

Review

Research Progress on Sesquiterpenes from the Genus *Ainsliaea*

Hui Zhang ^{1,†}, Ru-Ru Sun ^{1,†}, Ya-Feng Liu ^{1,2,3}, Xin Guo ^{1,2,3}, Chong-Long Li ^{1,2,3}, Ze-Dong Nan ^{1,2,3,*} and Zhi-Bo Jiang ^{1,2,3,*} 

¹ School of Chemistry and Chemical Engineering, North Minzu University, Yinchuan 750021, China; zh204017596@163.com (H.Z.); srr2375764@163.com (R.-R.S.); liuyf@nmu.edu.cn (Y.-F.L.); guoxin@nmu.edu.cn (X.G.); chlong@nmu.edu.cn (C.-L.L.)

² Ningxia Key Laboratory of Solar Chemical Conversion Technology, North Minzu University, Yinchuan 750021, China

³ Key Laboratory for Chemical Engineering and Technology, State Ethnic Affairs Commission, North Minzu University, Yinchuan 750021, China

* Correspondence: nanzdchem@163.com (Z.-D.N.); j836811@163.com (Z.-B.J.)

† These authors contributed equally to this work.

Abstract: Sesquiterpenes constitute the principal components of the genus *Ainsliaea*, encompassing guaiane, germacrane, eudesmane, and polymer sesquiterpene lactones types. These secondary metabolites exhibit diverse pharmacological activities, including antitumor, antibacterial, anti-inflammatory, antiviral, antioxidant, hepatoprotective, and neuroprotective effects. Through a comprehensive literature search of the Web of Science, PubMed, SciFinder, and CNKI databases, it was discovered that there are as many as 145 main sesquiterpenoids in the genus *Ainsliaea*. However, the nuclear magnetic resonance (NMR) data for the sesquiterpenes in this genus have not been systematically compiled and summarized. Therefore, this review aims to highlight the chemical structures, NMR data, and pharmacological activities of sesquiterpenes in *Ainsliaea*. By meticulously analyzing published scholarly literature, our goal is to provide a solid foundation for further exploration of new sesquiterpenes and extensive utilization of this genus.

Keywords: *Ainsliaea*; sesquiterpenes; nuclear magnetic resonance (NMR); structure analysis



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1. Introduction

Ainsliaea is a perennial herb, with over seventy species mainly distributed in southeast Asia. In China, there are forty-four species and four varieties. The majority of these plants are found around the Yangtze River Basin, with only one species distributed in the northeast. They are typically harvested in summer and autumn, and the entire plants are used for Chinese medicinal purposes [1]. According to the ‘*Supplements to the Compendium of Materia Medica*’, it is documented that *Ainsliaea* has a sweet and mild taste, cold properties, and belongs to the lung, spleen, and large-intestine meridians. The ‘*Comprehensive Dictionary of Chinese Herbal Medicine*’ states that *Ainsliaea* has the functions of clearing heat, promoting diuresis, cooling blood, and detoxification. *Ainsliaea fragrans* Champ. Is the primary ingredient in the national protected Chinese medicine ‘Xingxiang Tu’erfeng’ herbal granules and herbal tablets.

Since the 1980s, several studies have been conducted on the chemical composition and pharmacological activities of the genus *Ainsliaea*, leading to the discovery of over 400 compounds. The chemical constituents of this genus mainly include sesquiterpenoids, triterpenoids, steroids and their derivatives, phenolic acids, flavonoids, anthraquinones, coumarins, lignans, essential oils, and other components. Chemical studies have revealed that sesquiterpenes are the characteristic components of *Ainsliaea* plants [2,3]. The investigation has shown that sesquiterpenes in *Ainsliaea* plants mainly consist of guaiane, germacrane, eudesmane, polymer sesquiterpene lactones, and others [4]. However, NMR spectroscopy data for these sesquiterpenes derivatives have not been reported. This paper

aims to provide references for the analysis and identification of new structural compounds by summarizing the ^1H - and/or ^{13}C -NMR data of 145 sesquiterpenes from the genus *Ainsliaea* between 1979 and 2022 through consulting the relevant literature.

2. Guaiane-Type Sesquiterpene

Guaiane sesquiterpenes are a class of compounds with three isoprene units consisting of 5,7 fused rings, which are substituted by 4,10-dimethyl-7-isopropyl moieties as the basic nucleus. These compounds possess antibacterial, anti-inflammatory, antitumor, neuroprotective, and other biological activities [5]. Thus far, a total of 63 guaiane sesquiterpenes have been reported in this genus, mainly 12,6 guaican-type sesquiterpene lactones. The structures and detailed information are listed in Figure 1 and Table 1.

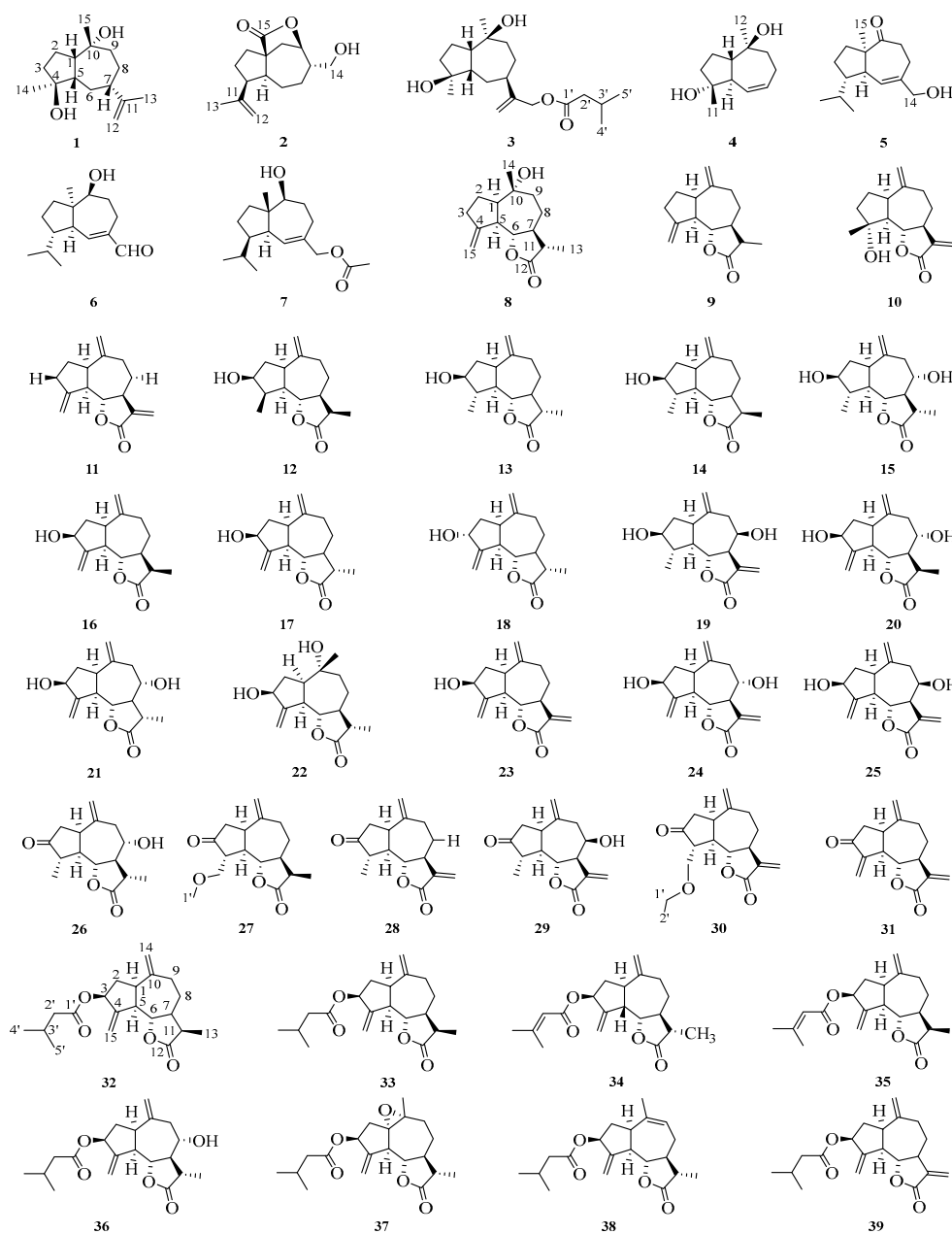


Figure 1. Cont.

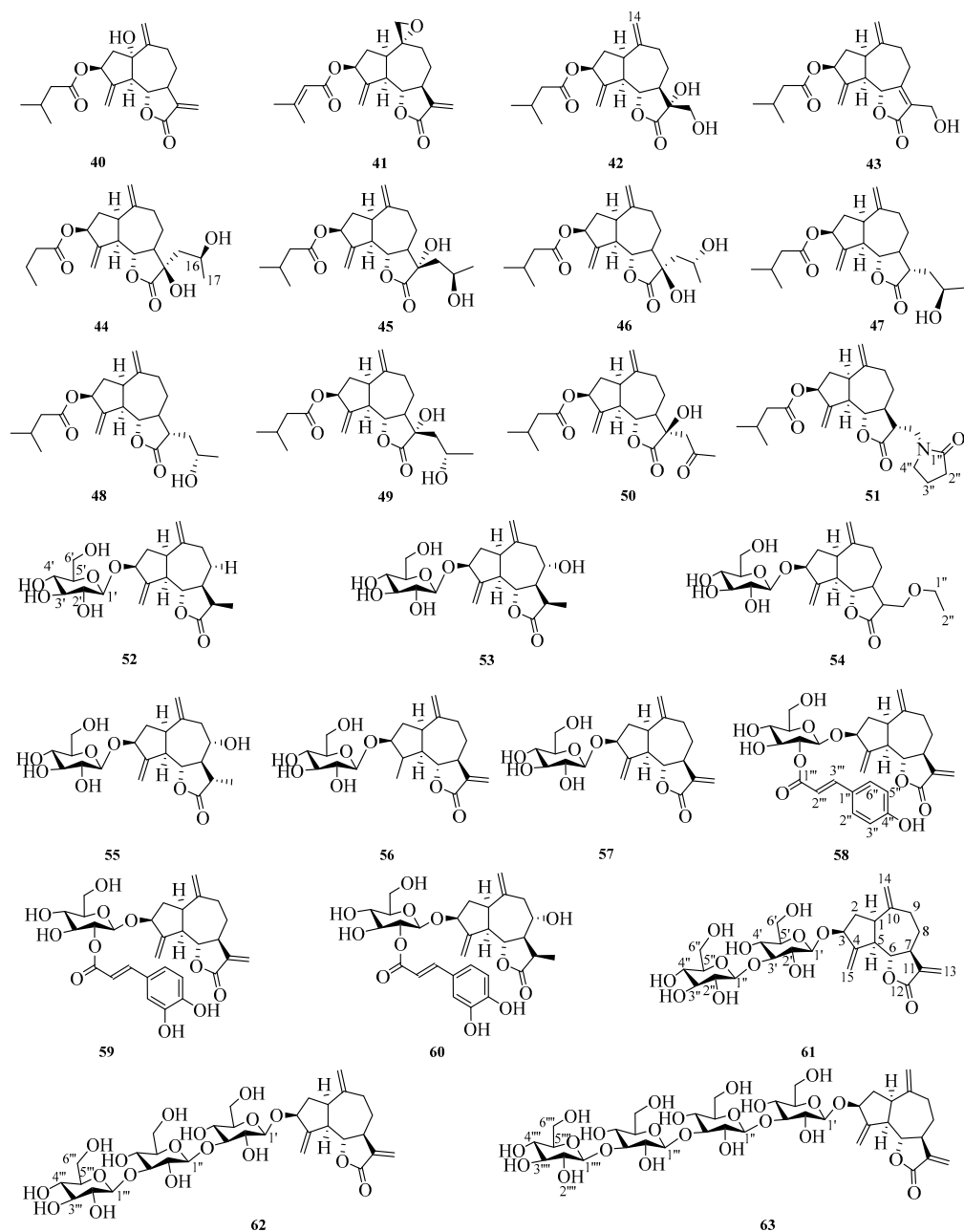


Figure 1. Chemical structures for compounds 1–63.

Table 1. The compound name, molecular formula, and NMR test reagent of guaianane-type sesquiterpene.

No.	Compound Name	Molecular Formula	Solvent	Ref.
1	Epi-guaiadiol A	C ₁₅ H ₂₆ O ₂	CD ₃ OD	[6]
2	Ainslide A	C ₁₅ H ₂₂ O ₃	CDCl ₃	[7]
3	Spicatene B	C ₂₀ H ₃₄ O ₄	CDCl ₃	[8]
4	4β,10α-Dimethyl-1β,5α-bicyclo[3,5,0]dec-6-en-4α,10β-diol	C ₁₂ H ₂₀ O ₂	CDCl ₃	[9]
5	Aphanamol I	C ₁₅ H ₂₄ O ₂	CDCl ₃	[10]
6	Aphanamol II	C ₁₅ H ₂₄ O ₂	CDCl ₃	[10]
7	Yunnanol A	C ₁₇ H ₂₈ O ₃	CDCl ₃	[11]
8	Ainslide E	C ₁₅ H ₂₂ O ₃	CDCl ₃	[7]

Table 1. Cont.

No.	Compound Name	Molecular Formula	Solvent	Ref.
9	4(15),10(14)-Guaiadien-12, 6-olide mokkolactone	C ₁₅ H ₂₀ O ₂	CDCl ₃	[12]
10	4-Hydroxy-10(14),11(13)-guaiaadien-6,12-olide	C ₁₅ H ₂₀ O ₃	CDCl ₃	[13]
11	Dehydrocostuslactone	C ₁₅ H ₁₈ O ₂	CDCl ₃	[14]
12	Ainslide F	C ₁₅ H ₂₂ O ₃	CDCl ₃	[7]
13	Dihydroestafiatol	C ₁₅ H ₂₂ O ₃	CDCl ₃	[15]
14	4β,15,11β,13-Tetrahydrozaluzanin C	C ₁₆ H ₂₂ O ₂	CDCl ₃	[16]
15	Isolipidiol	C ₁₅ H ₂₂ O ₄	CD ₃ OD	[17,18]
16	11α,13-Dihydrozaluzanin C	C ₁₅ H ₂₀ O ₃	CDCl ₃	[19]
17	11β,13-Dihydrozaluzanin C	C ₁₅ H ₂₀ O ₃	CDCl ₃	[15]
18	11β,13-Dihydro-3-epizaluzanin C	C ₁₅ H ₂₀ O ₃	CDCl ₃	[20]
19	8β-Hydroxy-4β, 15-dihydrozaluzanin C	C ₁₅ H ₂₀ O ₄	CDCl ₃	[21]
20	8α-Hydroxy-11α, 13-dihydrozaluzanin C	C ₁₅ H ₂₀ O ₄	CDCl ₃	[22]
21	11β,13-Dihydrodesacylynaropicrin	C ₁₅ H ₂₀ O ₄	CDCl ₃	[18,23]
22	10α-Hydroxy-10(14),11β(13)-tetrahydrozaluzanin C	C ₁₅ H ₂₂ O ₄	CDCl ₃	[24]
23	Zaluzanin C	C ₁₅ H ₁₈ O ₃	CDCl ₃	[25]
24	Desacylynaropicrin	C ₁₅ H ₁₈ O ₄	CDCl ₃ CD ₃ OD/CDCl ₃	[18,26]
25	8-Epidesacylnaropicrin	C ₁₅ H ₁₈ O ₄	C ₅ D ₅ N	[27]
26	Isoamberboin	C ₁₅ H ₂₀ O ₄	CDCl ₃	[28]
27	Ainslide D	C ₁₆ H ₂₂ O ₄	CDCl ₃	[7]
28	Estafiatone	C ₁₅ H ₁₈ O ₃	CDCl ₃	[29]
29	8-Epigrosheimin	C ₁₅ H ₁₈ O ₄	CDCl ₃	[30]
30	Ainsliaolide B	C ₁₇ H ₂₂ O ₄	CDCl ₃	[31]
31	Dehydrozaluzanin C	C ₁₅ H ₁₆ O ₃	CDCl ₃	[32]
32	Diaspanolide A	C ₂₀ H ₂₈ O ₄	CDCl ₃	[33]
33	Diaspanolide E	C ₂₀ H ₂₈ O ₄	CDCl ₃	[34]
34	Ainsliaolide A	C ₂₄ H ₂₆ O ₄	CDCl ₃	[35]
35	Ainsliaolide D	C ₂₀ H ₂₆ O ₄	CDCl ₃	[36]
36	8α-Hydroxy-diaspanolide A	C ₂₀ H ₂₈ O ₅	CDCl ₃	[8]
37	Yunnanolides H	C ₂₀ H ₂₈ O ₅	CDCl ₃	[37]
38	Yunnanolides I	C ₂₀ H ₂₈ O ₄	CDCl ₃	[37]
39	Diaspanolide B	C ₂₀ H ₂₆ O ₄	CDCl ₃	[33]
40	1α-Hydroxy-3-O-isobutyrate	C ₂₀ H ₂₆ O ₅	CDCl ₃	[38]
41	Ainslide C	C ₂₀ H ₂₄ O ₅	CDCl ₃	[7]
42	Yunnanolide J	C ₂₀ H ₂₈ O ₆	CDCl ₃	[11]
43	Spicatene A	C ₂₀ H ₂₆ O ₅	CDCl ₃	[8]
44	Yunnanolides A	C ₂₂ H ₃₂ O ₆	CDCl ₃	[37]
45	Yunnanolides C	C ₂₂ H ₃₂ O ₆	CDCl ₃	[37]
46	Yunnanolides D	C ₂₂ H ₃₂ O ₆	CDCl ₃	[37]
47	Yunnanolides E	C ₂₂ H ₃₂ O ₅	CDCl ₃	[37]
48	Yunnanolides F	C ₂₂ H ₃₂ O ₅	CDCl ₃	[37]
49	Yunnanolides B	C ₂₂ H ₃₂ O ₆	CDCl ₃	[37]
50	Pertyolide C	C ₂₂ H ₃₀ O ₆	CDCl ₃	[39]
51	Yunnanolides G	C ₂₄ H ₃₃ NO ₅	CDCl ₃	[37]
52	11α, 13-Dihydroglucozaluzanin C	C ₂₁ H ₃₀ O ₈	CD ₃ OD/C ₅ D ₅ N	[22]
53	8α-Hydroxy-11α, 13-dihydroglucozaluzanin C	C ₂₁ H ₃₀ O ₉	CD ₃ OD	[22]
54	13-Ethoxy-4(15),10(14)-dien-guai-6,12-olide-3-O-β-D-glucopyranoside	C ₂₃ H ₃₅ O ₉	CD ₃ OD	[40]
55	11β,13-Dihydro-8α-hydroxyglucozaluzanin C	C ₂₁ H ₃₀ O ₉	C ₅ D ₅ N	[41]
56	4β,15-Dihydrozaluzanin C	C ₂₁ H ₃₀ O ₈	DMSO	[42]
57	Glucozaluzanin C	C ₂₁ H ₂₈ O ₈	CDCl ₃ /C ₅ D ₅ N/ CD ₃ SOCD ₃	[12,22]
58	Ainsliaside C	C ₃₀ H ₃₄ O ₁₀	CD ₃ OD	[43]
59	Ainsliaside A	C ₃₀ H ₃₄ O ₁₁	C ₅ D ₅ N	[44]
60	2'-O-E-Caffeoyl-8α-hydroxy-11α,13-dihydro-3-β-O-β-D-glucozaluzanin C	C ₃₀ H ₃₆ O ₁₂	CD ₃ OD	[45]

Table 1. Cont.

