6-deoxy derivative swietemahonolide (161) were also obtained. Two new xyloccensins [I (162) and J (163)] have been isolated from *Xylocarpus granatum* and *X. moluccensis*.[110] They represent trivial ester changes from the original xyloccensin F (164).[141]

Full details of the structure elucidation of 6-deacetylnimbinal (165), nimbinol (166), and 28-deoxonimbolide (167) from


(168)


(169)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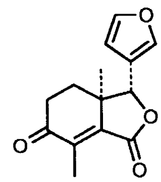
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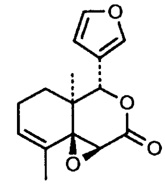
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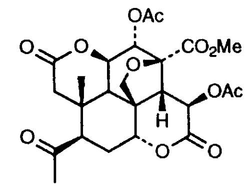
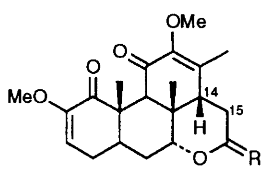
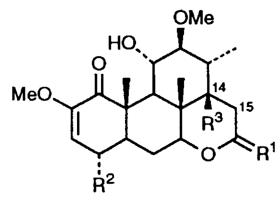
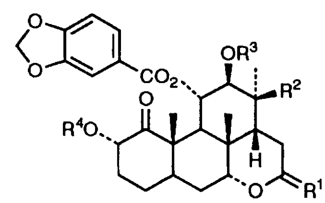
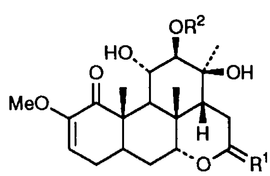
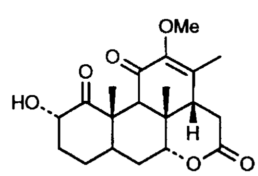

(173)

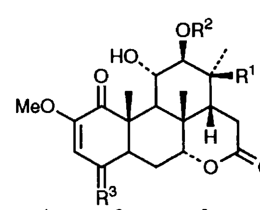

(174) R = H_2,OH
(176) R = O
(179) R = O; 14,15-didehydro

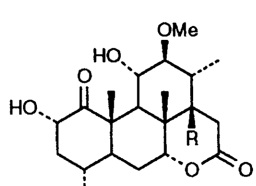

(175) R^1 = H, β-OMe; R^2 = R^3 = H;
(182) R^1 = H, α-OH; R^2 = R^3 = H
(184) R^1 = O; R^2 = R^3 = H; 14,15-didehydro
(185) R^1 = O; R^2 = H; R^3 = OH
(187) R^1 = O; R^2 = OH; R^3 = H
(192) R^1 = H, OH; R^2 = CH_3; R^3 = H

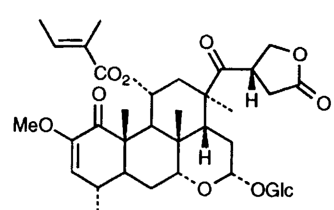

(177) R^1 = O; R^2 = H; R^3 = R^4 = Me
(180) R^1 = O; R^2 = OH; R^3 = R^4 = Ac
(181) R^1 = O; R^2 = H; R^3 = Me; R^4 = PhCO
(183) R^1 = O; R^2 = H; R^3 = Me; R^4 = Ac
(188) R^1 = O; R^2 = R^4 = H; R^3 = Me
(196) R^1 = H, α-OGlc; R^2 = OH; R^3 = R^4 = Ac
(199) R^1 = H, α-OGlc; R^2 = H; R^3 = Me; R^4 = Ac
(200) R^1 = H; α-OGlc; R^2 = H; R^3 = R^4 = Me
(201) R^1 = H, α-OGlc; R^2 = OH; R^3 = Ac; R^4 = H


(178) R^1 = O; R^2 = Me
(186) R^1 = H, α-OMe; R^2 = H
(197) R^1 = H, α-OGlc; R^2 = Me


(189)


(190) R^1 = H; R^2 = Me; R^3 = O
(191) R^1 = OH; R^2 = H; R^3 = O
(193) R^1 = OH; R^2 = Ac; R^3 = H, α-CH_3


(194) R = H
(195) R = OH

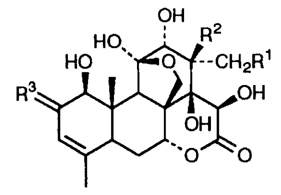

(198)

Azadirachta indica have appeared.[142] Several novel rearrangement reactions of azadirachtin and related derivatives have been described.[143] The epoxyhydroxyacetal (168) has been synthesized in optically active form both as a model compound for azadirachtin[144] and also as an intermediate [as (169)] for the synthesis of azadirachtin.[145,146] The highly functionalized decalin fragment (170) of azadirachtin has been synthesized.[147,148]

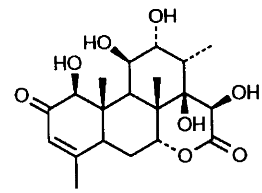
The mass-spectral fragmentation of some limonoids has been discussed.[149] The structure of fraxinellonone (171), a degraded limonid from *Fagaropsis glabra*, has been published.[150] Another degraded limonoid, calodendrolide (172), has been synthesized.[151]

4.2 Quassinoids

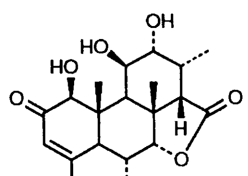
Javanicin (173) is a ring-A cleaved quassinoid from *Brucea javanica*.[152] Its structure was established by *X*-ray analysis. The javanicin series of norquassinoids from *Picrasma javanica* has been considerably extended[153] with the isolation of B (174), E (175), F (176), G (177), H (178), I (179), J (180), K (181), L (182), N (183), O (184), P (185), Q (186), R (187), S (188), T (189), X (190), and Y (191). Javanicins M (192), U (193), V (194), and W (195) still retain the 4α-methyl group. The 15-*O*-glucoside of javanicin B (174) is called javanicinoside C while javanicinoside B has structure (196). These authors should give more thought to their nomenclature. Other javanicinosides, which bear no relationship to the corresponding javanicin aglycones, are D



(202) $R^1 = R^2 = H$; $R^3 = H$, α-OH
(203) $R^1 = R^2 = H$; $R^3 = O$
(204) $R^1 = R^2 = OH$; $R^3 = O$



(205)



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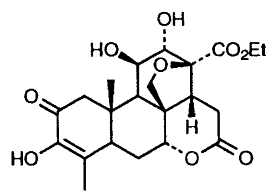

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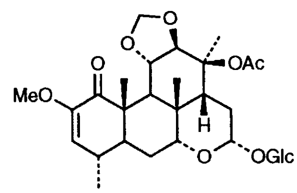

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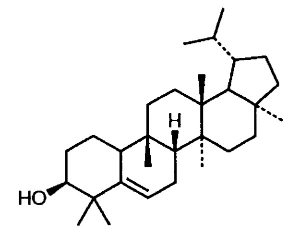
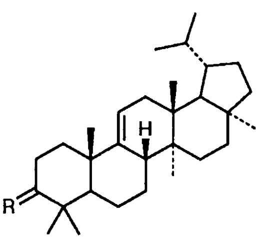
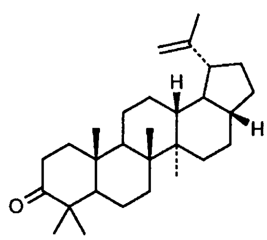


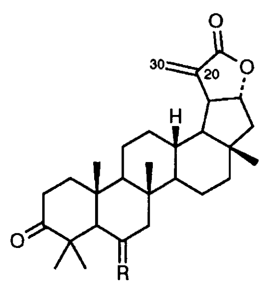
(209)



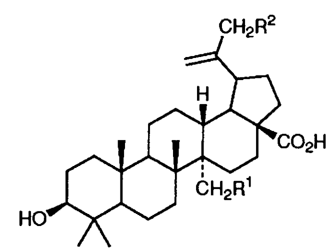
(210) R = O
(211) R = H, β-OH
(212) R = H, β-$O_2CC_{25}H_{51}$



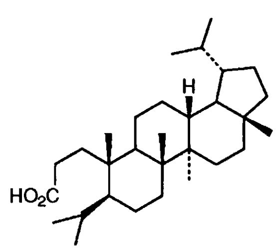
(213)



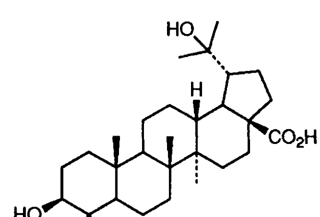
(214) R = H,H
(215) R = H,H; 20,30-epoxide
(216) R = O



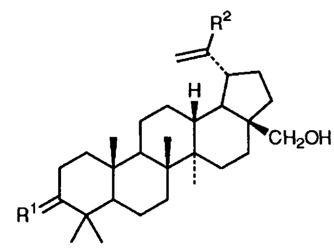
(217) $R^1 = OH$; $R^2 = H$
(219) $R^1 = H$; $R^2 = OH$



(218)



(220)



(221) $R^1 = H$, β-OH; $R^2 = CHO$
(222) $R^1 = O$; $R^2 = CH_3$

(197), E (198), F (199), G (200), and H (201).[154] 13β,18-Dihydroeurcomanol (202),[155] the corresponding 2-ketone, 13β,18-dihydroeurycomanone (203), 13β,18-dihydroxyeurycomanone (204), 14β,15β-dihydroxyklaineanone (205), and longilactone (206) from *Eurycoma longifolia*,[156] bruceine I (207) from *Brucea javanica*,[157] and picrasinoside H (208) from *Picrasma ailanthoides*[158] are further new quassinoids.

