Steroids and Terpenoids of the Genus *Ajuga*

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Summary: The biological and physiological activities exhibited by various species of *Ajuga* has created wide interest in its phytochemistry. This article describes the steroids and terpenoids reported so far from *Ajuga* species.

Introduction

Ajuga is one of the important genus of the family "*labiatae*". It is a large family comprising of about 170 genera and 3,000 species of world wide distribution, growing under great variety of soils and climates but more abundant in Mediterranean regions and in the hills. *Ajuga* genus contains 40 species and *Ajuga bracteosa* is one of them [1], which is found in Pakistan. Another species of this genus which is also found in Pakistan is *Ajuga parviflora*.

The family consists of aromatic annual or perennial glandular and hairy herbs but rarely shrubs. Leaves are nearly alternate, simple exstipulate with hairs. Flowers are in varticillaster inflorescence [2].

The genus *Ajuga* is of great medicinal and economic importance. For example *Ajuga bracteosa* is used in fever.

Ajuga genus is also used in other medicines i.e., as astringent [2] and in swollen wounds and as febrifuge in stomach ache, diarrhea, rheumatic, fevers, burns, tumours, bites of insects, eye trouble as well as diseases of bladder [3]. It is also used in females menstral disorder and urinary obstructions.

Ajuga spectabalis exhibits a stimulating activity on smooth and cardiac muscles [4].

Ajuga reptans has been considered good for the bad effect of excessive drinking, for lowering the pulse and lessening its frequency. It is also employed for cough and allays irritation of the throat [5].

Table-1: Steroids isolated from *Ajuga* species

No.	Name of Compound (Structure number)	Physical data (M.P.=°C)	Source	Ref. No.
1.	Ecdysone (1) $C_{27}H_{44}O_6$ MW = 464		A.rep.	[21]
2.	20-Hydroxyecdysone. (2) $C_{27}H_{44}O_7$ MW = 480		"	"
3.	Polypodine. (3) $C_{27}H_{44}O_8$ MW = 496		"	"
4.	Cyasterone (4) $C_{29}H_{44}O_8$ MW = 520	M.P. 164-166 $[\alpha]_D$ + 64.5 (c=1.0, H_2O)	"	"
5.	29-Norcyasterone (5) $C_{28}H_{42}O_8$ MW = 520		"	"
6.	29-norsengosterone (6) $C_{28}H_{42}O_9$ MW = 522		"	"
7.	Ajugalactone (7) $C_{29}H_{40}O_8$ MW = 516	M.P. 225-235	"	"
8.	Ponesterone-A (8) $C_{29}H_{44}O_7$ MW = 480		A.jap.	[22]
9.	Ajugasterone-C (9) $C_{29}H_{44}O_6$ MW = 464			
10.	Makisterone-C (10) $C_{29}H_{48}O_7$ MW = 508			
11.	Makisterone-D (11) $C_{28}H_{48}O_7$ MW = 508		"	[23]
12.	Ajugasterone-B (12) $C_{28}H_{46}O_7$ MW = 506	M.P. 240	A.Inc.	"
13.	Ajugasterone-B (13) tetra acetate $C_{37}H_{53}O_{11}$ MW = 677	M.P. 165-167	"	"
14.	β-Sitosterol (14) $C_{29}H_{50}O$ MW = 414.69	M.P. 136-137 $[\alpha]_D$ - 35° ($CHCl_3$)	A.bra.	[24,25]
15.	α-Sitosterol (15) $C_{29}H_{48}O$ MW = 412	M.P. 147-148 $[\alpha]_D$ -47.7° ($CHCl_3$)	"	"
16.	Turkesterone (16) $C_{27}H_{44}O_{28}$ MW = 496		A.tur.	[15]
17.	Ecdysterone (17) $C_{28}H_{44}O_7$ MW = 480	M.P. 240-241	A.dec. A.Inc. A.rep	[2-]
18.	Makisterone-A (18) $C_{28}H_{46}O_7$ MW = 494	M.P. 263-265	"	[27]
19.	22-Oxocyasterone. (19) $C_{29}H_{42}O_8$ MW = 518		"	[28]
20.	24,25-dehydroprecyasterone. (20) $C_{29}H_{42}O_8$ MW = 518		"	"

