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NATURALLY OCCURRING CADINENES

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Key Word Index—Cadinenes; amorphane; bulgarane; cadinane; muurolane; cadalene; calamenene; norcadinenes.

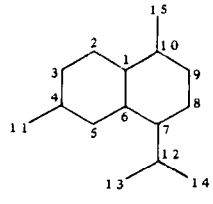
Abstract—Naturally occurring cadinenes appearing in the literature up to the end of 1986 have been reviewed.

INTRODUCTION

The first report on the isolation of a cadinene† [1] albeit in impure form, dates back to the first half of the last century [2]. Since then several other cadinenes have been found to be widely distributed in nature and at present naturally occurring compounds of this class number *ca* 165.‡ Recent isolation of artemisinin, a highly potent antimalarial drug from the traditional Chinese medicinal herb *Artemisia annua* L. (*vide infra*), has prompted us to review the cadinenes isolated from nature.

Structure **1** represents the basic skeleton of cadinenes. They have been further subdivided into four classes based on the nature of the ring fusion and the orientation of the isopropyl group at C-7.

(i) Amorphanes: the basic skeleton of this group is (1*S*,6*S*,7*S*)-7-(2-propyl)-4,10-dimethylbicyclo[4.0.4] decane (**2**) or its mirror image. (ii) Bulgaranes: the basic skeleton of this group is (1*R*,6*S*,7*S*)-7-(2-propyl)-4,10-dimethylbicyclo[4.0.4]decane (**3**) or its mirror image. (iii) Cadinanes: The basic skeleton of this group is (1*S*,6*R*,7*S*)-7-(2-propyl)-4,10-dimethylbicyclo [4.0.4] decane (**4**) or its mirror image. (iv) Muurolanes: The basic skeleton of this group is (1*R*,6*R*,7*S*)-7-(2-propyl)-4,10-dimethylbicyclo[4.0.4]decane (**5**) or its mirror image. In addition to these four classes of cadinenes, aromatic compounds with a cadinene skeleton have also been found to be widely distributed in nature.



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The formation of these four classes of cadinenes has been rationalized by assuming a cyclization process involving a 1,3-hydride shift to the carbonium ion at C-12 of the germacrene **6** (Scheme 1), for muurolane and cadinane groups and by the double 1,2-hydride shift of the C-12 carbonium ion for the formation of amorphane and bulgarane groups [3, 4]. Although this scheme has not been verified experimentally, the laboratory conversions of some germacrenes to various cadinenes have been reported [5–9].

RESULTS AND DISCUSSION

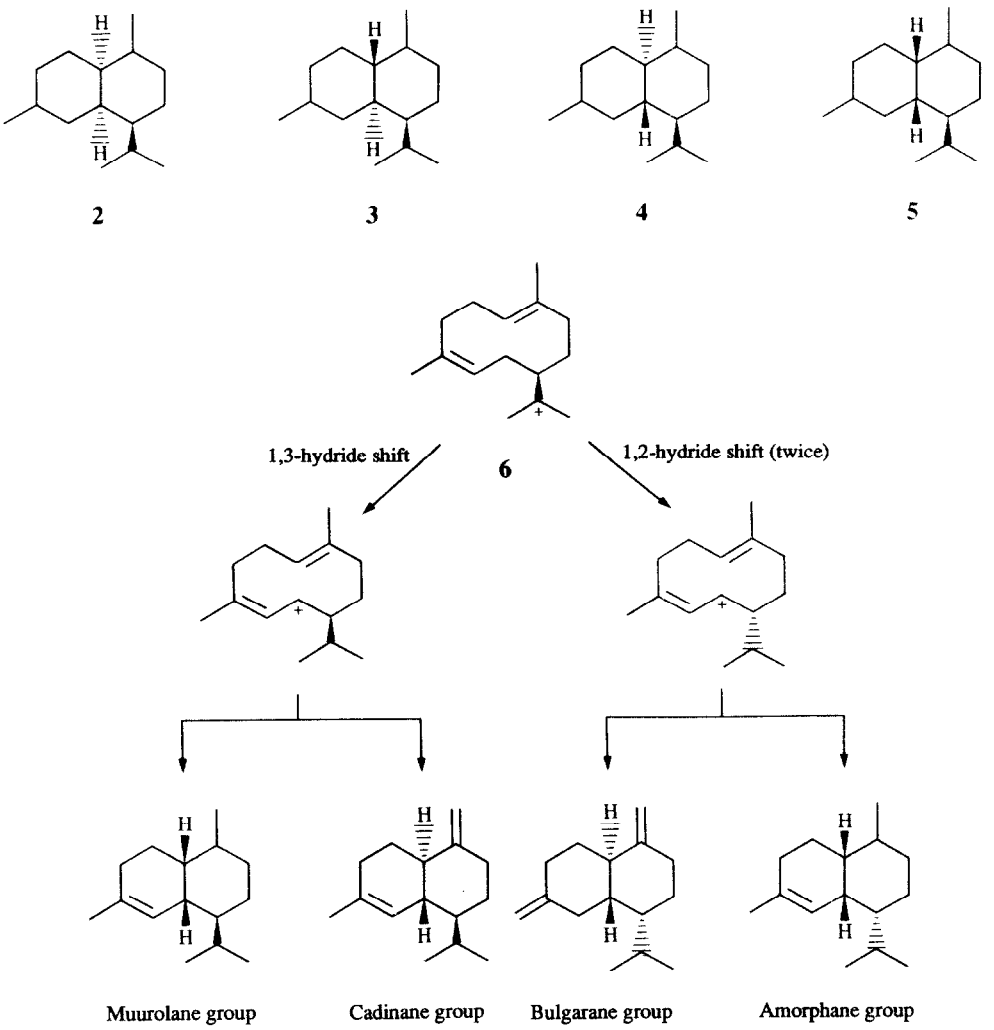
Amorphanes

So far 21 compounds of this group have been identified from various sources, among which (−)-α-amorphene (**7**), (+)-α-amorphene (**8**) and (−)-γ-amorphene (**9**) are widely distributed in nature [10–16]. Total synthesis has confirmed the structure of (±)-α-amorphene [17, 18]. A compound known as zizanene isolated from the oil of *Vetiveria* species was found to be identical with (+)-α-amorphene (**8**) [19, 20]. The absolute stereochemistry of (−)-α-amorphene **7** was established by chemical correlations [5]. (−)-γ-Amorphene isolated from *Agathis australis* Salsb. [21] and *Amorpha fruticosa* [22] and (−)-γ-muurolene isolated from *Pinus sylvestris* [23–25] were shown to possess the structure and absolute stereochemistry as represented in **9** [21, 23].

Of the six new cadinenes isolated from the aerial parts of *Ageratina adenophora* by Bohlmann and Gupta, compounds **10**, **11** (X=O; Y=H) and **11** (X=β-OH, H; Y=H) have been placed under this group (for others, see Miscellaneous); their structures and relative stereochemistries were deduced from spectral analysis [26]. Compounds **12–14** were isolated from *Eupatorium trapezoideum* Kunth [27] of which compounds **12** and **13** were found to be identical with compounds **11** (X=O; Y=H)

†Until now, all the naturally occurring or synthetic compounds mentioned in the literature having the basic skeleton **1** have been arbitrarily referred to as cadinenes. We have given this general name in order to avoid confusion, as otherwise it implies a double bond-containing compound of cadinane skeleton which only encompass one group of compounds out of the four groups *inter alia* classified as cadinenes. The name cadinene was given by Wallach [1].

‡This review covers literature up to 1986 and only naturally occurring cadinenes are included.

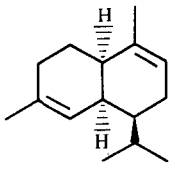


Scheme 1.

and **11** (X = β-OH, H; Y = H) respectively. The absolute stereochemistry of compound **12** was established by the application of Horeau's method, CD spectrum as well as by chemical correlation studies [28, 29]. Finally this has been confirmed by X-ray analysis of the *p*-bromobenzoate of **12**. The absolute stereochemistry of **13** and **14** was determined by chemical correlation with **12**. Compound **12** shows appreciable antifeedant properties against *Philasomia ricini* Hutt. [29].

Three interesting compounds of this group are **15**–**17** isolated from the marine sponge *Halichondria* species [30]. Their structures were deduced from spectral analysis and stereochemistry was established by chemical correlation. Sesquiterpenes containing isocyanide or isothiocyanate groups are uncommon and obviously pose interesting biogenetic problems. The co-occurrence of these three compounds containing isocyanide, isothiocyanate and formamide groups from the same source presents indirect support for the proposed biosynthetic relationship between the isocyanide and formamide group [31]. Compound **18** isolated from *Chromolaena arnottiana* is the only amorphene derivative with a primary alcohol function [32].

Artemisia annua L., a herb used as a Chinese traditional folk medicine, is found to be a prodigious source for many polyoxygenated amorphenes [33, 34]. Ten compounds, namely, qinghaosu acid (artemisinic acid) (**19**) [34], qinghaosu II (arteannuin B) (**20**) [34, 35], compounds **21** [36] and **22** [35, 37], qinghaosu I (**23**), qinghaosu V (**24**), deoxyartemisinin (qinghaosu III) (**25**), qinghaosu IV (**26**), qinghaosu (arteannuin, artemisinin) (**27**) [34] and artemisitene (**28**) [38] have been isolated from this Chinese herb. *Artemisia annua* has been used by the Chinese as a cure for malaria and it has been established that artemisinin (**27**) is the active constituent which responds to chloroquine-sensitive as well as resistant strains of *Plasmodium falciparum* and many other *Plasmodium* species [39–42]. The structure of this interesting sesquiterpene lactone endoperoxide was ascertained on the basis of spectral analysis [33]. The relative [43, 44] and absolute [45] stereochemistry of **27** was confirmed and the *trans*-configuration of the lactone ring was determined by comparison of its ORD spectrum [33] with that of arteannuin B (**20**) [35]. The structure of **27** was further confirmed by total synthesis [46–49] and detailed analysis of its ^{13}C NMR signals has been published recently



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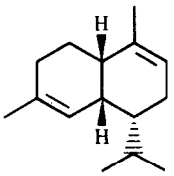

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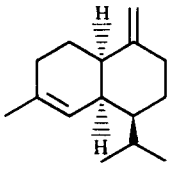

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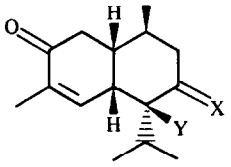
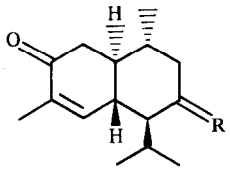
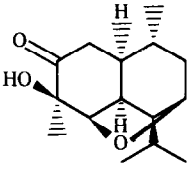


10 X = O, Y = OH
11 X = β-OH,H; Y = H
X = O, Y = H



12 R = β-OH,H
13 R = O



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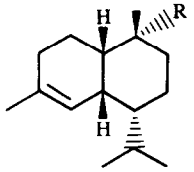
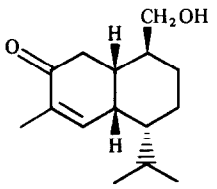


15 R = NC
16 R = NCS
17 R = NHCHO



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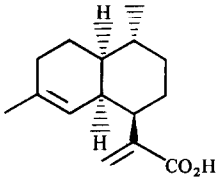