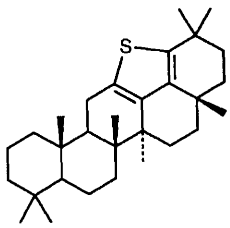
Synthetic approaches to quassinoids[159] and total syntheses of chaparrinone[160] and amarolide[161] have been reported. An enzyme-linked immunosorbent assay has been developed for the determination of picomole quantities of quassinoids.[162] The quassinoids have been reviewed.[163]

5 The Lupane Group

Hancokinol (209), hancolupenone (210), and hancolupenol (211) and its hexacosanoate (212) from *Cynanchum hancokianum* represent new skeletal types.[164] The structures of hancokinol (209) and hancolupenone (210) were established by *X*-ray analysis.

28-Norlup-20(29)-en-3-one (213) occurs in *Pistacia lentiscus* resin.[165] The γ-lactone ochraceolide A (214), its epoxide ochraceolide B (215), and the 6-ketone ochraceolide C (216) have been isolated from *Kokoona ochracea*.[166] The C-27-oxygenated lupane cylicodiscic acid (217) is the genin[167] of the saponin cylicodiscoside[168] from *Cylicodiscus gabunensis*. Other lupanes include dihydrocanaric acid (218) and its methyl ester from *Hoya naumanii*;[169] 3β,29-dihydroxylup-20(30)-en-28-oic acid (219) from *Relhania genistifolia* and 3β,20-dihydroxy-lupan-28-oic acid (220) from *R. calycina*;[170] 3β,28-dihydroxy-lup-20(30)-en-29-al (221) and betulone (222) from *Betula lenta*;[171] and betulinic acid and epibetulinic acid 3-*O*-sulfates from *Schefflera octophylla*.[172] The following lupane saponins have been investigated: leucasin from *Leucas nutans*;[173] menyanthoside from *Menyanthes trifoliata*;[174] two glycosides from *Paliurus ramosissimus*;[175] anemosides A_3 and B_4,[176] pulchinenosides A, B, and C,[177] and another saponin[178] from *Pulsatilla chinensis*; and saponins from *Schefflera octophylla*.[179-182]

Lupane, 24-norlupane, 28-norlupane, and 24,28-dinorlupane have been synthesized from betulin[182] as standards for the identification of lupanes in oil samples.[184]



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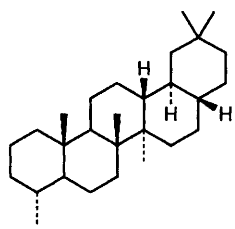
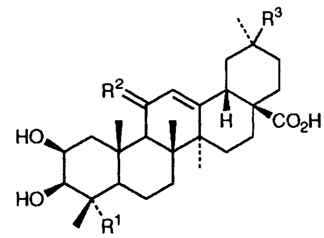
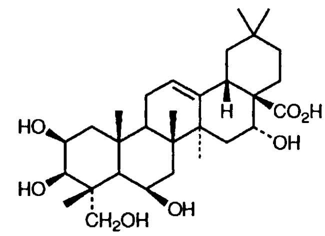


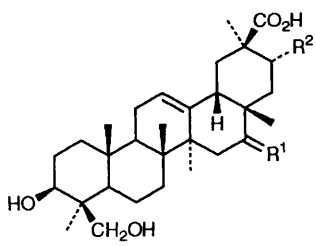
(224)



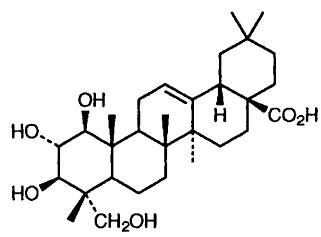
(225) $R^1 = CH_2OH$; $R^2 = H,H$; $R^3 = CH_2OH$
(226) $R^1 = CH_2OH$; $R^2 = O$; $R^3 = CO_2Me$
(227) $R^1 = CO_2Me$; $R^2 = H,H$; $R^3 = CO_2Me$



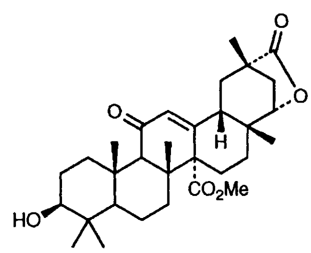
(228)



(229) $R^1 = O$; $R^2 = H$
(230) $R^1 = H,H$; $R^2 = OH$



(231)



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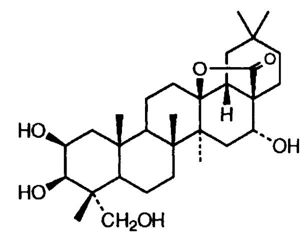
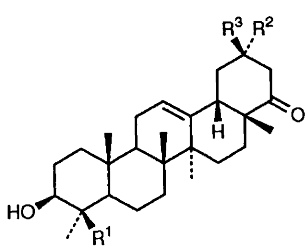
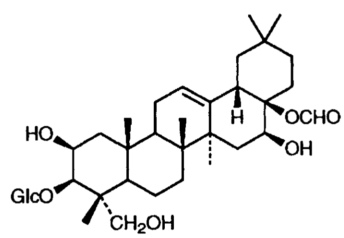


(233)



(234) $R^1 = R^2 = R^3 = CH_2OH$
(235) $R^1 = R^2 = CH_2OH$; $R^3 = CH_3$
(236) $R^1 = R^2 = CH_3$; $R^3 = CO_2H$
(237) $R^1 = R^3 = CH_3$; $R^2 = CO_2H$



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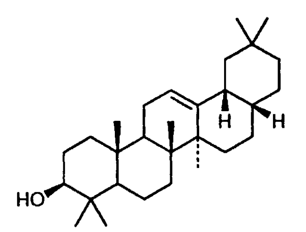
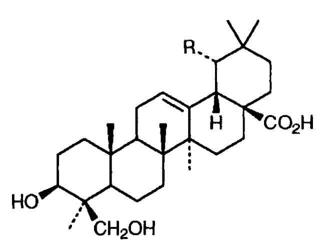
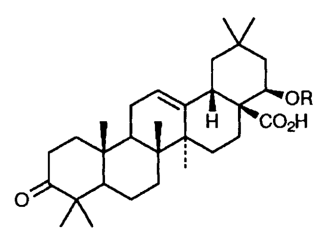


(239)



(240) R = OH
(243) R = H



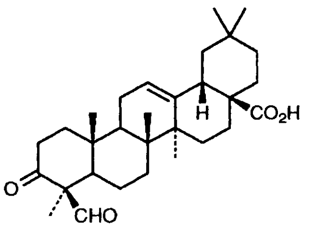
(241) R = Angeloyl
(242) R = $OCCH(CH_3)_2$

6 The Oleanane Group

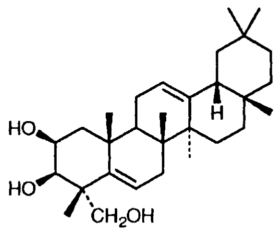
The novel thiophene derivative oleana-12,18-diene-12,19-thiirane (223) has been detected in sediments deposited under anaerobic evaporitic conditions.[185] The thiophene (223) can be prepared by heating β-amyrin acetate with elemental sulfur followed by removal of the 3-oxygen function. 24,28-Dinor-18α-oleanane (224) is a constituent of Egyptian petroleum.[186] A series of 24-noroleanenes has been synthesized by acid-catalysed rearrangement of 24-norolean-12-ene and identified in oil samples.[184]

The genins of the saponins esculentosides J and M from *Phytolacca esculenta* are esculentagenic acid (225)[187] and esculentagenin (226),[188] respectively. Acinospesigenin (227) is a new genin from *P. acinosa*[189] whereas 16α-hydroxyprotobassic acid (228) is the genin of tridesmosaponins A and B, complex saponins from *Tridesmostemon claessenssi*.[190] Glyyunnan-sapogenins A (229) and B (230) have been isolated from *Glycyrrhiza yunnanensis*[191] and mucunagenin a (231) has been found in *Mucuna birdwodiana*.[192] Two γ-lactone derivatives, glyuranolide (232) from *Glycyrrhiza uralensis*[193] and grindeliasapogenin D (233) from *Grindelae herba*,[191] have been isolated. Subprogenin B (234) is unusual in having three primary hydroxyl groups. It occurs in the root of *Sophora subprostrata* with subprogenins A (235), C (236), and D (237).[195] Vicoside A (238) from *Vicoa indica* is a 28-noroleanane glycoside with a 28-*O*-formyl group.[196] The formation of 28-nor compounds could involve such intermediates. 28-Norolean-12-en-3β-ol (239) has been reported from *Pistacia lentiscus* resin[165] and its structure confirmed by *X*-ray analysis of the corresponding 3-ketone, which had previously been isolated from sediments.[197] The 19α-hydroxyoleanane derivative spathodic acid (240) is a constituent of *Spathodea campanulata*.[198]