Table-1: . Continued

No.	Name of Compound (Structure number)	Physical data (M.P.=°C)	Source	Ref. .No.
21.	HO-cyasterone. $C_{29}H_{44}O_9$ MW = 536	M.P. 165-167	"	[27]
22.	Sengosterone $C_{29}H_{44}O_9$ MW = 536	$[\alpha]_D$ -47.5 (c=1.0, $CHCl_3$)	A.rep. A.Inc.	[21,23,30]
23.	Ajugasterone-A $C_{28}H_{42}O_7$ MW = 478		A.rep. A.Inc.	[21,23,30]
24.	Ajugasterone-D $C_{28}H_{42}O_7$ MW = 478		"	"
25.	Clerosterol		A.rep.	[15]
26.	22,23-Didehydro-clerosterol		"	"
27.	C-29 Monohydroxylated sterol.		"	"

Table-2: Terpenoid isolated from *Ajuga* species.

No.	Name of Compound (Structure number)	Physical data (M.P.=°C)	Source	Ref.No.
1.	Ajugamarin-A1 (21) $C_{29}H_{40}O_{10}$ MW = 548		A.nip	[31]
2.	Ajugamarin-B1 (22) $C_{24}H_{34}O_9$ MW = 550		"	"
3.	Ajugamarin-Cl (23) $C_{24}H_{34}O_9$ MW = 468	$[\alpha]_D$ +5.5° ($CHCl_3$)	"	"
4.	Ajugamarin-B2 (24) $C_{31}H_{44}O_{11}$ MW = 593	M.P. 154-155 [α] - 25° ($CHCl_3$)	"	"
5.	Ajugamarin-B3 (25) $C_{22}H_{32}O_8$ MW = 424	[α] +22° ($CHCl_3$)	"	"
6.	Ajugamarin-D1 (26) $C_{29}H_{44}O_{11}$ MW = 569	[α] +14° ($CHCl_3$)	"	"
7.	Ajugamarin-A2 (27) $C_{31}H_{42}O_{11}$ MW = 590		A.dec.	[19]
8.	Ajugamarin-G1 (28) $C_{34}H_{48}O_{11}$ MW = 632	M.P. 165-167 [α] -37° ($CHCl_3$)	"	"
9.	Ajugamarin-H1 (29) $C_{34}H_{48}O_{11}$ MW = 632	M.P. 75-79 [α] -11° ($CHCl_3$)	"	"
10.	Ajugamarin-F4 (30) $C_{29}H_{42}O_9$ MW = 535	M.P. 160-163 [α] -32° ($CHCl_3$)	"	"
11.	Ajugamarin-B4 (31) $C_{27}H_{40}O_9$ MW = 509	[α] + 20.2° ($CHCl_3$)	A.cil	[32]
12.	Ajugamarin-B5 (32) $C_{29}H_{42}O_{10}$ MW = 551	[α] -7.3° ($CHCl_3$)	"	"
13.	Ajugamarin-E1 (33) $C_{27}H_{40}O_9$ MW = 509	[α] -19° ($CHCl_3$)	"	"
14.	Ajugamarin-E2 (34) $C_{27}H_{40}O_9$ MW = 509	[α] -19° ($CHCl_3$)	"	"
15.	Ajugamarin-E3 (35) $C_{29}H_{42}O_{10}$ MW = 551	[α] - 37° ($CHCl_3$) M.P. 161-163	"	"
16.	Ajugamarin-F1 (36)	$[\alpha]_D$ -17.9° ($CHCl_3$)	"	"
17.	Ajugamarin-F3 (37) $C_{27}H_{41}O_8$ MW = 493	$[\alpha]_D$ -1.4° ($CHCl_3$)	"	"