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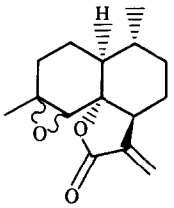
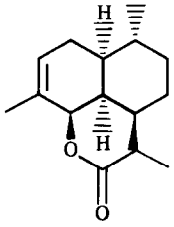


20 α-epoxy
21 β-epoxy
22 4,5-ene



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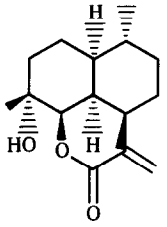

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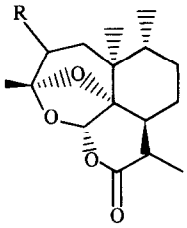
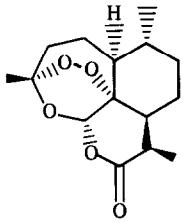
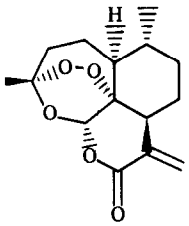


25 R = H
26 R = α-OH



27



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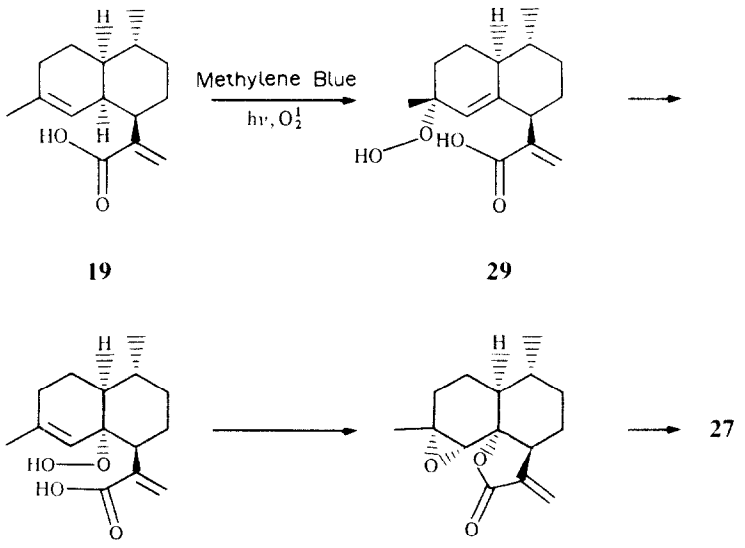
[50]. The *cis* ring junction of **19** was established on the basis of a Cotton effect exhibited by the 4-oxo esters derived from 4,5-epoxide of **19** [51]. Deoxyartemisinin. (**25**) was prepared from **19** via an allylic hydroperoxide and its structure and absolute configuration has been confirmed by X-ray crystallography [52]. Structures and stereochemistry of **20**–**22** were established by synthesis and X-ray analysis [37, 53–55].

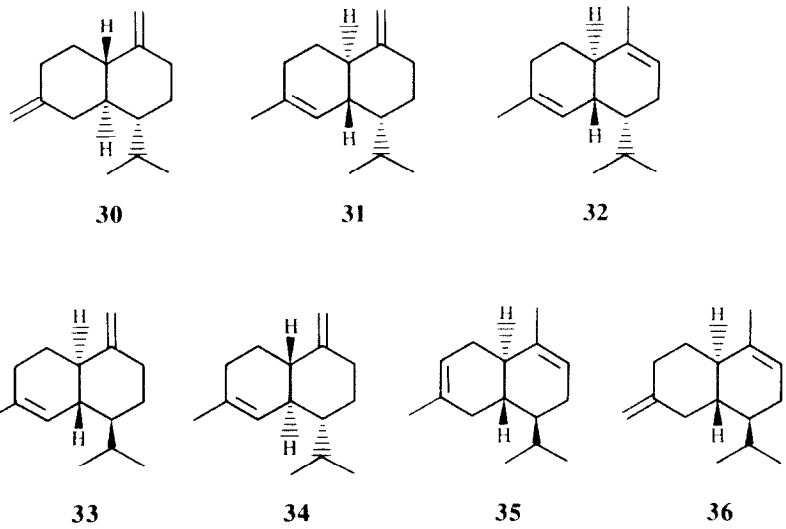
It has been suggested that compound **19**, which co-occurs with other polyoxygenated compounds in *Artemisia annua*, is the biogenetic precursor of all these compounds. This postulation has been supported by the formation of **27** from **19** via allylic hydroperoxide (**29**) (Scheme 2) [56]. Recently Popli and Thakur *et al.* have suggested through radioactive labelling studies that artemisinin is formed from farnesylpyrophosphate via germacrene skeleton→dihydrocostunolide→cadinanolide→arteannuin B [57].

Bulgaranes

Cadinenes belonging to the bulgarane group are relatively rare in nature. Only three compounds have been detected so far. The first compound of this (−)-ε-bulgarene **30** was isolated from *Mentha piperita* oil of Bulgarian origin. Its structure was determined by spectral analysis and chemical transformation [58]. The other two compounds γ-bulgarene and β-bulgarene, isolated



Scheme 2.



from the essential oil of *Juniperus oxycedrus*, were shown to be **31** and **32** respectively by spectroscopic methods [59]. The absolute stereochemistry of these three compounds has not yet been established.

Cadinenes

This group of cadinenes comprises the largest number of compounds widely distributed in nature. So far 37 compounds have been isolated from various sources. Many compounds belonging to this group are comparatively ubiquitous in nature, e.g. (+)-γ-cadinene (**33**) has been detected in more than 70 different sources (see Table 1). Its structure was established by Herout's group [60, 61]. Its antipode (−)-γ-cadinene (**34**) has also been isolated [62]. Synthesis of (±)-γ-cadinene was reported [63, 64].

(−)-β-Cadinene **35** [65, 66], (−)-γ_1-cadinene (**36**) [67–70] and ε-cadinene **37** [60, 71, 72] have also been isolated and a number of syntheses have been reported for compound **37** [73–76]. Absolute stereochemistry of these three compounds was determined by correlation with cadinene dihydrochloride (whose X-ray analysis [77] gave relative stereochemistry) and ORD studies and degradation to D-(+)-isopropylsuccinic acid [60, 78].

Isolation and syntheses of α-cadinene (**38**) [79, 80], δ_1-cadinene (**39**) [69, 81, 82] (antipode of compound **36**) and γ_2-cadinene (**40**) [76, 83–85] have been reported. Stereospecific synthesis of (−)-γ_2-cadinene (**40**) was also described [74, 86]. (+)-α-Cadinol (**41**), khusinol (**42**), epikhusinol (**43**), khusinodiol (**44**), khusol (**45**), cadina-4α,10β-diol (**46**) and a cadinene epoxide isokhusinoloxide (**47**) were isolated from the north Indian vetiver oil (*Vetiveria zizanioides*) [87, 88]. Khusinol was earlier described to have the absolute configuration as represented by **42** with the hydroxyl group at C-3 instead of C-2 [68], but Tirodkar *et al.* have shown khusinol to be as **42** [89]. Khusinodiol (**44**) was also found to contain a β-hydroxyl

Table 1. Sources of cadinenes

Source	Compound isolated	Formula	Reference
Agaricaceae (Fungi)			
Clitocybe illudens (Schwein) Sacc.	torreyol	**74**	378
Lentinus lepideus Fries	γ-muurolene	**9**	338
	α-muurolene	**70**	338
	δ-cadinene	**100**	338
	calamenene	**165**	338
Anacardiaceae (Angiosperms)			
Mangifera indica L.	δ-cadinene	**100**	340
Schinus molle L.	δ-cadinene	**100**	355
	α-cadinol	**49**	355
Apiaceae (Angiosperms)			
Bunium cylindricum (Boiss. & Hoh), Drude	cadinene		414
Carum carvi L.	(±)-cadinene		412
Daucus carota L.	(−)-γ-cadinene	**34**	258
Ferula costata	δ-cadinene	**100**	261
	γ-cadinene	**33**	261
	torreyol	**74**	261
	cadinol		261
Ferula iliensis Krasn.	β-calacorene	**138**	365
	α-cadinol	**49**	365
Laserpitium siler L.	calamenene	**165**	362
	calacorene	**137**	362
Platytaenia lasiocarpa	γ-cadinene	**33**	292
(Boiss.) Roch. & Riedl.	α-cadinene	**38**	292
Stewartiella baluchistanica E. Nair.	γ-muurolene	**9**	300
Apoidea (Insect)			
Bee honey comb (*Apies* spp.)	α-muurolene	**70**	424
	γ-cadinene	**33**	424
	calamenene	**165**	424
	α-calacorene	**137**	424
Araceae (Angiosperms)			
Acorus calamus L.	δ-cadinene	**100**	304, 305
var. *angustatus*	calamenene	**165**	304, 305
	calacorene	**137**	304
	calamenenediol	**54**	100
	cadala-1,4,9-triene	**103**	305
Acorus gramineus Soland.	δ-cadinene	**100**	306
	calamenene	**165**	306
Araliaceae (Angiosperms)			
Aralia cordata Thunb.	δ-cadinene	**100**	309
Dendropanax trifidus Makino	δ-cadinene	**100**	199
	(−)-γ-cadinene	**34**	199
	α-muurolene	**70**	199
	β-muurolene	**35**	199
Fatsia japonica Deene et Planch	epicubenol	**77**	324
	δ-cadinene	**100**	324
	α-muurolene	**70**	324
	calamenene	**165**	324
Tetrapanax papyriferum K Koch.	δ-cadinene	**100**	335
Araucariaceae (Gymnosperms)			
Agathis australis (Salisb.) Steud.	γ-muurolene	**9**	388
Araucaria araucana (Mol.)	(+)-α-cadinene	**38**	368
K. Koch (= *Araucaria imbricata*)	(−)-α-cadinol	**49**	368
Asteraceae (Angiosperms)			
Acritopappus longifolius	γ-cadinene	**33**	242
(Gardner) K. et R.	δ-cadinene	**100**	242
Ageratum conyzoides L.	δ-cadinene	**100**	16
	γ-cadinene	**33**	16
Ageritina adenophora		**10**	26
(Spreng.) K. et R.		**11**	26
		121	26
Arnica longifolia (DC.) Eaton	T-muurolol	**76**	395, 397