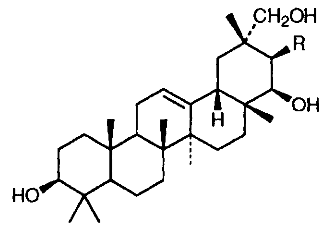
The structure of lantadene A (241) from the red variety of *Lantana camara* has been confirmed by *X*-ray analysis.[199] Lantadene D (242) is the corresponding isobutyrate from *L. camara* var. *aculeata*.[200] 3β,24-Dihydroxyolean-12-en-28-oic acid (243) from *Lantana indica*[201] appears to be identical with



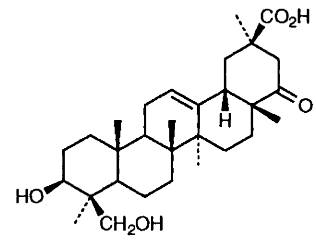
(244)



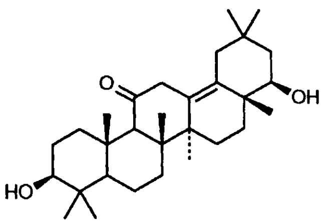
(245)



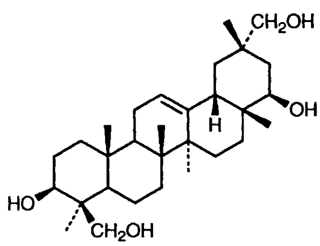
(246) R = H
(247) R = OH



(248)



(249)



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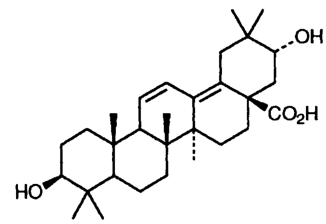

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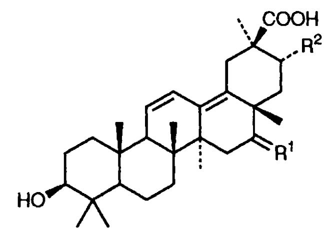
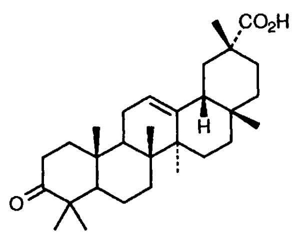


(252) R^1 = O; R^2 = H
(253) R^1 = H,H; R^2 = OH



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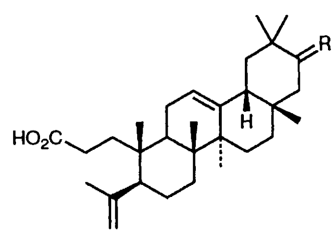
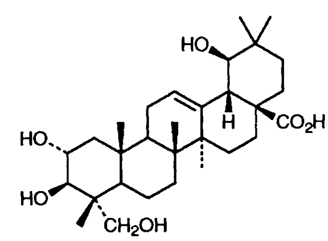
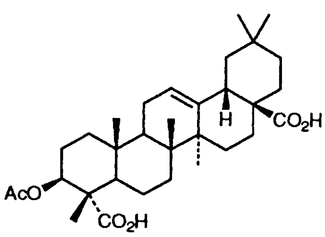


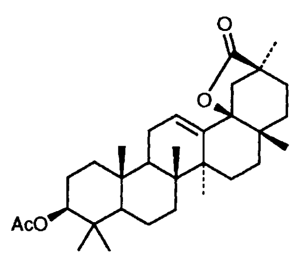
(255) R = O
(256) R = H,H



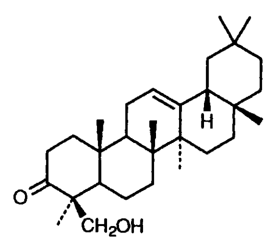
(257)



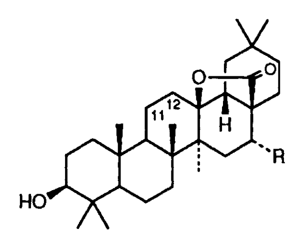
(258)



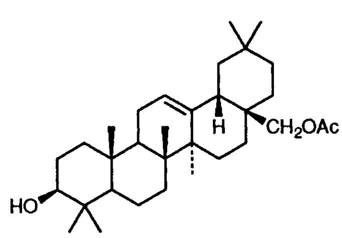
(259)



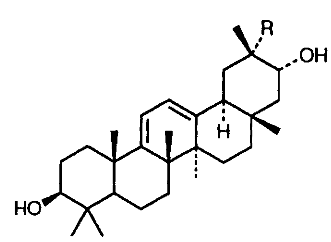
(260)



(261) R = H; 11,12-didehydro
(262) R = OH



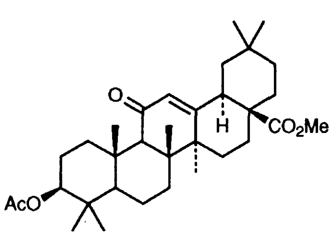
(263)



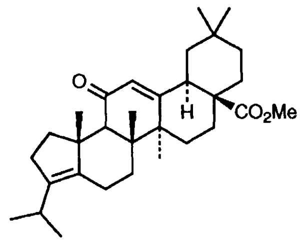
(264) R = CH_2OH
(265) R = CO_2H

bredemolic acid isolated from *Bredemeyera floribunda* in 1960.[202] The corresponding 3,24-dioxo derivative (244) has also been isolated from *L. indica*.[203] Butyraceol (245) is an oleana-5,12-diene from *Madhuca butyracea*.[204] Abrisapogenols A (246), B (247), and I (248) have been obtained from *Abrus cantoniensis* where they occur with abrisaponin I whose genin is (248).[205] Abrisapogenol J (249) has been obtained from the seeds of *A. precatorius*.[206] During the study of the saponins of *Oxytropis glabra*, a new aglycone, oxytrogenol (250), was obtained.[207] The 11,13(18)-diene (251) has been found in *Glycyrrhiza echinata* roots[208] and the 11,13(18)-dienes glyyunnansapogenins C (252) and E (253) are constituents of *G. yunnanensis*.[209] Katononic acid (254) has been reported from *Austroplenckia populnea*.[210] The 3,4-seco derivative (255) has been isolated from *Dacryodes normandii*[211] and the 3,4-seco derivatives dihydronyctanthic acid (256) and its methyl ester have been found in *Hoya naumanii*.[169]

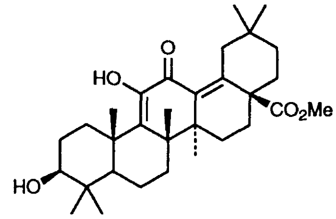
Crystal structure of tomentosic acid (257) from *Terminalia bellerica*[212] and gypsogenic acid acetate (258) from *Opilia celtidifolia*[213] have been published. A detailed spectroscopic study of soyasapogenol B has appeared.[214] Other new oleananes include 3β-acetoxyolean-12-en-30,18β-olide (259) from *Cornulaca monacantha*,[215] 24-hydroxyolean-12-en-3-one (260) from *Symplocos racemosa*,[216] 3β-hydroxyolean-11-en-28,13β-olide (261) from *Hyptis albida*,[217] leucolactone (262) from *Leucas aspea*,[218] and 28-acetoxyerythrodiol (263) from *Erythrina eriotriocha*.[219] The 18α-oleanadienes glyyunnansapogenins G (264) and H (265) have been obtained from *Glycyrrhiza*



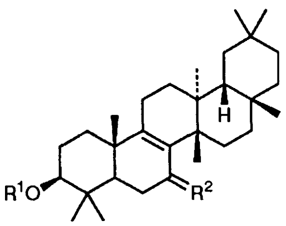
(266)



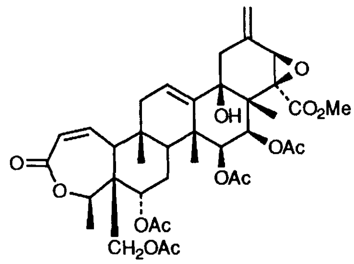
(267)



(268)



(269) $R^1 = Ac$; $R^2 = H,H$
(270) $R^1 = H$; $R^2 = O$



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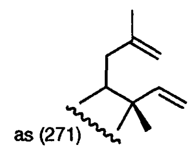
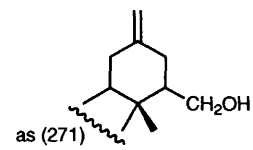


(272)



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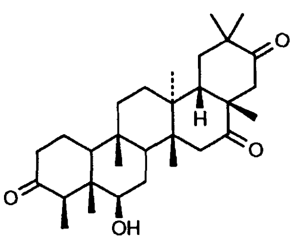

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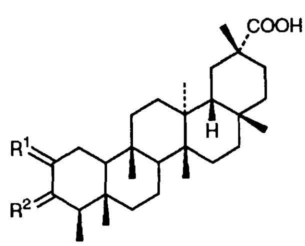
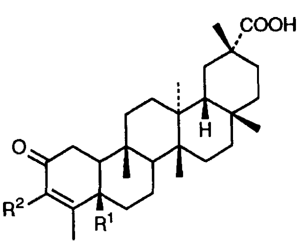
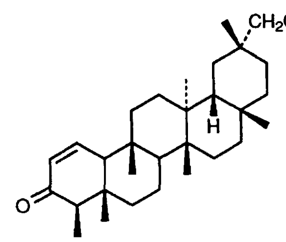