No.	Compound Name	Molecular Formula	Solvent	Ref.
61	Macroclinside B	C ₂₇ H ₃₈ O ₁₃	DMSO CD ₃ OD	[25,46]
62	Macroclinside I	C ₃₃ H ₄₈ O ₁₈	DMSO	[25,46]
63	ZaluzaninC-3-O-β-glucopyranosyl-(1→3)-β-glucopyranosyl-(1→3)-β-glucopyranosyl-(1→3)-β-glucopyranoside	C ₃₉ H ₅₈ O ₂₃	DMSO	[46]

2.1. NMR Data of Guaiane Sesquiterpenes (1–63)

The ¹H and ¹³C NMR spectroscopy results were summarized in Tables 2–11. Additionally, this paper provides a summary of the nuclear magnetic data testing instrument used for compounds 1–63. NMR data for compounds 2, 8, 12, 27, and 41 were obtained with Bruker AV-400 HD spectrometers (Bruker, Byersbin, Switzerland). The ¹H and ¹³C data of compounds 3, 7, 36–38, 42–49, and 51 were obtained by a Bruker Ascend-500 spectrometer (Bruker, Nasdaq, New York, NY, USA). For compounds 11, 25, 55, and 59, the NMR data were recorded on a JEOL FX-90Q spectrometer (JEOL, Tokyo, Japan). Compounds 20, 52, 53, and 57 had their ¹H and ¹³C data taken with a Varian Mercury Plus 400 instrument (Varian, Palo Alto, CA, USA). Compounds 23, 61, and 62 were measured by a Varian Inova 400 instrument (Varian, Palo Alto, CA, USA). The ¹H and ¹³C data of compounds 61, 62, and 63 were recorded using a unity Bruker AV500 instrument (Bruker, Switzerland). NMR data of compounds 9 and 57 were obtained using the Bruker AMX 500 (Bruker, Zurich, Switzerland) and Varian Unity Inova 500 instruments (Varian, USA). The ¹H and ¹³C data of compounds 30 and 35 were run on a Bruker Avance 600 spectrometer (Bruker, Germany). The ¹H- and ¹³C-NMR data of compounds 32 and 39 were collected by a Bruker DRX-500 spectrometer (Bruker, Switzerland). Nuclear magnetic data of compounds 1, 4, 14, 15, 16, 18, 28, 31, 33, 54, 56, 58, and 60 were recorded on the following instruments: VNS-600 (Varian, Palo Alto, CA, USA), Bruker ACF-500 NMR (Bruker, Germany), Bruker Avance DRX 500, Bruker Avance II 800 (Bruker, Switzerland), Bruker ARX-300 NMR (Bruker, Switzerland), Bruker Avance 400 (Bruker, Zug, Switzerland), Varian Inova 500 (Varian, Palo Alto, CA, USA), Bruker AC 200 (Bruker, Karlsruhe, Germany), Bruker Advance 500 (Bruker, Germany), Bruker AV-600 (Bruker, Switzerland), Varian 500 MHz (Varian, Palo Alto, CA, USA), and Bruker AV500-III (Bruker, Switzerland), Varian VNS600 (Varian, USA), Bruker Avance 300 (Bruker, Switzerland), and Bruker Avance 500 (Bruker, Switzerland). The ¹H and ¹³C spectrums of compounds 5–6 were tested at 360 and 25 MHz, respectively; 10, 24, and 40 were run at 400MHz; 13 and 17 were recorded at 200 MHz for ¹H and 50 MHz for ¹³C NMR; The ¹H-NMR spectra of 19 and 29 were measured on 500.13 MHz; 21 and 26 were tested in the 270 MHz; 22 was taken with 300 MHz; 34 and 50 were collected in the 500 MHz. The carbon spectrum of compound 24 was determined at 100 MHz, compound 19 was recorded at 125.76 MHz, and compound 34 was recorded at 125 MHz. Carbon spectrum data for compounds 10, 21, 22, 26, 40, and 50 have not been reported in the literature.

Table 2. ¹H-NMR data of compounds 1–8.

NO.	1 [6]	2 [7]	3 [8]	4 [9]	5 [10]	6 [10]	7 [11]	8 [7]
	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	2.54, ddd, 10.8, 8.4, 8.4	—	2.74, q, 9.3	2.05, m	—	—	—	2.30, q, 7.6
2 α	1.81–1.85, m	1.94–2.02, m	1.95, m	1.76, dd, 11.7, 6.4	2.08	—	1.63, m	1.78–1.87, m
2 β	1.51–1.54, m	1.59–1.70, m	1.49, m	1.66, dd, 11.7, 7.3	1.35	—	1.42, m	1.90–1.98, m
3 α	1.67, br d, 10.4	2.47–2.60, m	1.69, m	1.70, dd, 7.3, 6.4	1.8	—	—	2.38–2.48, m
3 β	1.66, dd, 10.4, 3.6	1.86–1.97, m	—	—	1.35	—	1.43, m	—
4	—	2.99–3.08, m	—	—	1.66	—	1.63, m	—
5	2.00, ddd, 13.2, 10.8, 3.6	2.07, td, 12.1, 2.4	2.03, m	2.24, dd, 11.5, 2.5	2.27	—	2.03, m	2.76, t, 8.9
6 α	1.60, dd, 13.2, 3.6	1.76–1.85, m	1.78, m	5.74, br d, 11.0	5.51	6.62, d, 5	5.44, d, 4.5	4.08, t, 9.9
6 β	1.35, ddd, 13.2, 13.2, 10.8	1.25–1.37, m	1.64, m	—	—	—	—	—
7 α	—	1.86–1.97, m	2.14, m	5.80, ddd, 11.0, 5.3, 2.4	—	—	—	2.03–2.09, m
7 β	2.12, ddd, 10.8, 10.8, 4.2	1.15–1.27, m	—	—	—	—	—	—
8 α	1.72, ddd, 13.8, 7.2, 4.2, 3.6	—	1.72, m	1.98, m	2.54	—	—	1.99–2.07, m
8 β	1.49, dddd, 13.8, 13.8, 10.8, 4.2	2.13–2.22, m	1.86, m	2.30, m	2.29	—	2.17, m	1.30–1.39, m
9 α	1.89, ddd, 13.8, 7.2, 4.2	4.77–4.81, m	1.66, m	1.61, ddd, 14.0, 9.5, 1.8	2.81	—	—	1.87–1.94, m
9 β	1.61, ddd, 13.8, 13.8, 3.6	—	1.78, m	1.83, ddd, 14.0, 9.5, 2.0	2.41	—	1.76, m	1.58–1.67, m
10 α	—	2.47–2.57, m	—	—	—	3.40, dd, 11, 6	3.44, dd, 10.5, 6.0	—
10 β	—	1.95, d, 13.1	—	—	—	—	—	—
11	—	—	—	1.19, s	1.57	—	1.54, m	2.17–2.25, m
12 α	4.65, br s	4.79–4.84, m	5.03, br s	1.26, s	0.9	0.92, d, 7	0.89, d, 7.0	—
12 β	4.58, br s	—	5.01, br s	—	—	—	—	—
13	1.70, s	1.68, s	4.56, s	—	0.92	0.93, d, 7	0.89, d, 7.0	1.23, d, 6.9
14 α	1.13, s	3.62, dd, 10.7, 4.5	1.30, s	—	4.01	9.37, s	4.49, m	1.13, s
14 β	—	3.38, dd, 10.7, 7.6	—	—	—	—	—	—
15 α	1.20, s	—	1.25, s	—	1.27	1.04, s	0.97, s	5.11, s
15 β	—	—	—	—	—	—	—	4.95, s
2'	—	—	2.22, m	—	—	—	2.07, s	—
3'	—	—	2.11, m	—	—	—	—	—
4'	—	—	0.97, d, 6.6	—	—	—	—	—
5'	—	—	0.97, d, 6.6	—	—	—	—	—

Table 3. ¹H-NMR data of compounds 9–17.

NO.	9 [12]	10 [13]	11 [14]	12 [7]	13 [15]	14 [16]	15 [17]	16 [19]	17 [15]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	C ₅ D ₅ N	CDCl ₃	CDCl ₃
1	2.89, dt, 8.0, 4.5	3.02, br ddd, 12.5, 8, 8	—	2.69–2.76, m	—	2.76, m	2.79, m	2.83, m	—
2 α	1.95, m	1.82, m	—	1.92–2.06, m	—	1.75, m	2.02, m	2.34, m	—
2 β	1.87, m	—	—	—	—	2.15, m	2.24, m	—	—
3 α	2.49, m	1.82, m	—	4.21–4.28, m	3.71, m	3.72, q, 6.4	3.91, m	4.54, t, 6.0	4.54, t, 7.4
3 β	—	1.92, m	—	—	—	—	—	—	—
4	—	—	—	2.31–2.39, m	—	1.85, m	2.14, m	—	—
5	2.81, br dd, 9.5, 8.0	2.38, dd	—	2.22–2.31, m	—	1.75, m	1.98, m	—	—
6	3.93, t, 9.5	4.06, dd	3.98, t, 9	4.23, t, 9.7	3.93, t, 10	3.93, t, 9.7	3.93, m	4.13, t, 9.0	4.02, t, 9.5
7	2.12, m	2.77, ddddd	—	2.26–2.33, m	—	1.85, m	2.18, ddd, 10.4, 10.0, 9.6	—	—
8 α	1.94, m	2.26, dddd	—	1.83–1.91, m	—	2.10, m	—	1.41, m	—
8 β	1.32, m	1.38, dddd	—	1.33–1.42, m	—	1.23, m	3.83, m	—	—
9 α	2.22, dd, 12.0, 7.0	2.69, ddd	—	2.62–2.68, m	—	2.60, dt, 13.0, 4.0	2.35, dd, 12.8, 11.8	—	—
9 β	2.05, dt, 12.0, 5.0	1.94, br ddd	—	1.78–1.88, m	—	1.85, m	3.00, dd, 12.8, 4.8	—	—
11	2.49, m	—	—	2.66–2.71, m	2.12, m	2.20, m	2.77, m	—	2.14, qd, 11, 6.9
13 α	1.25, d, 7.0	6.24, d	6.25, d, 3.5	1.18, d, 7.8	1.21, d, 6.8	1.29, d, 7.0	1.68, d, 7.2	1.16, d, 6.0	1.23, d, 6.9
13 β	—	5.53, d	5.51, d, 3.2	—	—	—	—	—	—
14 α	4.89, br s	5.01, br s	4.91, s	5.00, s	4.95, s	4.92, s	4.99, br s	4.95, s	4.96, s
14 β	4.79, br s	4.97, br s	4.84, s	4.95, s	—	—	5.09, br s	4.93, s	4.93, s
15 α	5.21, d, 2.0	1.32, s	5.29, br s	0.97, d, 7.2	1.24, d, 6.3	1.20, d, 7.3	1.44, d, 6.4	5.40, s	5.38, t, 1.9
15 β	5.06, d, 2.0	—	5.09, br s	—	—	—	—	5.31, s	5.29, t, 1.9

Table 4. ¹H-NMR data of compounds 18–25.

NO.	18 [20]	19 [21]	20 [22]	21 [23]	22 [24]	23 [25]	24 [26]	25 [27]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N
1	—	2.83, br ddd, 11.1, 10.6, 6.6	2.91, m	2.92, m	1.9–2.2, m	—	2.98, ddd, 10.6, 9.0, 7.1	—
2 α	—	2.21, ddd, 13.0, 6.6, 6.6	1.71, m	2.25, m	1.9–2.2, m	—	1.73, ddd, 12.6, 11.0, 9.0	—
2 β	—	1.79, ddd, 13.0, 10.6, 8.8	2.29, m	1.77, m	2.32, ddd, 14, 8, 8	—	2.11, ddd, 12.6, 7.2, 7.0	—
3	4.53, t, 7.6	3.75, ddd, 8.8, 8.8, 6.6	4.52, m	4.55, br d, 8	4.53, br t, 8, 1.9	4.53, m	4.50, br dd, 9.1, 7.5	—
4	—	1.90, m	—	—	—	—	—	—
5	—	1.93, m	2.88, m	2.85, m	2.72, br t, 9, 1.9	—	2.88, m	—
6	4.12, dd, 10.0, 9.2	4.27, dd, 9.7, 9.7	4.12, dd, 9.9, 9.9	4.07, t, 10	4.25, t, 9.9	4.06, t, 9.2	4.17, dd, 10.4, 8.9	—
7	—	2.80, m	2.35, m	2.00, q, 10	1.9–2.2, m	—	2.89, m	—
8 α	—	4.34, br m	—	—	1.4–1.8, m	—	—	—
8 β	—	—	3.78, m	3.78, t, 9, 4.5	—	—	3.90, ddd, 9.5, 5.0, 4.8	—
9 α	—	2.37, dd, 13.5, 3.4	2.10, m	2.72, dd, 14, 5	1.4–1.8, m	—	2.70, dd, 13.6, 5.1	—
9 β	—	2.69, dd, 13.5, 4.9	2.72, dd, 12.5, 4.5	2.21, dd, 14, 7	—	—	2.27, dd, 13.6, 4.6	—
11	—	—	2.87, m	2.58, m	2.25, dq, 12, 6.8	—	—	—
13 α	1.14, d, 8.0	6.37, d, 3.6	1.29, d, 7.7	1.42, d, 3p, 7	1.22, d, 6.8	6.16, d, 3.2	6.15, dd, 3.2, 1.3	6.49, d, 3.5
13 β	—	5.61, d, 3.2	—	—	—	5.46, d, 3.2	6.12, dd, 3.5, 1.3	5.68, overlapped
14 α	4.94, s	5.16, br s	5.08, br s	5.11, br	1.17, s	4.95, br s	5.08, d, 1.7	5.70, s
14 β	4.91, s	5.03, br s	5.01, br s	5.00, br	—	4.90, br s	4.97, d, 2.0	—
15 α	5.38, s	1.22, d, 6.3	5.42, dd, 1.7, 1.6	5.41, t, 1	5.24, t, 1.9	5.42, br s	5.37, d, 2.3	5.68, overlapped
15 β	5.30, s	—	5.32, dd, 1.7, 1.6	5.32, t, 1	5.20, t, 1.9	5.29, br s	5.30, d, 2.1	5.25, br s

Table 5. ¹H-NMR data of compounds 26–33.

NO.	26 [28]	27 [7]	28 [29]	29 [30]	30 [31]	31 [32]	32 [33]	33 [34]
	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	3.12, m	3.08, td, 8.5, 3.1	3.10, td, 8.5, 2.0	3.05, ddd, 8.4, 8.0, 3.1	3.10, t, 9.0	3.12, ddd	2.89, m	2.89–2.94, m
2 α	2.25, c	2.50–2.55, m	2.16, t, 8.5	2.60, dd, 19.3, 8.4	2.48, d, 16.8	2.68, dd	2.46, m	1.98–2.03, m
2 β	2.25, c	2.41–2.50, m	—	2.54, ddd, 19.3, 3.1, 1.4	2.55, dd, 16.8, 9.0	2.56, dd	—	1.74–1.80, m
3	—	—	—	—	—	—	5.54, m	5.56, dd, 6.3, 2.1
4	—	2.31–2.38, m	2.30, d, 7.5	2.33, ddq, 10.3, 6.9, 1.4	2.30, m	—	—	—
5	2.5, c	2.78–2.85, m	2.31, m	2.28, ddd, 10.3, 9.2, 8.0	2.91, q, 9.0	3.27, tdd	2.83, m	2.81–2.86, m
6	3.93, t, 9	4.06, t, 9.6	4.40, 8.8	4.55, dd, 9.2, 9.2	3.98, t, 9.0	4.01, t	4.08, t, 9.8	4.10, t, 9.7
7	2.05, q, 10	2.53–2.61, m	3.00, dddd, 8.0, 8.0, 3.0, 3.0	3.16, dddd, 9.2, 3.5, 3.0, 2.0	3.01, m	3.03, m	2.67, m	2.37–2.42, m
8 α	—	1.93–2.02, m	2.35, m	4.46, br m	1.47, m	3.03, m	—	1.87–1.94, m
8 β	—	1.38–1.49, m	1.48, m	—	2.33, m	1.46, m	—	1.37–1.46, m
9 α	2.82, dd, 13, 6	2.55–2.63, m	2.63, m	2.69, dd, 13.9, 3.0	2.22, m	2.20, m	2.46, m	—
9 β	2.25, c	2.03–2.12, m	2.22, m	2.50, dd, 13.9, 4.2	2.61, m	2.60, m	—	2.44–2.49, m
11 α	—	2.71–2.80, m	—	—	—	—	—	2.66–2.72, m
11 β	2.5, c	—	—	—	—	—	—	—
13 α	1.44, d, 7	1.19, d, 7.7	6.30, d, 3.5	6.45, d, 3.5	5.57, d, 3.6	6.30, d	1.15, d, 7.8	—
13 β	—	—	5.58, d, 3.5	5.68, d, 3.0	6.29, d, 3.6	5.58, d	—	1.17, d, 7.8
14 α	5.06, br	4.93, s	5.02, br s	5.09, br s	4.66, brs	4.94, s	4.89, d, 9.0	4.91, s
14 β	4.76, br	4.63, s	4.69, br s	4.84, br s	4.98, brs	4.60, s	—	4.92, s
15 α	1.24, d, 7	3.84, dd, 8.9, 3.1	1.28, d, 6.5	1.28, d, 6.9	3.71, dd, 9.0, 3.0	6.25, dd	5.25, t, 2.0	5.27, t, 2.1
15 β	—	3.61, dd, 8.9, 3.1	—	—	3.98, dd, 9.0, 3.0	5.87, dd	5.39, t, 2.0	5.42, t, 2.2
1'	—	3.30, s	—	—	3.48, q, 7.2	—	—	—
2'	—	—	—	—	1.14, t, 7.2	—	—	2.24, dd, 7.1, 1.7
3'	—	—	—	—	—	—	—	2.09–2.17, m
4'	—	—	—	—	—	—	0.96, d, 6.6	0.98, d, 6.6
5'	—	—	—	—	—	—	0.96, d, 6.6	0.98, d, 6.6

Table 6. ¹H-NMR data of compounds 34–42.

NO.	34 [35]	35 [36]	36 [8]	37 [37]	38 [37]	39 [32]	40 [38]	41 [7]	42 [11]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	2.89, m	2.93, m	2.95, q, 8.4	—	2.18, m	2.94, m	—	2.44–2.52, m	2.90, q, 8.0
2 α	2.46, m	2.48, m	2.44, m	2.23, m	2.67, m	1.78, m	2.43, br dd, 14, 8	2.25–2.37, m	2.49, m
2 β	1.79, m	1.79, m	1.77, m	—	1.51, m	—	2.18, dd, 14, 7	1.52–1.62, m	1.80, m
3	5.55, dd, 8, 6	5.57, ddt, 8, 6, 2	5.56, m	5.72, m	5.54, m	5.56, m	5.75, tt, 8, 7, 1.5	5.50, t, 6.7	5.56, m
5	1.93, m	2.85, m	2.82, m	2.66, d, 11.5	2.55, m	2.85, m	2.80, br d, 10, 1.5	2.89–2.97, m	2.04, m
6	3.99, t, 9	4.11, t, 10	4.00, t, 9.9	3.88, t, 10.5	4.07, t, 10.0	4.06, dd, 16.9, 7.6	3.91, t, 10	4.22, t, 9.5	4.39, t, 10.0
7	2.81, m	2.42, m	2.01, m	1.69, m	1.76, m	2.85, m	3.05, dddd, 10, 10, 4, 3.5, 3	2.92–3.01, m	2.76, t, 9.5

Table 6. Cont.

NO.	34 [35]	35 [36]	36 [8]	37 [37]	38 [37]	39 [32]	40 [38]	41 [7]	42 [11]
8 α	2.11, m	1.92, m	—	1.45, m	2.40, m	—	2.30, m, 4, 4	2.24–2.33, m	1.80, m
8 β	1.31, m	1.42, m	3.77, m	—	1.97, m	—	1.45, m, 10, 9	1.44–1.55, m	—
9 α	2.49, m	2.52, m	2.71, dd, 13.1, 5.0	1.45, m	5.54, m	2.46, m	2.63, ddd, 12, 9, 4	1.88–1.98, m	2.48, m
9 β	2.03, m	2.01, m	2.23, m	—	—	—	2.30, m	1.78, dt, 14.3, 5.0	2.04, m
11	2.20, m	2.69, p, 8	2.57, m	2.32, m	2.29, m	—	—	—	—
13 α	1.22, d, 7	1.16, d, 8	1.42, d, 7.0	1.25, m	1.23, d, 7.0	5.49, d, 3.1	6.21, d, 3.5	6.27, d, 3.0	3.79, d, 12.0
13 β	—	—	—	—	—	6.21, d, 3.5	5.49, d, 3	5.55, d, 3.0	3.65, d, 11.0
14 α	4.91, s	4.91, br s	5.05, s	1.35, s	1.80, br s	4.97, d, 6.4	5.20, br s	2.75, d, 4.4	4.93, d, 6.5
14 β	—	4.92, br s	4.99, s	—	—	—	5.09, br s	2.54, d, 4.4	—
15 α	5.26, brt, 2	5.28, br t, 2	5.43, t, 2.2	5.50, s	5.44, br s	5.27, t, 2.1	5.53, t, 1.5	5.45, s	5.41, br t, 2.0
15 β	5.38, brt, 2	5.41, br t, 2	5.28, t, 2.2	5.41, s	5.33, br s	5.45, t, 2.0	5.36, t, 1.5	5.25, s	5.30, br t, 2.0
2' α	5.71, s	5.72, br s	2.23, m	2.17, m	2.55, m	—	2.59, qq, 7, 7	5.69, s	2.23, dd, 7.5, 2.0
2' β	—	—	—	—	2.18, m	—	—	—	—
3'	—	—	2.13, m	2.09, m	2.09, m	—	—	—	2.13, m
4'	1.89, s	1.91, br s	0.97, d, 6.6	0.95, d, 6.5	0.95, d, 6.5	0.96, d, 6.6	1.20, d	1.91, s	0.97, d, 6.5
5'	2.17, s	2.19, br s	0.97, d, 6.6	0.95, d, 6.5	0.95, d, 6.5	0.96, d, 6.6	1.19, d	2.18, s	0.97, d, 6.5

Table 7. ¹H-NMR data of compounds 43–51.