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
Artemisia annua L.	artemisinic acid (qinghao acid)	**19**	34
	arteannuin B (qinghao II)	**20**	35
	arteannuin C	**21**	37
		22	37
	qinghaosu I	**23**	34
	qinghaosu V	**24**	34
	qinghaosu III	**25**	34
	qinghaosu IV	**26**	34
	qinghaosu (artemisinin)	**27**	33
	artemisitene	**28**	38
Artemisia absinthium L.	α-cadinene	**38**	382
Artemisia pallens Wall (Davana)	γ-cadinene	**33**	244
Artemisia rehan Chiov.	cadinene		423
Artemisia scoparia Waldst & Kit.	δ-cadinene	**100**	310
Artemisia vestita Wall	α-cadinene	**38**	180
	cadalene	**126**	180
Artemisia vulgaris L.	δ-cadinene	**100**	311
Artemisia vulgaris var. *vulgaritisima*	α-cadinol	**49**	367
Aster subulatus Michx.	α-muurolene	**70**	387
Brachylaena hutchinsii	α-amorphene	**7**	10
Hutchinson	α-muurolene	**70**	12
	(+)-γ-amorphene	**9**	12
	(−)-δ-cadinene	**100**	12
	(+)-calamenene	**165**	12
	cubenol	**52**	12
Callilepis laureloa DC.	α-cadinol	**49**	369
Chromolaena arnotiana	chromoarnottion	**18**	32
(Griseb) King & Robinson		**182**	157
Chromolaena sp.		**109**	157
		110	157
		111	157
		112	157
		113	159
		114	159
		115	159
		116	160
		177	160
		178	237
Chrysanthemum indicum L.	T-muurolol	**76**	251
	α-cadinol	**49**	251
	γ-cadinene	**34**	251
	calamenene	**165**	251
	calacorene	**137**	251
Chrysanthemum cuneifolium Kitam	T-muurolol	**76**	251
Chrysanthemum japonese	γ-cadinene	**34**	252
Maxim.	calamenene	**165**	252
	T-muurolol	**76**	252
	α-cadinol	**49**	252
Chrysanthemum shiwogiku Kitam (= *Dendranthema shiwogiku* Kitam)	ε-cadinene	**37**	399
Dilophus fasciola	calamenene	**165**	140
	4,10-epoxy muurolene	**91**	140
	epibicyclosesquiphellandrene	**105**	140
	δ-cadinene	**100**	140
	cubenol	**52**	140
Eupatorium trapezoideum Kunth.		**12**	27
		13	27

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
		14	27
		84	27
		121	27
		190	29
Heterotheca grandiflora Nutt. epicubenol		77	131
		80	131
		85–88	131
		94–97	131
		126, 127	131
		131, 137	131
		138	131
		179–181	131
		191–199	131
Heterotheca grandiflora Nutt. (from Hawaii)		**174–176**	236
Heterotheca inuloides Cass.	α-calacorene	**137**	202–205
Heterotheca parvifolia		**128**	186–188
Heterotheca subaxillaries	δ-cadinene	**100**	421
(Lam.) Britton & Rusby.	calamenene	**165**	131
	epicubenol	**77**	131
	α-cadinol	**49**	131
		108	131
		177	237
		178	237
Inula royleana DC. & Clarke	δ-cadinene	**100**	330
Jasonia tuberosa DC.	3-β-hydroxy-T-cadinol	**51**	97
Laggera aurita Sch. Bip.	δ-cadinene	**100**	332
	α-cadinol	**49**	332
Leucanthemopsis pulverulenta (Lag.) Heywood		**64–69**	109
Matricaria chamomilla L.	γ-cadinene	**33**	401
Mikania micrantha HBK	δ-cadinene	**100**	342
Santolina chamaecyparissus L.	γ-cadinene	**33**	296
Santolina virdis L.	γ-cadinene	**33**	296
Saussurea sacra Edgew.	1-cadinol		405
Senecio fuchsii	γ-muurolene	**9**	297
Zlatnik & Gmelin	γ-cadinene	**33**	297
	δ-cadinene	**100**	297
	calamenene	**165**	297
	calacorene	**137**	297
Senecio jacobaea D. Don.	γ-cadinene	**33**	297
	calacorene	**137**	297
	α-cadinol	**49**	297
Senecio vulgaris L.	α-muurolene	**70**	297
	γ-cadinene	**33**	297
	δ-cadinene	**100**	297
Stevia rebaudiana Bertoni	δ-cadinene	**100**	334
Trichogonia grazielae K. et R.	γ-muurolene-11-oic acid	**98**	146
Verbesina occidentalis (L.) Walt.		**117–120**	160
Xanthium canadense Mill.	δ-cadinene	**100**	337
Bombacaceae (Angiosperms) *Bombax* sp.	isohemigossypol	**146**	209
	isohemigossypol-2-methyl ether	**147**	209
	isohemigossypolone	**150**	193
	isohemigossypolone-3-methyl ether	**151**	209
Montezuma speciossima Sesse & Moc ex DC. (= *Thespesia grandiflora* DC)	gossypol	**143**	208
Burseraceae (Angiosperms)			
Boswellia sacra Flueckig	α-muurolene	**70**	308
(Arabic olibanum)	δ-cadinene	**100**	308
Canarium album Raesch.	α-cadinol	**49**	247
	δ-cadinol	**74**	248
	δ-cadinene	**100**	248, 312
	γ-cadinene	**34**	248

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
	calamenene	**165**	248
	γ-muurolene	**9**	248
	α-muurolene	**70**	248
Pimeta dioica L.	10-cadinol	**50**	392
	α-cadinol	**49**	392
Calycanthaceae			
Calycanthus floridus L.	α-muurolene	**70**	246
	γ-muurolene	**9**	246
	δ-cadinene	**100**	246
	γ-cadinene	**34**	246
	calamenene	**165**	246
	α-cadinol	**49**	246
Cupressaceae (Gymnosperms)			
Chamaecyparis formosensis	T-muurolol	**76**	316
Matsum.	(+)-δ-cadinene	**100**	316
Chamaecyparis nootkatensis	δ-cadinene	**100**	250
Spack.	γ-cadinene	**33**	250
	α-cadinol	**49**	250
Cupressus sempervirens L.	ε-cadinene	**37**	371
Cupressus fastigata DC.	α-cadinol	**49**	371
Juniperus sp. (central Asian)	δ-cadinene	**100**	264
	α-calacorene	**137**	264
	β-calacorene	**138**	264
	calamenene	**165**	264
Juniperus communis L.	δ-cadinene	**100**	331
	α-muurolene	**70**	361
	γ-muurolene	**9**	361
	calamenene	**165**	361
	trans-calamenene	**166**	361
Juniperus formosana Hayata	δ-cadinol	**74**	379
Juniperus macropoda Boiss.	cadinene		400
Juniperus oxycedrus L.	ε-cadinene	**37**	265
	γ-cadinene	**33**	266
	cis-calamenene	**165**	266
	trans-calamenene	**166**	266
	δ-cadinene	**100**	266
	β-cadinene	**35**	39
	γ-muurolene	**9**	266
	α-muurolene	**70**	266
	β-bulgarene	**32**	266
	γ-bulgarene	**31**	266
	T-muurolol	**76**	266
	α-cadinol	**49**	266
	cubenol	**52**	266
	epi-cubenol	**77**	266
Juniperus rigida Wall.	epi-cubenol	**77**	425
	cadinenol	**77**	425
Juniperus thurifera L.	α-cadinol	**49**	375
Cyperaceae (Angiosperms)			
Cyperus difformis L.	α-cadinol	**49**	374
Cyperus globosus Allioni	α-cadinol	**49**	374
Cyperus iria L.	γ-cadinene	**34**	257
	α-cadinol	**49**	372
	γ-muurolene	**9**	257
	δ-cadinene	**100**	257
	calamenene	**165**	257
Cyperus microiria Steud.	α-cadinene	**38**	372
	α-cadinol	**49**	372
Cyperus monophyllus Vahl.	δ-cadinene	**100**	321
Cyperus pilosus Vahl.	α-cadinol	**49**	373
Cyperus polystachyos Rottb.	α-cadinol	**49**	374

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
Cyperus rotundus L.	δ-cadinene	**100**	358
	calamenene	**165**	358
Cyperus serotinus Rottb.	δ-cadinene	**100**	358
	calamenene	**165**	358
Dictyotaceae (Algae)			
Dictyopteris zonarioides	epizonarene	**122**	162
	zonarene	**123**	162
	ent-10-epizonarene	**124**	162
	ent-zonarene	**125**	162
Dictyopteris divericata	δ_1-cadinene		178
	cadalene	**126**	178
	δ-cadinol	**74**	178
	cubenol	**52**	416
	epicubenol	**77**	416
Dipterocarpaceae			
Dipterocarpus Sp. (Gurjum Balsam)	δ-cadinene	**100**	327
Ericaceae (Angiosperms)			
Ledum groenlandicum L. (= *Ledum canadense*)	α-amorphene	**7**	14
Ledum palustre L.	α-amorphene	**7**	14
Rhododendron adamsii Rehd.	δ-cadinene	**100**	352
Rhododendron capitatum Maxim.	δ-cadinene	**100**	353, 354
Rhododendron dahuricum L.	δ-cadinene	**100**	352
Rhododendron tsinghaiense	δ-cadinene	**100**	293
Ching.	γ-cadinene	**33**	293
	α-cadinol	**49**	293
	(−)-torreyol	**74**	293
	isocalamenediol	**55**	293
Euphorbiaceae (Angiosperms)			
Croton sondrianus Muell. Arg.	calamenene	**165**	254
Euphorbia monostyla Prokh	α-cadinene	**38**	383
	β-cadinene	**35**	383
Geoglossaceae (Fungi)			
Sclerotinia fruticola	sclerosporin	**92**	141
(Wint.) Rehm.	sclerosporal	**93**	141
Geraniaceae (Angiosperms)			
Geranium macrorrhizum L.	γ-muurolene	**9**	326
	δ-cadinene	**100**	325
	calamenene	**165**	325
Mansonia altissima	mansonone A	**153**	214
A. Chevalier	mansonone B	**154**	215
	mansonone C	**155**	215
	mansonone D	**156**	215
	mansonone E	**157**	215
	mansonone F	**158**	215
	mansonone G	**162**	218
	mansonone H	**161**	218
	mansonone I	**159**	216
	mansonone L	**160**	217
Gorgonaceae (Horny Coral)			
Subergorgia hicksoni	2-methoxy calamenene	**170**	231–233
	5-hydroxy-2-methoxy calamenene	**171**	231–233
Hepaticae (Bryophyta)			
Barbilophozia sp.	calamenene	**165**	356
Conocephalum conicum (L.) Underwood	δ-cadinene	**100**	318, 319
Lophocolea heterophylla	calacorene	**137**	182
(Schrad.) Dum.	calamenene	**165**	182
	cadalene	**126**	182
Hydrocharitadaceae (Angiosperms)			
Vallisneria denseserrulata Makino	δ-cadinol	**74**	381
Lamiaceae (Angiosperms)			
Agastache rugosa Kuntze	δ-cadinene	**100**	307