Table-2 Continued

No.	Name of Compound (Structure number)	Physical data (M.P.=°C)	Source	Ref.No.
18.	Deacetylajuarin-iv (38) $C_{21}H_{32}O_8$ MW = 363	M.P. 2.6-207 $[\alpha]_D$ -16° ($CHCl_3$)	"	"
19.	Ajugarepttansin (39) $C_{29}H_{44}O_{10}$ MW = 552	M.P. 210-212 $[\alpha]_D$ -28° (CCl_4)	A.rep.	[33]
20.	Ajugapitin (40) $C_{29}H_{42}O_{10}$ MW = 551	M.P. 196-198 [α] -70.3° ($CHCl_3$)	A.cha.	[26]
21.	15-ethoxy-14- (41) hydroajugapitin	$[\alpha]_D$ -3.53° ($CHCl_3$)	"	"
22.	14-hydro-15- (42) hydroxyajugapitin $C_{29}H_{44}O_{11}$ MW = 568		"	"
23.	Chamaepitin (43) $C_{21}H_{46}O_{13}$ MW = 626		"	"
24.	Ajugavensin-A (44) $C_{29}H_{43}O_9$ MW = 535	M.P. 139.41 $[\alpha]_D$ -42.9° ($CHCl_3$)	A.Jen	[16]
25.	Ajugavensin-B (45) $C_{29}H_{41}O_9$ MW = 533	M.P. 143-145 $[\alpha]_D$ -53.2° ($CHCl_3$)	"	"
26.	Ajugavensin-C (46) $C_{27}H_{38}O_8$ MW = 490	M.P. 140-42 $[\alpha]_D$ + 1.0° ($CHCl_3$)	"	"
27.	Ajugarin-I (47) $C_{24}H_{37}O_7$ MW = 434		A.rem	[34]
28.	Ajugarin-11 (48) $C_{22}H_{32}O_6$ MW = 392		"	"
29.	Ajugarin-111 (49) $C_{24}H_{36}O_8$ MW = 452		"	"
30.	Ajugarin-iv (50) $C_{23}H_{34}O_6$ MW = 452	M.P. 119-120 $[\alpha]_D$ -57.5° ($CHCl_3$)	"	[35]
31.	Ajugarin-v (51) $C_{22}H_{32}O_5$ MW = 406	M.P. 217-218 $[\alpha]_D$ -13.5 ($CHCl_3$)	"	"
32.	2-Acetylivain-1 (52) $C_{30}H_{44}O_{11}$ MW = 580	$[\alpha]_D$ + 15.9° ($CHCl_3$)	A. Pse	[36]
33.	14,15-dihydro- (53) ajgapitin $C_{29}H_{44}O_{10}$ MW = 552		A.iva	[37]
34.	Ivain-I (54) $C_{28}H_{40}O_{10}$ MW = 536		A.iva	[37]
35.	Ivain-II (55) $C_{28}H_{40}O_9$ MW = 520		"	"
36.	Ivain-III (56) $C_{29}H_{44}O_{10}$ MW = 553		"	"
37.	Ivain-IV (57) $C_{29}H_{42}O_{10}$ MW = 551		"	"
38.	Ajuga cumbin-3 (58) $C_{25}H_{38}O_7$ MW = 550		A.dec.	[20]
39.	Ajugacumbin-F (59) $C_{31}H_{40}O_{12}$ MW = 604		"	"

Table-2 Continued

No.	Name of Compound (Structure number)	Physical data (M.P.=°C)	Source	Ref.No.
40.	Ajugareptansone-A (60) $C_{29}H_{40}O_{10}$ MW = 548		A.rep.	[38]
41.	Ajugareptansone-B (61) $C_{24}H_{30}O_8$ MW = 440		"	"
42.	Betulinic acid (62) $C_{30}H_{48}O_3$ MW = 456	M.P. 293-297 $[\alpha]_D$ -11° (CH_3OH)	A.brac	[39]

