Diterpenoids of terrestrial origin

Journal:	*Natural Product Reports*
Manuscript ID	NP-REV-08-2015-000087
Article Type:	Review Article
Date Submitted by the Author:	04-Aug-2015
Complete List of Authors:	Hanson, James; University of Sussex, Department of Chemistry

Diterpenoids of terrestrial origin

James R. Hanson

Received

Covering January to December 2014. Previous review, *Nat. Prod. Rep.*, 2015, **32**, 76 - 87.

This review covers the isolation and chemistry of diterpenoids from terrestrial as opposed to marine sources and includes, labdanes, clerodanes, abietanes, pimaranes, kauranes, cembranes and their cyclization products. There are 200 references.

1 Introduction

This report follows the pattern of its predecessors[1] covering the identification and chemistry of diterpenoids of terrestrial as opposed to marine origin. The latter are thoroughly reviewed in the articles on marine natural products.[2] In many cases diterpenoids from marine sources have carbon skeleta that are very different from those of terrestrial origin. A number of reviews have appeared on various aspects of diterpenoid chemistry, [3–6] biosynthesis,[7,8] and biotransformation.[9]

2 Acyclic and related diterpenoids

The nemoralisins E - J have been isolated[10,11] from *Aphanamixis grandifolia* (Meliaceae). The structure of nemoralisin B has been revised to **1**.[11] The aphanamixins A - F are similar compounds which were obtained[12] from the bark of *A. polystacha*.

3 Bicyclic diterpenoids

3.1 Labdanes

Copalyl diphosphate plays a central role in the biosynthesis of the diterpenoids. The structure particularly of the active site, and the mechanism of action of *ent*-copalyl diphosphate synthase have been examined.[13,14] A number of labdanes including eperuic acid (*ent*-labd-8(17)-en-15-oic acid) which possess anti-microbial activity, have been isolated[15] from a Brazilian plant, *Microlicia hatschbachii* (Melastomataceae). The inhibition of acetylcholinesterase by derivatives of cativic acid has been examined[16] in the context of the treatment of Alzheimer's disease. Extraction of the wood of the Taiwan fir, *Cunninghamia konishii* (Cupressaceae) has afforded[17,18] some 12,13-epoxylabdane 18- and 19-oic acids. 15-Acetoxy- and 15-chloro-14-hydroxysclareol have been reported[19] as constituents of *Salvia*

reuterana (Lamiaceae) but may be artefacts formed from the corresponding epoxide. Further labdanes including labda-12,14-dien-6β,7β,8α-triol, solidagol, have been isolated[20] from Canada golden rod, *Solidago canadensis* (Asteraceae). A lactam, amomax A 2 together with the corresponding lactone, has been obtained from the roots of a Chinese plant *Amomum maximum* (Zingiberaceae) which has been used to treat stomach disorders. Further labdanes have been obtained from other Chinese medicines including *Scoparia dulcis* (Scrophulariaceae),[22] *Lagopsis supina* (Lamiaceae)[23] and *Leonurus japonicus* (Lamiaceae)[24,25] which have been used for the treatment of inflammation. These include the lagopsins and leoheteronins. Examination of the Chinese liverwort, *Heteroscyphus tener* afforded[26] the heteroscyphins A - D (e.g. A 3).

A number of 3,4-*seco*-labdanes including callicarpaolide **4** have been obtained[27,28] from *Callicarpa nudiflora* (Verbenaceae) which is used in Chinese traditional medicine as an anti-inflammatory agent. These compounds inhibit NO production. Ring A has also been oxidized to a lactone in rhizomucronol A 5 which is a constituent of the South-East Asian medicinal plant *Rhizophora mucronata* (Rhizophoraceae).[29]

Andrographolide has continued to attract interest as a tumour inhibitory agent. It has been reviewed,[30] more relatives have been isolated[31] from *Andrographis paniculata* and its biological activity has been examined further.[32] An unusual heptacyclic *bis*-labdane pahangensin, has been isolated[33] from the rhizomes of the wild ginger, *Alpinia pahangensis* (Zingiberaceae) which is found in Malaysia.