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
Elsholtzia densa Benth.	γ-cadinene	**33**	258–260
Levendula vera DC.	γ-cadinene	**33**	270
	δ-cadinene	**100**	271
		188, 189	106
	β-cadinene	**35**	272
	δ-cadinene	**100**	272
Mentha arvensis L.	γ-cadinene	**33**	276
var. *glabrata*	δ-cadinene	**100**	276
	ε-cadinene	**37**	276
Mentha candicans Miller	γ-muurolene	**9**	389
Mentha gentilis L.	δ-cadinene	**100**	341
var. *Lanata* (Rydb.)	α-cadinene	**38**	341
Fujita et Fujita	α-muurolene	**70**	341
(Comb. nov.)	calamenene	**165**	341
	calacorene	**137**	341
Mentha piperita L. & Huds.	α-cadinene	**38**	13
	α-amorphene	**7**	13
	ε-muurolene	**71**	398
Mentha piperita var. *officinalis*, Bulgarian	γ-muurolene	**9**	277
	ε-muurolene	**71**	278
	α-muurolene	**70**	278
	δ-cadinene	**100**	278
	γ-cadinene	**33**	278
	ε-bulgarene	**30**	278
	calamenene	**165**	278
	w-cadinene	**101**	149, 150
Nepeta leucophylla Benth.	γ-cadinene	**33**	404
Ocimum basilicum L.	10-cadinol	**50**	392
	α-cadinol	**49**	392
Ocimum gratissimum L.	γ-muurolene	**9**	344
	δ-cadinene	**100**	344
Sideritis arborescens	α-muurolene	**70**	333
var. *arborescens*, Salzm.	δ-cadinene	**100**	333
Teucrium polium L.	γ-cadinene	**33**	301
	δ-cadinene	**100**	301
Thymus caespititius Brot.	α-muurolene	**70**	302
	γ-cadinene	**33**	302
Thymus quinquecostatus (Celak) Kitamura (= *Thymus serpyllum* Hook f. non L.)	γ-cadinene	**33**	303
Lauraceae (Angiosperms)			
Cinnamomum camphora	T-cadinol	**50**	408
(Nees & Eberm) Sieb.	T-muurolol	**76**	408
	cubenol	**52**	408
	epicubenol	**77**	408
	(+)-cadinenol		408
Litsaea gerciae Vidal	γ-cadinene	**33**	273
Litsaea japonica (Thunb.) Juss.	α-amorphene	**7**	15
	α-muurolene	**70**	15
	β-cadinene	**35**	15
	γ-cadinene	**33**	15
	α-cadinol	**49**	15
Litsaea kostermania Cheng	γ-cadinene	**33**	273
Machilus thumbergii Sieb. et Zuce	δ-cadinene	**100**	339
Neolitsea soricea Koidz.	γ-cadinene	**33**	281
	δ-cadinene	**100**	281
Parabenzoin trilobum Nakai	γ-cadinene	**33**	281
	δ-cadinene	**100**	281
Leguminosae (Angiosperms)			
Amorpha fruticosa L.	γ-amorphene	**9**	22
	γ-cadinene	**33**	22
	calamenene	**165**	22

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
Copaifera sp.	4-cadinene		413
Hymenaea courbarial L.	α-muurolene	**70**	328
	δ-cadinene	**100**	329
	γ-muurolene	**9**	329
Sindora glabra Prain	γ-muurolene	**9**	298
	δ-cadinene	**100**	298
	(−)-calamenene	**166**	298
	γ-cadinene	**33**	298
	α-muurolene	**70**	298
Lepidoptera (Insect)			
Atrophaneura larva	δ-cadinene	**100**	422
Lepidoziaceae (Bryophyta)			
Bazzania japonica (Lac.) Lindb.	calamenene	**165**	357
Bazzania tricenta (Wahl.), Trev.	calamenene	**165**	419
Bazzania tridens (Review *et al.*) Trev.	calamenene	**165**	419
Bazzania trilobata (L.) S. Grey	calamenene	**165**	419
Liliaceae (Angiosperms)			
Yucca gloriosa L.	α-muurolene	**70**	263
	γ-muurolene	**9**	263
	γ-cadinene	**33**	263
	δ-cadinene	**100**	263
Malvaceae			
Gossypium sp. (Bracts, leaves)	2,7-dihydroxy cadinene	**132**	192, 191
	2-hydroxy-7-methoxy cadalene	**133**	192
	lacinilene C	**134**	194
	lacinilene C-7-methyl ether	**135**	194
	hemigossypol	**140**	194
	6-hydroxy hemigossypol	**141**	206
	6-methoxy gossypol	**142**	206
	gossypol	**143**	207
	6-methoxygossypol	**144**	208
	6,6′-dimethoxy gossypol	**145**	209
Magnoliaceae (Angiosperms)			
Magnolia stellata (Sieb. et Zuce) Maxim	δ-cadinene	**100**	322
	α-cadinol	**49**	322
Magnolia wilsonii Rehd.	δ-cadinol	**74**	380
Michelia alba DC.	δ-cadinene	**100**	418
Schizandra nigra Max.	schizandronol I	**63**	108
Marchantiales (Bryophyta)			
Dumortiera hirsuta (Swartz) Nees	γ-muurolene	**9**	385
	α-muurolene	**70**	385
Meliaceae (Angiosperms)			
Cedrela odorata Hort. Kew	epicubenol	**77**	249
	T-muurolol	**76**	249
	α-muurolene	**70**	249
	β-cadinene	**35**	249
	δ-cadinene	**100**	249
	γ-cadinene	**33**	249
	α-calacorene	**137**	249
	calamenene	**165**	249
Cedrela toona Roxb.	T-muurolol	**76**	94
	epicubenol	**77**	94
	cubenol	**52**	94
Dysoxylum fraseranum Benth.	δ-cadinene	**100**	148
Melia azedarach L.	γ-cadinene	**33**	274
var. *japonica* Makino	δ-cadinene	**100**	275
	γ-muurolene	**9**	275
	α-cadinol	**49**	275
	α-muurolene	**70**	275
	epicubenol	**77**	275

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
	cubenol	**52**	275
	T-cadinol	**50**	275
	T-muurolol	**76**	275
Moraceae (Angiosperms)			
Hop aroma in American beer	T-cadinol	**50**	415
(*Humulus lupulus* L.)	δ-cadinol	**74**	415
Humulus lupulus L.	α-cadinene	**38**	79
Humulus japonicus	γ-cadinene	**33**	394
Sieb. & Zuce			
Musci (Bryophyta)			
Diphophyllum albicans (L.) Dumortier.	calamenene	**165**	359–360
Myoporaceae (Angiosperms)			
Eremophila drummondii S. Moore	3-hydroxy calamenene	**167**	229
Myrtaceae (Angiosperms)			
Baeckea frutescens L.	δ-cadinol	**74**	377
	calamenene	**165**	377
Eucalyptus saligna Sm.	δ-cadinene	**100**	323
Myrica gale L.	γ-cadinene	**33**	279
	δ-cadinene	**100**	279
Myrtus pimenta L.	α-muurolene	**70**	346
	δ-cadinene	**100**	346
Myristicaceae (Angiosperms)			
Myristica womerslveyi Sincl.	δ-cadinene	**100**	343
	calamenene	**165**	343
Ongraceae (Angiosperms)			
Oenothera biennis L.	γ-muurolene	**9**	280
	δ-cadinene	**100**	280
	γ-cadinene	**33**	280
	calamenene	**165**	280
Pinaceae (Gymnosperms) *Abies* sp.	cadinene		420
	calamenene		420
Abies alba Mill.	α-muurolene	**70**	241
	γ-muurolene	**9**	241
	ε-muurolene	**71**	241
	δ-cadinene	**100**	241
	γ-cadinene	**33**	241
Abies gracilis Hort.	α-muurolene	**70**	241
ex Lavallee	γ-muurolene	**9**	241
	ε-muurolene	**71**	241
	δ-cadinene	**100**	241
	γ-cadinene	**33**	241
Abies myriana Myabe & Kudo	α-muurolene	**70**	241
	γ-muurolene	**9**	241
	ε-muurolene	**71**	241
	δ-cadinene	**100**	241
	γ-cadinene	**33**	241
Abies sachalensis Mast.	α-muurolene	**70**	241
	γ-muurolene	**9**	241
	ε-muurolene	**71**	241
	δ-cadinene	**100**	241
	γ-cadinene	**33**	241
Cedrus deodora (Roxb.)	α-muurolene	**70**	315
Loud.	δ-cadinene	**100**	315
Larix olgensis A. Henry	α-muurolene	**70**	269
	γ-muurolene	**9**	269
	ε-muurolene	**71**	269
	γ-cadinene	**33**	269
	δ-cadinene	**100**	269
	ε-cadinene	**37**	269
	calamenene	**165**	269
Larix sibirica Ledeb.	ε-muurolene	**71**	267
	α-muurolene	**70**	267, 386

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
	γ-muurolene	**9**	267, 386
	δ-cadinene	**100**	268
	γ-cadinene	**33**	268
	amorphene		268
	calamenene	**165**	268
Picea ajaensis Fisch.	cadalene	**126**	179
	γ-cadinene	**33**	283
	δ-cadinene	**100**	283
	α-muurolene	**70**	283
	γ-muurolene	**9**	283
	T-cadinol	**50**	283
	T-muurolol	**76**	283
Picea amorica Pancic ex Steiu	cadinene		410
Pinus Sp.	γ-cadinene	**33**	284
	δ-cadinene	**100**	284
	α-muurolene	**70**	390
Pinus clausa Vasey	cadinene		402
Pinus contorta Dougl.	mixture of cadinenes		393
var. *latifolia*	cadinols & muurolols		393
Pinus edulis Engelm.	α-amorphene	**38**	285
	γ-amorphene	**9**	285
	β-cadinene	**35**	285
	γ-cadinene	**33**	285
	δ-cadinene	**100**	285
	α-muurolene	**70**	285
	ε-cadinene	**37**	285
	γ-muurolene	**9**	285
Pinus eladrica Medw.	α-muurolene	**70**	289
	δ-cadinene	**100**	289
	γ-cadinene	**33**	289
Pinus elliotti Engelm.	cadinene		402
Pinus kochiana Griff.	α-muurolene	**70**	289
	δ-cadinene	**100**	289
	γ-cadinene	**33**	289
Pinus mugo Turra	γ-cadinene	**33**	286
	α-muurolene	**70**	286
	γ-muurolene	**9**	286
Pinus pallasiana L.	α-muurolene	**70**	289
	δ-cadinene	**100**	289
	γ-cadinene	**33**	289
Pinus pinaster Soland.	α-muurolene	**70**	287
	γ-muurolene	**9**	287
	γ-cadinene	**33**	287
	δ-cadinene	**100**	287
	α-cadinol	**49**	287
Pinus pinea L.	α-muurolene	**70**	391
Pinus pungeus Lamb.	γ-muurolene	**9**	347
	δ-cadinene	**100**	347
	α-cadinol	**49**	347
	δ-cadinol	**74**	347
Pinus rigida Mill.	γ-muurolene	**9**	422
	δ-cadinene	**100**	422
	α-cadinol	**49**	422
	δ-cadinol	**74**	422
Pinus serotina Michx.	cadinene		402
Pinus sibirica Mayr	α-muurolene	**70**	351
	γ-muurolene	**9**	351
	δ-cadinene	**100**	351
Pinus sibiricus	α-muurolene	**70**	348
	δ-cadinene	**100**	348
	γ-cadinene	**33**	348
Pinus silvestris L.	α-muurolene	**70**	288