(275) $R^1 = O$; $R^2 = \beta$-OH, H
(276) $R^1 = H,H$; $R^2 = O$



(277) $R^1 = CH_3$; $R^2 = OH$
(278) $R^1 = CHO$; $R^2 = OH$
(280) $R^1 = CH_3$; $R^2 = H$



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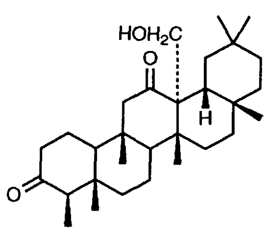
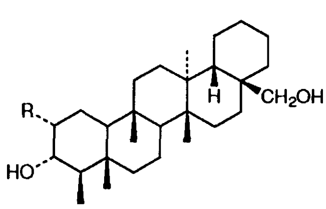
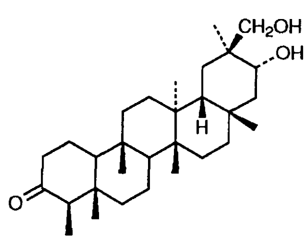


(281)



(282) R = OH
(283) R = H



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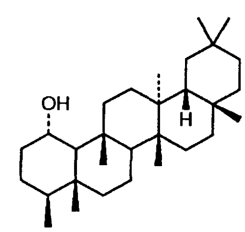


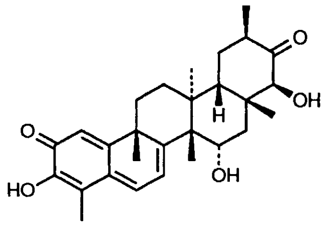
(285)

yunnanensis.[220] During the preparation of methyl 3β-acetoxy-11-oxo-18α-olean-12-en-28-oate (266) by treatment of the corresponding 18β-compound with HBr, the ring-A contracted product (267) was obtained. Alkaline hydrolysis of (266) afforded the diosphenol (268) in addition to the expected product. The structure of the diosphenol (268) was confirmed by *X*-ray analysis.[221] A similar ring-A contracted derivative has been obtained from glycyrretic acid during studies on the preparation of glycosides of glycyrretic acid for immunoassay purposes.[222]

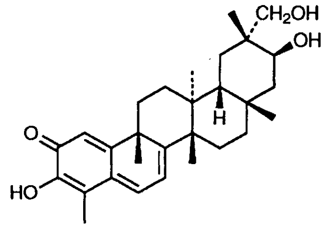
Taraxeryl *cis*-*p*-hydroxycinnamate has been reported from *Rhizophora apiculata*.[223] The *trans*-isomer, careaborin, is already known.[224] Isomultiflorenyl acetate (269) has been found in *Benincasa cerifera*[225] and 3β-hydroxymultiflor-8-en-7-one (270) in *Euphorbia supina*.[226]

The unusual friedelane derivative (271), with a rearranged ring E, has been isolated from *Lophanthera lactescens*.[227] A Prins reaction of a ring-E cleaved derivative [(272) $\rightarrow$ (273)] is proposed for the biosynthetic origin of this system. *X*-Ray crystal structures of several friedelanes have appeared, including maytensifolin (274) from *Maytenus diversifolia*;[228] 3β-hydroxy-2-oxofriedelan-29-oic acid (275),[210] 3-oxofriedelan-29-oic acid (276),[229] and 3-hydroxy-2-oxofriedel-3-en-29-oic acid (277)[230] from *Austroplenckia populnea*; and cangoronine (278) from *Maytenus ilicifolia*. Cangoronine (278) is accompanied by ilicifoline (279).[231] Among other new friedelanes are 2-oxofriedel-3-en-29-oic acid (280) from *Schaefferia cuneifolia*;[232] pristimeronol (281)[233] and pristriol (282) and its 3-deoxy-derivative (283)[234] from *Pristimera grahamii*; 21α,30-dihydroxyfriedelan-3-one (284) from *Salacia reticulata*;[235] and friedelan-1α-ol (maculaniol) (285) from the fungus *Leptosphaeria maculans*.[236]

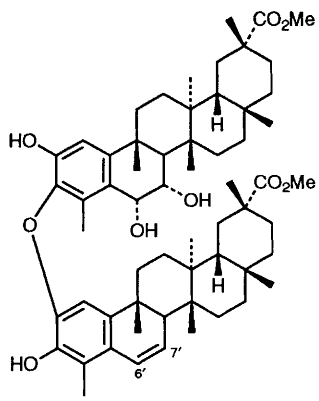
Further evidence for the structure of celastranhydride (see *Nat. Prod. Rep.*, 1993, **11**, 107) from *Kokoona zeylanica* has



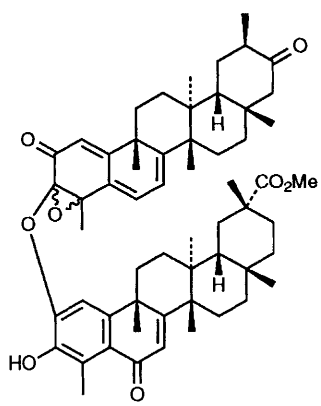
(286)



(287)



(288)
(289) 6′,7′-dihydro



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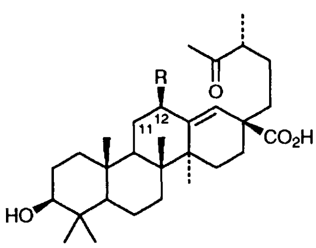
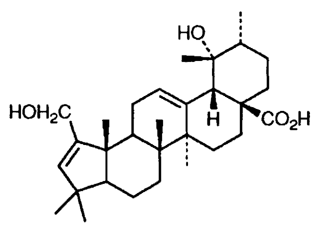


(291) R = H; Δ^{11}
(292) R = OH



(293)

been presented.[237] The new quinone methides 15α,22β-dihydroxytingenone (286)[238] and excelsine (287)[239] have been obtained from *Cassine balae* and *Hippocratea excelsa* respectively. Several bis-friedelanes have been isolated from *Maytenus ilicifolia*, including cangorisin A (288), its atropisomer, atropcangorisin A, dihydroatropcangorisin A (289), and cangorisin B (290).[240] Complete ^{1}H and ^{13}C NMR assignments[241] and a crystal structure[242] of friedelin and ^{13}C NMR assignments of some friedelanes and secofriedelanes[243] have been published

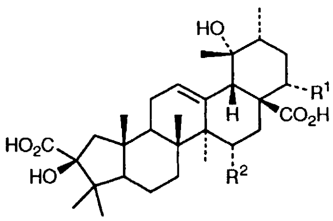
The 3-*O*-glucopyranoside of phytolaccagenic acid has been isolated from *Diploclisia glaucescens*[244] and the 28-*O*-glucopyranosyl ester of barrinic acid from *Barringtonia acutangula*.[245] New oleanane saponins that have been isolated are detailed in Table 1. New oleanane saponins have also been isolated from the following species: *Albizzia lucida*,[307] *Amaranthus hypochondriacus*,[308] *Anagallis arvensis*,[309] *Aralia elata*,[310] *Astrantia major*,[311] *Calendula arvensis*,[312] *Centipeda minima*,[313] *Chenopodium guinoa*,[314] *Crossopteryx febrifuga*,[315] *Crotalaria albida*,[316] *Cylicodiscus gabunensis*,[317] *Deeringia amaranthoides*,[318] *Dipsacus asper*,[319] *Eleutherococcus senticosus*,[320] *Glycine max*,[321,322] *Gypsophila paniculata* and *G. arrostii*,[323] *Isertia haenkeana*,[324] *Kalopanax pictum*,[325] *Lanenaria breviflora*,[326] *Lonicera japonica*,[327] *Mimosa tenuiflora*,[328] *Oxytropis glabra*,[77] *Polyscias dichroostachya*,[329] *Randia dumetorum*,[330] *Solidago canadensis*,[331] and *Wedelia calendulaceae*.[322]