NO.	43 [8]	44 [37]	45 [37]	46 [37]	47 [37]	48 [37]	49 [37]	50 [39]	51 [37]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	2.90, m	2.86, q, 8.5	2.89, m	2.90, q, 8.5	2.90, q, 8.0	2.90, q, 3.0	2.89, m	2.88, q, 8.4	2.13, m
2 α	2.21, m	2.44, m	2.57, dt, 13.0, 4.0	2.46, m	2.03, m	2.02, m	2.57, dt, 13.0, 4.0	2.44, m	2.44, m
2 β	1.84, m	1.76, m	1.96, m	1.78, m	1.80, m	1.82, m	1.99, m	1.76, m	—
3	5.61, t, 7.6	5.54, m	5.54, m	5.55, m	5.57, m	5.57, m	5.56, m	5.56, m	5.56, m
5	2.62, m	2.71, t, 9.5	2.89, m	2.73, t, 9.5	2.80, t, 9.5	2.82, t, 9.2	2.89, m	2.75, t, 9.2	2.79, t, 9.5
6	4.81, d, 11.2	4.29, t, 9.5	4.01, t, 9.5	4.36, t, 9.5	4.08, t, 10.0	4.05, t, 9.5	3.91, t, 9.5	4.37, t, 9.2	4.02, t, 9.5
7	—	1.87, m	2.35, m	2.07, m	2.03, m	2.13, m	2.34, m	1.94, m	2.45, m
8 α	3.05, m	2.10, m	2.10, m	2.12, m	2.15, m	2.13, m	2.12, m	1.64, m	—
8 β	2.50, m	—	1.39, m	1.30, m	1.39, m	1.35, m	1.35, m	1.81, m	2.38, q, 8.0
9 α	2.52, m	2.44, m	2.49, dt, 14.0, 8.0	2.46, m	2.47, m	2.49, m	2.50, dt, 14.0, 8.0	2.03, m	2.03, m
9 β	2.17, m	2.01, m	1.75, m	2.07, m	—	—	1.75, m	2.50, m	1.78, dt, 14.0, 6.5
11	—	—	—	—	2.46, m	2.49, m	—	—	2.89, q, 8.0
13 α	4.38, dd, 16.6, 13.3	—	1.76, m	1.96, m	1.80, m	1.82, m	1.76, m	2.62, d, 16.7	3.69, dd, 4.5, 4.5
13 β	—	1.64, dd, 15.0, 2.5	1.68, m	1.78, m	1.69, m	1.67, m	1.57, dd, 15.0, 2.5	2.80, d, 16.7	3.57, dd, 5.0, 5.0
14 α	5.01, s	4.91, s	4.93, s	4.93, s	4.93, s	4.92, s	4.93, s	4.93, s	4.92, s

Table 7. Cont.

NO.	43 [8]	44 [37]	45 [37]	46 [37]	47 [37]	48 [37]	49 [37]	50 [39]	51 [37]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
14β	4.94, s	4.88, s	4.89, s	4.91, s			4.90, s	4.91, s	4.90, s
15α	5.42, br s	5.41, br t, 1.5	5.39, br t, 2.0	5.42, br t, 2.0	5.39, br t, 2.0	5.40, t, 2.0	5.41, br t, 2.0	5.43, br s	5.37, br t, 2.0
15β		5.27, br t, 1.5	5.29, br t, 2.0	5.29, br t, 2.0	5.27, br t, 2.0	5.26, t, 2.0	5.29, br t, 2.0	5.30, br s	5.25, br t, 2.0
16	—	4.97, m	4.27, m	4.16, m	3.97, m	4.21, m	4.31, m	—	—
17	—	1.22, d, 6	1.23, d, 6.0	1.30, d, 8.5	1.24, d, 6.0	1.24, d, 6.0	1.23, d, 6.0	2.32, s	—
2'	2.23, m	2.21, dd, 7.5, 1.5	2.23, dd, 7.5, 2.0	2.22, dd, 7.5, 2.0	2.22, dd, 8.0, 2.0	2.23, dd, 8.0, 1.5	2.22, dd, 8.0, 2.0	2.23, dd, 7.1, 1.7	2.23, dd, 8.0, 1.5
3'	2.11, m	2.15, m	2.10, m	1.96, m	2.15, m	2.13, m	2.12, m	2.12, m	2.13, m
4'	0.97, d, 6.6	0.95, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.96, d, 7.0
5'	0.97, d, 6.6	0.95, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.97, d, 6.5	0.96, d, 7.0

Note: The ¹H-NMR data of 2''α, 2''β, 3'', and 4'' for **51** were recorded as 2.30, m; 1.32, m; 2.03, m; and 3.45, m, respectively.

Table 8. ¹H-NMR data of compounds 52–57.

NO.	52 [22]	52 [22]	53 [22]	54 [40]	55 [41]	56 [42]	57 [12]	57 [22]
	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N	DMSO	CDCl ₃	C ₅ D ₅ N
1	2.96, m	2.75, m	3.00, m	2.97, q, 8.4	—	2.76, m	2.80, br t, 10.0	2.77, m
2α	2.35, m	2.22, m	2.30, m	2.36, m, overlapped	—	1.69, dd, 12, 10	2.39, m	2.30, m
2β	1.96, m	1.81, m	1.96, m	1.96, dt, 14.0, 6.9	—	2.14, dd, 13.5, 7.0	1.98, m	1.92, m
3	4.62, m	4.41, m	4.61, m	4.63, dd, 7.8, 5.9	4.84, br t, 7	3.56, dd, 15.5, 8.0	4.65, br dd, 6.0, 6.0	4.81, dd, 7.3, 1.5
4	—	—	—	—	—	1.85, m	—	—
5	2.79, dd, 9.5, 9.5	2.68, m	2.83, dd, 9.9, 9.9	2.75, dd, 9.9, 8.4	—	1.91, m	3.01, dd, 17.5, 8.5	2.74, m
6	4.33, dd, 9.9, 9.5	4.33, dd, 9.9, 9.5	4.38, dd, 10.6, 9.9	4.23, t, 9.9	—	3.93, t, 10	4.28, dd, 9.0, 9.0	4.26, m
7	2.43, m	2.33, m	2.36, m	2.39, m, overlapped	—	2.78, m	2.89, m	2.70, m
8α	1.88, m	1.58, m	—	2.16, tt, 10.0, 5.0	—	2.25, m	2.28, m	1.97, m
8β	1.44, m	1.20, m	3.72, m	1.39, m	—	1.24, m	1.46, m	1.16, m
9α	2.03, m	2.10, m	2.15, dd, 12.6, 8.0	2.08, ddd, 13.7, 9.3, 5.1	—	2.54, m	2.21, m	2.10, m
9β	2.53, m	2.36, m	2.71, dd, 12.6, 5.0	2.52, m, overlapped	—	1.97, m	2.52, ddd, 13.0, 6.5, 6.5	2.37, m
11	2.67, m	2.64, m	2.77, m	2.54, dt, 11.8, 3.7	—	—	—	—
13α	1.12, dd, 7.7, 1.5	1.05, d, 7.7	1.23, d, 7.7	3.71, dd, 9.9, 3.7	1.65, d, 7	5.59, d, 2	6.12, d, 3.0	5.53, br d, 1.5
13β	—	—	—	3.63, dd, 9.9, 3.7	—	6.00, d, 2.5	5.57, d, 3.0	5.87, br d, 1.5
14α	4.99, s	5.00, s	5.09, s	4.99, s	5.14, br s	4.95, d, 5	5.01, br s	5.02, d, 1.1
14β	4.91, s	4.83, s	4.98, s	4.91, s	5.01, br s	4.99, d, 5	4.94, br s	4.83, d, 1.1
15α	5.40, s	5.82, br s	5.36, d, 1.3	5.42, d, 1.7	5.87, br s	1.15, d, 10	5.44, br s	6.23, d, 3.4
15β	5.31, s	5.50, br s	5.32, d, 1.3	5.30, d, 1.7	5.54, br s	—	5.35, br d, 1.0	5.38, d, 3.4

Table 8. Cont.

NO.	52 [22]	52 [22]	53 [22]	54 [40]	55 [41]	56 [42]	57 [12]	57 [22]
	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N	DMSO	CDCl ₃	C ₅ D ₅ N
1'	4.46, d, 7.7	5.04, d, 7.9	4.44, d, 7.7	4.45, d, 7.8	5.06, d, 7	4.19, d, 7.5	4.47, d, 7.5	5.05, d, 7.9
2'	3.24, m	3.94, m	3.20, m	3.23, m	—	2.95, m	3.20–3.40, m	3.96, m
3'	3.36, m	4.24, m	3.31, m	3.36, m	—	3.16, m	3.87, dd, 10.0, 10.0	4.24, m
4'	3.28, m	4.06, m	3.26, m	3.28, m	—	3.04, m	3.20–3.40, m	4.08, m
5'	3.28, m	4.22, m	3.24, m	3.27, m	—	3.08, m	3.67, dd, 12.0, 5.5	4.22, m
6'α	3.66, dd, 11.9, 5.0	4.36, m	3.64, dd, 12.0, 6.0	3.88, dd, 12.0, 1.9	—	3.42, dd, 11.5, 6.0	3.20–3.40, m	4.40, dd, 11.8, 5.5
6'β	3.86, br d, 11.9	4.56, dd, 11.9, 2.0	3.87, dd, 12.0, 2.0	3.67, dd, 12.0, 5.2	—	3.65, dd, 9.5, 4.5	—	4.57, dd, 11.8, 2.4

Note: The ¹H-NMR data of 1'' and 2'' for 54 were recorded as 3.51, q, 7.0 and 1.17, t, 7.0.

Table 9. ¹H-NMR data of compounds 58–61.

NO.	58 [43]	59 [44]	60 [45]	61 [46]	NO.	58 [43]	59 [44]	60 [45]	61 [46]
	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD	DMSO		CD ₃ OD	C ₅ D ₅ N	CD ₃ OD	DMSO
1	2.99, dd, 16.8, 8.4	—	2.99, t, 9.1	—	15α	5.43, br s	5.45, br s	5.32, d, 1.1	5.38, br s
2α	2.33, m	—	2.26, m	—	15β	5.35, br s	—	5.27, d, 1.1	5.20, br s
2β	1.96, m	—	1.98, m	—	1'	4.45, d, 7.2	—	4.66, d, 8.1	4.40, d, 7.8
3	4.63, m	—	4.64, m	4.50, m	2'	3.87, m	5.65, br t, 10	4.85, m	—
5	2.78, dd, 9.6, 9.6	—	2.85, br d, 9.4	—	3'	3.37, t, 8.4	—	3.58, t, 8.6	—
6	4.26, dd, 9.6, 9.3	—	4.04, t, 10.2	4.13, dd, 10.2, 8.9	4'	3.28, m	—	3.38, m	—
7	2.89, m	—	2.34, dd, 8.1, 2.1	—	5'	3.26, m	—	—	—
8α	2.26, m	—	3.50, m	—	6'α	3.86, dd, 12.0, 2.4	—	3.91, dd, 12.0, 2.1	—
8β	1.45, m	—	—	—	6'β	3.68, dd, 12.0, 5.4	—	3.70, dd, 12.0, 5.8	—
9α	2.18, m	—	2.06, m	—	1''	—	—	—	4.32, d, 7.8
9β	2.50, m	—	2.58, dd, 11.5, 4.7	—	2''	7.56, d, 7.8	6.9–7.5, m	7.02, d, 2.0	—
11	—	—	2.72, t, 7.8	—	3''	7.37, d, 7.8	—	—	—
13α	6.09, d, 3.0	—	1.09, d, 7.7	6.02, d, 3.5	5''	7.37, d, 7.8	6.9–7.5, m	6.78, d, 8.1	—
13β	5.56, d, 3.0	—	—	5.61, d, 3.2	6''	7.56, d, 7.8	6.9–7.5, m	6.94, dd, 8.2, 1.9	—
14α	5.01, s	5.13, br s	5.11, br s	4.91, br s	2'''	6.48, d, 15.6	6.37, d, 15	6.24, d, 15.9	—
14β	4.91, s	—	4.90, br s	4.88, br s	3'''	7.58, d, 15.6	7.82, d, 15	7.55, d, 15.9	—

Table 10. ¹H-NMR data of compounds 61–63.

NO.	61 [25]	62 [46]	62 [25]	63 [46]	NO.	61 [25]	62 [46]	62 [25]	63 [46]
	CD ₃ OD	DMSO	DMSO	DMSO		CD ₃ OD	DMSO	DMSO	DMSO
1	—	—	4.88, d, 7.6	2.92, m	1'	4.54, d, 8.0	4.40, d, 7.8	5.01, d, 8.0	4.40, d, 7.8
2 α	—	—	—	2.25, m	2'	—	—	—	3.43, m
2 β	—	—	—	1.77, m	3'	—	—	—	3.40, m
3	4.64, m	4.50, t, 7.4	4.64, m	4.51, br s	4'	—	—	—	3.21, m
5	—	—	—	2.80, m	5'	—	—	—	3.18, m
6	4.27, t, 9.6	4.13, t, 9.5	4.11, t, 9.6	4.14, t, 9.6	6' α	—	—	—	3.36, m
7	—	—	—	2.91, m	6' β	—	—	—	3.29, m
8 α	—	—	—	2.23, m	1''	4.51, d, 8.0	4.47, d, 7.8	4.98, d, 7.6	4.46, d, 7.7
8 β	—	—	—	1.35, m	2''	—	—	—	3.28, m
9 α	—	—	—	2.11, m	3''	—	—	—	3.47, m
9 β	—	—	—	2.41, m	4''	—	—	—	3.21, m
11	—	—	—	—	5''	—	—	—	3.28, m
13 α	6.10, d, 3.6	6.02, d, 3.5	6.01, d, 3.6	6.02, d, 3.5	6'' α	—	—	—	3.36, m
13 β	5.57, d, 3.6	5.61, d, 3.1	5.59, d, 3.6	5.61, d, 3.5	6'' β	—	—	—	3.29, m
14 α	5.02, br s	4.91, s	5.19, br s	4.91, br s	1'''	—	4.36, d, 7.8	—	4.52, d, 7.8
14 β	4.93, br s	4.88, s	5.08, br s	4.88, br s	2''—OH	—	—	—	5.10, d, 4.1
15 α	5.40, br s	5.38, br s	5.36, br s	5.38, br s	2''—OH	—	—	—	5.24, d, 3.2
15 β	5.35, br s	5.20, br s	—	5.20, br s	2'''—OH	—	—	—	5.26, d, 3.4

Note: The 1''', 2''', 3''', 4''', 5''', 6''' α , and 6''' β data of compound 63 were 4.36, d, 7.9; 3.05, m; 3.18, m; 3.04, m; 3.24, m; 3.42, m; and 3.29, m.

Table 11. ¹³C-NMR data of compounds 1–63.

NO.	1 [6]	2 [7]	3 [8]	4 [9]	5 [10]	6 [10]	7 [11]	8 [7]	9 [12]	11 [14]	12 [7]	13 [15]	14 [16]	15 [18]	15 [17]	16 [19]	17 [15]
	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N	CDCl ₃	CDCl ₃
1	52.7	53.1	52.1	50.6	59	47.9	46.3	52.5	47.3	47.6	41.4	42.1	42.2	43.6	43	43.3	43.5
2	26.4	34.2	26.2	21.6	40	39.7	39.7	26	30.5	32.5	34.8	38.3	38.3	35.5	39.6	38.6	38.7
3	40.6	30.2	39.2	40.3	27.1	27	27.3	31	32.8	30.2	73.9	78.3	78.4	79.1	78.1	73.6	73.5
4	81.9	49.4	83.5	80	56.1	55	55.9	152	152	151.1	40.6	47	47	48.2	47.9	153.3	153.2
5	53.4	58.2	49.1	51.2	51.5	53.3	51.5	51.2	52.2	52	47.8	50.6	52.9	52.3	51.5	49.7	49.5
6	32.8	24.9	29.3	130.1	141.8	159.8	135	84	85.6	85.1	83.4	85.9	85.9	83.9	82.5	83.7	83.7
7	48.6	28.9	39	131.6	132.7	142	133.2	48.6	42.3	45.1	46.3	52.9	50.6	60	59.2	39.3	50.8
8	31.5	45.2	32	23.5	25	19.2	25.3	26.5	32.8	30.8	29.2	32.7	32.8	77.1	76.3	28.7	32.3
9	42.6	78.5	36.6	42.7	67	27.5	27.7	40.6	37.9	36.1	39.4	37	37	48.9	48.9	36	35.9
10	75.5	36.4	75	75	213.8	75.8	76.5	74.7	150.2	149.2	148.5	149.2	149.3	146.3	146.1	149	148.8
11	153.5	146.6	149.5	22.8	33	32.8	33.1	42.8	50.1	139.7	39.7	42	42.1	43.5	42.9	46.3	42
12	108.3	114.2	111.1	21.7	22.1	19.9	19.8	178.8	179	169.5	180.3	178.6	178.6	181.9	179.5	179.7	179.8
13	20.3	22.7	66.1	—	24.7	22.1	22.1	13.2	13.5	120	11.8	13	13.1	16.8	19	11.4	13.1
14	23.8	65	25.4	—	34.6	193	68.7	27.1	112.1	112.5	111.8	112.5	112.5	115.1	114.2	113.4	113.5

Table 11. Cont.

15	26.3	180.1	32.3	—	19.9	19.8	20	107.9	109.5	109.5	8.3	14.1	18.1	18.8	17	111.4	111
1'	—	—	173	—	—	—	171	—	—	—	—	—	—	—	—	—	—
2'	—	—	43.6	—	—	—	21	—	—	—	—	—	—	—	—	—	—
3'	—	—	25.8	—	—	—	—	—	—	—	—	—	—	—	—	—	—
4'	—	—	22.6	—	—	—	—	—	—	—	—	—	—	—	—	—	—
5'	—	—	22.6	—	—	—	—	—	—	—	—	—	—	—	—	—	—
NO.	18 [20]	20 [22]	21 [18]	23 [25]	24 [18]	24 [18]	25 [27]	27 [7]	28 [29]	30 [31]	31 [32]	32 [33]	33 [34]	34 [35]	35 [36]	36 [8]	37 [37]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N	CDCl ₃	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	43.3	43.6	44.2	43.9	45.2	46	44.7	40.1	39.8	40	39.6	43.7	43.7	44.1	43.8	44.6	62.9
2	38.6	38.6	39	38.7	39.2	40	39.9	45.4	44	45.7	44.6	36.2	36.2	36.6	36.3	36.7	38.1
3	73.6	73.7	73.6	73.2	73.7	73.1	73.1	217.6	219.2	217.7	204.4	74.4	74.4	73.8	73.7	74.4	74
4	153.2	152.7	153	152.7	152.4	154.3	155.2	53.3	50.6	52.9	144.4	148.9	148.9	148.8	148.8	148.3	145.1
5	49.7	49.9	50.7	45.4	51.3	51.9	50.5	45.4	47.1	44.6	48.6	50.1	50.1	50.4	50	51.2	53.1
6	83.6	78.9	79.1	83.7	79	80.9	78.8	88.5	88.8	88.8	86.8	83.7	83.7	83.9	83.8	79.1	81.8
7	39.3	53.4	56	49.6	51	51.7	50.1	43.7	44	44.2	44	45.7	45.7	50	45.6	55.8	55.5
8	28.7	69.9	74.9	28.8	71.9	74.1	66	29.3	31.8	31.8	31.6	28.7	28.7	32.4	28.7	75.1	24.9
9	36	45	44.8	32.6	41.3	42.9	44.2	38.5	38.7	38.4	38.2	36.2	36.2	36.3	36.3	45.3	37.4
10	148.9	143.1	143.2	147.8	142.7	144.7	145.1	149.6	148.7	149.1	148.2	148.4	148.4	148.8	148.9	143.1	70
11	46.3	38.1	42	139.6	138.1	140.6	137.7	39.3	138.7	139	138.6	39.2	39.2	42.1	39.2	42.1	42.4
12	179.6	179.1	178.6	170.3	169.9	172	170.1	179.8	169.9	169.9	not detected	179.6	179.6	178.4	179.6	178.6	177.6
13	11.4	11.2	15.9	120.2	123.2	122.9	120.9	11.6	121.3	121.2	121.4	11.4	11.4	13.2	11.3	16.1	12.4
14	113.3	115.9	116.2	115.2	117.1	117	115.9	112.6	113.1	113.1	113.6	113.4	113.4	113.5	113.2	116.4	20.7
15	111.3	112.6	112	110.9	113.2	112.1	109	70.5	14.2	68.1	122.1	113.2	113.2	112.9	113.1	114.3	118.3
1'	—	—	—	—	—	—	—	59.3	—	—	66.7	—	172.8	172.8	166.3	166.2	173
2'	—	—	—	—	—	—	—	—	—	—	14.9	—	43.6	43.6	116	115.9	43.8
3'	—	—	—	—	—	—	—	—	—	—	—	—	25.7	25.7	157.3	157.2	25.9
4'	—	—	—	—	—	—	—	—	—	—	—	—	22.4	22.4	27.4	27.4	22.6
5'	—	—	—	—	—	—	—	—	—	—	—	—	22.4	22.4	20.3	20.2	22.6
NO.	38 [37]	39 [33]	41 [7]	42 [11]	43 [8]	44 [37]	45 [37]	46 [37]	47 [37]	48 [37]	49 [37]	50 [39]	51 [37]	52 [22]	52 [22]	53 [22]	54 [40]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD
1	43.9	44.6	43.1	44.2	45	44.2	43.5	44.3	44.1	44.1	43.5	44.3	45.9	45.8	44	46.3	45.7
2	37.3	34.6	32.2	36.2	35.7	36.2	36	36.3	36.4	36.5	36.3	36.4	36.4	38.5	37.6	38.4	38.4
3	73.5	74.3	73.4	74.3	74.3	74.3	74.4	74.3	74.3	74.3	74.4	75.5	74.3	81.7	80.4	81.6	81.4
4	148.4	147.8	148.7	148.1	147.1	148.5	149	148.3	148.4	148.6	149.2	148.4	148.6	151.5	150.7	151	151.1
5	50.4	50.2	48.8	47.8	49.7	50.3	50	50.3	50	50	50.1	50.3	49.9	52	50.2	52.5	51.4
6	83.6	83.9	83.9	83.7	81.5	82.2	81.2	83.3	84.8	84.3	81	83	84.2	85.7	83.6	81.2	85.1
7	49.2	45.2	44.7	50.1	165.3	52.7	55	51.9	48.9	47.9	53.4	52	46.7	41.4	39.6	54.7	45.6
8	29.9	30.6	26.8	25	29.9	24.9	27.4	25.5	32.4	32.2	27.1	25.2	30.6	30	28.6	70.9	33.1
9	122.2	36.6	34	34.7	30.7	34.6	36	34.4	35.9	36.1	36.1	34.6	36.4	37	36	46	36.1
10	137	148.2	57.3	148	147.9	148.2	147.7	148.1	148.3	148.5	148	148.2	148.5	151.4	—	145.9	150.8
11	42.2	139.5	139.3	75	125.7	75.7	76.4	76.4	46.4	43.5	77.5	76.5	43.9	47.7	45.5	40.5	49.5
12	178.3	170	169.9	177.5	173.4	177.2	180.3	177.7	179.6	179	177.8	176	176.5	183.1	179.6	182.5	178.9
13	12.9	120.3	121	64.3	55	41.2	40.9	42.8	38.3	37.1	38	44.4	40.8	12.2	11.4	11.8	67.6
14	27.9	114.3	50.3	114	114.3	113.5	113.4	113.8	113.7	113.6	113.3	114	113.5	114.2	113	116.4	114
15	116.8	113.4	112.6	114.1	118.3	114	114.4	114.2	113.4	113.2	113.9	114.5	113	114.2	112.2	115.7	113.2

Table 11. Cont.