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
	γ-muurolene	**9**	288
	γ-cadinene	**33**	288
Pinus sosnowsky Nakai	α-muurolene	**70**	289
(= *P. hamata* (Stev.) Sosn.]	δ-cadinene	**100**	289
	γ-cadinene	**33**	289
Pinus strobus L.	γ-muurolene	**9**	349
	δ-cadinene	**100**	349
	α-cadinol	**49**	349
	δ-cadinol	**74**	349
Pinus sylvestris L.	α-muurolene	**70**	350
	γ-muurolene	**9**	289
	ε-muurolene	**71**	351
	δ-cadinene	**100**	351
Pinus taeda L.	cadinene		402
Pinus virginiana Mill.	γ-muurolene	**9**	349
	δ-cadinene	**100**	349
	α-cadinol	**49**	349
	δ-cadinol	**74**	349
Pinus sp. (Bulgarian)	α-muurolene	**70**	290
	γ-muurolene	**9**	290
	γ-cadinene	**33**	290
	δ-cadinene	**100**	290
Pinus sp. (Chinese gum terpentine)	δ-cadinene	**100**	291
	γ-cadinene	**33**	291
	calamenene	**165**	291
Pinus sp. (Chinese terpentine oil)	γ-cadinol	**49**	376
	α-cadinol	**49**	376
Siberian spruce	γ-cadinene	**33**	299
Taiwania cryptomerioides Hayata		**62**	107
		81–83	133
	T-cadinol	**50**	96
	α-cadinol	**49**	96
	δ-cadinol	**74**	96
	T-muurolol	**76**	96
		56	102
		57	102
Piperaceae			
Piper sp. (Pepper)	δ-cadinene	**100**	345
	calamenene	**165**	345
Piper sp. (Japanese pepper)	γ_2-cadinene	**40**	282
	α-muurolene	**70**	282
	δ-cadinene	**100**	282
	γ-cadinene	**33**	282
	calamenene	**165**	282
	α-calacorene	**137**	282
Poaceae			
Avena sativa L. cv. Victory	γ-muurolene	**9**	384
Cymbopogon distans	γ-amorphene	**9**	255
(Nees ex Steud) Walt.	α-muurolene	**70**	255
	δ-cadinene	**100**	255
	γ-cadinene	**34**	255
Cymbopogon flexuosus Stapf.	δ-cadinene	**100**	256
	γ-cadinene	**34**	256
Cymbopogon flexuosus var. Egyptian	β-cadinene	**35**	406
Cymbopogon flexuosus var. Malabar	cadinene		407
Cymbopogon jwarancusa Schutt.	α-muurolene	**70**	320
	δ-cadinene	**100**	320
	calamenene	**165**	320
Cymbopogon winterianus Jowitt	γ-cadinene	**34**	253
	δ-cadinene	**100**	253
	1-cadinol		403
Vetiveria zizanioides (L.)	α-cadinol	**49**	88, 87

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
	khusinol	**42**	88, 89
	epi-khusinol	**43**	88, 87
	khusol	**45**	88, 87
	cadina-4α,10β-diol	**46**	88, 87
	khusinodiol	**44**	88, 87
	isokhusinoloxide	**47**	88, 87
	khusinoloxide	**48**	91
	khusilol	**183**	238
	khusitone	**184**	238
	khusitene	**185**	238
	khusitoneol	**186**	238
	methyl khusilate	**187**	239
Porifera (Marine sponge) *Halichondria* sp.	—	**15–17**	30
Rosaceae (Angiosperms)			
Rosa damascena Mill.	γ-cadinene	**33**	294
	δ-cadinene	**100**	249
	α-muurolene	**70**	294
Rosa jundzillii Bess.	γ-cadinene	**33**	295
	δ-cadinene	**100**	295
	α-muurolene	**70**	295
Rufaceae (Angiosperms)			
Boenninghausenia albiflora Reichb.	γ-cadinene	**33**	245
	cadalene	**126**	181
Scapaniaceae (Bryophyta)			
Scapania parvitexta	calamenene	**165**	364
Scapania robusta Horik	α-amorphene	**7**	363
	calamenene	**165**	363
Scapania undulata (L.)	(+)-*ent*-epicubenol	**78**	128
	γ-cadinene	**33**	129
Taxodiaceae (Gymnosperms)			
Cunninghamia konishii Hayata	α-cadinol	**49**	370
Metasequoia glyptostroboides	calamenene	**165**	366
Hu et Cheng	α-calacorene	**137**	366
	α-cadinol	**49**	366
Termitidae (Isoptera-Insect)			
Amitermis excellens	γ-cadinene	**33**	243
Reticulitermes flavipes	γ_1-cadinene	**36**	81
Kollar	γ_1-cadinene aldehyde	**59**	81
Reticulitermes virginicus	γ_1-cadinene	**36**	81
(Banks)	γ_1-cadinene aldehyde	**59**	81
Ternstroemiaceae (Angiosperms)			
Camellia sinensis (L.)	α-muurolene	**70**	314
Staph. (Black tea aroma)	δ-cadinene	**100**	314
	cadinol		314
Trichocoleaceae (Bryophyta)			
Neotrichocolea bissethii (Mitt.) Hatt.	calamenene	**165**	222
Trichocoleopsis sacculata (Mitt.) O Kam	calamenene	**165**	222
Trichocoleopsis tomentella (Ehrh.) Dum.	calamenene	**165**	222
Turneraceae (Angiosperms)			
Turnera diffusa Willd.	δ-cadinene	**100**	396
	calamenene	**165**	396
Turnera diffusa Willd.	δ-cadinene	**100**	396
var. *aphrodisiaca*	calamenene	**165**	396
Urticaceae (Angiosperms)			
Cecropia adenopus Martitus	δ-cadinene	**100**	313
	calamenene	**165**	313
Ulmus glabra Huds.		**127**	185
Ulmus rubra Muhl.		**127–130**	183
Verbenaceae			
Lippia americana L.	cadin-4-en-1-ol	**79**	130
Verbena sp.	T-muurolol	**76**	409
Vitex rotundifolia	α-muurolene	**70**	336

Table 1. *Continued*

Source	Compound isolated	Formula	Reference
L. Fill.	δ-cadinene	**100**	336
	α-cadinol	**49**	336
Vitaceae			
Vitis vinifera L.	α-muurolene	**70**	262
	γ-muurolene	**9**	262
	γ-cadinene	**33**	262
	δ-cadinene	**100**	262
	calamenene	**165**	262
Xenidae (Soft corals)			
Heteroxenia fuscescens	α-muurolene	**86**	134
	7-hydroxy muurolene	**87**	134
	7-acetoxy muurolene	**88**	134
Zingiberaceae			
Amomum globosum Lour	α-amorphene	**7**	11
(= *Alpania globosa* Horan)	δ-cadinene	**100**	11
Curcuma zedoaria Rosc.	pyrecurzenone	**136**	200
Hedychium spictum Ham ex Sm var. *acuuminatum*	γ-cadinene	**33**	411
Stahlianthus involucratus	stahlianthusone	**163**	219
	dihydrostahlianthusone	**164**	219
Zingiber officianale Rosc.	calamenene	**165**	417

group at C-2 [88] instead of an α-hydroxyl at C-3 as reported earlier [90]. β-Orientation of the hydroxyl group at C-10 in **44** seems to have biogenetic relationship with khusinoloxide (**48**) isolated from the same north Indian vetiver oil, in which the epoxide group is β-oriented [91]. Khusinol **42** has been converted into epikhusinol supporting structure **43** for the later [92]. Other hydroxycadinenes reported so far are (−)-α-cadinol (**49**) [60, 72, 93] (antipode of compound **41**), (+)-T-cadinol (**50**) [93–96], 3β-hydroxy-T-cadinol (**51**) [97], cubenol (**52**) [94, 98] and its mirror image (**53**) [99], calamenediol (**54**) [9, 100, 101], isocalamenediol (**55**) [100], cadina-4β,10β-diol (**56**) [102], cadinan-4β,10α-diol (**57**) [102] and a triol cadinan-4β,5α,10α-triol (**58**) [103]. The absolute stereochemistry of (−)-α-cadinol (**49**) was deduced from chemical correlation [60, 72] and its synthesis reported [104]. Yamamura *et al.* have reported the stereochemistry of **54** and **55** [105].

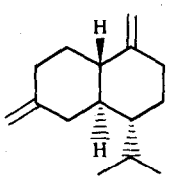
γ_1-Cadinene aldehyde (**59**) was isolated from the soldier termites of *Reticulitermes flavipes* and *R. virginicus* [81] and another aldehyde **60** was isolated along with the ketone **61** [106] from lavender oil. A ketocadinol **62** was isolated from *Taiwania cryptomerioides*, its stereochemistry was established by converting α-cadinol (**49**) to it [107] and by synthesis [104]. Schizandronol (**63**) was isolated from *Schizandra nigra* Max. and its structure deduced from spectral analysis and chemical correlation [108]. This is the second cadinene containing a primary alcohol group in it.

Six cadinenic acid methyl esters **64–69** were isolated from *Leucanthemopsis pulverulenta* and their structures were determined by spectral analysis and chemical correlations. The absolute configuration of all the six methyl esters was determined by chemical correlations and CD studies [109]. Compounds **34**, **37**, **39–48**, **53**, **59**, **63** are all antipodal cadinenes.

Muurolanes

Twenty-three compounds of this group have been isolated from various sources. Westfelt had isolated α-muurolene (**70**), γ-muurolene [(+)-γ-amorphene **9** [21]], and ε-muurolene (**71**) as the major components of *Pinus sylvestris* and Swedish sulphate turpentine and characterized them by spectral analysis and chemical transformations [20–23, 71, 73]. ε-Muurolene (**71**) was synthesized starting with trimethylsilylmethyl butadiene [76]. Stereochemistry of α-muurolene **70** was completely assigned from the X-ray studies of its diepoxides **72** and **73** [110].

One of the most important compounds of this group is torreyol [111] isolated from various natural sources. It occurs in both dextro and laevo rotatory forms and has been described under several names such as albicaulol [112–114], pilgerol [115], δ-cadinol [116, 117] and sesquigoyol [118], and its structure remained controversial for many years. Westfelt reviewed all the structural investigations and proposed structure **74** [111] for torreyol. The following year Lin *et al.* [119] reopened the controversy concerning the structure of (−)-torreyol. They interpreted some chemical and spectroscopic data and suggested that it should be the amorphane derivative **75**, but their evidence for this assignment still did not exclude Westfelt's [111] structure **74**. Rezvuchin *et al.* proposed (+)-torreyol as the enantiomer of **74** from LIS NMR and from conformational analysis studies [120–122]. Synthesis of (+)-torreyol [123, 124] provided additional information concerning the relative configurations at C-1, C-6 and C-7. Finally the configuration and conformation of (−)-torreyol have been established by a rigorous ^{1}H NMR study using LIS reagents as **74** [93]. Stereospecific total synthesis fully confirmed the structure **74** [124]. Torreyol has been detected by GC-MS in the wings of the male butterfly *Lycaeides argyrognomon*



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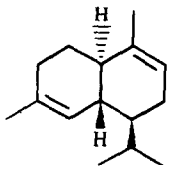

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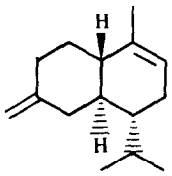

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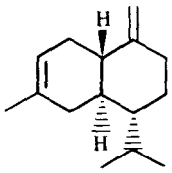

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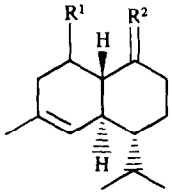
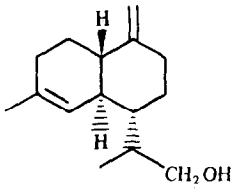