Table 1 New oleanane saponins

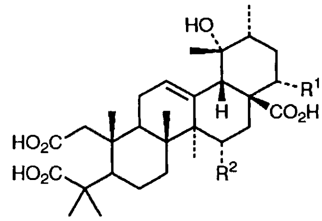
Compound(s)	Source	Ref.
Acutosides A–I	*Luffa acutangula*	246, 247
Amaranthus saponins I–IV	*Amaranthus hypochondacus*	248
Anagallisins A, B, D, E	*Anagalus arvensis*	249
Araloside D	*Aralia chinensis*	250
Astersaponins E, F	*Aster tataricus*	251
Astersaponins Ha–Hd	*Aster tataricus*	252
Bellissaponins BA_1, BA_2	*Bellis perennis*	253
Boussingosides A_1, A_2, B, C, D_1	*Boussingaultia baselloides*	254, 255
Buddlejasaponins I–IV	*Buddleja japonica*	256
Clematoside S	*Clematis grata*	257
Clemontanoside B	*Clematis montana*	258
Crocosmiosides C–I	*Crocosmia crocosmiiflora*	259
Denticin, Denticulin	*Primula denticulata*	260
Deutziacosides A, B	*Deutzia corymbosa*	261
Dubiosides D–F	*Theandiantha dubia*	262
Emarginatosides B, C	*Sapindus emarginatus*	263
Esculentosides K and L	*Phytolacca esculenta*	264
Flaccidosides I–III	*Anemone flaccida*	265, 266
Gigantea saponins 1–4	*Solidago gigantea*	267
Glyeurysaponin	*Glycyrrhiza eurycarpa*	268
Guaiascins C–F, H–L	*Guaiacum officinale*	269, 270
Gymnemasaponins I–V	*Gymnema sylvestre*	271
Gymnemic acids VIII, IX	*Gymnema sylvestre*	272
Hederasaponins E, F, H, I	*Hedera helix*	273
Hederosides A_1, A_2, C, D_1, D_2, E_1, G, H_1, H_2, I	*Hedera taurica*	274–276
Helianthosides 1–3	*Helianthus annuus*	277
Hemslosides G1, G2	*Hemsleya graciliflora*	278
Hispidacin	*Medicago hispida*	279
Kalopanax saponin C	*Kalopanax septemlobus*	280
Kalopanax saponin G	*Glycyrrhiza uralensis*	281
Kalopanax saponins JLa, JLb	*Kalopanax pictus*	282
Licorice saponins F2, G2, H2, J2, K2	*Kalopanax pictus*	283
Lucyosides N, P	*Luffa cylindrica*	284
Macranthoside I	*Lonicera macranthoides*	285
Malonylsaikosaponins a, d	*Bupleurum falcatum*	286
Medicoside L	*Medicago sativa*	287
Mimonosides A, B	*Mimosa tenuifolia*	288
Mubenoside A	*Stauntonia hexaphylla*	289
Periandradulcins A–C	*Periandra dulcis*	290
Phaseoluside A	*Phaseolus vulgaris*	291
Polycarponoside A	*Polycarpone loeflingiae*	292
Polysciasaponin P_1	*Polyscias scutellaria*	293
Pseudoginsenoside RI_2	*Panax pseudo-ginseng*	294
Pulsatilosides A–C	*Pulsatilla campanella*	295
Scaberosides A_1–A_4, B_1–B_6	*Aster scaber*	296, 297
Sigmoisides A, B	*Erythrina sigmoidea*	298
Solidagosaponins I–IX	*Solidago virga-aurea*	299
Songarosaponins A–C	*Verbascum songaricum*	300
Tragopogonsaponins A–R	*Tragopogon porrifolius*	301
Triplosides A–C	*Triplostegia grandiflora*	302
Wistariasaponins D, G	*Wistaria brachybotrys*	303
Yemuosides I, YM_8, YM_9	*Stauntonia chinensis*	304, 305
Yiyeliangwanosides I and II	*Nothopana davidii*	306

7 The Ursane Group

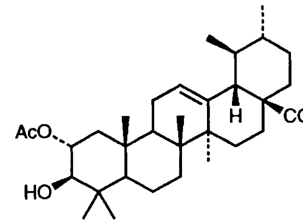
The unusual 18,19-cleaved ursanes α-ilexanolic acid (291) and β-ilexanolic acid (292) are the genins of a series of glycosides, ilexosides A–I, from the fruits of *Ilex crenata*.[333,334] Some of the ilexosides exhibit antiallergenic activity. The ring-A contracted structure (293) has been assigned both to hyptadienic acid from *Hyptis suaveolens*[335] and to coleonolic acid from *Coleus forskohlii*.[336] Chronologically the former name should take



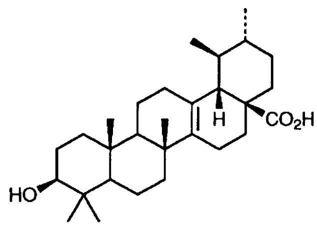
(294) R^1 = OH; R^2 = H
(295) R^1 = H; R^2 = OH



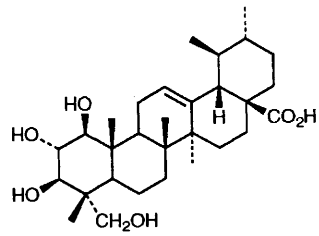
(296) R^1 = OH; R^2 = H
(297) R^1 = H; R^2 = OH



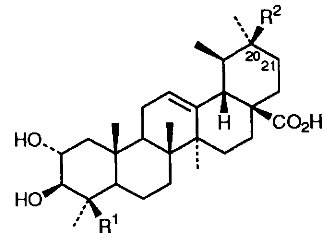
(298)

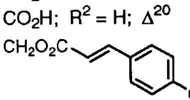


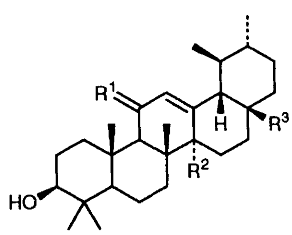
(299)

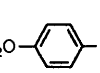
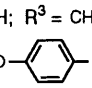


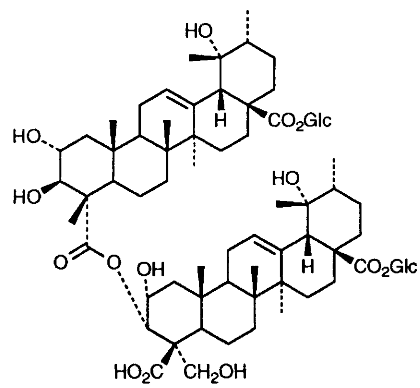
(300)



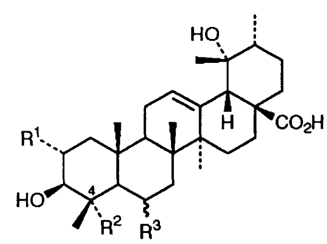
(301) $R^1 = CO_2H$; R^2 = OH
(302) $R^1 = CO_2H$; R^2 = H; Δ^{20}
(303) $R^1 = CH_2O_2C$  OH ; R^2 = H



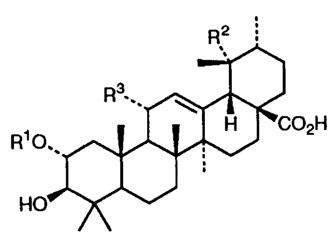
(304) R^1 = O; $R^2 = CH_3$; $R^3 = CO_2H$
(305) R^1 = H,H; $R^2 = CO_2H$; $R^3 = CH_2O$  OH
(306) R^1 = H,H; $R^2 = CH_2O$  OH $R^3 = CO_2H$



(307)



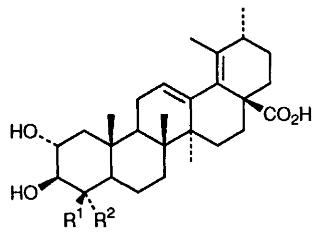
(308) R^1 = OH; $R^2 = CO_2H$; R^3 = H
(309) R^1 = H; $R^2 = CH_3$; R^3 = α-OH
(310) R^1 = H; $R^2 = CH_3$; R^3 = β-OH
(311) R^1 = H; R^2 = CHO; R^3 = β-OH
(312) $R^1 = R^2$ = H; R^3 = β-OH; $\Delta^{4(24)}$
(313) R^1 = H; $R^2 = CO_2H$; R^3 = β-OH



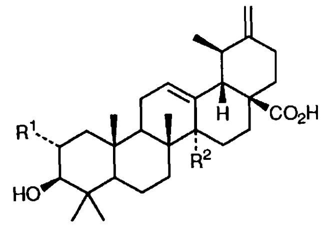
(314) R^1 = H; $R^2 = R^3$ = OH
(315) $R^1 = CH_3$; $R^2 = R^3$ = H

preference. A different kind of contracted ring A is found in musancropic acids A (294) and B (295) from *Musanga cecropioides*.[337] Presumably a benzilic acid rearrangement of a 2,3-diketone precursor is involved in the ring-contraction process. The same species, *M. cecropioides*, is the source of the 2,3-seco compounds musangic acids A (296) and B (297)[338] (*cf.* cecropiacic acid, see *Nat. Prod. Rep.*, 1989, **6**, 493) and cecropic acid (298).[339] Pyrocincholic acid (299), a 27-norursane derivative, occurs in *Isertia haenkeana* as a glycoside.[340] Mucunagenin b (300) occurs in *Mucuna birdwoodiana* with four ursane saponins.[192] A 20β-hydroxyursane, cordepressic acid (301), and its 24-*O*-β-D-galactosyl ester, cordepressin, have been isolated from *Corchorus depressus* together with cordepressenic acid (302), the anhydro derivative of (301).[341] Four ursanes, obtusinin (303), obtusilin (304), and the *p*-hydroxyphenyl ethers obtusidin (305) and obtusinidin (306), have been obtained from *Plumeria obtusa*.[342] Obtusilin (304) together with its acetate have also been reported from *Bursera delpechiana*.[343]