16	—	—	—	—	—	64.8	63.5	64.9	67	64.9	65.7	210.4	—	—	—	—	—			
17	—	—	—	—	—	24.9	24.4	24.9	24.3	23.9	24.6	32.3	—	—	—	—	—			
1'	173	172.8	166.2	173	173.1	173	172.9	172.9	172.8	172.8	172.8	173.1	172.8	103.3	103.8	102.5	103.2			
2'	43.6	43.6	115.9	43.6	43.8	43.6	43.6	43.6	43.6	43.6	43.6	43.8	43.6	75.7	75.3	75.8	75.3			
3'	25.8	25.8	157.9	25.8	25.9	25.7	25.7	25.8	25.8	25.8	25.7	25.9	25.8	78.7	78.6	78.8	78.2			
4'	22.4	22.4	27.6	22.4	22.6	22.4	22.4	22.4	22.4	22.4	22.4	22.5	22.4	72.2	71.7	72.4	71.8			
5'	22.4	22.4	20.5	22.4	22.6	22.4	22.4	22.4	22.4	22.4	22.4	22.6	22.4	78.4	78.4	78.4	77.9			
6'	—	—	—	—	—	—	—	—	—	—	—	—	—	63.3	62.9	63.4	62.9			
NO.	55 a [41]	56 b [42]	57 c [12]	57 a [22]	57 d [22]	58 e [43]	59 a [44]	60 e [45]	61 b [46]	61 e [25]	62 b [46]	62 b [25]	63 b [46]	NO.	58 e [43]	59 a [44]	60 e [45]	62 b [46]	62 b [25]	63 b [46]
1	44.4	42.1	45.4	44.5	43.7	46.2	45.9	46.1	43.5	46.2	43.4	43.4	43.9	1''	130.9	126.7	127.8	103.9	103.9	103.9
2	37.9	37.3	38	38	37.3	38.6	37.3	37.8	36.9	38.5	36.9	36.9	37.4	2''	129	114.9	115.4	72.4	72.4	73.4
3	80.5	86.2	80.7	80.5	79.7	81.3	79.5	80.9	83.3	81.4	83.2	83.2	79.8	3''	130	148.2	149.7	73.8	73.8	86.6
4	150.8	44.1	150.2	150.6	150.3	150.8	150.2	150.1	150	150.6	149.9	150	150.4	4''	150.1	147.3	146.8	79.4	79.4	68.9
5	50.8	49.5	50.7	50.1	49.1	51.8	51.6	52.4	48.8	51.7	48.8	48.8	49.2	5''	130	116.3	116.5	76.1	76.1	73.2
6	79.4	86.2	84.7	83.6	83.6	85.2	83.1	80.8	76.9	85.2	86.6	86.6	83.7	6''	129	122.1	123	60.8	61.1	63.5
7	56.3	46.8	45.8	45.1	44.6	46.4	45.9	54.4	44.3	46.5	44.3	44.3	44.7	1'''	172.3	166.4	168.3	103.4	103.4	103.5
8	75.1	30.6	31	30.6	30.4	31.6	30.4	70.6	30.1	31.6	30.1	30.1	30.5	2'''	121.7	115.6	115.3	72.7	72.7	73
9	46.6	35.7	33.9	34.2	34.1	34.4	33.8	46.4	33.8	34.4	33.8	33.7	34.2	3'''	144.6	145.9	147.3	76.9	76.9	87.2
10	145.1	149.5	149.3	148.9	149.1	150	149	144.8	148.7	150	148.7	148.8	149.2	4'''	—	—	—	68.5	68.5	69
11	42.3	140.1	141.5	141	140.5	142.1	140.5	39.9	140.2	142.1	140.1	140.1	140.6	5'''	—	—	—	76.3	76.1	76.6
12	179	169.6	171.6	170	169.9	172.3	169.9	181.9	169.6	172.2	169.5	169.5	170	6'''	—	—	—	61.1	61	61.3
13	16.5	119.5	120	119.4	120.1	120.4	119.5	11.5	119.8	120.4	119.7	119.9	120.2	—	—	—	—	—	—	—
14	115.1	112.7	114.1	113.9	113.9	114.7	114.9	116.6	113.6	114.7	113.6	113.6	114	NO.	61 b [46]	61 e [25]	—	—	—	—
15	112.3	18.1	112.8	112.1	111	113.6	114.9	116.2	110.8	113.8	110.8	110.8	111.1	1''	104.1	105.3	—	—	—	—
1'	104	104.1	102.3	104.3	102.9	103.1	98.5	99.4	101.7	102.3	101.7	101.7	102.2	2''	72.3	75.6	—	—	—	—
2'	75.3	73.6	74.3	75.3	73.9	78.2	76.1	75.3	70.2	74.6	70.1	70.1	72.8	3''	73.9	78.3	—	—	—	—
3'	78.2	76.8	77.5	78.6	77.2	75.3	74.9	76.4	88.3	88.3	88	88	88.4	4''	79.4	71.5	—	—	—	—
4'	71.8	70.2	71.1	71.7	70.5	71.8	71.8	72.1	68.5	70.2	68.4	68.4	68.9	5''	76.1	77.8	—	—	—	—
5'	78.5	76.8	77.2	78.5	77.2	77.9	78.2	78.1	76.3	77.5	76.3	76.3	76.8	6''	61.1	62.7	—	—	—	—
6'	62.9	61.2	62.1	62.8	61.5	62.8	62.6	62.8	60.9	62.6	60.8	60.9	63.5	—	—	—	—	—	—	—

Note: The 1'', 2'', 3'', and 4'' data of compound **51** were 176.3; 32.4; 18.3; and 48.8, respectively. The ¹³C-NMR data of 1'' and 2'' of **54** were recorded as 67.8 and 15.3. a: Measured in C₅D₅N. b: Measured in DMSO. c: Measured in CDCl₃. d: Measured in CD₃SOCD₃. e: Measured in CD₃OD.

2.2. Bioactivity of Guaiane Sesquiterpenes

2.2.1. Anti-Inflammatory

Nitric oxide (NO) is a related target of inflammation, and inhibiting the release of NO can treat inflammatory diseases. Dihydroestafiatol (**13**), zaluzanin C (**23**), and dehydrozaluzanin C (**31**) strongly inhibited the production of nitric oxide in RAW264.7 macrophages stimulated with lipopolysaccharide (LPS), with IC₅₀ values of 7.11, 2.50, and 0.82 μM [31]. From the bioassay results, three exocyclic double bonds in guaianolides play a key role in the inhibition of the production of nitric oxide (NO), and a reduction in exocyclic double bonds will lower the inhibitory effect. Under the condition of the presence of three exocyclic double bonds, the hydroxylation of C-1 will enhance the inhibitory activity. Zaluzanin C (**23**) showed a potent inhibitory effect against NO production in LPS-stimulated RAW264.7 macrophages with an IC₅₀ value of 6.54 ± 0.16 μM [34]. It may be speculated that the α-methylene-γ-lactone moiety of zaluzanin C (**23**) has a key role in its inhibition of NO release. Moreover, other functional groups, especially hydroxyl, have a great influence on the inhibitory effect of NO production. Zaluzanin C (**23**) showed remarkable inhibition against NO release in LPS-induced RAW264.7 macrophages, possibly because zaluzanin C (**23**) had an α-methylene-γ-lactone moiety and the large isovaleroxyl at C-3 hinders the binding of the compound to related proteins [37]. Guailactone can be structurally modified to obtain compounds containing the α-methylene-γ-lactone part. It is also used to introduce hydroxyl groups into guaiacols containing three outer-ring double bonds to enhance the inhibitory ability of these compounds against NO production and achieve anti-inflammatory effects.

Ainslide C (**41**), ainsliaolide A (**34**), diaspanolide B (**39**), zaluzanin C (**23**), and estafiatone (**28**) inhibit NLRP3 inflammasome activity by inhibiting the LDH release rate. Meanwhile, a Western blot assay showed that compound **41** inhibited the activity of inflammasome by inhibiting the production of Caspase-1 and IL-1β induced by LPS and Nigericin. Among them, the substituents of compounds **41**, **34**, **39**, **23**, and **28** are terminal double bonds, and the α-methylene-γ-butyrolactone structure seems to be the key to inhibiting LDH release activity [7]. Glucozaluzanin C (**57**) and dihydroestafiatol (**13**) showed significant anti-inflammatory activity by inhibiting the expression of nuclear factor kappa B (NF-κB) in the 293-NF-κB-luciferase reporter cell line and the production of TNF-α, IL-1β, IL-6, and IL-10 in RAW264.7 macrophages induced by lipopolysaccharide (LPS) [47].

8α-Hydroxy-11α,13-dihydrozaluzanin C (**20**) showed moderate COX-1-inhibiting activity with an IC₅₀ value of 78.8 μM, comparable to that of the representative anti-inflammatory drug aspirin with an IC₅₀ value of 77.2 μM. 8α-Hydroxy-11α,13-dihydrozaluzanin C (**20**) and 2'-O-E-Caffeoyl-8α-hydroxy-11α,13-dihydro-3-β-O-β-D-glucozaluzanin C (**60**) displayed potent COX-2 inhibitory activities with IC₅₀ values ranging from 12.5 to 57.9 μM, in comparison with that of aspirin with an IC₅₀ value of 87.6 μM [45].

2.2.2. Antitumor and Cytotoxic

8-Epidesacylcinaropicrin (**25**) exhibited moderate activity toward the human tumor cell lines MDA-MB-231 and HepG2, with IC₅₀ values of 18.91, and 11.16 μM, respectively [8]. Mokko lactone (**9**), zaluzanin C (**23**), and glucozaluzanin C (**57**) showed non-specific significant cytotoxicity against the A549 (non-small cell lung adenocarcinoma), SK-OV-3 (ovarian), SK-MEL-2 (skin melanoma), XF498 (CNS), and HCT15 (colon) cell lines with ED₅₀ values ranging from 0.36 to 5.54 μg/mL [12]. Dehydrozaluzanin C (**31**) is a guaicol lactone, which has significant cytotoxicity to RAW264.7 macrophages. In the presence of three outer-ring double bonds, the carbonylation of C-1 may result in a high cytotoxicity to RAW264.7 macrophages [31].

2.2.3. Antiobesity

Ainsliaside A (**59**) isolated from *Ainsliaea acerifolia* had significant inhibitory activity on pancreatic lipase with a semi-inhibitory concentration of 15.3 ± 0.7 μM. In addition,

ainsliaside A (59) also exhibited potent inhibitory effects against 3T3-L1 adipocyte cells and can be used as a potential antiobesity agent [43].

3. Germacrane-Type Sesquiterpenes

Germacrane sesquiterpenes represent a class of sesquiterpenes that are extensively distributed in Compositae plants, characterized by the formation of a substantial ten-membered ring structure at the 5 and 10 positions. Currently, all twelve germacrane-type sesquiterpenes reported from this genus are lactones, with lactone rings predominantly located at the C-6, C-7 and C-7, C-8 positions. Detailed information is presented in Figure 2 and Table 12.

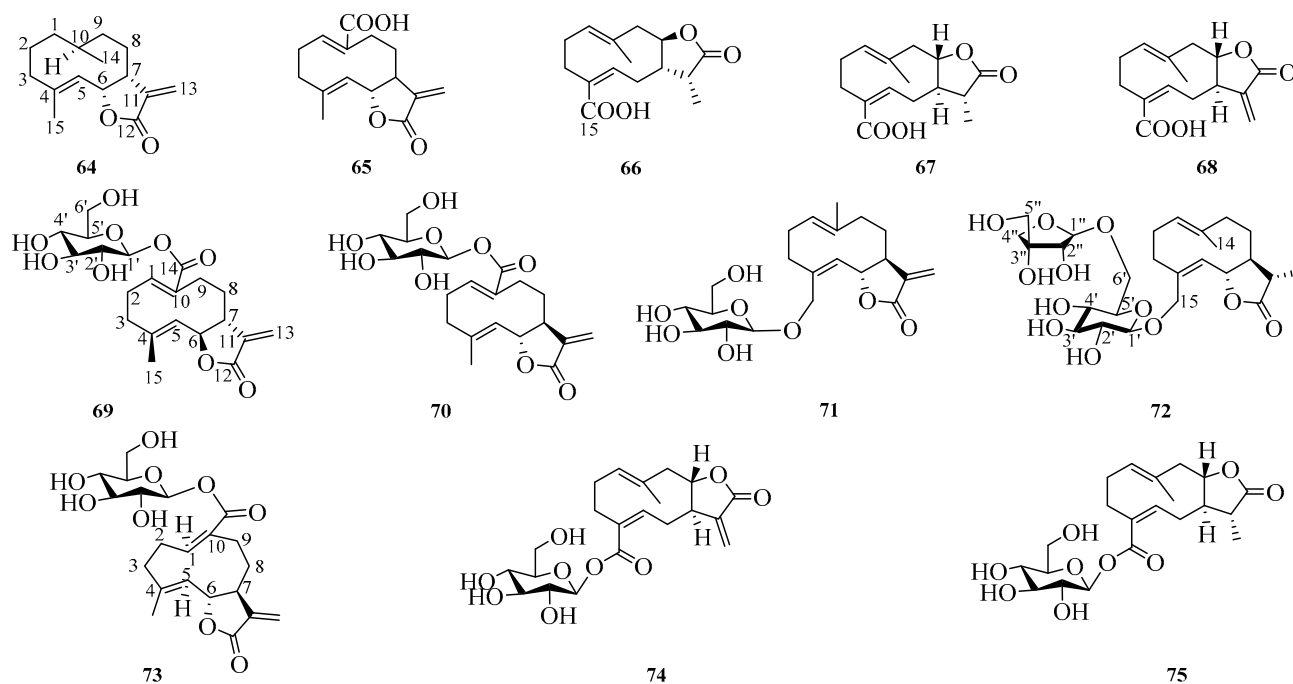


Figure 2. Chemical structures for compounds 64–75.

Table 12. The compound name, molecular formula, and NMR test reagent of germacrane-type sesquiterpenes.

No.	Compound Name	Molecular Formula	Solvent	Ref.
64	Isodihydrocostunolide	C ₁₅ H ₂₂ O ₂	CDCl ₃	[48]
65	Taraxinic acid	C ₁₅ H ₁₈ O ₄	CDCl ₃	[49]
66	Yunnanolide K	C ₁₅ H ₂₀ O ₄	CDCl ₃	[11]
67	Germacra-1(10), 4-diene-11 α -methyl-12,8 α -olide-15-acid	C ₁₅ H ₂₀ O ₄	DMSO/CD ₃ OD	[34,50]
68	Germacra-1(10),4,11(13)-triene-12,8 α - olide-15-acid	C ₁₅ H ₁₈ O ₄	CD ₃ OD	[50]
69	Ainsliaside B	C ₂₁ H ₂₈ O ₉	C ₅ D ₅ N CD ₃ OD	[44]
70	Taraxinsaure-1'-O- β -D-glucopyranoside	C ₂₁ H ₂₈ O ₉	CD ₃ OD	[51]
71	Picriside B	C ₂₁ H ₃₀ O ₈	C ₅ D ₅ N	[27]
72	Ainsliaolide C	C ₂₆ H ₄₀ O ₁₂	DMSO	[31]
73	Taraxic acid-1'-O- β -D-glucopyranoside	C ₂₁ H ₂₈ O ₉	CD ₃ OD/C ₅ D ₅ N	[2]
74	Germacra-1(10),4,11(13)-triene-12,8 α - olide-15-oic	C ₂₁ H ₂₈ O ₉	CD ₃ OD	[50]
75	acid(15-1')- β -D-glucopyransyl ester Ainsliaea latifolia A	C ₂₁ H ₃₀ O ₉	CD ₃ OD	[52]

3.1. NMR Data of Germacrane-Type Sesquiterpene (64–75)

The NMR spectrum data for both ^1H and ^{13}C are presented in Tables 13 and 14. A summary of the test instruments used to obtain the NMR data for compounds 64–75 is provided. The ^1H and ^{13}C data of compounds 67, 68, and 74 were measured with the Bruker Avance III-500 instrument (Bruker, Switzerland). The nuclear magnetic data of compounds 69 and 71 were obtained by the JEOL FX-90Q instrument (JEOL, Duzhao, Japan). The NMR spectra of compounds 65, 66, 67, 72, 73, and 75 were recorded on various instruments including the Brukerspeckospin AC-600P (Bruker, Germany), Bruker Ascend-500 spectrometer (Bruker, Germany), Bruker Advance 500 (Bruker, Germany), Bruker Avance 600 (Bruker, Biel, Switzerland), JNM-FX-100 (JEOL, Japan), and Bruker Avance-500 (Bruker, Karlsruhe, Germany), respectively. The ^1H -NMR data of compound 64 were measured at a frequency of 300 MHz; however, no literature reports exist regarding the proton data for compound 70. The carbon spectra for compounds 64 and 70 were acquired at 75 MHz and 25.2 MHz, respectively.

3.2. Bioactivity of Germacrane-Type Sesquiterpene

Isodihydrocostunolide (64) showed moderate cytotoxicity against the human cancer cell lines MDA-MB-231 ($\text{IC}_{50} = 18.2 \mu\text{M}$) and HepG2 ($\text{IC}_{50} = 12.2 \mu\text{M}$), respectively [11]. Ainsliaea latifolia A (75), isolated from Ainsliaea latifolia, exhibited moderate activity against the HCT116 and SMMC-7721 human tumor cell lines when adriamycin was used as the positive control, with IC_{50} values of 14.72 and 10.53 μM [52].

Table 13. ¹H-NMR data of compounds 64–75.