The readily available bicyclic diterpenoids, manool and sclareol, have continued to provide the starting materials for synthetic studies for analogues of Ambrox®[34] and for other diterpenes,[35,36].

3.2 Clerodanes

The ^{1}H and ^{13}CNMR spectra of the neoclerodane, scutecyprin, have been

assigned.[37] Further studies on the biological activity of salvinorin A and its relatives have been carried out.[38] The scutebatas S and T[39] and barbatellarine F[40] were new neoclerodanes obtained from the Vietnamese medicinal plant, *Scutellaria barbata* (Lamiaceae) whilst the scutagalerins A and B were isolated[41] from *S. galericulata.* Other members of the Lamiaceae which have yielded new clerodanes during the year include *Ajuga decumbens* (ajugacumbin J),[42] *Stachys aegyptica,*[43] and *Leonurus japonicus* (leojapononin A).[44] Sapindaceae species have also been a source of clerodanes and further examples have been isolated[45,46] from *Dodonaea viscosa* and *D. polyandra.* The lactone, crotonolide A **6** was amongst the new clerodanes that were isolated[47] from *Croton laui* (Euphorbiaceae) whilst crotopene A which has nerve growth potentiating activity, was obtained[48] from *C.yanhui.*

A number of *cis*-clerodanes have been isolated including some glycosides from the Himalayan plant, *Nannoglottis carpesioides* (Asteraceae),[49], the balanspenes A–H (e.g. A,7) from *Casearia balansae* (Flacourtiaceae),[50] the zuelaguidins A–H (e.g. E, **8**) from the Costa Rican shrub, *Zuelania guidonia,*[51], the stephanialides A–E from the Chinese liverwort, *Scapania stephanii,*[52] and further compounds from the tubers of *Dioscorea bulbifera.*[53]

The salvimicrophyllins A–D from *Salvia microphylla*[54] include some 5,10-*seco* compounds (e.g. A, **9**) whilst some rearranged clerodanes which were obtained from *Solidago shortii* (e.g.**10**) may arise by an aldol condensation of a 5,10-*seco*-5,10-diketone.[55]

4 Tricyclic diterpenoids

4.1 Abietanes

The biological activity of derivatives of abietane diterpenoids[56] and of the tanshinones[57] have been reviewed. Two genes involved in the biosynthesis of abietanes in rosemary, have been characterized.[58]

Dehydroabietic acid has been identified[59] in *Boswellia thurifera* (Burseraceae)

resin (frankincense). Further abietanes which possess anti-fungal activity against the rice pathogen, *Magnaporthe grisea*, have been found[60] in *Oryza sativa* (rice) husks. 12-Hydroxy-7-oxoabieta-8,11,13-trien-18-oic acid (abietapinoic acid) and 13-hydroxy-7-oxopodocarpic acid (podopinoic acid) have been isolated[61] from *Pinus massoniana* (Pinaceae). Similar abietic acid derivatives have been obtained[62] from a Chinese traditional medicine, fenxiangzhi, which is the resin of *Liquidamber formosana* (Hamamalidaceae). Extraction of the leaves and twigs of *Drypetes perreticulata* (Euphorbiaceae) gave[63] the cytotoxic diterpenoids, dryperreins A-D (e.g.A, **11**). Anti-viral norabietanes have also been isolated from *Flueggea virosa* (Euphorbiaceae)[64] and *Gaultheria yunnanensis* (Ericaceae).[65]

Highly oxidized and rearranged abietanes are common constituents of the Lamiaceae. Some abietane dimers such as broussonetone A have been obtained[66] from root cultures of *Salvia broussonetii* whilst the salyunnanins (e.g.A **12**) were modified abietanes which were isolated[67] from *S. yunnanensis*. Other *nor*- and *seco*-abietanes have been detected in further studies of *S. miltiorrhiza* (e.g. salvialba acid **13**),[68] *S. przewalksii*[69] and *S. viridis*.[70] The 12-glucoside of 3β,12-dihydroxyabieta-8,11,13-trien-7-one was detected[71] in *Clinopodium chinense*.