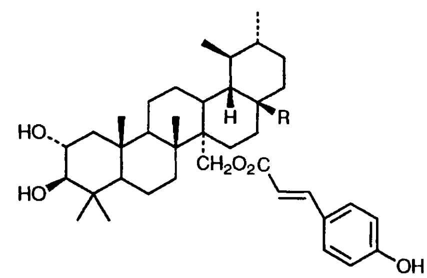
Coreanoside F1 (307) is a bis-ursane glycoside from *Rubus coreanus*.[344] Other 19α-hydroxyursanes include vismiafolic acid (308) from *Vochysia vismiafolia*;[345] 3β,6α,19α-trihydroxyurs-12-en-28-oic acid (309) from *Eriobotrya japonica*;[346] the 6β-epimer (310) of (309), the corresponding 23-aldehyde (311), and the 23-nor derivative (312),[347] 3β,6β-dihydroxyurs-12-ene-23,28-dioic acid (313), and a quinovic acid saponin[348] from *Uncaria tomentosa*; and 11α-hydroxytormentic acid (314), the 6-*O*-methyl-β-D-glucopyranosyl ester of tormentic acid, and nepetoic acid (315) from *Rosa laevigata*.[349] Nepetoic acid (315) was



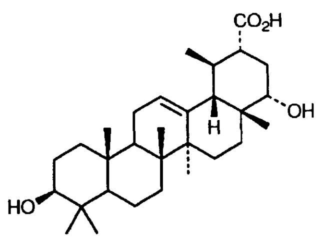
(316) $R^1 = R^2 = CH_3$
(317) $R^1 = CH_3$; $R^2 = H$
(318) $R^1 = H$; $R^2 = CH_3$



(319) $R^1 = OH$; $R^2 = CH_3$
(320) $R^1 = H$; $R^2 = CO_2H$



(321) $R = CO_2H$
(322) $R = CH_3$



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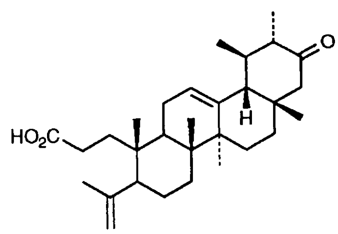

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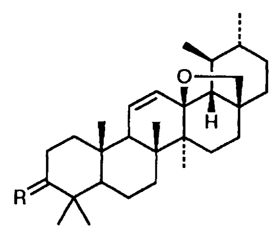
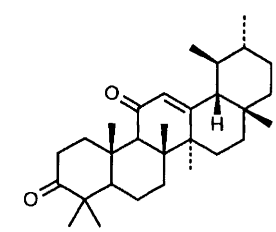


(325) R = O
(326) R = β-OH, H



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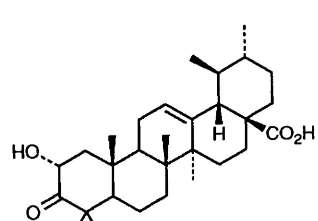

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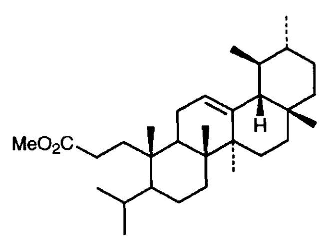

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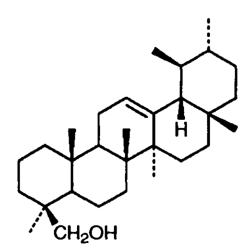

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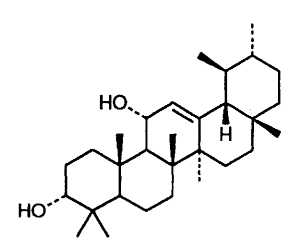

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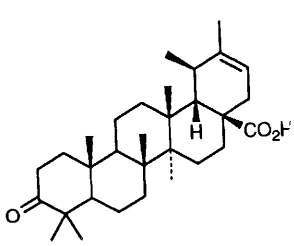

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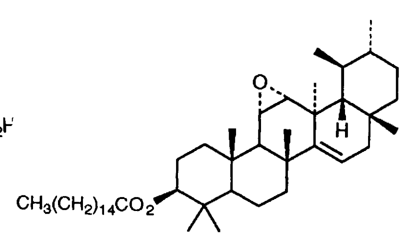
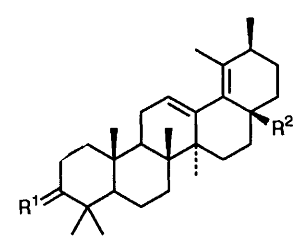
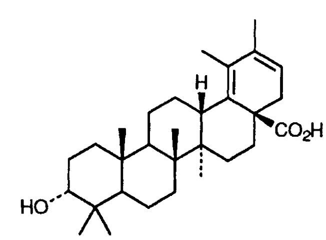


(333)



(334) R^1 = β-OAc, H; $R^2 = CH_2OH$
(335) R^1 = O; $R^2 = CO_2H$
(336) R^1 = β-OH, H; $R^2 = CO_2H$

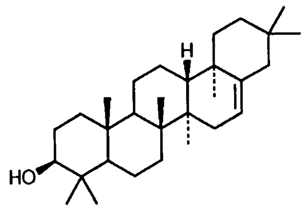


(337)

first reported from *Nepeta eriostachia*.[350] Three ursadienes, goreishic acids I (316), II (317), and III (318), have been isolated from the Chinese medicine Goreishi, which comprises the faeces of *Togopterus xanthipes*.[351] 2α-Hydroxymicromeric acid (319) has been found in *Terminalia chebula*.[352] A glycoside from *Crossopteryx febrifuga* has the related 12,20(30)-diene aglycone (320).[353] Coumarobtusanoic acid (321) and coumarobtusane (322) are 27-*O*-*p*-hydroxycinnamates from *Plumeria obtusa*.[354] Tripterygic acid (323) is a 30-carboxylic acid from *Trypterygium wilfordii*.[355] 24-Norurs-12-ene has been synthesized and identified in oil samples.[184] Other new ursanes include 21-oxo-3,4-secoursa-4(23),12-dien-3-oic acid (324) from *Dacryodes normandii*;[211] 13,28-epoxyurs-11-en-3-one (325), the corresponding 3β-alcohol (326), and urs-12-ene-3,11-dione (327) from *Salvia mellifera*;[356] 2α-hydroxy-3-oxours-12-en-28-oic acid (328) from *Relhania calycina*;[170] dihydroroburic acid methyl ester (329) from *Hoya naumanii*;[169] urs-12-en-24-ol (330) (pakistanol) from *Abutilon pakistanicum*;[357] and urs-12-ene-3α,11α-diol (331) from *Salvia willeana*.[358] Structure (332), with an extra tertiary methyl group, has been proposed for a compound from *Curculigo orchioides*.[359] Prolonged treatment of acetyl methyl ursolate with hydrogen peroxide in acetic acid results in 15α-hydroxylation.[360]

The quinoasaponins 6–10 from *Chenopodium quinoa*[361] and zygophyllosides A and B from *Zygophyllum propinquum*[362] are ursane saponins. Further new saponins have been obtained from *Centipeda minima*,[313] *Isertia haenkeana*,[324] *Rubus ellipticus*,[363] and *Uncaria guianensis*.[364]

Ecdysanthera rosea contains 11α,12α-epoxy-D-friedours-14-en-3β-yl palmitate (333)[365] and the 3-palmitate of taraxast-20-ene-3β,11β-diol has been found in *Chrysanthemum morifolium*.[366] The taraxastane derivatives (334) and (335) have been identified in *Symplocos racemosa*. The structure of (335) was confirmed by conversion to the known vanguerolic acid (336).[216] The 18,20-diene (337) from *Leptosphaeria maculans* can be considered as either a taraxastane or an ursane derivative.[236]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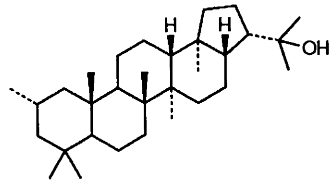
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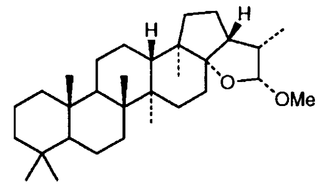
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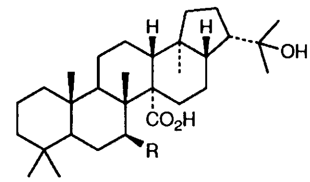
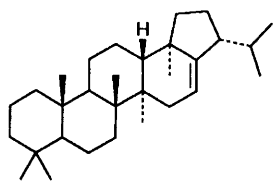