NO.	64 [48]	65 [49]	66 [11]	67 [34]	67 [50]	68 [50]	69 [44]	71 [27]	72 [31]	73 [2]	74 [50]	75 [52]
	CDCl ₃	CDCl ₃	CDCl ₃	DMSO	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N	C ₅ D ₅ N	DMSO	C ₅ D ₅ N	CD ₃ OD	CD ₃ OD
1	1.68–1.40, m	5.68, dd, 13.0, 3.7	5.09, dd, 12.0, 5.0	5.06, dd, 11.7, 4.5	5.06, dd, 11.7, 4.5	5.06, dd, 11.7, 4.5	6.72, br t, 7	—	4.81, m	5.64, dd, 11.0, 4.0	5.13, m	5.16, dd, 11.8, 4.7
2 α	1.68–1.40, m	3.38, m	2.31, m	2.16, t, 12.3, 4.9	2.13, m	2.13, m	—	—	2.17, m	2.14, m	—	2.38–2.42, m
2 β	—	—	2.18, m	2.02–2.06, m	2.02, m	2.02, m	—	—	2.26, m	3.54, m	—	2.14–2.20, m
3 α	2.10, d, 2.7	2.25, m	2.93, m	2.66–2.72, overlapped	2.69, m	2.69, m	—	—	1.83, m	2.3–2.0, m	—	2.92, dd, 12.5, 3.9
3 β	1.9–1.7, m	2.35, m	1.88, m	1.78, t, 12.3, 5.2	1.78, m	1.78, m	—	—	2.61, m	—	—	1.90–1.98, m
5	5.15, d, 6	4.91, d, 10.0	5.51, dd, 11.0, 2.5	5.38, dd, 11.1, 2.1	5.38, dd, 11.1, 2.1	5.38, dd, 11.1, 2.1	4.81, d, 10	—	4.75, br s	4.94, dd, 10.0, 1.2	5.74, m	5.65, dd, 11.3, 2.6
6 α	—	—	3.13, m	3.01, t, 16.1, 10.8	3.01, m	3.01, m	4.58, dd, 10, 9	—	—	—	—	3.19–3.24, m
6 β	4.8, m	4.58, dd, 10.0, 9.0	2.49, d, 16.5	2.41–2.55, overlapped	2.44, m	2.44, m	—	—	4.77, d, 9.6	4.72, dd, 10.0, 10.0	—	2.55–2.65, m
7	3.6, m	2.56, m	2.31, m	1.82–1.90, m	1.86, m	1.86, m	—	—	1.63, m	2.54, m	—	1.92–2.01, m
8 α	1.68–1.40, m	2.19, m	4.39, t, 11.5	—	—	—	—	—	1.72, m	2.3–2.0, m	—	—
8 β	—	—	—	4.19–4.25, m	4.22, m	4.22, m	—	—	2.06, m	—	4.27, m	4.27–4.37, m
9 α	1.68–1.40, m	2.90, m	2.93, m	2.66–2.72, overlapped	2.66, m	2.66, m	—	—	2.24, m	2.86, m	—	2.83, d, 12.3
9 β	—	2.15, m	2.28, m	2.27, t, 11.8	2.27, m	2.27, m	—	—	1.96, m	2.3–2.0, m	—	2.33–2.38, m
10	2.5, m	—	—	—	—	—	—	—	—	—	—	—
11	—	—	2.78, m	2.41–2.55, overlapped	2.51, m	—	—	—	2.33, m	—	—	2.54–2.58, m
13 α	6.2, d, 2	6.24, d, 3.4	1.26, d, 7.5	1.11, d, 7.0	1.11, d, 7.0	6.08, m	—	5.51, d, 3.2	1.09, d, 6.6	6.23, d, 3.5	6.23, dd, 18.8, 3.0	1.28, d, 7.0
13 β	5.65, d, 2	5.51, d, 2.9	—	—	—	5.79, m	—	6.35, d, 3.6	—	—	5.74, m	—
14	1.1, d, 8	—	1.35, s	1.27, s	1.27, s	1.31, s	—	1.37, br s	1.30, s	—	1.41, s	1.41, s
15 α	1.2, s	1.60, s	—	—	—	—	1.71, br s	—	3.78, br s	1.72, d, 1.2	—	—
15 β	—	—	—	—	—	—	—	—	4.40, br s	—	—	—
1'	—	—	—	—	—	—	6.26, d, 8	4.96, d, 7.5	4.17, d, 7.8	6.18, d, 7.6	5.52, d, 7.9	5.56, d, 7.8
2'	—	—	—	—	—	—	—	—	2.97, m	4.44–3.80, m	—	3.43, overlapped
3'	—	—	—	—	—	—	—	—	3.13, m	4.44–3.80, m	—	3.43, overlapped
4'	—	—	—	—	—	—	—	—	2.99, m	4.44–3.80, m	—	3.43, overlapped
5'	—	—	—	—	—	—	—	—	3.23, m	4.44–3.80, m	—	3.43, overlapped
6' α	—	—	—	—	—	—	—	—	3.44, d, 11.2, 7.2	4.44–3.80, m	3.81, m	3.84, dd, 12.0, 2.0
6' β	—	—	—	—	—	—	—	—	3.81, d, 11.2, 1.8	4.44–3.80, m	3.69, m	3.75, dd, 12.0, 4.4

Note: The ¹H-NMR data of 1'', 2'', 4'' α , and 4'' β and 5'' of 72 were recorded as 4.88, br s; 3.74, m; 3.57, d, 9.6; 3.83, d, 9.6; and 3.33, s, respectively.

Table 14. ¹³C-NMR data of compounds 64–75.

NO.	64 [48]	65 [49]	66 [11]	67 [34]	67 [50]	68 [50]	69 [44]	70 [51]	71 [27]	72 [31]	73 [2]	74 [50]	75 [52]
	CDCl ₃	CDCl ₃	CDCl ₃	DMSO	CD ₃ OD	CD ₃ OD	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N	DMSO	CD ₃ OD	CD ₃ OD	CD ₃ OD
1	16.7	149.9	128.8	128.9	128.9	130.4	143	149.7	126.9	125.9	149.6	130	129.8
2	32.6	26.7	26.8	27.1	27.1	27.6	26.2	27.6	27.7	25.9	27.6	27.7	27.9
3	42.6	39.2	34.8	34.9	34.9	35.7	38	39.9	36	34.9	40	35.5	35.7
4	148.9	139.8	124.1	125.1	125.1	126.6	141.3	141.8	141	138.8	141.8	126.6	125.5
5	118.5	126	150.7	148.6	148.6	149.2	127.1	127.2	130.1	130.1	127.1	151.6	152
6	76.4	82.1	28.6	31.1	31.1	32.8	82.8	83.8	80.3	78.7	83.8	32.8	32.4
7	39.5	50.3	48.3	52.6	52.6	50.1	46.7	51.1	50.8	53.7	51.1	50.1	54.4
8	41.7	30.1	82.4	82.6	82.6	85.1	24.2	31.3	27.1	27	31.3	85	84.5
9	32.7	36.7	46.2	45.5	45.5	47.2	26.4	37.2	41.1	40.7	37.2	47.2	46.8
10	37.5	130.3	134.2	134	134	134.3	135.1	131.9	137.6	137	131.9	135.1	135.6
11	139.9	143.2	40.7	41.4	41.4	141.2	139	144.5	141.2	41	144.4	141.1	43
12	170.4	170.6	178.7	178	178	171.7	172.4	172.7	170.3	178.1	172.6	171.7	180.5
13	121.5	120.1	11.7	13.1	13.1	121.2	119.4	120.4	119.1	12.8	120.4	121.2	13.2
14	22.5	173.3	16.8	16.8	16.8	17.1	167.6	169.8	16.3	15.8	167.8	17.4	17.3
15	28.6	17	171.9	169	169	167.5	17.2	17.3	67.8	65.8	17.3	167.5	167.7
1'	—	—	—	—	—	—	95.7	95.3	105.3	103.1	95.3	95.7	95.7
2'	—	—	—	—	—	—	73.9	73.9	75.2	73.3	73.9	74	74
3'	—	—	—	—	—	—	78.6	78.3	78.6	76.6	78.3	78.7	78.4
4'	—	—	—	—	—	—	71	71	71.8	70.2	71	71	71
5'	—	—	—	—	—	—	77.9	78.7	78.6	75.8	78.7	78.4	78.7
6'	—	—	—	—	—	—	62.4	62.3	62.9	67.5	62.4	62.2	62.2

Note: The ¹³C NMR of 1'', 2'', 3'', 4'', and 5'' of 72 were 109.3; 75.6; 78.7; 73.2; and 63.3, respectively.

4. Eudesmane Sesquiterpenes

The basic skeleton of eudesmane-type sesquiterpenes consists of two six-membered rings comprising a total of 15 carbon atoms. These compounds exhibit a diverse range of biological activities, including anti-inflammatory, cytotoxic, antibacterial, antimalarial, insecticidal, and neuroprotective activities [53]. Up to now, 35 eudesmane-type sesquiterpenes have been reported within this genus. Their structures and detailed information are shown in Figure 3 and Table 15.

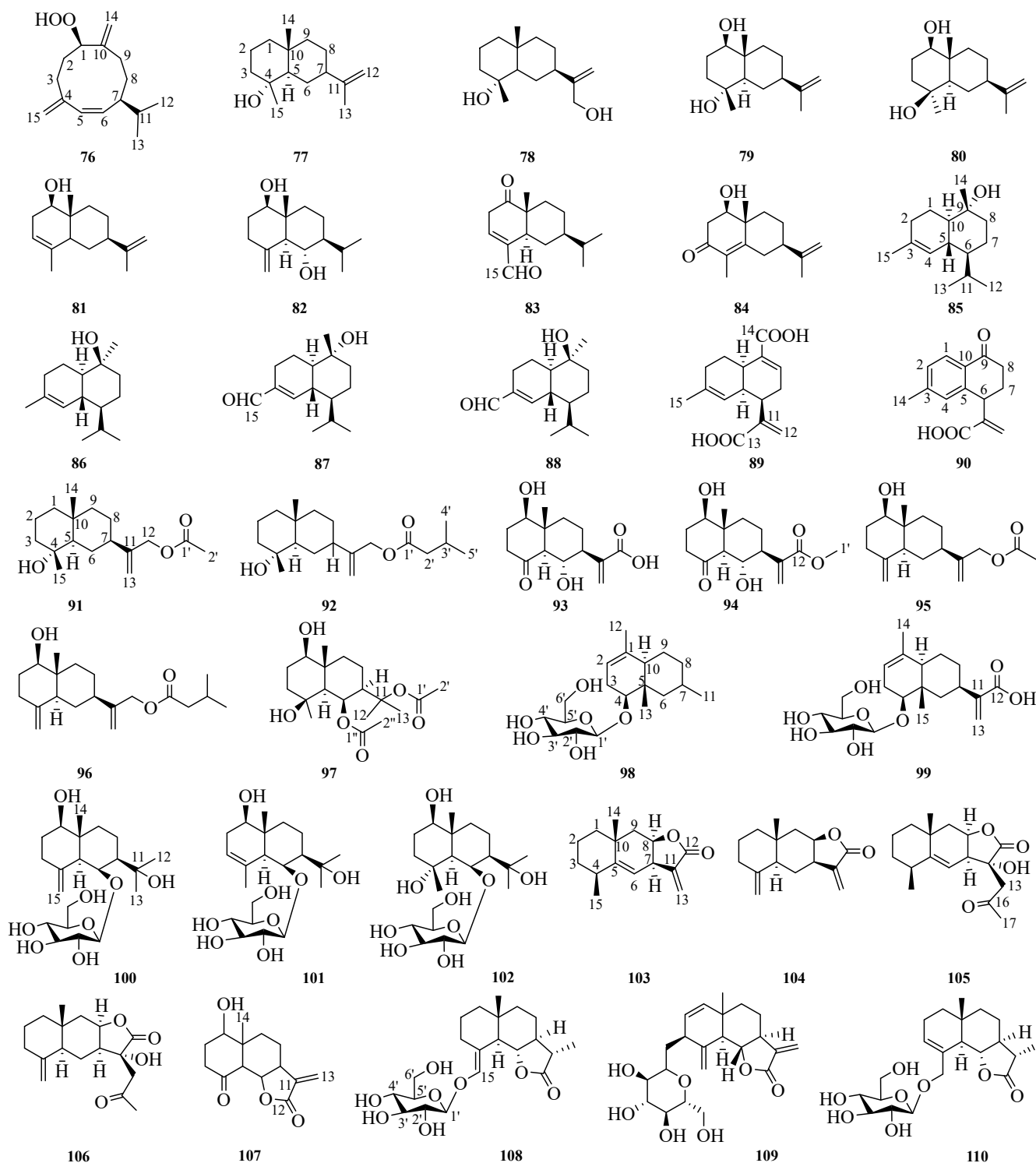


Figure 3. Chemical structures for compounds 76–110.

Table 15. The compound name, molecular formula, and NMR test reagent of eudesmane sesquiterpenes.

No.	Compound Name	Molecular Formula	Solvent	Ref.
76	1 β -Hydroperoxygermacra-4(15),5,10(14)-triene	C ₁₅ H ₂₄ O ₂	CDCl ₃	[12]
77	Selin-11-en-4 α -ol	C ₁₅ H ₂₆ O	CDCl ₃	[54]
78	4 α -Hydroxy-4 β -methyl-dihydrocostol	C ₁₅ H ₂₆ O ₂	CDCl ₃	[55]
79	Cyperusol C	C ₁₅ H ₂₆ O ₂	CDCl ₃	[56]
80	1 β ,4 β -Dihydroxyeudesman-11-ene	C ₁₅ H ₂₆ O ₂	CDCl ₃	[57]
81	α -Dictyoptero	C ₁₅ H ₂₄ O	CDCl ₃	[58]
82	1 β ,6 α -Dihydroxy-4(15)-eudesmane	C ₁₅ H ₂₆ O ₃	CDCl ₃	[59]
83	1-oxo-5 α ,7 α H-eudesma-3-en-15-al	C ₁₅ H ₂₂ O ₂	CDCl ₃	[60]
84	1 β -Hydroxy- α -cyperone	C ₁₅ H ₂₂ O ₂	CDCl ₃	[61]
85	(-)- α -Cadinol	C ₁₅ H ₂₆ O	CDCl ₃	[62,63]
86	T-Cadinol	C ₁₅ H ₂₆ O	CDCl ₃	[63,64]
87	10-Hydroxyl-15-oxo- α -cadinol	C ₁₅ H ₂₄ O ₂	CDCl ₃	[65,66]
88	15-oxo-T-cadinol	C ₁₅ H ₂₄ O ₂	CDCl ₃	[66]
89	Ainsliaea acid B	C ₁₅ H ₁₈ O ₄	CD ₃ OD	[67]
90	4-Acrylic-6-methyl- α -tetralone	C ₁₄ H ₁₄ O ₃	CDCl ₃	[67]
91	4 α -Hydroxy-12-acetoxy-eudesm-11(13)-en	C ₁₇ H ₂₈ O ₃	—	[68]
92	4 α -Hydroxy-eudesm-11-en-12-isovaleroxyl	C ₂₀ H ₃₄ O ₃	CDCl ₃	[34]
93	Ainsliatone A acid	C ₁₄ H ₂₀ O ₅	CD ₃ OD	[42]
94	Ainsliatone B	C ₁₅ H ₂₂ O ₅	CDCl ₃	[31]
95	Ainslide B	C ₁₇ H ₂₆ O ₃	CDCl ₃	[7]
96	Spicatene C	C ₂₀ H ₃₂ O ₃	CDCl ₃	[8]
97	6,11-Diacetoxy-1,4-dihydroxyeudesmane	C ₁₉ H ₃₂ O ₆	CD ₃ OD	[40]
98	Alatoside N	C ₂₀ H ₃₄ O ₆	CD ₃ OD	[42]
99	Alatoside M	C ₂₁ H ₃₂ O ₈	CD ₃ OD	[42]
100	Ainsliaside C	C ₂₁ H ₃₆ O ₈	C ₅ D ₅ N	[69]
101	Ainsliaside D	C ₂₁ H ₃₆ O ₈	C ₅ D ₅ N	[69]
102	Ainsliaside E	C ₂₁ H ₃₈ O ₉	C ₅ D ₅ N	[69]
103	Alantolactone	C ₁₅ H ₂₀ O ₂	CDCl ₃	[70]
104	Isoalantolactone	C ₁₅ H ₂₀ O ₂	CDCl ₃	[70]
105	Pertyolides B	C ₁₇ H ₂₄ O ₄	CDCl ₃	[39]
106	Pertyolides A	C ₁₇ H ₂₄ O ₄	CDCl ₃	[39]
107	Ainsliatone A	C ₁₄ H ₁₈ O ₄	CDCl ₃	[71]
108	4(15)-En-eudesma-6,12-olide-15-O- β -D-glucopyranoside	C ₂₁ H ₃₂ O ₈	CD ₃ OD	[40]
109	Ixerin W	C ₂₂ H ₃₀ O ₇	C ₅ D ₅ N	[27]
110	3(4)-En-eudesma-6,12-olide-15-O- β -D-glucopyranoside-O- β -D-glucopyranoside	C ₂₁ H ₃₂ O ₈	CD ₃ OD	[40]

4.1. NMR Data of Eudesmane-Type Sesquiterpene (76–110)

The ¹H and ¹³C spectrum data are shown in Tables 16–20. An overview of the testing instruments used for the NMR data of compounds 76–110 is provided. The NMR data for compounds 82 and 87 were measured with the Bruker DRX-500 spectrometer (Bruker, Germany). The ¹H and ¹³C spectra of compounds 85 and 86 were obtained by the JEOL MN 100 instrument (JEOL, Japan). For compounds 87 and 88, NMR data were recorded on the Bruker AM 400 spectrometer (Bruker, Switzerland). The NMR data for compounds 93, 98, and 99 were taken with the Varian 500 (Varian, Palo Alto, CA, USA) and Bruker AV500-III instruments (Bruker, Switzerland). Compounds 97, 108, and 110 had their NMR data obtained on the Bruker AV-600 spectrometer (Bruker, Switzerland). The ¹H and ¹³C data of compounds 100, 101, 102, and 109 were measured using the JEOL FX-90Q instrument (JEOL, Japan). The NMR data for compounds 100, 101, and 102 were recorded by the GSX-270 (JEOL, Japan) and GSX-500 NMR instruments (JEOL, Japan). The ¹H and ¹³C spectra of compounds 103 and 104 were analyzed using the Bruker Avance DRX 500 spectrometer (Bruker, Germany). NMR data for compounds 76, 77, 79, 80, 83, 85, 92, 94, 95, and 96 were obtained from various instruments including Bruker AMX 500 (Bruker, Zurich, Switzerland) and Varian Unity Inova 500 (Varian, Palo Alto, CA, USA),

Bruker DRX-300 (Bruker, Karlsruhe, Germany), JEOL JNM LA-500 (JEOL, Japan), Varian Mercury-300 BB (Varian, San Jose, USA), Varian Mercury Plus 400 (Varian, USA), NT-200 (University of California, Davis, CA, USA), Bruker Avance 500 (Bruker, Germany), Bruker Avance 600 (Bruker, Biel, Switzerland), Bruker AV-400 HD (Bruker, Byersbin, Switzerland), Bruker Ascend 500 (Bruker, Zurich, Switzerland), and other instruments. Compound **78** was measured at 200 MHz for ^1H NMR. Compounds **84** and **107** were tested at 400 MHz. Compounds **89** and **90** were recorded at 600 MHz. Compound **91** was run at 60 MHz. Compounds **105** and **106** were operated at 500 MHz. No test instrumentation has been reported in the literature regarding NMR spectrum data for compound **81**. Furthermore, no hydrogen spectrum data of compound **86** have been reported in the existing literature. The carbon spectrum data for compound **84** were measured at 50.32 MHz; the ^{13}C data collection for compound **107** occurred at 100 MHz; compound **86** was recorded at 75 MHz for ^{13}C NMR; while the carbon spectrum data of compounds **89** and **90** were measured at 150 MHz. Compounds **78**, **91**, **105**, and **106** have not been described within any available literature concerning their ^{13}C NMR spectra.

4.2. Bioactivity of Eudesman-Type Sesquiterpene

Eucalyptane sesquiterpenes exert anti-inflammatory effects by inhibiting the activity of the NLRP3 inflammasome and inhibiting the production of NO in RAW264.7 macrophages. Cyperusol C (**79**) derived from *Ainsliaea pertyoides* can inhibit NLRP3 inflammasome activity by inhibiting the LDH release rate [7]. Ainsliatone B (**94**) strongly inhibited the production of nitric oxide in RAW264.7 macrophages stimulated with lipopolysaccharide (LPS), with IC_{50} values of 8.78 μM [31].

Isoalantolactone (**104**) remarkably inhibited the proliferation of MGC803 cell lines with IC_{50} values of $2.2 \pm 0.2 \mu\text{M}$. Double-bond moieties may be necessary for its cytotoxicity [34]. Alantolactone (**103**) exhibited significant inhibition against the human tumor cell lines A549, HCT116, MGC803, and CCRF-CEM with IC_{50} values of 3.56, 2.23, 2.89, and 14.67 μM , respectively [39].

Table 16. ¹H-NMR data of compounds 76–84.

NO.	76 [12]	77 [54]	78 [55]	79 [56]	80 [57]	81 [58]	82 [59]	83 [60]	84 [61]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	4.15, dd, 12.0, 3.5	—	—	3.32, dd, 11.2, 4.4	3.27, dd, 11.4, 4.8	3.49–3.56, m	3.43, dd, 11.7, 4.6	—	3.83, dd, 13, 5.5
2 α	2.05, m	—	—	1.62, m	1.96, m	1.45–1.55, m	1.86, m	2.54, m	2.64, dd, 16.5, 5.5
2 β	—	—	—	1.72, m	1.91, m	—	1.55, m	—	2.56, dd, 16.5, 13
3 α	2.27, ddd, 2.5, 5.5, 13.0	—	—	1.52, ddd, 13.5, 12.0, 3.5	1.16, m	5.26, s	2.07, m	6.64, br d, 5.2	—
3 β	2.46, td, 13.0, 5.0	—	—	1.79, ddd, 12.0, 3.5, 3.0	1.09, m	—	2.33, m	—	—
5	6.04, d, 16.0	1.81, dddd	—	1.28, m	1.24, dd, 9.6, 3.6	1.10–1.38, m	1.75, m	2.21, dd, 10.8, 4.8	—
6 α	5.46, dd, 16.0, 10.5	—	—	1.26, m	1.27, m	1.10–1.38, m	—	1.44, m	2.19, ddd
6 β	—	—	—	1.84, m	1.22, m	—	3.72, dd, 9.8, 9.8	1.87, m	2.08, ddd, 14, 12, 1.5
7	1.5–1.86, 2.62, m	1.96, dddd, br	—	1.94, m	1.58, m	1.84–1.92, m	1.28, m	1.81, m	2.02, br dddd, 13.5, 12, 3.5, 2.5
8 α	1.5–1.86, 2.62, m	—	—	1.61, m	1.78, m	1.84–1.92, m	1.53, m	1.58, m	1.75, m
8 β	—	—	—	1.38, dddd, 17.0, 13.5, 13.0, 3.5	1.71, m	—	1.21, m	—	1.59, dddd, 13.5, 13.5, 13.5, 3.5
9 α	1.5–1.86, 2.62, m	—	—	1.13, ddd, 13.5, 13.0, 4.0	1.88, m	1.84–1.92, m	1.17, m	2.43, ddd, 14.4, 10.8, 3.6	1.35, ddd, 13.5, 13.5, 4
9 β	—	—	—	1.90, ddd, 13.5, 3.5, 3.5	1.86, m	—	1.92, m	2.86, ddd, 14.4, 5.4, 5.4	2.16, ddd, 13.5, 3.5, 3
11	1.5–1.86, 2.62, m	—	—	—	—	—	2.24, m	1.67, m	—
12 α	0.83, d, 6.5	—	4.12, s	4.72, m	4.74, br s	4.71, s	0.95, d, 6.9	0.94, d, 6.8	4.78, m
12 β	—	—	—	—	4.71, br s	—	—	—	—
13 α	0.92, d, 6.5	1.75, s	5.00, d, 1.4	0.89, s	1.76, s	1.73, s	0.87, d, 7.1	0.94, d, 6.8	1.78, dd
13 β	—	—	4.90, d, 1.0	—	—	—	—	—	—
14 α	5.21, br s	1.12, s	0.88, s	1.75, s	1.05, s	1.58, s	0.71, s	1.33, s	1.18, s
14 β	5.34, br s	—	—	—	—	—	—	—	—
15 α	4.89, br s	0.89, s	1.09, s	1.11, s	1.16, s	0.76, s	5.02, s	9.35, s	1.73, d, 1
15 β	4.97, br s	—	—	—	—	—	4.74, s	—	—

Table 17. ¹H-NMR data of compounds 85–92.