The *bis-seco*-abietane, hyptisolide A **14** which was obtained[72] from *Hyptis crenata* (Lamiaceae), may arise by the cleavage of rings B and C of rosmanol **15** between C-7:C-8 and C-11:C-12. Further abietanes including some glycosides have been isolated from *Lycopus lucidus*,[73] *Clerodendrum bungei*,[74,75] and *C. trichotomum*.[76] *Seco*-abietanes such as merrilliadione (a 9,11-*seco*-abietane)[77] and wardinol A (a 9,10 *seco*-abietane)[78] have been obtained from *Illicium merrillianum* and *I. wardii* respectively. Examination[79] of the trunk of *Abies holophylla* (Pinaceae) yielded a pinacolic rearrangement product holophyllin A **16** whilst a spiro-lactone structure **17** has been assigned[80] to decandrinin which was obtained from the mangrove plant, *Ceriops decandra*.

The ready availability of abietic acid and some other resin acids coupled with an interest in the biological activity of the diterpenoids, has led to their use as starting

materials for the synthesis of triptolide analogues,[81,82] jiadifenoic acid[83] and taepeenin D analogues.[84] The anti-malarial activity of abietane analogues[85] and the anti-microbial activity of abietane:carbazole derivatives[86] has been examined. The preparation of a dicyanoabietane[87] and the cleavage of ring C in the synthesis of 9:13-spirolactones[88] have been reported.

4.2 Pimaranes, cassanes and related diterpenoids

Circular dichroism measurements have established[89] the configuration of the pimarane 17-hydroxy-*ent*-pimara-8(14),15-diene-3-one, which was isolated from *Sclerocroton integerrimus* (Euphorbiaceae). The potential of different strains of *Streptomyces griseus* to produce diterpenoids has been examined.[90] *Ent*-pimara-8(14),15-diene-19-oic acid from *Vigueria avenaria*[91] and the corresponding 8-en-7,11-dione from *Acanthopanax gracilistylus*[92] have anti-inflammatory activity whilst kiernol has been shown[93] to stimulate osteoblast differentiation. Further pimarane members of the jiadifenoic acid series (J-P) have been isolated[94] from *Illicium jiadifengpi*.

A number of fungal metabolites have been shown to possess a pimarane skeleton including xyllantin A **18** from *Xylaria allantoidea*,[95] some hymatoxin relatives from an endophytic *Xylaria* species,[96] and a 20-norpimarane, diplopimarane **19** from the oak pathogen, *Diplodia quercivora*.[97] 20-Norpimaranes have been detected[98] in a strain of *Aspergillus wentii* that had been obtained from a brown alga, *Sargassum fusiforme*. Ring A has undergone ring contraction during the formation of clerospicasin J **20** which was isolated[99] from *Clerodendranthus spicatus* (Lamiaceae).

The ebractenoids (e.g. A **21**) form a group of rosanes and related norditerpenes which were isolated[100] as anti-inflammatory constituents of the roots of *Euphorbia ebractolata* which are used in Chinese traditional medicine. The notolutesins A-J (e.g. A **22**) which were obtained[101] from the Chinese liverwort, *Notoscyphus lutescens*, have a dolabrene skeleton.

A large number of cassane diterpenoids have been obtained from *Caesalpinia* (Fabaceae) species including the caesalis A–M (e.g. A **23**) from *C. bonduc*,[102,103] the echinalides A–G from *C. echinata*,[104] and the caesalfurfuric acids from *C. furfuracea*.[105] Various studies of *C. minax* have afforded the caesalpins I and J,[106] neocaesalpins N and AF–AH,[107,108] the anti-malarial lactam, caesalminine A **24**,[109] and the unusual *nor*-cassane, norcaesalpinin **25**.[110] Other cassanes have been obtained from *C. sappan*[111,112] whilst some cassane amides were isolated from *Erythrophleum fordii*[113] and *E. suaveolens*.[114] The hawaiinolides A–G, which were obtained from the entomogenous fungus, *Paraconiothyrium hawaiiense* have been assigned[115,116] cleistanthane (e.g. A **26**) and cassane skeleta. Some erythroxan diterpenoids have been isolated[117] from *Fagonia mollis*.