41 R^1 = H, R^2 = Me, β-OH
42 R^1 = β-OH, R^2 = CH_2
43 R^1 = α-OH, R^2 = CH_2
44 R^1 = β-OH, R^2 = Me, β-OH



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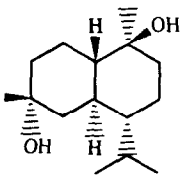

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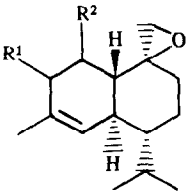
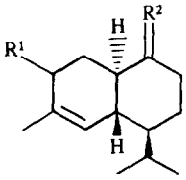
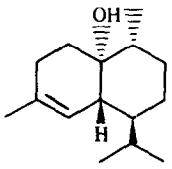


47 R^1 = H, R^2 = β-OH
48 R^1 = α-OH, R^2 = H



49 R^1 = H, R^2 = Me, α-OH
50 R^1 = H, R^2 = Me, β-OH
51 R^1 = β-OH, R^2 = Me, β-OH



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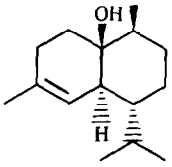

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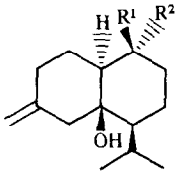
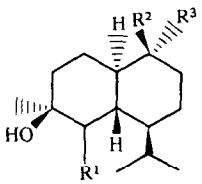
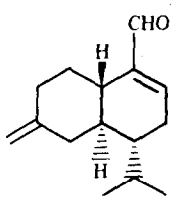


54 R^1 = OH, R^2 = Me
55 R^1 = Me, R^2 = OH



56 R^1 = H, R^2 = OH, R^3 = Me
57 R^1 = H, R^2 = Me, R^3 = OH
58 R^1 = α-OH, R^2 = Me, R^3 = OH



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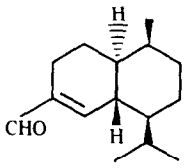

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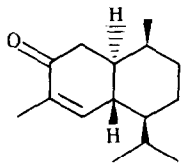
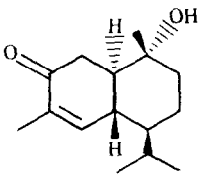


61

(Lepidoptera: Lycaenidae) and has been described to play an important role in the insect-courtship behaviour [125].

The C-10 epimer of (+)-torreyol, (−)-T-muurolol (**76**) was isolated from several sources as a mixture with T-cadinol (**50**) [96, 126, 127], its structure was assigned on the basis of chemical and spectroscopic studies and further supported by partial synthesis [94]. Extensive ^{1}H NMR studies using LIS reagents conclusively supported the structure **76** for (−)-T-muurolol [93]. (−)-Epicubenol **77** from commercial cubeb oil [98] and its enantiomer (+)-*ent*-epicubenol (**78**) from the liverwort *Scapania undulata* [128] were also isolated from *Alurolaxis selaginoides* Dax [129]. Isolation of two more muurolenic alcohols cadin-4-en-1-ol **79** [130] from the essential oil of *Lippia americana* and cadin-3-en-1-ol (isoepicubenol) (**80**) from *Heterotheca grandiflora* [131] were reported. Bohlmann and Giencke have synthesized **80** and found that the synthetic compound is not identical with isoepicubenol isolated from nature; therefore they have revised its structure as epicubenol (**77**) [132]. Two diols muurol-4-en-3α,10β-diol (**81**), muurol-4-en-3β,10β-diol (**82**) and an α,β-unsaturated keto alcohol muurol-4-en-10β-ol-3-one (**83**) were isolated from the wood of *Taiwania cryptomerioides*. Spectroscopic studies and chemical correlation of these three compounds with (−)-T-muurolol (**76**) have established their structures and stereochemistries [133]. Another α,β-unsaturated ketone (**84**) was reported [27] and its absolute stereochemistry was determined by chemical correlation with (**12**) [29]. A muurolenic angelate, **85** [131] and three muurolenes, **86–88** [134] were isolated and their structures and rela-



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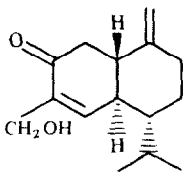

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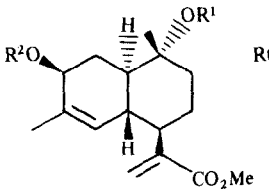
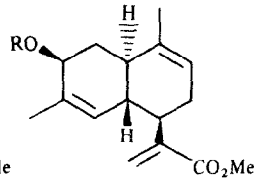
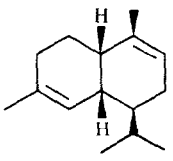


4 R^1 = H, R^2 = Ac
R^1 = H, R^2 = H
R^1 = Et, R^2 = Ac
R^1 = Et, R^2 = H



68 R = Ac
69 R = H



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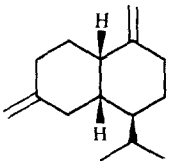

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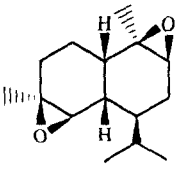

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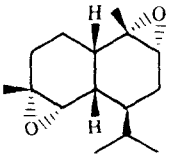

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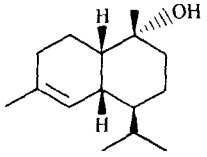

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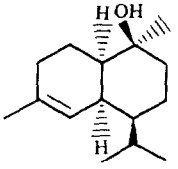

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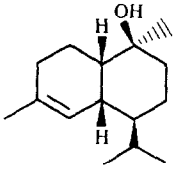

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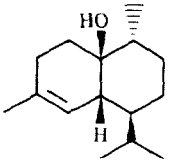

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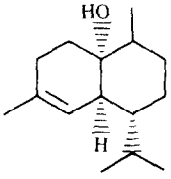

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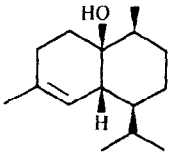

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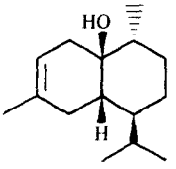

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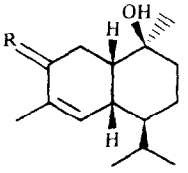


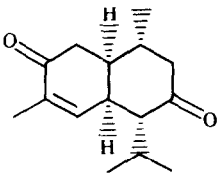
81 R = H, α-OH
82 R = H, β-OH
83 R = O

tive stereochemistries follow from spectral analysis and chemical correlations.

An α,β-unsaturated ketone named chiloscyphone was isolated from *Chiloscyphus polyanthus* [135] and its stereostructural assignments were made as **89** [136, 137]. In 1981 Grass reported the total synthesis of compound **89** but comparison of the spectral data of synthetic **89** and different α-methylenecyclohexanones led to the conclusion that chiloscyphone could not be represented by **89** [138]. This controversy was finally resolved by the isolation of a compound named chiloscypholone from *Chiloscyphus pallescens* which on dehydration gave compound **90** [139] identical with the naturally occurring compound from *C. polyanthus* [137].

4,10-Epoxymuurolene (**91**) was isolated from *Dilophus fasciola* [140]. Two other important compounds of this group are sclerosporin and sclerosporal isolated from *Sclerotinia fruticola* [141]. Initially these compounds were proposed to have the guaiane skeleton [142] which later were revised and structures **92** for sclerosporin and **93** for sclerosporal were assigned on the basis of high field NMR study and total syntheses [143–145]. It has been argued that synthetic compounds **92** and **93** are the natural enantiomers on the basis of their bioassay and CD spectral comparison [144]. *Heterotheca grandiflora* afforded four muurolenic acid methyl esters **94–97**. Extensive CD studies and chemical correlation studies showed **94** and **95** to be (1*S*,3*S*,6*S*,7*R*), and **96** and **97** to be (1*S*,3*S*,6*S*,7*R*,10*S*) [109]. Another muurolenic antipodal acid, γ-muurolene-11-oic acid (**98**) was isolated from *Trichogonia grazielae* and its stereochemistry established by extensive NMR and degradative studies [146].

Recently Extine *et al.* have carried out X-ray crystallographic studies of muurolene dihydrochloride showing its absolute stereostructure as **99** [147]. Muurolene dihydrochloride is a characteristic well defined crystalline deriva-



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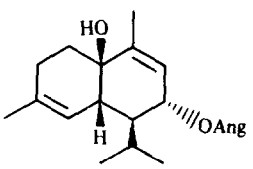

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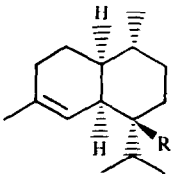
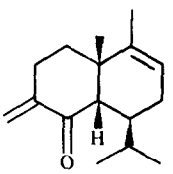


86 R = H
87 R = OH
88 R = OAc



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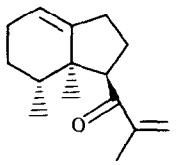

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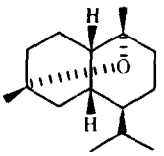

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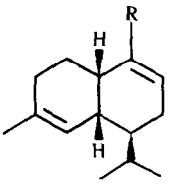
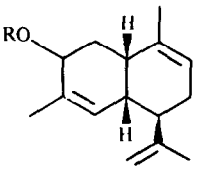
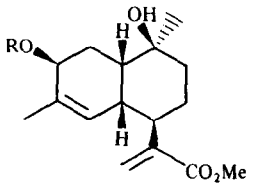


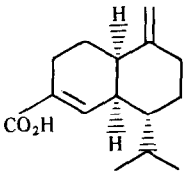
92 R = CO_2H
93 R = CHO



94 R = H
95 R = Ac



96 R = H
97 R = Ac



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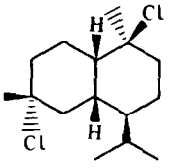

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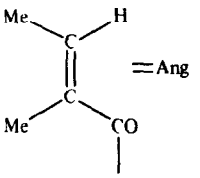

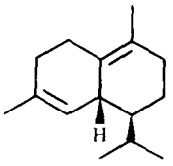

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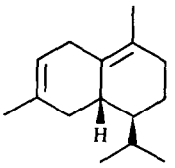

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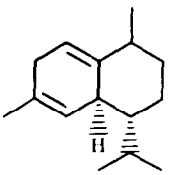

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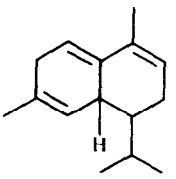

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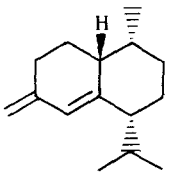

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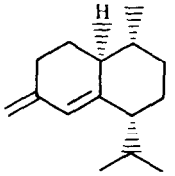

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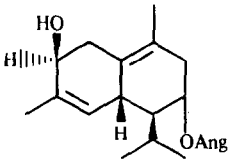

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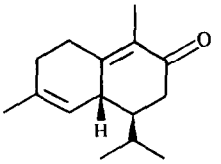


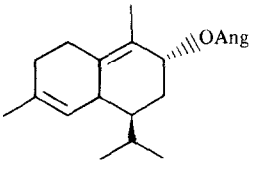
107

tive of the natural muurolenes **70** [23], **71** [71], and muurolols **76** and **77** [94].