		A.rem. = *Ajuga remota*	
		A.pse. = *Ajuga pseduoiva*	
Ref.	= Reference	A.dec. = *Ajuga decumbens*	
A.nip.	= *Ajuga nipponensis*	A.tur. = *Ajuga turkestanica*	
A.	= *Ajuga*	A.rep. = *Ajuga reptans*	
A.cil	= *Ajuga ciliata*	A.inc. = *Ajuga incisa*	
A.bra.	= *Ajuga bracteosa*		
A.japn.	= *Ajuga japonica*		
A.cha.	= *Ajuga chamaepytis*		
A.jen.	= *Ajuga jenevensis*		

Insect antifeedant activity of clerodane diterpenoids from *Ajuga chamaepytis*, *Ajuga iva* and *Ajuga reptans* has been studied against larvae of *Spodoptera littoralis*. Antifeedant activities of nine clerodane diterpenoids, isolated from different species of *Ajuga* plants, were studied against larvae of the Egyptain cotton leaf worm *Spodoptera littoralis* by the application of leaf disk method [6].

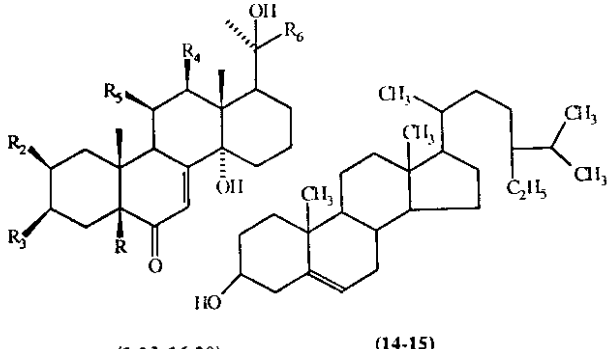
Phytoecdysones have been reported from *Ajuga remota* as insect ecdysis inhibitors and feeding deterrants. β-Ecdysone, cyasterone, ponasterone-A, ajugasterone-C and ajugalactone were separated by droplet counter current chromatography from *Ajuga remota*, a plant resistant to insects. Ponasterong A strongly inhibited the growth and ecdysis of *Pectinophora gossypiella* and *Bombyx mori* larvae. Ajugalactone was inactive and the three other compounds showed moderate activity. β-Ecdysone or ponsterone A treatment of *Bombyx mori* had little effect on the ascorbic acid content and decreased the phenoloxidase activity in the molting fluid. Ajugasterone C was 710-fold potent than cyasterone as a deterrant for *Schizaphis graminum* when added to the diet [7]. Herbicidal and pesticidal activities have also been reported from *Ajuga reptans* and *Ajugá lupulina* respectively [8,9]. Cyasterone, an insect molting hormone, has been isolated from *Ajuga chia* [10,11].

β-Sitosterol, 8-O-acetyl harpagide and betulinic acid were isolated from the aerial parts of *Ajuga bracteosa* [39] while ajugalactone, cyasterone, makisterone A and β-ecdysone were found in *Ajuga chamaepytis* [12]. Clerosterol, two C_{23}-monohydroxylated sterols, 22, 23-didehydro clerosterol have been reported from *Ajuga reptans* [13].

From the leaves of *A. turkestanica*, cyasterone and ecdysterone while from its roots turkesterone have been reported [14,15].

Three neo-clerodane diterpenoids, Ajugavensins A,B and C have been isolated from the ethyl acetate extract of the aerial parts of *Ajuga jenevensis* [16]. Four bitter neo-clerodane, ajugamarins CI, B2, B3 and D1 and ajugarin i, ii and iii have been isolated from *Ajuga remota* [17].