(341) R = H
(342) R = OAc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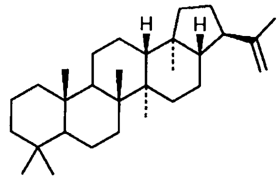
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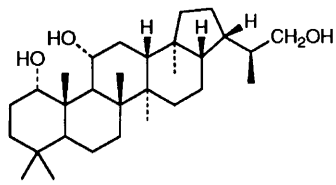
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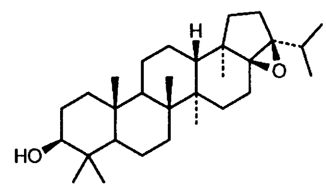
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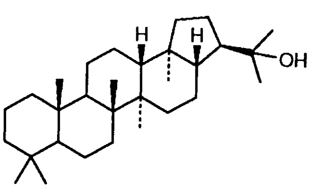
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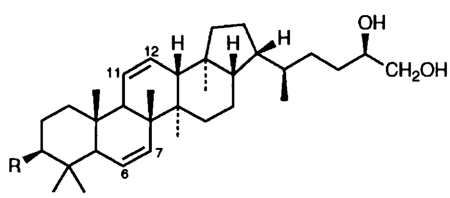
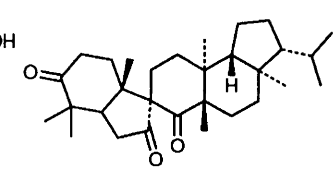

(348) R = H
(349) R = H; 11,12-dihydro
(350) R = CH_3
(351) R = CH_3; 11,12-dihydro
(352) R = CH_3; 6,7-dihydro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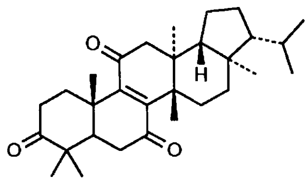
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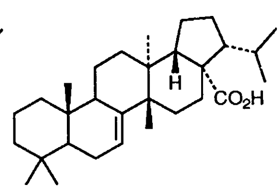
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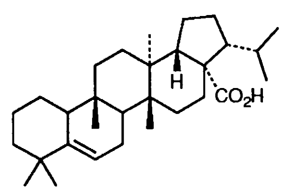
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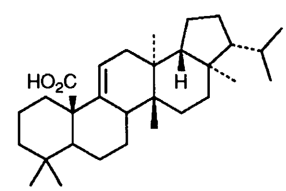
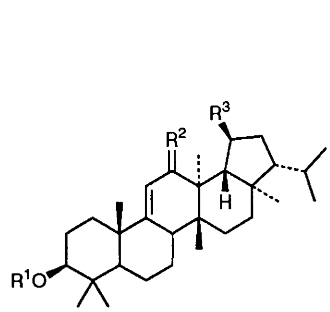
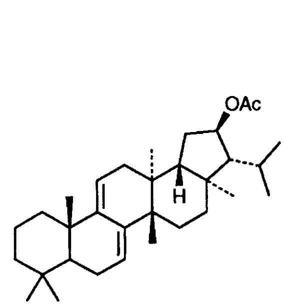

(357)

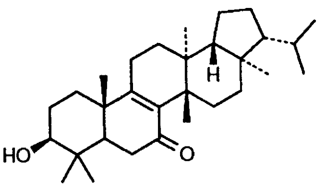
8 The Hopane Group

Chiratenol (338), from *Swertia chirata*, has a novel rearranged hopane skeleton.[367] *Methylobacterium organophilum* produces 2α-methyldiplopterol (339)[368] in addition to the previously isolated 2β-methyl derivative (see *Nat. Prod. Rep.*, 1986, **3**, 437). The unusual orton acetal (340) occurs in *Polypodium polypodioides*.[369] Two further phlebic acids, C (341) and D (342), have been found in *Peltigera aphthosa*.[370] Two hydrocarbons, hop-16-ene (343) and 21αH-hop-22(29)-ene (344), have been isolated from *Davallia mariesii*.[371] Other new hopanes include hopane-1α,11α,29-triol (345) from *Cheiropleuria bicuspis*,[372] 17β,21β-epoxyhopan-3β-ol (346) from *Euphorbia supina*,[373] and 21αH-hopan-22-ol (347) from *Polypodium vulgare* and *P. virginium*.[60] The 34,35-dinorbacteriohopanes (348)–(352), some with an extra methyl group at C-3, have been isolated from *Acetobacter aceti*.[374]

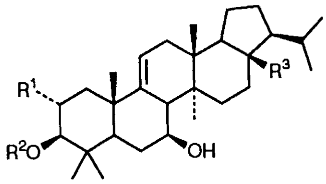

(358) R^1 = Ac; R^2 = O; R^3 = H
(359) R^1 = Ac; R^2 = β-OH, H; R^3 = H
(360) R^1 = H; R^2 = β-OH, H; R^3 = H
(361) R^1 = Ac; R^2 = H,H; R^3 = OH


(362)

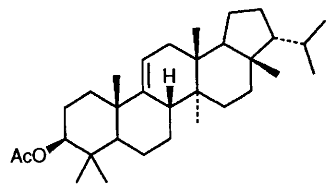
The novel rearranged fernene neospirosupinanetrione (353) has been found in *Euphorbia supina* together with supinenolone D (354).[375] Fern-7-en-28-oic acid (355) and its Δ^8- and $\Delta^{9(11)}$-isomers co-occur in *Microsorium brachylepis* and *M. normale* together with adian-5-en-28-oic acid (356).[376] New fern-9(11)-



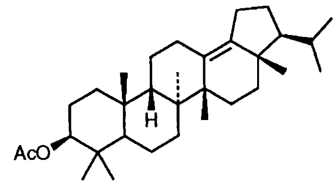
(363)



(364) $R^1 = R^2 = H$; $R^3 = CH_2OH$
(365) $R^1 = R^2 = H$; $R^3 = CH_3$
(366) $R^1 = OAc$; $R^2 = H$; $R^3 = CH_3$
(367) $R^1 = OH$; $R^2 = Ac$; $R^3 = CH_3$
(368) $R^1 = OH$; $R^2 = H$; $R^3 = CH_3$
(369) $R^1 = OH$; $R^2 = H$; $R^3 = CH_2OH$



(370)



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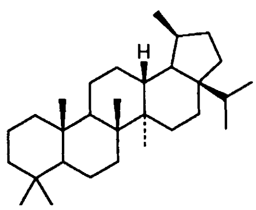
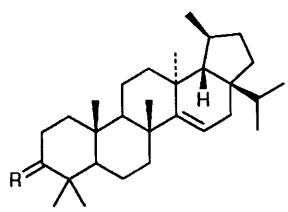
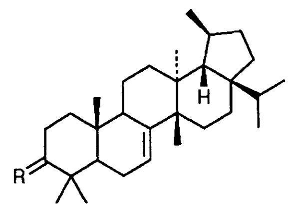


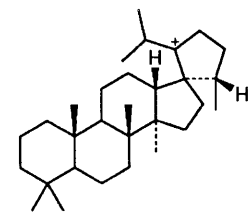
(372)



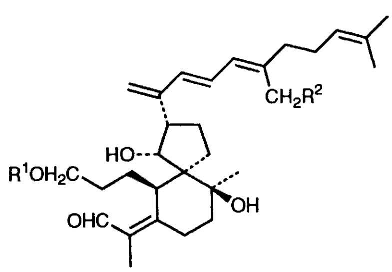
(373) R = β-OH, H
(375) R = O



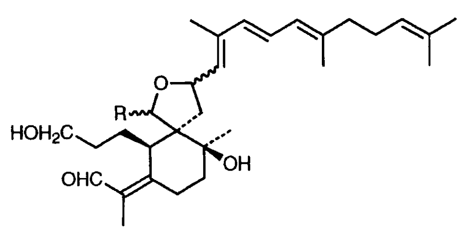
(374) R = β-OH, H
(376) R = O



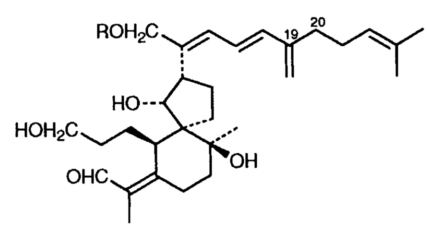
(377)



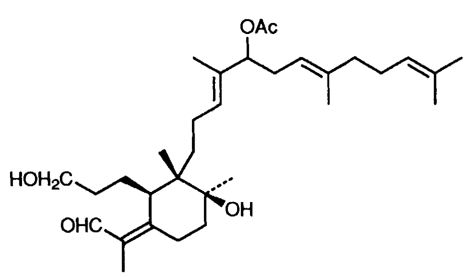
(378) $R^1 = H$; $R^2 = OAc$
(379) $R^1 = acyl$; $R^2 = OAc$
(382) $R^1 = R^2 = H$



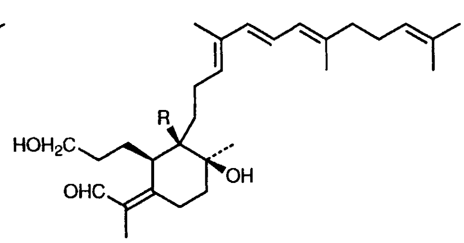
(380) R = H
(381) R = OH



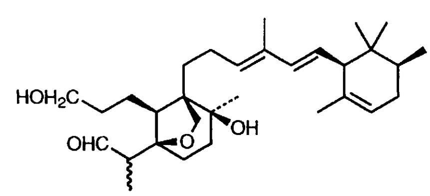
(383) R = Ac
(384) R = H
(386) R = H; Δ^{19}



(385)



(387) $R = CH_3$
(388) $R = CH_2OH$



(389)

enes include the 25-carboxylic acid (357) from *Adiantum venustum*,[377] and the 12-oxygenated derivatives (358)–(360) and the 19-alcohol (361) from *Pseudocyphellaria aurata*.[378] Sericostinyl acetate (362) from *Sericostoma pauciflorum* has a 20β-acetate group.[379] 3β-Hydroxyfern-8-en-7-one (363) has been isolated from *Euphorbia supina*.[226]