Miscellaneous

Many other cadinenes have been isolated from natural sources which cannot be placed under any of the above four groups as only one or two out of the three asymmetric centres (used for subdivision) are present in them. However their stereochemical assignments are based on biogenetic considerations as most of them co-occur with other cadinenes of established stereochemistry. (+)-δ-cadinene (**100**) [60, 94, 148], ω-cadinene (**101**) [149], (+)-*ent*-cubebene (**102**) [128] and (−)-cadala-1,4,9-triene (**103**) [150] were isolated and their structures deduced from spectral analysis and chemical correlations. Absolute stereostructure [22, 60, 72] and synthesis [151, 152] of (+)-δ-cadinene **100** have also been reported. A compound isolated from Bulgarian *Mentha piperita* oil was described as δ-cadinene [149] which was later found to

be ω-cadinene **101** [94, 148]. δ-Cadinene **100** has been detected in more than one hundred sources (see Table 1). Structures of two other dienes bicyclosesquiphelandrene (**104**) and epibicyclosesquiphellandrene (**105**) were assigned on the basis of spectral studies and stereochemistry was deduced from chemical evidence [153] and confirmed by synthesis [154, 155]. The isopropyl group stereochemistry in **105** was established as pseudoequatorial [156]. Aerial parts of *Heterotheca grandiflora* provided two cadinenes, **106** and **107** and *H. subaxillaris* afforded **108** [131]. Eight furanocadinenes, **109** and **110** [157], 1-α-hydroxyverboccidentafuran (**111**) and verboccidentafuran (**112**) [158], **113**–**115** [159], and **116** [160] were isolated and their structures and relative stereochemistry followed from spectroscopic studies and chemical transformations. *Verbesina occidentalis* has provided four oxygenated cadinenes **117**–**120** whose relative stereochemistries were deduced from spectral analysis [158]. Compound **121** ($R^1 = H$, β-OAc, $R^2 = H$) isolated from *Eupatorium trapezoideum* [29] was found to be



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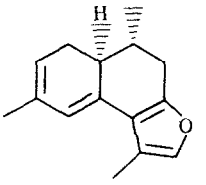

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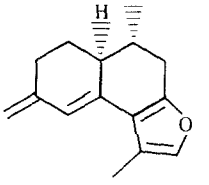

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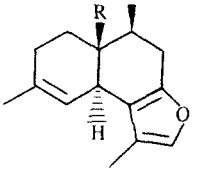
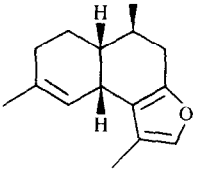


111 R = OH
112 R = H



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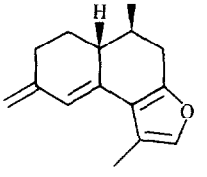

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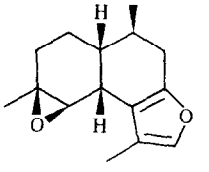

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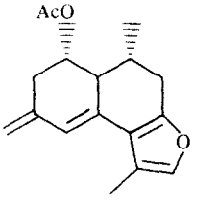

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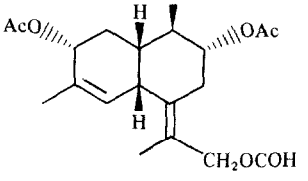

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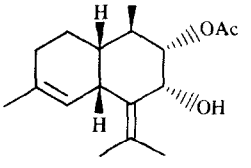

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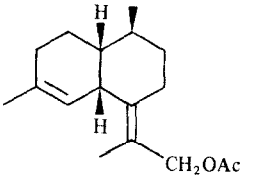

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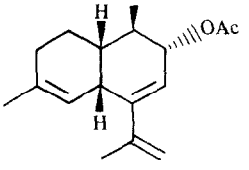

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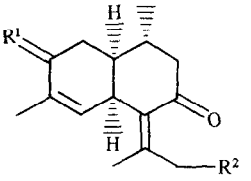

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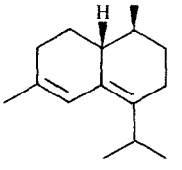
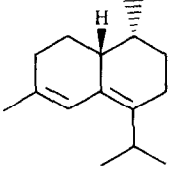


122

R^1 = OAc,H; R^2 = H
R^1 = O, R^2 = H
R^1 = O, R^2 = OH



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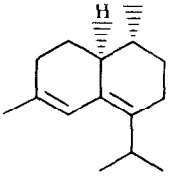
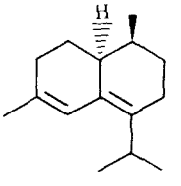


124



125

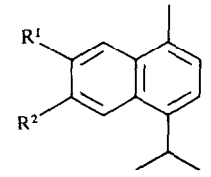
identical with one of the compounds from *Ageratina adenophora* (for others see under Amorphanes [26] and as it co-occurs with compounds **12–14**, its absolute stereochemistry is based on biogenetic considerations. The same biogenetic considerations could be advanced to assign the absolute stereochemistry to two other compounds **121** ($R^1=O$, $R^2=H$) and **121** ($R^1=O$, $R^2=OH$) isolated from *A. adenophora* [26].

Epizonarene (**122**) zonarene (**123**), with heteroannular conjugated diene system and their enantiomeric forms **124** and **125** respectively have been shown to occur widely in several essential oils [161] and in the brown seaweed *Dictyopteris zonarioides* [162]. A number of syntheses [28, 163, 164] and transformation [165] to zonarenes have been reported. Zonarenes are also produced in several rearrangement studies [166, 167]. Absolute stereochemistry of all the four isomers of zonarenes has been elucidated by Anderson *et al.* from CD studies by the application of transoid diene chirality rule [161, 168].

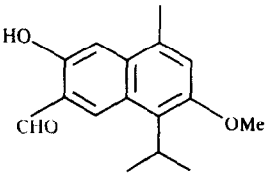
Cadalenes

Cadinenes containing both rings aromatized [napthalene derivatives] are known as cadalenes. So far 22 aromatic cadinenes have been detected which are widely distributed in nature. The detection of first aromatic cadinene cadalene (**126**) was intimately associated with the structure establishment of the first cadinene [2] and dates back to the early 1930s [169, 170]. The first cadinene and many other sesquiterpenes on dehydrogenation yielded cadalene (**126**) [5-(2-propyl)-3,8-dimethylnaphthalene]* which was the central skeleton of many sesquiterpenes. The structure of **126** was confirmed by synthesis [171–177]. It was also isolated from a number of natural sources [131, 178–182]. Rowe *et al.* [183, 184] and Lindgren and Svahn [185] have reported the iso-

*Numbering as naphthalene derivative.



126 R^1 = H, R^2 = Me
127 R^1 = OH, R^2 = Me
128 R^1 = OH, R^2 = CHO



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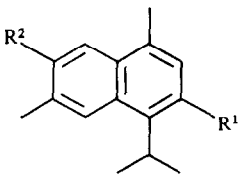
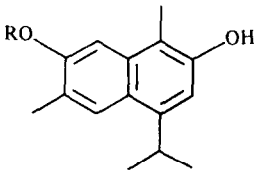
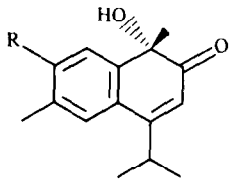


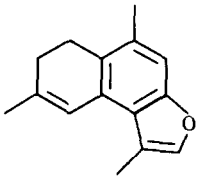
130 R^1 = OH, R^2 = H
131 R^1 = H, R^2 = OMe



132 R = H
133 R = Me



134 R = OH
135 R = OMe



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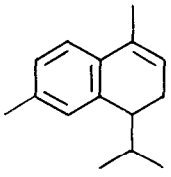

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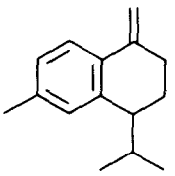

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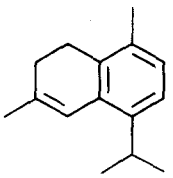

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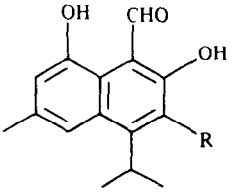
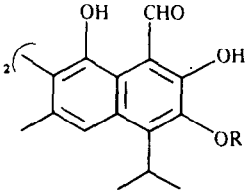
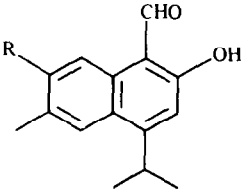


140 R = H
141 R = OH
142 R = OMe



143 R = H
144 1R = H, 1R = Me
145 R = Me



146 R = OH
147 R = OMe

lation of phenolic cadalenes, **127–130**, from elm woods *Ulmus rubra*, Muhl and *U. glabra* Huds. Compound **128** was also isolated from *Heterotheca parvifolia* [186–188]. Synthesis of compounds **127**, **128** [189, 190] and **129** [187] has also been described. Compounds **127** and 2-methoxycadalene **131** were isolated from *H. grandiflora* which co-occur with other cadinenes and norcadinenes, and their structures follow from spectroscopic studies [131].

Two cadalenic napthols 2,7-dihydroxycadalene (**132**) and 2-hydroxy-7-methoxycadalene (**133**) have been isolated from green and field-dried cotton bracts [191, 192] and have been synthesized [192, 193]. Compounds **132** and **133** autoxidize very rapidly on silica gel to lacinilene C (**134**) and lacinilene C- 7-methyl ether (**135**) respectively. Compounds **134** and **135** were also isolated from field-dried cotton leaves and bracts [194, 195]. Synthesis [196–198] confirmed the revised structures [193, 195, 197] **134** and **135**. Compounds **134** and **135** isolated from field-dried cotton leaves and bracts were optically active indicating that they are formed enzymatically from the naphthols **132** and **133** respectively. Compound **135** has been implicated as a causative of byssionosis, a chronic respiratory distress [199]. The 1,2-dihydrofuranocadalene pyrecurzenone was isolated from the rhizomes of *Curcuma zedoaria* and structure **136** was assigned to it [200] which was confirmed by synthesis [201] 5,6-Dihydrocadalene (α-calacorene) (**137**) and 5,6,7-trihydrocadalene (β-calacorene) (**138**) were isolated from *Heterotheca inuloides* and *H. grandiflora* and α-corocolene (**139**) was isolated from Japanese hop (*Humulus lupulus*). Their structures follow from spectral analysis and synthesis [202–205]. Calacorenes were also detected in several other sources (see Table 1).

Hemigossypol (**140**), 6-hydroxyhemigossypol (**141**) and 6-methoxyhemigossypol (**142**) were isolated from cotton plants (*Gossypum* sp.) infected with *Verticillium albo-atrum* [206]. Compounds **140** and **141** were also isolated from healthy cotton plants where they co-occur with dimeric gossypol (**143**), 6-methoxygossypol (**144**) and 6,6′-dimethoxygossypol (**145**) [207–209]. Isohemigossypol (**146**) and its 2-methyl ether (**147**) were isolated from *Bombax* sp. but further study is needed before their structures can be definitely ascertained [183, 209]. Compound **143** was found to be toxic to swine, poultry and rodents [210] and to insects [211]. It was also suggested to be an antitumour constituent of the Puerto Rican plant *Montezuma speciossima* [208]. It shows potent male fertility regulating activity and can be used as potential male oral contraceptive [208]. Gossypol was shown to induce persistent DNA-strand breaks in human leucocytes [212].