5 Tetracyclic Diterpenoids

The biochemical characterization of *ent*-kaurene synthase and a quantum-chemical evaluation of the intermediates in tetracyclic diterpene biosynthesis, have been reported.[118,119] Further labelling studies on the biosynthesis of the terpenoid metabolites of *Fusarium* (*Gibberella*) *fujikuroi* have been described.[120] The gibberellin oxidase activity in a soil bacterium, *Bradyrhizobium japonicum*, which is found in the root nodules of the soya bean, has been examined.[121]

The balance between the methylerythritol and mevalonate pathways in *Stevia rebaudiana* has been examined.[122] The steviol glycosides were formed by the MEP pathway. Further minor diterpene glycosides (rebaudiosides) have been detected[123] in the leaves of *S. rebaudiana* and their quantification in different extracts has been examined.[124] In order to reduce the bitter after-taste of stevioside, various modifications of the glycosides including trans glycosylation and biotransformation have been reported.[125,126] The sterebins O, P_1 and P_2 were isolated when the crude plant extract was fermented with *Saccharomyces cerevisiae*.[127] The ease of isolation of isosteviol by treatment of the crude extracts of *S. rebaudiana* with acid,

has led to a number of studies of its chemistry including the formation of glucuronic acid conjugates,[128] macrocyclic and polyethylene glycol esters,[129,130], triazole conjugates,[131], urea[132] and isoniazid derivatives.[133]

A number of new tetracyclic diterpenoids have been isolated including 14β-hydroxy-3-oxo-*ent*-kaur-16-ene from *Croton kongensis* (Euphorbiaceae),[134] 1α,3α,7β,18-tetrahydroxy-*ent*-kaur-16-ene (sideripullol A) from *Sideritis pullulans*, (Lamiaceae),[135] 3-keto-6β,19-dihydroxy-*ent*-kaur-16-ene from a Saudi Arabian propolis,[136] 7α,15β-dihydroxy-*ent*-kaur-16-ene from *Hyalis argentea* (Asteraceae)[137] and a glycoside related to atractyligenin from *Vigna angularis* (Leguminoseae).[138] A further group of diterpenoid glycosides related to cafestol have been obtained[139] from coffee beans.

Isodon species have continued to be the source of highly oxygenated *ent*-kaurenes and ring B *seco*-kaurenes. New members of this series have been isolated from *I. adenantha*,[140] *I. eriocalyx varn. laxiflora*,[141] *I excisoides*,[142] *I. japonicus*,[143,144] *I. sculponeatus*,[145,146] *I. tenuifolius*,[147] and *I. wikstroemioides*.[148,149] A review has appeared of the biological activity of glaucocalyxin A[150] and a number of its derivatives have been examined as anti-cancer agents.[151] The anti-mycobacterial activity of this series has been evaluated.[152] The cleavage of ring D of 7α,14β-dihydroxy-*ent*-kaur-16-en-15-ones under Mitsunobu conditions to form *ent*-abietanes (e.g. **27** - **28**) has been examined.[153]

The microbiological hydroxylation of grandiflorenic acid at C-12α by *Fusarium graminearum* has been reported.[154] The anti-cancer activity of some unsaturated ketones derived from gibberellic acid has been described.[155] The absolute configuration of the scopadulane diterpenes from *Calceolaria* species has been established.[156]

The Ericaceae, particularly *Rhododenron* and *Leocothoe* species are noted for the production of toxic kalmane and grayane diterpenoids. The constituents of Chinese species have been reviewed[157] and further examples of these compounds including

the interesting *seco*-rhodomollone **29** and a C-*nor*-D-*homo*-grayane **30** have been reported from *R. molle*[158,159] and *R. principis*.[160] Methods for the determination of grayanotoxins in honey have been described.[161]

6 Macrocyclic diterpenoids and their cyclization products

The chandonanones A–F (e.g. A, **31**) are cembranes which were isolated[162] from the Chinese liverworts *Chandonanthus birmensis* and *C. hirtellus*. The absolute configuration of the cembrane diterpenoids which were isolated from *Bursera multijuga*, has been established.[163] Examination of *Croton insularis* has afforded[164,165] a group of casbenes including the endoperoxide **32** and a *seco*-casbene **33**.