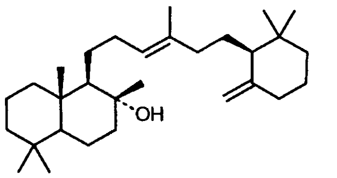
Rubia cordifolia var. *pratensis* and *R. oncotricha* contain a series of arborinane derivatives, rubiaarbonols A (364), B (365), C (366), D (367), E (368), and F (369).[380] Sorghumol acetate (370) and boehmerol acetate (371) have been found in *Pluchea lanceolata*.[381]

Bicyclic intermediates representing the AB and DE fragments of isoarborinol have been synthesized.[382] Hopane triterpenoids are regioselectively oxidized by *m*-chloroperbenzoic acid at unactivated positions yielding 17-or 21-hydroxyderivatives or 17,21-epoxides.[383] On treatment with bromine, *N*-bromosuccinimide, or molten sulfur, 17βH,21βH-hopanes are converted into 17αH,21βH- and 17βH,21αH-isomers, a result of possible geochemical significance.[384] The use of ozone for remote oxidation of hopanes has been investigated.[385]

9 Miscellaneous Compounds

A new skeletal type of triterpene, madeirane (372), has been proposed as the parent of four compounds isolated from *Euphorbia mellifera*, a species endemic to Madeira and two of the Canary islands.[386] The compounds are D-friedomadeir-14-en-3β-ol (373) and D:C-friedomadeir-7-en-3β-ol (374), and the corresponding ketones (375) and (376). Confirmation of structure was obtained by *X*-ray analyses of (374) and the acetate of (375). Formally the biosynthesis of madeirane (372) (as the C-18 cation) proceeds *via* the spirodammaranyl C-24 cation (377). Subsequent backbone rearrangement affords (373) and (374).

Several new spiro-bicyclic iridals have been reported, including (378)-(381) from *Iris pseudocorus*;[387] (382) from *I. foetidissima*;[388] belamcandal (383) and desacetyl belamcandal (384) from *Belamcanda chinensis* and *I. japonica*, together with 16-acetyl isoiridogermanal (385);[389] and (386) from *I. pallida* and *I. foetidissima*, together with the iridals (387)–(389).[390] The iridal group has been reviewed.[391]



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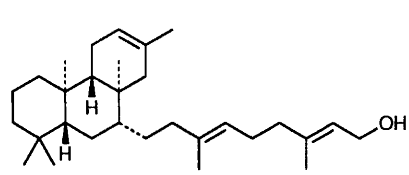

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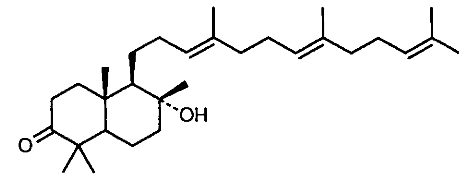

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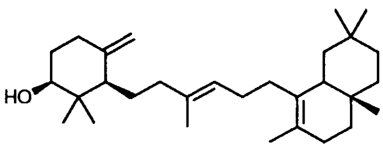

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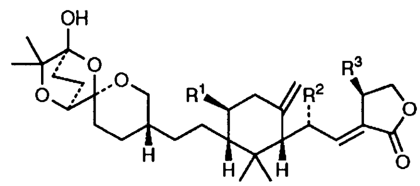
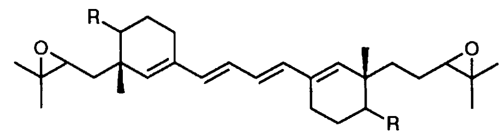
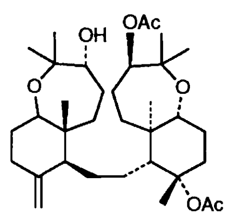


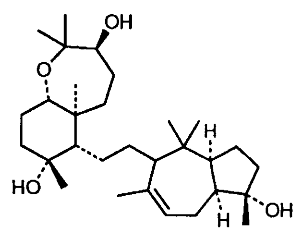
(394) $R^1 = R^2 = H$; $R^3 = OH$
(395) $R^1 = R^2 = R^3 = H$
(396) $R^1 = H$; $R^2 = R^3 = OH$
(397) $R^1 = R^3 = OH$; $R^2 = H$



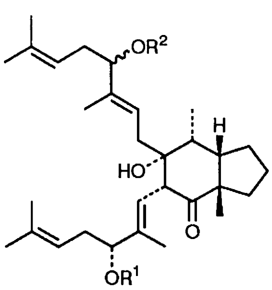
(398) R = β-OH
(399) R = α-OH



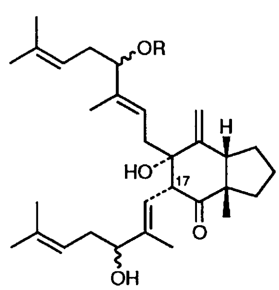
(400)



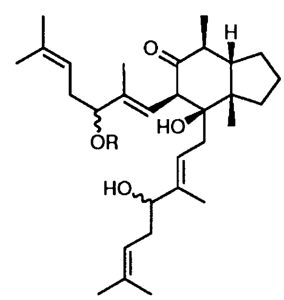
(401)



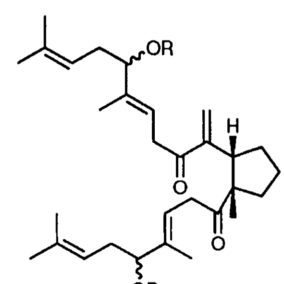
(402) R^1 = Rh; R^2 = Rh(1→4)Fu
(403) R^1 = H; R^2 = Rh(1→4)Rh(1→4)Fu



(404) R = Rh(1→4)Fu
(405) R = Rh(1→4)Rh(1→4)Fu
(406) R = Rh(1→4)Fu; 17-epimer



(407) R = Rh(1→4)Fu

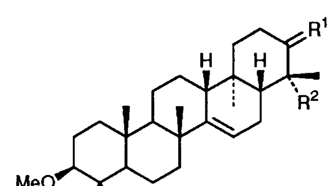


(408) R = Rh(1→4)Fu

Rh = α-L-Rhamnopyranosyl
Fu = β-D-Fucopyranosyl

The first total synthesis of ambrein (390)[392] and a synthesis of (*Z*,*Z*)-tricyclohexaprenol (391)[393] have been published. It has been shown that (*E*,*E*)-tricyclohexaprenol and octaprenol, two of the 'primitive' amphiphilic lipids, improve phospholipidic membranes.[394] 8α-Hydroxypolypoda-13,17,21-trien-3-one (392) is a constituent of *Pistacia lentiscus* resin.[165] A cyclized achillane, achilleol B (393), has been found in *Achillea odorata*[395] (see *Nat. Prod. Rep.*, 1994, **11**, 111). The saponaceolides A (394),[396] B (395), C (396), and D (397)[397] have been isolated from the fungus *Tricholoma saponaceum*. The structure of saponaceolide A (394) was established by *X*-ray analysis and the absolute configuration determined.

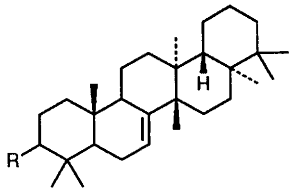
New squalene-derived metabolites from sponges include naurols A (398) and B (399) from an unidentified sponge[398] (*cf.* limatulone *Nat. Prod. Rep.*, 1986, **3**, 438) and raspacionin (400) from *Raspaciona aculeata*.[399] The structure of raspacionin (400) was confirmed by *X*-ray analysis.[400] The absolute stereochemistry of sipholenol A (401) has been assigned.[401] *Xestospongia vanilla* is proving to be a very rich source of triterpenoid glycosides. Seven more metabolites, xestovanins B



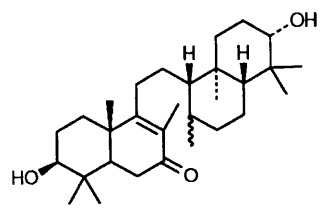
(409) R^1 = α-OAc, H; $R^2 = CH_3$
(410) R^1 = O; $R^2 = CH_2OH$
(411) R^1 = O; R^2 = CHO
(412) R^1 = α-OAc, H; R^2 = CHO

(402) and C (403), dehydroxestovanins A (404) and B (405), epidehydroxestovanin A (406), isoxestovanin A (407), and secodehydroxestovanin A (408), have been isolated.[402] Isoxestovanin A (407) has a novel carbon skeleton and presumably arises from secoxestovanin A (see *Nat. Prod. Rep.*, 1994, **11**, 111) by an aldol condensation.

The new serratanes (409)–(412) from *Pinus armandii* have



(413) R = β-OH
(414) R = α-OH



(415)

been described.[403] Swertenol (413) and episwertenol (414) from *Swertia chirata* are further examples of the swertane carbon skeleton[404] (see also *Nat. Prod. Rep.*, 1994, **11**, 110). *Cissus quadrangularis* yielded 3β,21α-dihydroxyonocer-8-en-7-one (415).[405]

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