NO.	85 [63]	86 [63]	87 [66]	88 [66]	89 [67]	90 [67]	91 [68]	92 [34]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃	—	CDCl ₃
1 α	—	—	2.16, m	2.24, m	2.34, m	7.97, d, 8.0	—	1.36–1.46, overlapped
1 β	—	—	1.21, m	1.21, m	1.69, m	—	—	1.12, m
2 α	—	—	2.06, m	2.06, m	1.65, m	7.17, d, 8.0	—	1.53–1.62, overlapped
2 β	—	—	2.46, m	2.45, m	1.80, m	—	—	—
3 α	—	—	—	—	—	—	—	1.77–1.81, m
3 β	—	—	—	—	—	—	—	1.36–1.46, overlapped

Table 18. ¹H-NMR data of compounds 93–100.

NO.	93 [42]	94 [31]	95 [7]	96 [8]	97 [40]	98 [42]	99 [42]	100 [69]
	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N
1 α	3.80, dd, 10.0, 5.0	3.86, dd, 11.7, 5.1	3.43, dd, 11.6, 4.6	3.43, dd, 11.6, 4.6	3.12, dd, 11.0, 3.4	—	—	3.58, dd, 11, 4
1 β	—	—	—	—	—	—	—	—
2 α	2.08, m	1.89, m	1.78–1.87, m	1.57, m	1.46, m	5.30, br s	5.31, br s	—
2 β	1.84, m	2.14, m	1.55–1.60, m	1.82, m	1.93, m	—	—	—
3 α	2.21, m	2.42, m	2.32, ddd, 13.6, 4.9, 2.2	2.10, m	1.49, m	2.00, m	2.03, m	—
3 β	2.53, m	—	2.06–2.17, m	2.32, m	1.58, m	2.40, m	2.41, m	—
4	—	—	—	—	—	3.67, dd, 10.0, 6.5	3.72, dd, 10.0, 6.5	—
5	1.64, m	2.24, d, 9.9	1.75–1.82, m	1.77, m	0.96, br s	—	—	2.84, d, 6
6 α	—	—	1.67–1.74, m	1.25, m	5.78, s	1.25, m	1.26, m	4.92, br t, 5.5
6 β	4.14, t, 10.0	4.22, dd, 10.2, 9.9	1.39–1.46, m	1.67, m	—	2.37, m	2.40, m	—
7	2.41, m	2.54, m	1.98–2.08, m	2.00, m	2.11, d, 13.6	1.95, m	2.02, m	—
8 α	1.64, m	1.67, m	1.64–1.71, m	1.35, m	1.56, m	1.57, m	1.50, m	—
8 β	—	1.74, m	1.33–1.40, m	1.69, m	1.87, m	1.72, m	1.67, m	—
9 α	1.36, m	1.40, m	1.97, t, 3.3	1.21, m	1.09, m	1.25, m	1.26, m	—
9 β	1.81, m	1.87, m	1.17–1.27, m	1.98, m	1.96, m	1.72, m	1.84, m	—
10	—	—	—	—	—	2.02, m	2.04, m	—
11	—	—	—	—	—	1.13, d, 7.0	—	—
12	—	—	4.59, s	4.59, s	1.41, s	1.58, s	—	1.57, s
13 α	6.23, s	6.27, br s	5.07, d, 0.9	5.10, br s	1.45, s	0.81, s	5.58, s	1.58, s
13 β	5.67, s	5.69, br s	5.02, s	5.00, br s	—	—	6.14, s	—
14	0.76, s	0.88, s	0.71, s	0.71, s	1.31, s	—	1.58, s	1.08, s
15 α	—	—	4.77, d, 1.5	4.50, br s	1.28, s	—	0.85, s	5.03, br s
15 β	—	—	4.50, d, 1.5	4.77, br s	—	—	—	5.17, br s
1'	—	3.76, s	—	—	—	4.30, d, 7.5	4.30, d, 8.0	5.09, d, 8
2'	—	—	2.10, s	2.23, m	1.98, s	3.14, m	3.14, m	3.96, t, 8.5
3'	—	—	—	2.13, m	—	3.31, m	3.30, m	4.20, t, 9
4'	—	—	—	0.97, d, 6.6	—	3.27, m	3.27, m	4.24, t, 9
5'	—	—	—	0.97, d, 6.6	—	3.22, m	3.25, m	3.77, m
6' α	—	—	—	—	—	3.67, dd, 12.0, 6.5	3.65, dd, 12.0, 6.5	4.34, dd, 12, 4
6' β	—	—	—	—	—	3.85, dd, 11.5, 2.0	3.85, dd, 11.5, 2.0	4.37, dd, 12, 2

Note: The ¹H-NMR data of 2'' of compound 97 were 1.98, s.

Table 19. ¹H-NMR data of compounds 101–110.

NO.	101 [69]	102 [69]	103 [70]	104 [70]	105 [39]	106 [39]	107 [71]	108 [40]	109 [27]	110 [40]
	C ₅ D ₅ N	C ₅ D ₅ N	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD
1 α	3.78, dd, 8, 7	3.63, dd, 10, 5	1.40–1.82, m	1.50–2.20, m	1.15, m	1.23, m	3.96, dd, 11.0, 4.8	1.38, m	5.46, dd, 10, 2	1.52, m
1 β	—	—	—	—	1.62, m	1.53, m	—	1.45, m	—	1.44, dd, 13.1, 7.0
2 α	—	—	1.40–1.82, m	1.50–2.20, m	1.40, m	1.26, m	1.91, m	1.55, m	5.86, dd, 10, 3	2.15, m
2 β	—	—	—	—	1.80, m	1.60, m	2.21, m	—	—	2.20, m
3 α	5.43, br s	—	1.40–1.82, m	1.50–2.20, m	1.56, m	2.00, m	2.40, m	1.53, m	6.36, br s	5.81, s
3 β	—	—	—	—	—	2.34, m	2.51, m	3.00, d, 11.7	6.36, br s	5.81, s
4	—	—	2.43, m	—	2.46, m	—	—	—	—	—
5	3.25, br s	2.66, d, 3	—	2.34, m	—	1.81, d, 12.4	2.62, d, 11.0	2.08, d, 11.0	2.55, br d, 11	2.33, d, 10.5
6 α	4.91, t, 3	5.09, t, 3	5.13, d, 8	1.71, m	4.94, d, 3.5	1.06, q, 12.4	—	4.06, t, 11.0	4.22, overlapped	4.25, t, 10.5
6 β	—	—	—	1.24, m	—	1.42, m	4.11, t, 11.0	—	—	—
7	—	—	3.56, m	2.97, m	3.01, dd, 5.4, 3.5	2.38, m	—	1.65, overlapped	2.50, m	1.61, m
8 α	—	—	4.80, m	4.48, m	5.13, dt, 5.4, 3.0	5.04, m	1.59, m	1.79, m	—	1.81, d, 11.7
8 β	—	—	—	—	—	—	2.10, m	1.58, m	—	1.63, m
9 α	—	—	2.09, dd, 6, 6	1.99, m	2.14, dd, 14.9, 3.0	1.46, dd, 15.6, 4.5	1.50, m	1.55, m	—	1.36, d, 11.2
9 β	—	—	1.53, m	1.39, m	1.51, dd, 14.9, 3.0	2.20, dd, 15.6, 2.6	2.06, m	1.35, m	—	1.58, m
11	—	—	—	—	—	—	—	2.43, dq, 10.6, 6.9	—	2.37, m
12	1.49, s	1.52, s	—	—	—	—	—	—	—	—
13 α	1.61, s	1.63, s	6.17, d, 4	6.11, d, 2	2.65, d, 17.5	2.64, d, 17.5	5.41, d, 3.0	1.15, d, 6.9	5.36, d, 3.1	1.16, d, 6.8
13 β	—	—	5.60, d, 4	5.57, d, 2	2.95, d, 17.5	3.02, d, 17.5	6.09, d, 3.0	—	6.15, d, 3.2	—
14	1.20, s	1.33, s	1.17, s	0.82, s	1.22, s	0.78, s	0.86, s	0.88, s	0.92, s	0.97, s
15 α	2.15, br s	1.47, s	1.07, s	4.76, d, 3	1.13, d, 7.7	4.79, s	—	6.16, br s	5.18, br s	4.38, d, 11.7
15 β	—	—	—	4.43, d, 3	—	4.43, s	—	—	5.13, br s	4.15, d, 11.7
17	—	—	—	—	2.33, s	2.34, s	—	—	—	—
1'	5.15, d, 8	5.30, d, 8	—	—	—	—	—	4.48, d, 7.7	5.06, d, 7	4.39, d, 7.7
2'	4.01, t, 8.5	4.01, t, 8.5	—	—	—	—	—	3.28, m	—	3.17, m
3'	4.25, t, 8.5	4.19, t, 9	—	—	—	—	—	3.35, m	—	3.33, m
4'	4.20, t, 9	3.94, t, 9	—	—	—	—	—	3.35, m	—	3.28, m
5'	3.92, m	4.06, dt, 8, 2	—	—	—	—	—	3.28, m	—	3.25, m
6' α	4.31, dd, 12, 5	4.16, dd, 11, 8.5	—	—	—	—	—	3.84, dd, 12.1, 2.3	—	3.86, dd, 11.9, 2.0
6' β	4.42, dd, 12, 2	4.67, dd, 11, 1.5	—	—	—	—	—	3.69, dd, 12.1, 5.0	—	3.67, dd, 11.9, 5.1

Table 20. ¹³C-NMR data of compounds 76–110.

NO.	76 [12]	77 [54]	78 [55]	79 [56]	80 [57]	82 [59]	83 [60]	84 [61]	85 [62]	86 [64]	87 [65]	88 [66]	89 [67]	90 [67]	92 [34]	93 [42]	94 [31]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃
1	89.9	43.3	42.2	79.3	79.7	79.1	212.2	74.4	21.9	22.6	21.3	21.2	26.5	127.5	41	77.3	77.2
2	29.3	20.1	20.1	28.5	25.6	31.9	38.9	42.4	30.9	30.9	22.2	22	27.4	128.6	20.1	31.6	30
3	30.7	44.6	43.5	40.8	39.5	35.1	158.6	197.5	135	134.3	142.4	141.1	138.2	145.1	43.4	40.6	39.2
4	146.4	72.2	72.2	71.6	71.3	146.1	143.7	129.5	122.3	122.6	151.6	152.8	119.6	129.6	72.1	212.6	212.1
5	129.6	54.9	55	52.9	50.4	55.9	53.1	161.9	39.8	37.7	41.4	39.3	38	142	54.9	62.2	61.3
6	138.1	26	27.3	25.7	26.4	67	25	32.8	46.7	46.6	45.6	45.6	38.9	40.4	26.2	67.6	67.5
7	52.7	46.3	41.1	45.7	46.1	49.3	55.8	45.1	22.7	19.8	22.1	26.4	26.6	27.5	42.6	48.4	46
8	35.6	26.8	26.6	26.4	26.8	18.1	26.8	26.5	42.2	40.3	41.8	40	140.8	35	27.1	28.1	26.6
9	36.5	41	44.7	40.5	39.3	36.3	35.1	37.7	72.4	70.7	72.1	70.6	134	198.1	44.6	37.5	36
10	148	34.6	34.7	38.9	38.9	41.7	59.6	41.3	50	47.9	49.6	47.6	36.1	130.5	34.6	45.7	43.8
11	31.9	150.7	154.1	150.3	150.6	26	32.3	148.9	25.9	26.1	26.2	28.6	143.8	144.7	148.8	125.9	141.9
12	20.5	108.1	65.3	108.3	108.6	21.1	19.4	109.4	21.1	21.4	21.4	21.2	124.9	130.7	65.8	170.5	167.6
13	20.7	22.7	107.9	21	20.7	16.2	21.9	20.6	15.1	15.2	15.2	15.2	170.5	171.3	110.7	144.1	125
14	114.6	18.6	18.7	13	12.5	11.6	19.6	16.3	20.8	28.4	20.5	19.9	171.5	21.9	18.7	12.2	11.9
15	113.2	21	22.7	22.7	29.7	107.8	192.7	11	23.8	23.7	194.5	194.6	23.9	—	22.7	—	—
1'	—	—	—	—	—	—	—	—	—	—	—	—	—	—	172.8	—	51.9
2'	—	—	—	—	—	—	—	—	—	—	—	—	—	—	43.5	—	—
3'	—	—	—	—	—	—	—	—	—	—	—	—	—	—	25.7	—	—
4'	—	—	—	—	—	—	—	—	—	—	—	—	—	—	22.4	—	—
5'	—	—	—	—	—	—	—	—	—	—	—	—	—	—	22.4	—	—
NO.	95 [7]	96 [8]	97 [40]	98 [42]	99 [42]	100 [69]	101 [69]	102 [69]	103 [70]	104 [70]	105 [39]	106 [39]	107 [71]	108 [40]	109 [27]	110 [40]	
	CDCl ₃	CDCl ₃	CD ₃ OD	CD ₃ OD	CD ₃ OD	C ₅ D ₅ N	C ₅ D ₅ N	C ₅ D ₅ N	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CD ₃ OD	C ₅ D ₅ N	CD ₃ OD	
1	79.4	79.4	81.2	136.3	136.2	79.4	76.6	79	41.7	42.5	42.3	42.2	76.4	43.2	127.1	38.7	
2	31.6	31.6	27.6	120.6	120.6	33.3	34.3	29.9	16.7	22.7	16.9	22.8	30.5	22.7	138.3	23.9	
3	34.3	34.3	42.3	29	29.9	33.8	122	42.5	32.7	36.8	32.9	36.9	38.6	26.7	76.2	128.2	
4	148.6	148.8	72.1	82.2	82.2	148.5	136.3	72.3	37.5	148.9	38.7	149.4	206.3	118.1	141.1	134.4	
5	47.7	47.7	54.6	37.9	37.9	53.2	53.6	59.4	149	46.2	152.8	46.7	58.4	53	49	49.9	
6	27	27	70	36.3	36.5	78	79.9	80.9	118.8	27.5	113.7	21.2	76.6	81.1	77.3	82.8	
7	41.5	41.6	51.1	42.5	41.3	45.4	44.8	45.5	39.4	40.5	47.1	47	48.9	54.1	51.7	55.1	
8	29.3	29.3	20.1	25.3	28.1	19.8	17.8	18.1	76.4	76.6	77.2	77.7	20.9	24.1	20.4	23.9	
9	37	37.1	40.7	29.8	30.2	36.4	30.8	34.6	42.6	41.3	42.7	41.4	36.1	41	39.5	40.7	
10	40.4	40.4	41	48.3	37.9	41.2	38.8	40.4	32.6	34.3	33.1	34.7	45.5	39.4	35.7	36.9	
11	148.7	148.6	85.9	14.7	147.9	72.9	72.4	72.1	139.8	142.2	79.2	79.7	138.3	42.2	139.1	41.9	
12	66.4	66.1	24.6	21	170.9	29.6	30.2	30.4	170.3	170.5	175.5	175.5	170.2	182.1	169.4	182.4	
13	111.3	111.2	25.6	10.7	123	31.3	30.7	30.7	121.5	119.9	43.6	42.2	117.2	12.6	115.1	12.6	
14	10.4	10.4	14.3	—	21	15.7	12.6	16.5	22.5	17.6	28.8	18	12.3	18.3	19.6	17.7	
15	107.2	107.2	29.8	—	10.9	107.2	23.1	23.8	28.5	106.6	23.2	106.5	—	139.1	107.5	73.4	
16	—	—	—	—	—	—	—	—	—	—	210.4	210.5	—	—	—	—	
17	—	—	—	—	—	—	—	—	—	—	32	32	—	—	—	—	
1'	171	173	172.5	101.5	101.5	104.4	105.6	105.6	—	—	—	—	—	104.5	104.2	103.8	
2'	21.2	43.7	22.2	75.2	75.2	75.2	74.1	75.2	—	—	—	—	—	74.9	74.4	75.3	
3'	—	25.9	—	78.2	78.2	78.2	78.7	78.8	—	—	—	—	—	78.2	77.5	78.3	
4'	—	22.6	—	71.9	71.9	71.5	71.8	72.4	—	—	—	—	—	71.2	70.8	71.7	
5'	—	22.6	—	77.8	77.8	78.3	78.3	78.5	—	—	—	—	—	78	77.5	77.8	
6'	—	—	—	63	63	62.6	62.8	63	—	—	—	—	—	62.4	61.8	62.8	

Note: The 1'' and 2'' data of compound 97 were 172.7 and 22.5.

5. Polymer Sesquiterpene Lactones

In *Ainsliaea* plants, polymer sesquiterpene lactones are typically formed through the polymerization of two or three sesquiterpene units; most monomeric precursors belong to guaiane sesquiterpenes. The structures of the 25 reported polymer sesquiterpene lactones can be found in Figure 4 and Table 21.

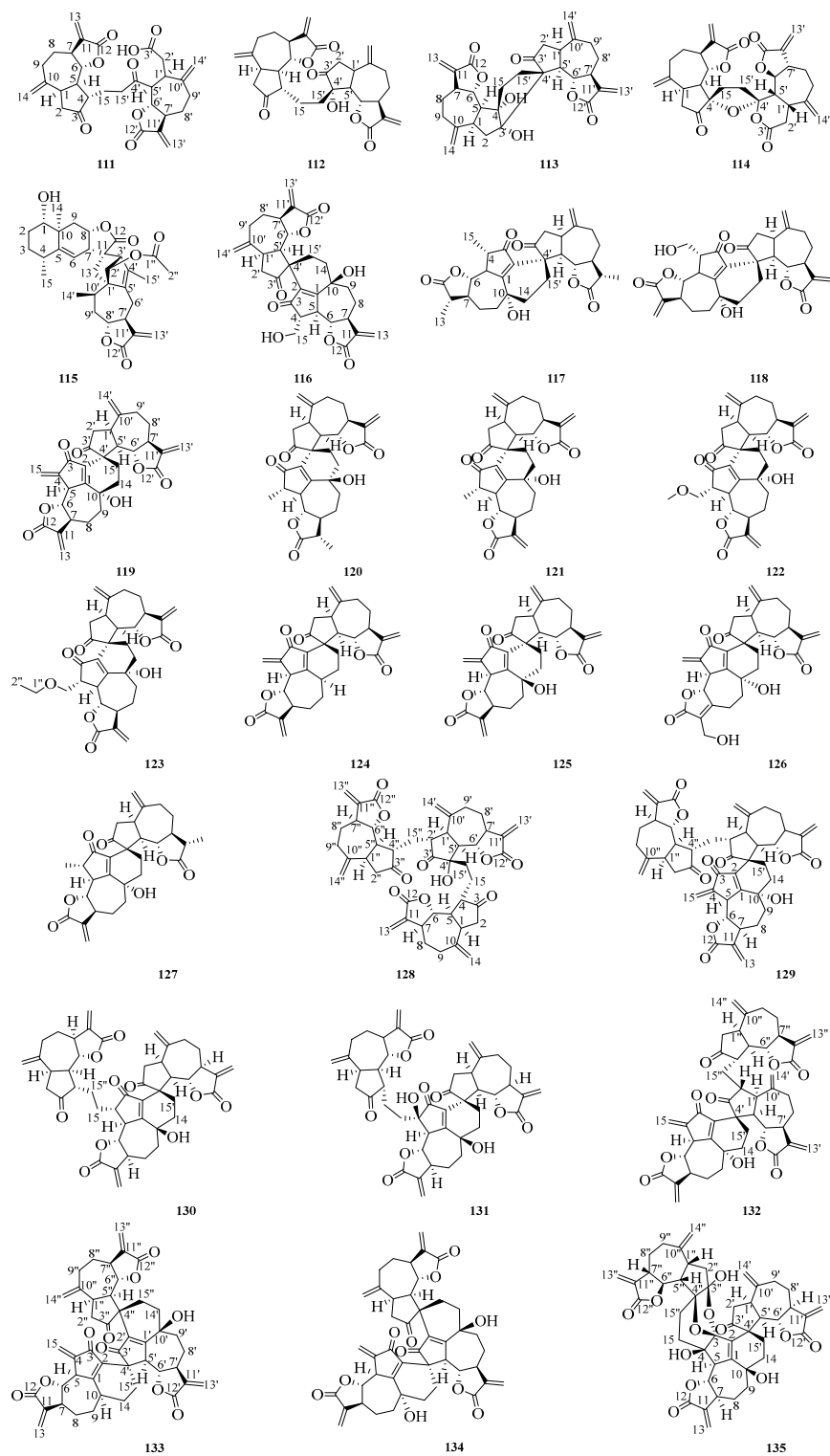


Figure 4. Chemical structures for compounds 111–135.