Jatrophanes, lathyranes, tiglianes, ingenanes, daphnanes and related diterpenoids have continued to attract interest because of their anti-viral activity. A group of jatrophanes isolated from *Euphorbia amygdaloides ssp. semiperfoliata* were inhibitors of the chikungunya virus[166] whilst others from *E. lunulata* had anti-proliferative activity.[167] The euphosquamosins (.e.g. A, **34**) from *E. squamosa* were inhibitors of a multi-drug transporter from *Candida albicans*[168] whilst some jatrophanes from *E. helioscopia* inhibited[169] NO production. The japodagricanones A and B (e.g. A, **35**) from *Jatropha podagrica* may be derived[170] by cleavage of a lathyrane. A group of hydroxylated ingol derivatives, the euphorantins A–R, differing mainly in their esterifying groups, were obtained[171] from *E. antiquorum* whilst further compounds of this series have been isolated[172] from *E. lathyris*.

The excoecafolins A–C (e.g. A, **36**) which possess a tigliane skeleton were obtained[173] from *Excoecaria acertifolia*. Other anti-viral tiglianes have been isolated from *Croton mauritianus*,[174] *C. tiglium*,[175] *E. bupleuroides*,[176] and *E. stracheyi*.[177] Some daphnane glycosides have been obtained from *E. pilosa*[178] and *Daphne giraldii*.[179]

Ingenol mebutate has been used[180,181] as a treatment for a pre-malignant skin

condition. Structure:activity relationships of a series of 3-benzoates of ingenol have been evaluated[182] in this context.

The eurifoloids A-R (e.g. A, **37**) have been obtained[183] from *E. neriifolia*. Euphorbactin **38** from *E. micractina*[184] has an unusual 6:5:7:3 ring system which may be derived from a lathyrane. A myrsinene poly-ester has been isolated[185] from *Pycnocycla spinosa* (Umbelliferae).

Further examination of *Taxus chinensis varn. mairei* has afforded[186] a taxane with an unusual hydroxyl group at C-17.

7 Miscellaneous diterpenoids

The unusual bicyclic and tetracyclic diterpenoids **39** and **40**, have been isolated[187] as metabolites of a strain of the fungus *Trichoderma atroviridae* which was a symbiont of *Taxus baccata*. The initial cyclization to form the cyclododecadiene ring is reminiscent of that leading to a taxadiene. The cercosporenes A-F (e.g. A,**40**) from a *Cercospora* species[188] and the plicatilisins E-H from *Coprinus plicatilis*[189] are guanacastane fungal metabolites. The hypoestenonols from the shrub, *Hypoestes forskolei* have a fusicoccane skeleton.[190] Some further mulinanes including a compound with a *trans-syn-trans* tricyclic skeleton have been isolated[191] from the Chilean plant, *Azorella trifurcata*. A new dolabellane was obtained[192] from *Aglaia odorata*.

Examination of the medicinal plant, *Claoxylon polot* (Euphorbiaceae) has afforded[193] a group of prenylbisabolenes, the claoxylones A-I (e.g. A, **42**) which possess anti-viral activity. Some prenyleudesmanes, the dysoxydenones A-L have been obtained[194] from the bark of *Dysoxylum densiflorum* whilst addition[195] of the DNA methyl transferase inhibitor, 5-azacytidine, to the fungus *Penicillium funiculosum* altered its metabolic profile and led to the formation of prenyleudesmanes.

The unusual structure **43** has been assigned[196] to cubentriol which was a

metabolite of the endophytic fungus, *Xylaria cubensis* that was found on a Taiwanese plant, *Litsea akoensis*. A series of glycosides related to sordarin have been isolated[197] from the fungus, *Xylotumulus gibbisporus*. Cinncassiol F **44** which was isolated[198] from the dried bark of *Cinnamomum cassia* (Lauraceae), has been shown to have immunosuppressive activity. An unusual folding of geranylgeranyl diphosphate **45** accompanied by a series of hydrogen shifts, has been invoked to account for the carbon skeleton of vulgarisin A **46** which was obtained[199] from the Chinese medicinal plant, *Prunella vulgaris* (Lamiaceae). The biosynthesis of lycosantalonol, a *cis*-prenyl relative of santalene, has been examined.[200]

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