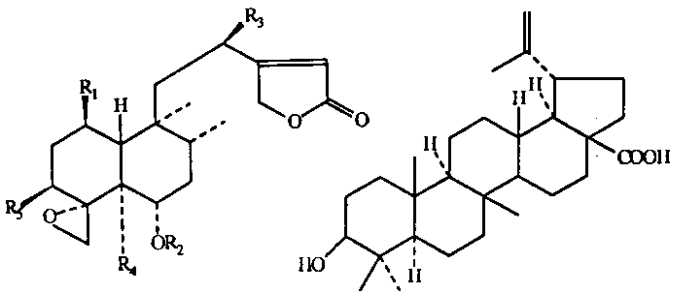
Three new bitter principals ajugamarin dihydroajugamarin and ajugamarin chlorohydrin were isolated from the leaves of *Ajuga nipponensis*. The absolute structure of ajugamarin was determined by x-ray analysis of ajugamarin-



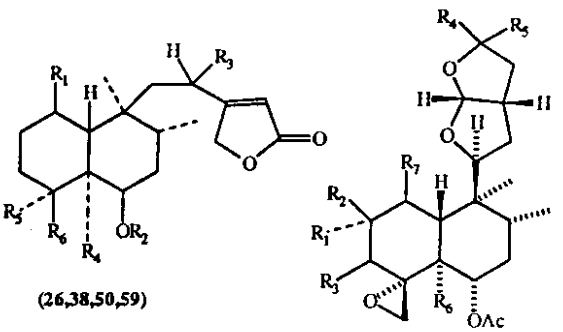
(1-13, 16-20) (14-15)

S.No.	R_1	R_2	R_3	R_4	R_5	R_6
1	H	OH	OH	H	H	CH_2 CH_2 CH_2 $C(CH_3)_2$ OH
2	H	OH	OH	H	H	CHOH CH_2 CH_2 $C(CH_3)_2$ OH
3	OH	OH	OH	H	H	CHOH CH_2 CH_2 $C(CH_3)_2$ OH
4	H	OH	OH	H	H	CHOH CH_2 CH CH(CH_3) OCOCH CH_3
5	H	OH	OH	H	H	CHOH CH_2 CH CH_2 OCOCHCH$_3$
6	OH	OH	OH	H	H	CHOH CH_2 CH CH_2 OCOCHCH$_3$
7	H	OH	OH	H	=O	CH CH_2 C(Et) $C(CH_3)$ COO
8	H	OH	OH	OH	H	CHOH CH_2 CH_2 CH $(CH_3)_2$
9	H	OH	OH	H	H	CHOH CH_2 CH_2 CH $(CH_3)_2$
10	H	OH	OH	H	H	CHOH CH_2 CH (Et)C$(CH_3)_2$OH
11	H	OH	OH	H	H	CHOH CH_2 CH [CH(CH_3) OH]CH $(CH_3)_2$
12	H	OH	OH	H	H	CHOH CH_2 CH (Et) C(CH_3) CH_2OH
13	H	OAc	OAc	H	H	CH_2OAc CH_2 CH (Et) C(CH_3) CH_2OAc
16	H	OH	OH	OH	H	CHOH CH_2 CH_2 C $(CH_3)_2$ OH
17	H	OH	OH	H	H	CHOH CH_2 CH_2 C $(CH_3)_2$ OH
18	H	OH	OH	H	H	CHOH CH_2 CH (CH_3) C $(CH_3)_2$OH
19	H	OH	OH	H	H	COCH$_2$ CH CH (CH_3) OCO CHCH$_3$
20	H	OH	OH	H	H	CHOH CH_2C=C (CH_3) OCO CH CH_3
14	-	-	-	-	-	-
15[a]	-	-	-	-	-	-

(a) C_4-C_5 double bond.