Table 21. The compound name, molecular formula, and NMR test reagent of polymer sesquiterpene lactones.

No.	Compound Name	Molecular Formula	Solvent	Ref.
111	Macrocephadiolide B	C ₃₀ H ₃₄ O ₈	CD ₃ OD	[72]
112	Ainsliadimer J	C ₃₀ H ₃₄ O ₇	CDCl ₃	[72]
113	Ainsliadimer A	C ₃₀ H ₃₄ O ₇	CDCl ₃	[73]
114	Macrocephadiolide A	C ₃₀ H ₃₂ O ₈	CDCl ₃	[72]
115	Japonicone A	C ₃₂ H ₄₀ O ₇	CDCl ₃	[74]
116	Ainsliadimer B	C ₃₀ H ₃₂ O ₈	CDCl ₃	[75]
117	Ainsliadimer C	C ₃₀ H ₃₆ O ₇	CDCl ₃	[31]
118	Ainsliadimer D	C ₃₀ H ₃₆ O ₈	DMSO	[31]
119	Gochnatolide A	C ₃₀ H ₃₀ O ₇	CDCl ₃	[76,77]
120	Ainsliadimer F	C ₃₁ H ₃₆ O ₆	CDCl ₃	[47]
121	Ainsliadimer I	C ₃₁ H ₃₄ O ₆	CDCl ₃	[47]
122	Ainsliadimer G	C ₃₂ H ₃₆ O ₇	CDCl ₃	[47]
123	Ainsliadimer H	C ₃₃ H ₃₈ O ₇	CDCl ₃	[47]
124	Gochnatolide C	C ₃₀ H ₃₀ O ₆	CDCl ₃	[76]
125	Gochnatolide B	C ₃₀ H ₃₀ O ₇	CDCl ₃	[76]
126	Gochnatolide E	C ₃₀ H ₃₀ O ₈	CDCl ₃	[78]
127	Gochnatolide F	C ₃₀ H ₃₄ O ₇	CDCl ₃	[78]
128	Macrocephatriolide B	C ₄₅ H ₅₀ O ₁₀	CDCl ₃	[79]
129	Macrocephatriolide A	C ₄₅ H ₄₆ O ₁₀	CDCl ₃	[79]
130	Ainsliatriolides A	C ₄₅ H ₄₈ O ₁₀	CDCl ₃	[80]
131	Ainsliatriolide C	C ₄₅ H ₄₈ O ₁₁	CDCl ₃	[81]
132	Ainsfragolide	C ₄₅ H ₄₆ O ₁₀	CDCl ₃	[78]
133	Ainsliatrimers A	C ₄₅ H ₄₄ O ₁₀	CDCl ₃	[75]
134	Ainsliatrimers B	C ₄₅ H ₄₄ O ₁₀	CDCl ₃	[75]
135	Ainsliatriolides B	C ₄₆ H ₅₀ O ₁₁	CDCl ₃	[80]

5.1. NMR Data of Polymer Sesquiterpene Lactones (111–135)

The ¹H and ¹³C NMR spectroscopy are summarized in Tables 22–26. This paper also provides an overview of the nuclear magnetic resonance testing instruments used for compounds 111–135. The ¹H and ¹³C spectra of compounds 111, 112, 114, 130, and 131 were measured using the Bruker Avance III-500 instrument. The NMR data for compounds 113, 117, 118, 120, 121, 122, and 123 were recorded on the Bruker Avance 600 spectrometer. For compounds 115 and 116, the respective ¹H and ¹³C spectra were obtained by the Bruker Avance 400 instrument. Compound 119's NMR data were acquired on the Bruker AC 200 spectrometer. NMR data for compounds 119, 124, and 125 were acquired using the Bruker AV500 instrument. The ¹H and ¹³C data of compounds 126, 127, and 132 were taken with the Bruker Avance AV500 spectrometer. Data for compounds 128, 129, and 135 were performed on the Bruker Avance III-600 instrument. NMR data collection for compounds 133 and 134 was conducted using the Bruker Avance 400 instrument.

5.2. Bioactivity of Polymer Sesquiterpene Lactones

5.2.1. Antitumor and Cytotoxic

Polymer sesquiterpene lactones inhibit the activity of tumor cell lines or cancer cell lines. The dimeric sesquiterpene lactone japonicone A (115) showed significant inhibitory activity against the three tested human tumor cell lines 95D, MDA-MB-231, and HepG2, with IC₅₀ values of 9.10, 3.82, and 1.43 μM, respectively [8]. Ainsliatriolide C (131), ainsliadimer B (116), and ainsliatrimers B (134) were isolated from *Ainsliaea yunnanensis* and showed very significant selective cytotoxic activities on MDA-MB-468, PANC-1, HEPG2, and A549 cells, and IC₅₀ values from 5.1 μM to 34.4 μM [81]. With DOX (doxorubicin) as the positive control, the antitumor activity of the isolated compounds against A549, LOVO, CEM, and MDA-MB-M-435 (MDA) was detected by an MTT assay. Both Ainsliatrimers A (133) and ainsliatrimers B (134) showed potent cytotoxicities against the LOVO and CEM cell lines [75]. Ainsliatriolide A (130) and ainsliatriolide B (135) exhibited stronger cytotoxicity on A-549, HT-29, BEL-7402, and HL-60 cancer cell lines, especially ainsliatriolide A (130) which displayed potent cytotoxicity with an averaged IC₅₀ value of 1.17 μM against four cancer cell lines [80].

Table 22. ¹H-NMR data of compounds 111–118.

NO.	111 [72]	112 [72]	113 [73]	114 [72]	115 [74]	116 [75]	117 [31]	118 [31]
	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	DMSO
1	3.24, dd, 8.0, 2.3	3.09, ddt, 20.6, 7.7, 4.2	3.15, dd, 12.0, 11.4	3.11, dd, 17.0, 7.0	3.28, dd, 11.7, 3.8	—	—	—
2 α	2.63, m	2.64–2.54, m	2.38, dd, 13.8, 12.0	2.68, m	1.82, m	—	—	—
2 β	2.43, m	2.29, tdt, 10.8, 5.0, 3.0	1.88–1.92	—	1.62, m	—	—	—
3 α	—	—	—	—	1.65, m	—	—	—
3 β	—	—	—	—	1.57, m	—	—	—
4	2.40, m	2.29, tdt, 10.8, 5.0, 3.0	—	—	2.45, m	2.68, m	2.50, m	2.37, m
5	2.54, q, 9.4	2.43–2.35, m	2.55, t, 11.4	2.53, t, 10.0	—	3.33, dd, 9.8, 4.3	2.78, dd, 11.4, 4.2	3.37, dd, 11.4, 4.8
6	4.08, t, 9.2	3.95, t, 9.2	4.07, dd, 11.4, 9.0	4.09, dd, 10.0, 8.8	5.37, d, 3.0	4.35, t, 9.8	4.29, t, 10.8	4.39, t, 10.8
7	3.16, m	2.92–2.81, m	2.72, m	2.95, tt, 8.8, 3.0	2.79, dd, 5.5, 3.2	2.80, m	1.82, overlap	2.84, m
8 α	2.38, m	2.12–1.91, m	2.22–2.24, m	2.34, m	—	2.05, m	2.00, overlap	2.04, m
8 β	1.49, m	1.55–1.41, m	1.37, dq, 12.6, 6.0	1.49, m	4.89, dd, 5.3, 2.7	2.10, m	1.82, overlap	1.90, m
9 α	2.63, m	2.64–2.54, m	2.67, m	2.71, m	2.59, dd, 14.8, 3.3	1.88, m	2.00, overlap	1.68, m
9 β	2.29, td, 12.5, 5.7	1.76, ddd, 14.0, 11.6, 5.0	1.88–1.92	2.16, td, 12.8, 5.0	1.53, dd, 15.1, 2.0	2.05, m	1.72, m	1.81, overlap
11	—	—	—	—	—	—	2.36, m	—
13 α	6.20, d, 3.2	6.26, dd, 5.1, 3.4	6.19, d, 3.0	6.26, d, 3.2	2.00, m	5.53, d, 3.0	1.27, d, 7.2	6.01, d, 3.0
13 β	5.68, d, 3.2	5.55, dd, 5.3, 3.1	5.46, d, 3.0	5.57, d, 3.2	1.88, m	6.21, d, 3.0	—	5.62, d, 3.0
14 α	5.00, s	5.05, d, 1.1	5.15, s	5.20, s	1.19, s	1.95, m	1.82, overlap	1.81, overlap
14 β	4.69, s	4.69, s	5.03, s	4.84, s	—	1.70, m	1.69, m	1.54, m
15 α	2.09, m	2.12–1.91, m	2.25–2.30	2.16, m	1.11, d, 7.6	3.94, dd, 11.0, 4.0	1.28, d, 7.2	3.88, m
15 β	1.90, m	1.65–1.56, m	2.16–2.21	—	—	—	—	3.62, m
1'	3.22, m	2.96, ddq, 12.0, 9.3, 3.2	3.04–3.06	2.97, q, 9.6	—	3.22, m	3.18, t, 9.0	3.27, t, 9.0
2' α	2.61, dd, 16.2, 5.5	2.50–2.46, m	2.91, dd, 16.8, 9.6	3.25, dd, 15.6, 10.2	4.60, s	3.23, m	3.22, m	3.27, overlap
2' β	2.46, dd, 16.2, 9.4	2.29, tdt, 10.8, 5.0, 3.0	2.45, d, 16.8	2.68, m	—	2.66, m	2.62, d, 18.6	2.49, overlap
3'	—	—	—	—	2.86, d, 1.4	—	—	—
5'	3.26, m	3.09, ddt, 20.6, 7.7, 4.2	3.04–3.06	2.53, t, 10.1	—	3.20, m	3.12, t, 9.6	2.89, t, 9.6
6' α	—	—	—	—	3.03, d, 15.5	—	—	—
6' β	4.39, t, 9.5	4.27, dd, 11.0, 8.8	4.12, dd, 10.2, 8.4	4.82, dd, 10.1, 9.0	2.08, m	4.12, t, 10.5	4.14, t, 9.6	4.25, t, 9.6
7'	3.07, m	2.92–2.81, m	3.04–3.06	2.89, ddd, 11.9, 9.0, 3.1	2.82, s	3.01, m	2.07, m	3.18, m
8' α	2.30, m	2.12–1.91, m	2.25–2.30	1.56, m	—	1.49, m	2.19, overlap	2.27, m
8' β	1.53, m	1.55–1.41, m	1.50, dq, 12.6, 3.6	2.28, m	4.21, ddd, 12.4, 8.4, 3.2	2.30, m	1.40, m	1.45, m
9' α	2.55, m	2.50–2.46, m	2.16–2.21	2.06, m	2.36, dt, 13.1, 4.1	2.21, m	2.58, m	2.49, br s
9' β	2.32, m	2.19, td, 12.5, 5.7	2.57, m	2.68, m	2.00, m	2.62, m	2.12, overlap	2.16, m
10'	—	—	—	—	2.15, m	—	—	—
11'	—	—	—	—	—	—	2.19, overlap	—
13' α	6.18, d, 3.2	6.26, dd, 5.1, 3.4	6.31, d, 3.0	6.33, d, 3.2	6.22, d, 3.3	5.57, d, 3.0	1.24, d, 6.6	6.04, d, 3.0
13' β	5.67, d, 3.2	5.55, dd, 5.3, 3.1	5.61, d, 3.0	5.57, d, 3.2	5.54, d, 3.1	6.26, d, 3.0	—	5.68, d, 3.0
14' α	5.05, s	5.14, s	4.96, s	5.19, s	1.05, d, 7.3	5.09, s	5.06, s	5.83, s
14' β	4.99, s	4.99, s	4.59, s	5.10, s	—	4.73, s	4.70, s	5.03, s
15' α	2.97, ddd, 18.3, 9.0, 5.3	2.72, ddd, 13.2, 4.8, 3.0	2.05, dd, 14.4, 5.4	2.80, m	1.67, d, 1.5	2.15, m	2.12, overlap	2.20, m
15' β	2.74, ddd, 18.3, 9.0, 6.2	2.64–2.54, m	2.01, dd, 14.4, 7.8	2.25, m	—	2.06, m	2.05, m	1.85, m

Note: The ¹H-NMR data of 2'' of 115 were 2.08, s.

Table 23. ¹H-NMR data of compounds 119–127.

NO.	119 [76]	120 [47]	121 [47]	122 [47]	123 [47]	124 [76]	125 [76]	126 [78]	127 [78]
	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
4	—	2.52, dd, 7.2, 4.2	2.56, dd, 7.1, 4.2	2.63, m	2.62, m	—	—	—	2.46, m
5	3.92, d, 10.4	2.80, dd, 11.1, 4.1	2.91, dd, 11.2, 4.2	3.52, dd, 12.3, 5.3	3.52, dd, 11.3, 4.1	3.55, d, 10.4	3.89, d, 10.8	3.62, d, 10.0	3.03, dd, 1.5, 10.2
6	3.76, t, 9.9	4.32, dd, 11.0, 9.6	4.33, dd, 11.1, 9.7	4.38, dd, 11.2, 9.8	4.36, dd, 11.3, 9.7	3.92, d, 9.9	4.36, t, 10.7	4.59, d, 10.0	3.75
7	3.88, m	1.84, m	2.75, mH	2.87, m	2.86, m	3.23–3.29, m	2.86, m	—	3.75
8 α	2.34–2.44, m	2.05, m	2.10, m	2.16, m	2.11, m	2.32–2.44, m	2.04–2.11, m	3.41	2.39, m
8 β	1.60–1.67, m	1.85, m	2.02, m	2.08, m	2.03, m	1.68–1.75, m	2.04–2.11, m	2.66	2
9 α	2.06–2.16, m	1.97, m	1.71, m	2.00, m	1.95, m	2.00–2.07, m	2.04–2.11, m	2.22	2.07, m
9 β	2.06–2.16, m	1.70, m	1.68, m	1.73, m	1.73, m	1.61–1.68, m	1.84–2.01, m	2.09	2.03
10	—	—	—	—	—	2.74, m	—	—	—
11	—	2.35, m	—	—	—	—	—	—	—
13 α	6.21, d, 3.7	1.27, d, 6.9	6.20, d, 3.2	6.23, d, 3.3	6.21, d, 3.3	6.28, d, 3.4	6.23, d, 3.4	4.43, dd, 13.3, 19.5	6.18, d, 3.2
13 β	5.49, d, 3.3	—	5.52, d, 3.1	5.56, d, 3.0	5.54, d, 3.1	5.57, d, 3.1	5.56, d, 3.1	—	5.45, d, 3.2
14 α	1.87–2.00, m	1.86, m	1.86, m	2.12, m	2.09, m	2.00–2.07, m	1.84–2.01, m	1.95	1.87
14 β	1.87–2.00, m	1.69, m	1.69, m	1.94, m	1.93, m	1.84–1.97, m	1.74, m	—	—
15 α	6.22, br s	1.27, d, 7.1	1.30, d, 7.2	3.94, dd, 9.5, 2.2	3.96, dd, 9.6, 2.3	6.18, br s	6.24, br s	6.26, s	1.23, d, 7.6
15 β	6.03, br s	—	—	3.74, dt, 9.5, 3.2	3.76, dt, 9.7, 3.1	6.00, br s	6.16, br s	6.05, s	—
1'	3.25, t, 9.6	3.22, m	3.22, t, 9.0	3.22, m	3.24, t, 9.3	3.23–3.29, m	3.27, m	3.27	3.17, t, 8.9
2' α	3.37, t, 10.0	3.22, m	3.23, m	3.22, m	2.68, m	3.33–3.40, m	3.38, m	3.32	3.23, m
2' β	2.63, m	2.63, m	2.64, m	2.63, m	2.62, m	2.62–2.66, m	2.70, m	2.68	2.61
5'	3.34, t, 10.5	3.20, d, 4.9	3.19, t, 8.7	3.25, t, 10.2	3.17, t, 9.3	3.33–3.40, m	3.28, m	3.39	3.11, t, 9.4
6'	4.24, t, 9.2	4.16, t, 9.4	4.17, t, 9.2	4.19, t, 9.2	4.18, t, 9.8	4.21, t, 9.5	4.21, m	4.26, t, 9.8	4.20, t, 9.4
7'	3.08, m	3.00, m	3.01, m	3.05, m	3.03, m	3.06, m	3.03, m	3.08, m	2.15
8' α	2.34–2.44, m	2.31, m	2.35, m	2.35, m	2.33, m	2.32–2.44, m	2.34, m	2.35, m	2.20, m
8' β	1.49–1.57, m	1.47, m	1.48, m	1.51, m	1.51, m	1.51, m	1.50, m	1.54, m	1.42, m
9' α	2.66, br s	2.64, m	2.65, m	2.65, m	2.64, m	2.62–2.66, m	2.66, s	2.66	2.57
9' β	2.34–2.44, m	2.20, m	2.22, m	2.24, m	2.23, m	2.26, m	2.25, m	2.26	2.13
11'	—	—	—	—	—	—	—	—	2.25, m
13' α	6.30, d, 3.4	6.26, d, 3.4	6.27, d, 3.4	6.29, d, 3.4	6.27, d, 3.4	6.29, d, 3.4	6.28, d, 3.4	6.29, d, 3.0	1.26, d, 7.0
13' β	5.64, d, 3.1	5.58, d, 3.0	5.58, d, 3.0	5.60, d, 3.0	5.59, d, 3.0	5.60, d, 3.0	5.60, d, 3.1	5.65, d, 3.0	—
14' α	5.10, s	5.09, br s	5.09, br s	5.12, br s	5.11, br s	5.08, s	5.12, s	5.11, s	5.05, s
14' β	4.71, s	4.72, br s	4.72, br s	4.75, br s	4.74, br s	4.73, s	4.75, s	4.75, s	4.67, s
15' α	2.06–2.16, m	2.13, m	2.11, m	2.12, m	2.12, m	2.00–2.07, m	2.04–2.11, m	2.15	2.1
15' β	1.87–2.00, m	2.02, m	1.97, m	2.07, m	2.07, m	1.84–1.97, m	2.04–2.11, m	1.92	1.85
1''	—	—	—	3.29, s	3.42, dd, 7.0, 3.5	—	—	—	—
2''	—	—	—	—	1.06, t, 7.0	—	—	—	—

Table 24. ¹H-NMR data of compounds 128–135.