Quinones

Cotton plants (*Gossypum* sp.) have also afforded cadalenic quinone aldehydes, *p*-hemigossypolone **148**) and its 6-methyl ether (**149**) [213]. The terpenoid aldehyde quinones of *Bombax* sp. are probably isohemigossypolone (**150**) and isohemigossypolone-2-methyl ether (**151**) [209]. A dark pigment gossyrubilene (**152**) was isolated from the cotton plant and its structure was deduced by spectral data and confirmed by partial synthesis from hemigossypolone (**148**) [209].

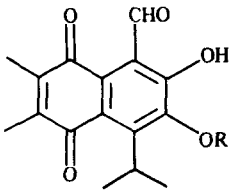
Twelve quinones have been isolated from *Mansonia altissima*. Mansonone A (**153**), mansonone B (**154**), mansonone C (**155**), mansonone D (**156**), mansonone E (**157**), mansonone F (**158**) were isolated by Marino Bettolo *et al.* [214, 215]. Mansonone I (**159**) was detected by Shimada *et al.* [216] and mansonone I, mansonone E, mansonone L (**160**), mansonone F, mansonone H (**161**), mansonone C and mansonone G (**162**) were detected by Galeffi *et al.* [217, 218]. Two other quinones stahlianthusone (**163**) and dihydrostahlianthusone (**164**) were detected in the essential oil from the bulb of *Stahlianthus involueratus* [219]. Synthesis of mansonone D (**156**) [220] and stahlianthusone (**163**) [221] has been reported. All these quinones are obviously derived from cadalene.

Calamenenes

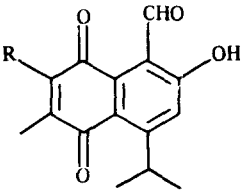
Calamenenes are A-ring aromatized cadinenes. The simplest calamenenes *cis*-calamenene (**165**) and *trans*-calamenene (**166**) were isolated from several essential oils [184, 222]. Many syntheses of calamenenes have been reported [223–227]. On the basis of extensive CD spectral studies and partial synthesis of calamenenes from sesquiterpenes of known absolute stereochemistry, the *cis*-structure **165** (7*R*,10*R*) was assigned to the major calamenene from Alaskan cedar and the *trans* structure **166** (7*S*,10*R*) to the common natural calamenene [184, 227, 228]. However, these results were based on the studies carried out with inseparable mixtures of *cis*- and *trans*-calamenenes. An Australian group of chemists, Croft *et al.* isolated calamenene and 3-hydroxycalamenene from *Eremophila drummondii*. This group then determined the absolute stereochemistry of 3-hydroxycalamenene as depicted in **167** from the X-ray crystallographic studies of its *p*-bromobenzoate [229]. Compound **167** was converted to the calamenene isolated from *E. drummondii* and therefore, has the complete structure **165** showing that the absolute configuration of the most common naturally occurring enantiomeric calamenene is (7*S*,10*S*) as in **168**. Their results also clarified the stereostructure of the synthetic calamenene obtained from dihydrokhusinol [230] and from carvone [227]. Rowe *et al.* have isolated 3-hydroxycalamen-11-al (**169**) from *Ulmus rubra* [183, 184]. 2-Methoxy- and 5-hydroxy-2-methoxycalamenenes have been detected in the marine gorgonian *Subergorgia hicksoni* and their structures were established as **170** and **171** respectively [231–233]. (10*R*,7*S*)-2-methoxycalamenene **172** was synthesized [234] from methyl dihydroxyserrulate (**173**)—a compound of known absolute stereochemistry. Compound **172** is the enantiomer of **170** isolated from *S. hicksoni* [231]. Several syntheses of **170** were also reported [232, 235]. Three 3,15-dihydroxycalamenene esters **174–176** were isolated from *Heterotheca grandiflora* collected from Hawaii [236]. Two calamenene angelate **177** and **178** have been detected in *H. subaxillaris* and their structures follow from spectroscopic analysis [237]. Other calamenenes isolated from *H. grandiflora* are **179–181** [131].

Norcadinenes

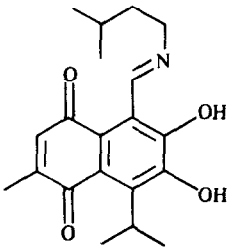
Nineteen norcadinenes **182–200** have been isolated from various sources. Chromoarnottion (**182**) was isolated from *Chromolaena arnottiana* [157], khusilal (**183**), khusitone (**184**), khusitene (**185**), khusitoneol (**186**) and khusilic acid methyl ester (**187**) [238, 239] were isolated from the oil of *Vetiveria zizaniodes* and compounds **188** and **189** were isolated from lavender oil [106]. Com-



148 R = OH
149 R = Me



150 R = OH
151 R = OMe



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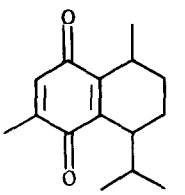

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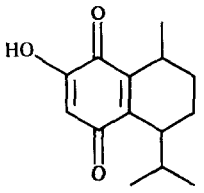

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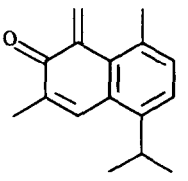

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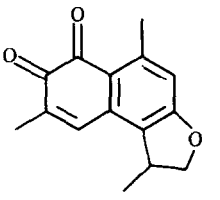

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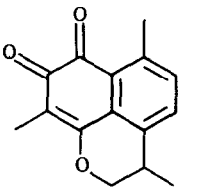

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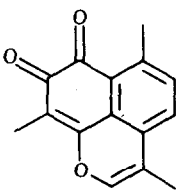

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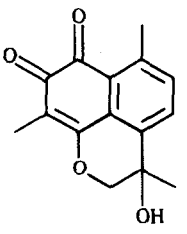

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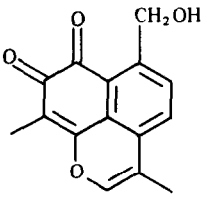

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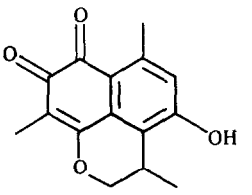

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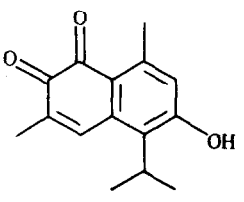

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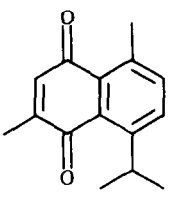

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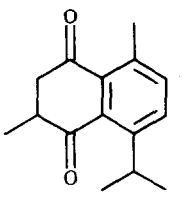

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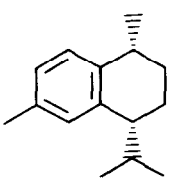

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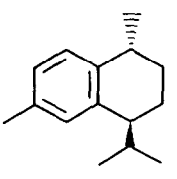

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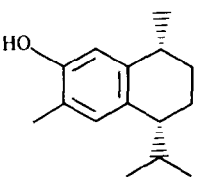

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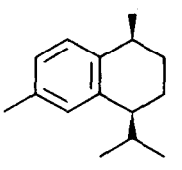

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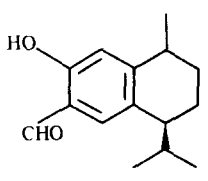

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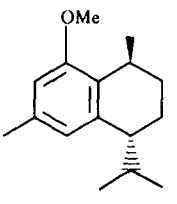

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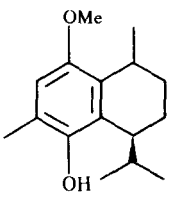

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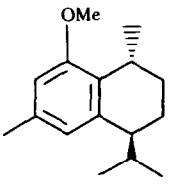

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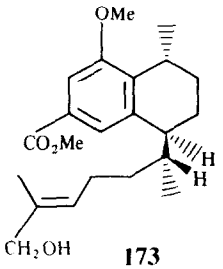

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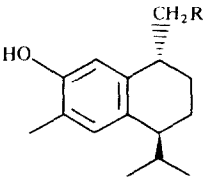
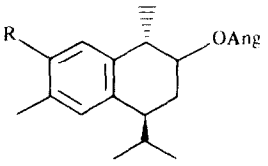
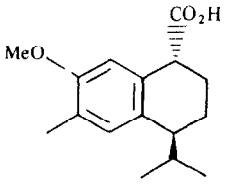


174 R = OAng
175 R = OMeBu
176 R = *iso*BuO



177 R = H
178 R = OH



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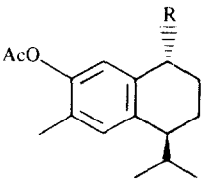
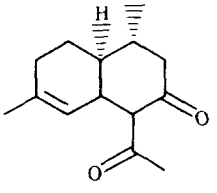


180 R = CO_2H
181 R = CH_2OAc

pound **190** co-occurs with **12–14** and **84** and therefore its absolute stereochemistry is based on biogenetic considerations. [29]. Degraded cadinenes **191–199** were isolated from *Heterotheca grandiflora* and their structures followed from spectral studies and chemical transformations. These norcadinenes are probably produced by decarboxylation of 3-methoxy-calamen-15-oic acid (**179**) [131]. Compound **200** is the only example of a norcadinene with a furan moiety. The absolute configuration of **200** was determined from the negative Cotton effect in CD [240]; this also supports the proposed stereochemistry of chromolaenin (**110**).



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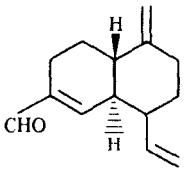

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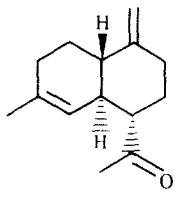

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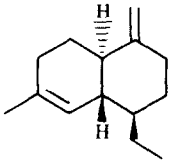

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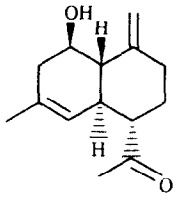

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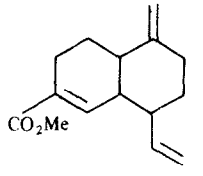

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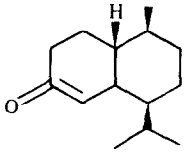

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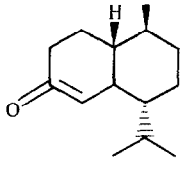

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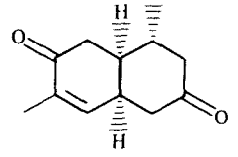

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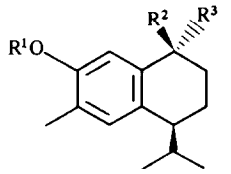

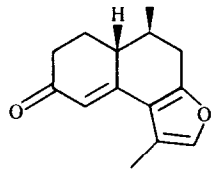

200

191 $R^1 = R^2 = R^3 = H$
192 $R^1 = Ac$, $R^2 = R^3 = H$
193 $R^1 = Me$, $R^2 = R^3 = H$
194 $R^1 = H$, $R^2, R^3 = O$
195 $R^1 = Me$, $R^2, R^3 = O$
196 $R^1 = R^2 = H$, $R^2 = OH$
197 $R^1 = H$, $R^2 = OH$, $R^3 = H$
198 $R^1 = Me$, $R^2 = H$, $R^3 = OH$
199 $R^1 = Me$, $R^2 = OH$, $R^3 = H$

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