(62)



(26,38,50,59)

(39-46,52-57)

(21-25,27-37,47-49,51,58,60-61)

St.No.	R_1	R_2	R_3	R_4	R_5
21	O-tigloyl	Ac	OH	CH_2OAc	H
22	O-Me Bu	Ac	OH	CH_2OAc	H
23	OH	Ac	OH	CH_2OAc	H
24	O-MeBu	Ac	OAc	CH_2OAc	H
25	O-MeBu	H	OH	CH_2OAc	H
27	O-tiglyl	Ac	OAc	CH_2OAc	OH
28	O-tiglyl	Ac	O-MeBu	CH_2OAc	H
29	O-MeBu	Ac	O-tigloyl	CH_2OAc	H
30	H	Ac	O-MeBu	CH_2OAc	H
31	O-MeBu	Ac	OH	CH_2OH	H
32	O-MeBu	Ac	OAc	CH_2OH	H
33	OH	Ac	O-MeBu	CH_2OH	H
34	OH	H	O-MeBu	CH_2OAc	H
35	OH	Ac	O-MeBu	CH_2OAc	H
36	H	H	O-MeBu	CH_2OH	H
37	H	H	H	CH_2OAc	H
47	H	Ac	H	CH_2OAc	H
48	H	Ac	H	CH_2OAc	H (6-OH)
49	H	Ac	H	CH_2OAc	H(4,17-Diol)
51	H	Ac	H	CH_3	H
58	OAc	Ac	H	$CH_2OCOCCH_3$ $CHMeOH$	CO_2 CHMeEt
60	=O	Ac	H	CH_2OAc	H
61[b]	=O	Ac	H	CH_2OAc	H
62	-	-	-	-	-

(b) C_3-C_7 double bond

St.No.	R_1	R_2	R_3	R_4	R_5	R_6	R_7
26	O-Me Bu	H	OH	Ac	OH	OAc	
38	H	H	H	Me	H	$COO CH_3$	
50	H	Ac	H	Me	H	$COO CH_3$	
59	H	H	H	CH_2OCOC Me CHMe	OH	CH_2OH	
39	H	H	OH	H	H	CH_2OAc	CO_2CHCH_3Et
40[c]	OH	H	$OCOCH_3$ CHEt	H	H	CH_2OAc	H
41	OH	H	$BrCHCH_3$ COO	H	OEt	CH_2OAc	H
42	OH	H	$BrCHCH_3$ COO	H	OH	CH_2OAc	H
43	OH	H	CH_3CHOAc $CHCH_3COO$	H	OH	CH_2OAc	H
44	H	H	H	H	H	CH_2OAc	$OCOCHCH_3Et$
45	H	H	H	H	H	CH_2OAc	$OCOCCH_3CHCH_3$
46	H	H	H	H	H	$CH_2OCOCCH_3$ $CHCH_3$	OH
52	H	MeCOO	$COOCH_3$ $CHCH_3$	H	H	$OCO CH_3$	H
53	OH	H	$COOCH_3$ CHEt	H	H	$OCO CH_3$	H
54	H	H	$COOCH_3$ $CHCH_3$	H	H	CH_2OAc	H
55	H	H	$COOCH_3$ $CHCH_3$	H	H	CH_2OAc	H
56	H	OH	$COOCH_3$ $CHCH_3$	Et	H	CH_2OAc	H
57	H	OH	$COOCH_3$ CHEt	H	H	CH_2OAc	H

(c) C_{14}-C_{15} double bond; St. No. = Structure number.

bromobenzoate. Ajugamarin chlorohydrin has been reported to have been formed by addition of hydrochloric acid to ajugamarin during the isolation procedure [18].

From *Ajuga decumbens* five neo-clerodane diterpenes, ajugamarin G1, A2, H1, F4 and B2 have been isolated [19]. Study on the chemical constituents of the *Ajuga decumbens* also resulted in the isolation of two new minor neo-clerodane diterpenes ajugacumbins E and F [20].

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