NO.	128 [79] CDCl ₃	129 [79] CDCl ₃	130 [80] CDCl ₃	131 [81] CDCl ₃	132 [78] CDCl ₃	133 [75] CDCl ₃	134 [75] CDCl ₃	135 [80] CDCl ₃	NO.	128 [79] CDCl ₃	129 [79] CDCl ₃	130 [80] CDCl ₃	131 [81] CDCl ₃
1	3.06, m	—	—	—	—	—	—	—	1'	2.72, m	3.07, dd, 10.6, 5.9	3.28, m	3.28, m
2	2.51–2.58, m	—	—	—	—	—	—	—	2'α	3.43, q, 7.3	3.36, dt, 8.5, 5.9	3.27, m	3.26, m
4	2.22, m	—	2.66, m	—	—	—	—	—	2'β	—	—	2.62, m	2.63, m
5	2.45, m	3.91, d, 10.3	3.23, dd, 11.0, 3.7	3.40, m	3.94, d, 9.0	3.43, d, 10.3	3.87, d, 10.3	3.07, m	5'	2.47, t, 11.1	3.43, m	3.34, m	3.26, m
6	3.92, t, 9.2	3.76, dd, 10.3, 10.3	4.21, dd, 11.0, 9.8	4.47, t, 10.0	3.80, d, 9.0	3.78, t, 10.3	3.64, t, 10.3	4.48, t, 10.5	6'	4.32, dd, 11.1, 8.7	4.25, dd, 11.0, 8.6	4.12, t, 9.4	4.12, t, 10.0
7	2.97, m	3.85, m	3.00, m	3.02, m	3.89, m	3.21, m	3.78, m	2.66, m	7'	2.79, m	2.99, m	3.12, m	3.12, m
8α	2.20, m	2.36, m	2.06, m	2.07, m	2.4	1.82, m	1.80, m	1.91, m	8'α	2.24, m	2.30, m	2.32, m	2.28, m
8β	1.44, m	1.56, m	2.02, m	1.98, m	1.61	2.35, m	2.34, m	1.95, m	8'β	1.50, m	1.46, m	1.48, m	1.48, m
9α	2.56, m	2.05, m	2.05, m	2.03, m	2.08, m	2.00, m	2.05, m	1.67, m	9'α	2.64, m	2.69, m	2.61, m	2.61, m
9β	2.18, m	1.92, m	2.01, m	1.75, m	1.95, m	2.38, m	2.49, m	1.92, m	9'β	1.97, td, 12.8, 4.5	2.18, m	2.27, m	2.21, m
10	—	—	—	—	—	2.77, m	—	—	13'α	6.23, d, 3.3	6.22, d, 3.2	6.15, d, 3.2	6.17, d, 3.4
13α	6.15, d, 3.2	6.16, d, 3.4	6.15, d, 3.2	6.11, d, 3.2	6.21, d, 3.2	5.57, d, 3.0	5.49, d, 3.4	5.43, d, 3.3	13'β	5.52, d, 3.3	5.58, d, 3.2	5.50, d, 3.2	5.52, d, 3.4
13β	5.49, d, 3.2	5.45, d, 3.4	5.50, d, 3.2	5.47, d, 3.2	5.48, d, 3.2	6.25, d, 3.0	6.19, d, 3.4	6.10, d, 3.3	14'α	5.37, br s	5.18, br s	5.08, br s	5.08, s
14α	4.96, br s	1.98, m	1.84, m	1.84, m	2	2.55, m	2.65, m	1.78, td, 13.9, 3.7	14'β	5.13, br s	5.10, br s	4.71, br s	4.74, s
14β	4.66, br s	1.88, m	1.70, m	1.73, m	1.93, m	1.64, m	1.93, m	1.56, m	15'α	2.04, m	2.28, m	2.18, m	2.13, m
15α	1.98, m	6.17, br s	1.91, m	2.15, m	6.22, br s	5.97, s	5.99, s	2.51, m	15'β	1.85, td, 12.8, 4.7	1.65, td, 14.3, 3.7	2.05, m	2.01, m
15β	1.43, m	5.97, br s	1.84, m	1.86, m	6.02, br s	6.26, s	6.30, s	2.13, m					
NO.	132 [78] CDCl ₃	133 [75] CDCl ₃	134 [75] CDCl ₃	135 [80] CDCl ₃	NO.	128 [79] CDCl ₃	129 [79] CDCl ₃	130 [80] CDCl ₃	131 [81] CDCl ₃	132 [78] CDCl ₃	133 [75] CDCl ₃	134 [75] CDCl ₃	135 [80] CDCl ₃
1'	3.10, m	—	—	3.06, m	1''	3.10, m	3.11, td, 8.7, 4.5	3.06, m	3.09, m	3.14, m	3.14, m	3.13, m	—
2'α	—	—	—	3.07, m	2''α	2.45–2.55, m	2.56, m	2.48, m	2.47, m	2.6	3.11, m	3.07, m	—
2'β	3.39, m	—	—	2.71, m	2''β	—	2.43, m	2.46, m	2.29, m	2.48	2.58, m	2.60, m	—
5'	3.46, t, 10.7	3.19, d, 10.0	3.17, d, 9.6	3.24, t, 9.3	4''	—	2.62, m	2.52, m	2.29, m	2.55	—	—	—
6'	4.29, dd, 10.7, 8.7	4.36, t, 10.0	4.35, t, 9.6	4.23, t, 9.3	5''	—	2.55, m	2.40, m	2.52, m	2.44	2.95, m	2.94, t, 9.6	2.37, m
7'	3.04	2.65, m	2.65, m	2.89, m	6''	3.96, t, 9.1	3.98, dd, 9.3, 9.3	3.94, t, 9.1	3.89, t, 10.0	4.02, t, 9.2	4.22, t, 9.5	4.22, t, 9.6	4.19, dd, 11.3, 8.8
8'α	2.32	1.99, m	1.99, m	1.50, m	7''	3.04	2.96, m	2.96, m	3.03, m	2.99	2.99, m	3.00, m	2.77, m
8'β	1.5	2.15, m	2.08, m	2.31, m	8''α	2.19, m	2.29, m	2.27, m	2.16, m	2.33	1.50, m	1.52, m	1.56, m
9'α	2.73, m	1.80, m	1.78, m	2.16, m	8''β	1.47, m	1.46, m	1.41, m	1.39, m	1.5	2.34, m	2.31, m	2.18, m
9'β	2.22	1.94, m	2.10, m	2.66, m	9''α	2.56, m	2.55, m	2.56, m	2.57, m	2.59	2.20, m	2.31, m	2.27, m
13'α	6.27, d, 3.2	5.48, d, 3.0	5.48, d, 3.2	5.59, d, 3.2	9''β	2.18, m	2.20, m	2.18, m	2.19, m	2.22	2.61, m	2.63, m	2.35, m
13'β	5.62, d, 3.2	6.10, d, 3.0	6.11, d, 3.2	6.28, d, 3.2	13''α	6.12, d, 3.2	6.23, d, 3.2	6.19, d, 3.2	6.17, d, 3.2	6.28, d, 3.0	6.27, d, 3.0	6.28, d, 3.2	6.16, d, 3.4
14'α	5.22, s	2.10, m	2.09, m	5.13, br s	13''β	5.47, d, 3.2	5.54, d, 3.2	5.50, d, 3.2	5.52, d, 3.2	5.57, d, 3.0	5.59, d, 3.0	5.59, d, 3.2	5.45, d, 3.4
14'β	5.14, s	1.80, m	1.79, m	4.79, br s	14''α	4.99, br s	4.98, br s	4.96, br s	4.94, s	5.02, s	5.09, s	5.10, s	4.93, br s
15'α	2.32	2.17, m	2.17, m	1.90, m	14''β	4.65, br s	4.73, br s	4.63, br s	4.59, s	4.77, s	4.76, s	4.77, s	4.89, br s
15'β	1.67	1.85, m	1.99, m	2.08, m	15''α	2.29, m	2.29, m	1.76, m	1.68, m	2.32	2.30, m	2.30, m	2.18, m
					15''β	1.67, ddd, 13.9, 7.5, 5.8	1.96, m	1.59, m	1.48, m	2.03	2.06, m	2.30, m	1.99, m

Table 25. ¹³C-NMR data of compounds 111–127.

NO.	111 [72]	112 [72]	113 [73]	114 [72]	115 [74]	116 [75]	117 [31]	118 [31]	119 [77]	120 [47]	121 [47]	122 [47]	123 [47]	124 [76]	125 [76]	126 [78]	127 [78]
	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	DMSO	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1	41	38.3	41.8	36.6	80.7	173.6	171.8	172.7	170.6	172	171.6	173.2	173.1	171	169	169.6	174
2	45.3	45.9	38.1	39.8	26	140.7	140.3	140.5	143.2	140.2	140.4	140.6	140.8	142.1	142.7	142.9	140.6
3	221	218.5	90.5	213.9	29.7	207.3	208.5	206.1	194.1	208.5	208.4	206.3	206.5	193.8	193.6	193.8	210
4	51.6	52.1	89.1	90.8	38.2	53.6	46.9	53.6	142	47	47	52	51.8	141.8	141.1	142.1	45
5	49.1	48.8	53.1	49.1	149.5	49	54.9	46.9	47.3	54.8	55.1	48.3	48.5	51	49.7	46.5	52.6
6	90.1	87.9	82.9	81.7	118.1	82.3	82.6	82.2	83.6	82.6	82.6	82.6	82.6	82.7	81.2	84	85
7	45.1	44.4	49	44.4	42.2	51.6	54.7	50.8	43.4	54.9	51.6	51.6	51.7	43.7	52.4	166	43.3
8	32.7	31.7	31.3	31.9	75.3	21	22.5	20.7	35.3	22.5	21	21	21	25.3	20.8	21.7	23
9	39	37.9	37.8	39.2	39.8	36	36.3	36.7	38.4	36.2	36.1	36.2	36.2	27.7	35.9	35.2	35
10	150.9	148.6	147.5	148	38.5	68.3	68.2	67.3	71.1	68.1	68.3	68.4	68.4	33.3	68.2	69.3	71.2
11	141.1	139	139.5	138.4	56.8	139.3	41.9	140	140.3	41.9	139.4	139.5	139.6	139.3	138.8	126	140.6
12	171.9	169.7	170	169.1	178.8	169.7	178.5	169.6	169.9	178.6	170	170.1	170.1	168.4	169.8	172.8	170
13	121.6	121.4	119.7	121.8	36.4	119.3	12.6	118.4	119.6	12.6	119.1	119.1	119.1	120.7	119.2	54.6	119.5
14	113.4	113.8	113.8	114.4	21.6	36.2	36.3	36	28.3	36.3	36.3	36	36	31.2	36.3	37.3	38.6
15	23.6	22.8	33.1	28.6	23	60.6	14.3	57.5	122.5	14.3	14.2	69.2	67.2	120.9	122.4	122.3	16.5
1'	41.1	39.6	50.1	36.1	62.3	40	40	38.7	39.9	40	40	40	40	40	40.1	39.8	39.7
2'	36.5	44.4	46.9	34.9	81.9	44.7	44.8	39.3	44.9	44.7	44.7	44.8	44.8	45.1	44.7	44.7	44.7
3'	175.1	215.3	224.9	172.4	56.2	222	222.1	220.4	219.7	222.3	222.1	222.5	222.4	220.4	222.1	219.4	219.8
4'	212.5	80.1	62.2	113.2	134.2	51	50.9	50.1	51	50.9	50.9	50.9	50.9	51	51.1	51.1	50.7
5'	57.4	51.4	38.9	47.3	136.4	49.6	50	50.8	49.1	49.6	49.5	49.6	49.7	49.4	49.6	48.8	49.7
6'	86	82.2	84	81.6	26	83.8	83.6	83.6	84.4	83.9	83.9	83.9	83.8	84.1	83.9	84.3	84
7'	44.4	40.1	43.7	45.3	45.3	43.5	47.8	42.5	43.5	43.5	43.5	43.4	43.5	43.5	43.5	43.6	47.8
8'	31.1	31.5	32.8	31.9	82.5	32	33	31.1	31.9	31.9	31.9	32	32	32	32	31.9	32.9
9'	37.9	36.5	38.9	40.1	36.1	39.5	39.8	39.2	39.4	39.5	39.5	39.5	39.5	39.5	39.4	39.5	39.8
10'	148.2	147.2	150	145.8	29.8	150.1	150.6	151.2	150.1	150.1	150.1	150.2	150.2	150.6	150.1	150.1	150.4
11'	140.8	138.7	138.6	138.8	139.4	138.5	41.8	139.3	138.1	138.5	138.4	138.6	138.5	138.6	138.4	138.1	41.8
12'	171.3	169.6	169	169.8	170	169.3	177.5	169.2	170.4	169.3	169.3	169.4	169.2	169.8	169.5	170.7	179
13'	121.4	121.4	122	120.8	119.5	121.7	13.4	120.6	121.8	121.6	121.7	121.5	121.5	121.6	121.8	122.7	13.3
14'	116	114.3	113.7	116.2	17	114.2	113.6	113.1	114.1	114.1	114.1	114.1	114.1	114	114.2	114.2	113.6
15'	42.9	31.2	26.3	37.1	14.3	25.8	26	25.7	23	25.8	25.8	25.9	25.9	26.2	25.8	27.6	28.4
1''	—	—	—	—	170	—	—	—	—	—	—	59.2	66.8	—	—	—	—
2''	—	—	—	—	21.2	—	—	—	—	—	—	—	15	—	—	—	—

Table 26. ^{13}C -NMR data of compounds 128–135.

NO.	128 [79]	129 [79]	130 [80]	131 [81]	132 [78]	133 [75]	134 [75]	135 [80]	NO.	128 [79]	129 [79]	130 [80]	131 [81]	132 [78]	133 [75]	134 [75]	135 [80]
	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3		CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3
1	40.1	170.4	173.4	172.9	170.1	173.8	172.4	149.6	1'	44.9	46.5	40.2	39.9	46.4	171.9	172.3	40
2	44.4	142.8	141.6	139.4	142.7	138.2	139.1	134.8	2'	42.7	48.6	44.6	44.6	48.6	140.3	140	44
3	218.7	193.6	207.8	205.2	193.6	193.9	194.2	115.4	3'	215	218.3	222.2	223	218.2	207.6	207.3	222
4	51.2	142.3	49.2	79	142.1	141.4	141.6	79.4	4'	80.9	52.8	51.2	51	52.7	52.3	51.9	52.7
5	49.1	47.6	51.2	53.6	47.5	51.2	46.9	56.9	5'	50.4	45.9	49.5	50.2	45.8	58.1	58.3	48.2
6	88.1	84	82.9	80.3	83.9	83.2	84	80.3	6'	81.9	83.7	84.2	84.2	83.6	80.7	80.6	83.9
7	44.2	43.7	51.5	51.4	43.6	44	43.5	50.5	7'	46.7	45.2	43.3	43.4	45	52.2	52.2	44.2
8	31.8	23.1	21	21.9	23	24.9	22.9	21.5	8'	31.3	32.1	31.9	31.8	32	21.2	21.1	32
9	38.1	35.2	35.8	36.3	35.1	28.9	34.2	36.9	9'	39.4	39.8	39.6	39.7	39.7	36.6	36.6	39.9
10	148.8	71.2	68.7	68	71.2	34.6	72.6	67.9	10'	146.2	148.9	150.4	150.1	148.8	68.3	68.3	149.6
11	139.4	140.6	140.1	139.3	140.4	139.3	140.2	139.9	11'	138.7	138.4	139.1	138.8	138.2	138.8	138.6	138.5
12	169.8	170.1	170.4	170.3	170	169.4	169.8	170.7	12'	169.8	170.9	169.5	169.6	170.8	169.4	169.3	169.9
13	120.8	119.6	118.7	118.5	119.5	120.5	119.6	118.4	13'	121.1	122.4	121	121.3	122.3	119.3	119.5	122.1
14	113.6	38.6	36.5	36.7	38.5	27.1	38.2	37.1	14'	114.8	114.6	114.2	114.5	114.5	36.7	36.6	114.9
15	23.6	121.6	25.2	31.7	121.7	122	123	27.8	15'	32.2	27	26.2	25.9	26.9	28.6	31.2	26.1
NO.	128 [79]	129 [79]	130 [80]	131 [81]	132 [78]	133 [75]	134 [75]	135 [80]									
	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3	CDCl_3									
1''	40.0	40.4	39.8	39.6	40.3	39.9	39.9	46.5									
2''	44.1	43.9	44.9	44.9	43.8	44.6	44.5	45.2									
3''	218.4	218.2	218.3	218.5	218.3	218.8	218.8	109.4									
4''	49.3	49.0	51.6	51.1	48.9	50.5	50.5	95.2									
5''	48.6	49.9	46.6	46.2	49.8	50.3	50.4	54.5									
6''	88.2	87.0	89.0	89.0	86.9	84.1	84.0	79.4									
7''	44.0	44.8	44.1	43.9	44.7	43.7	43.7	43.7									
8''	31.4	31.3	31.6	31.1	31.2	31.9	31.9	27.6									
9''	38.1	36.8	38.7	38.8	36.7	39.6	39.6	27.1									
10''	148.7	148.2	148.7	148.8	148.1	150.0	149.9	146.1									
11''	139.3	139.2	139.1	138.7	139.1	138.6	138.5	139.1									
12''	169.8	169.6	169.9	169.8	169.5	169.4	169.4	169.5									
13''	120.6	121.2	120.9	121.1	121.2	121.6	121.7	120.5									
14''	113.6	114.3	113.3	113.1	114.2	114.4	114.5	116.3									
15''	29.6	32.8	22.5	20.9	32.8	25.8	25.8	22.7									

With cisplatin as a positive control, the cytotoxicity of the compounds isolated from *Ainsliaea fragrans* was tested in the five cancer cell lines of C6 rat glioma cells, Huh1, HCC-LM3 human hepatocellular carcinoma cells, PANC-1 human pancreatic cells, and HeLa human cervical cancer cells. The cytotoxicity results showed that ainsfragolide (**132**) is an unusual guaianolide sesquiterpene trimer, which is generated by a new C2''–C15'' bond and has a significant inhibitory effect on five cancer cells with a half-inhibitory concentration value in the range of 0.4–8.3 μM . Three trimers ainsfragolide (**132**), ainsliatramer A (**133**), and ainsliatramer B (**134**) showed more potent cytotoxic effect against the five test cancer cells than the dimers. When compared with ainsliatramer B (**134**), the decreased activity of ainsliatramer A (**133**) indicated that the introduction of an extra hydroxy group at the C-10 position is important for the resultant cytotoxicity. Similarly, the dimers gochnatiolide A (**119**) and gochnatiolide B (**125**) with a C-10 OH group were more cytotoxic than gochnatiolide C (**124**), respectively. Furthermore, gochnatiolide A (**119**) with a β -configuration of OH-10 was about 2–10-fold more active than gochnatiolide B (**125**) with α -OH at C-10. In contrast, ainsliadimer B (**116**) with 10 β -OH and OH-15 groups showed reduced activity [78]. Therefore, we can try to obtain a trimer with a β -configuration hydroxyl at the C-10 position and no hydroxyl at the C-15 position by synthesis or structural modification, so as to improve the cytotoxic activity of polymer sesquiterpene lactones against cancer cells.

5.2.2. Anti-Inflammatory

Macrocephadiolide A (**114**) and macrocephadiolide B (**111**) showed a potent inhibitory effect on nitric oxide (NO) production, with IC₅₀ values of 0.99 and 6.13 μM , respectively, on lipopolysaccharide (LPS)-stimulated RAW264.7 macrophages. Macrocephadiolide A (**114**) dose-dependently suppressed the expression of inducible NO oxidase (iNOS) through inhibiting nuclear factor kappa B (NF- κ B) activation [72]. Ainsliadimer A (**113**) represents an unusual carbon skeleton with a cyclopentane system connecting the two monomeric sesquiterpene lactone units. This unique molecule exerted potent inhibitory activity against the production of nitric oxide in RAW264.7 cells stimulated by LPS, with an IC₅₀ value of 2.41 $\mu\text{g}/\text{mL}$ [73]. Gochnatiolide A (**119**) showed significant anti-inflammatory activity by inhibiting the expression of nuclear factor kappa B (NF- κ B) in the 293-NF- κ B-luciferase reporter cell line and the production of TNF- α , IL-1 β , IL-6, and IL-10 in RAW264.7 macrophages induced by lipopolysaccharide (LPS) [47]. The anti-inflammatory effects of polymer sesquiterpene lactones in *Ainsliaea* are achieved by inhibiting the production of nitric oxide (NO) in RAW264.7 macrophages, inhibiting the activity of nuclear factor kappa B (NF- κ B) in the luciferase 293-NF- κ B-luciferase reporter cell line, and inhibiting the expression of TNF- α , IL-1 β , IL-6, and IL-10 in RAW264.7 macrophages.

5.2.3. Other Biological Activities

The polymer sesquiterpene lactones also have antibacterial and blood-sugar regulation effects. Ainsliatramer B (**134**) also showed a medium inhibiting effect on *Bacillus subtilis* with a MIC value of 32 $\mu\text{g}/\text{mL}$ [81]. Macrocephatriolide B (**128**) showed potent inhibition against protein tyrosine phosphatase 1B (PTP1B) with an IC₅₀ value of $26.26 \pm 0.88 \mu\text{M}$. In insulin-stimulated C2C12 myotubes, macrocephatriolide B (**128**) dose-dependently enhanced glucose uptake by activating the insulin signaling pathway and might represent a new scaffold of insulin sensitizers [79].

6. Other Sesquiterpenoids

Beyond those mentioned above, there exist other categories of sesquiterpenoids and their derivatives, such as myrrhanes, lananes, and carabanes. And further details can be referenced in Figure 5 and Table 27.

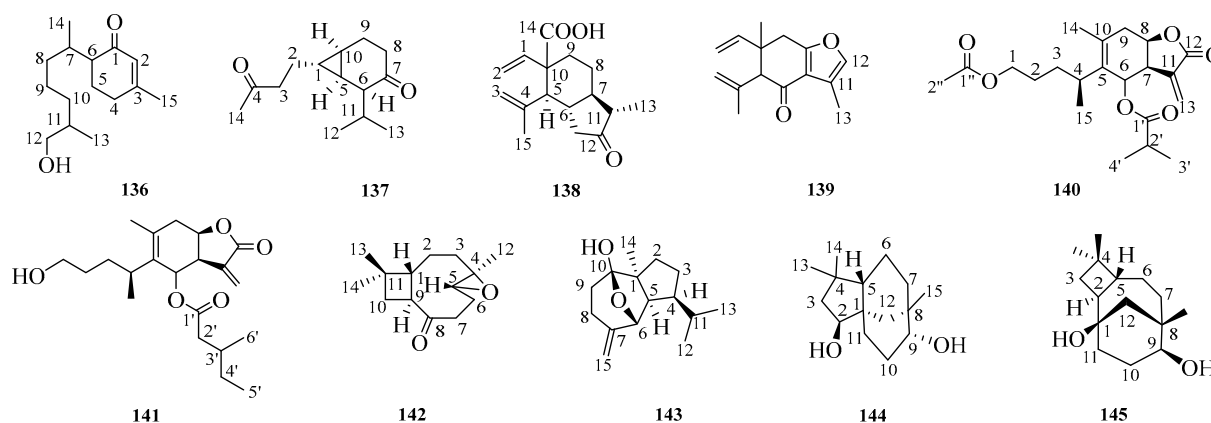


Figure 5. Chemical structures for compounds 136–145.

Table 27. The compound name, molecular formula, and test reagent of other sesquiterpenoids.

No.	Compound Name	Molecular Formula	Solvent	Ref.
136	1-Oxo-bisabol-2-ene-12-ol	C ₁₅ H ₂₆ O ₂	CDCl ₃	[82]
137	Pubescione	C ₁₄ H ₂₂ O ₂	CDCl ₃	[83]
138	Ainsliaea acid A	C ₁₆ H ₂₂ O ₃	CD ₃ OD	[67]
139	Curzerone	C ₁₅ H ₁₈ O ₂	CDCl ₃	[84]
140	1-O-Acetyl-6-O-isobutyrylbritannilactone	C ₂₀ H ₃₀ O ₆	CDCl ₃	[85]
141	6 α -(3-Methylvaleryloxy)-1-hydroxy-4 α H-1,10-secoeudesma-5(10),11(13)-dien-12,8 β -olide	C ₂₁ H ₃₂ O ₅	CDCl ₃	[86]
142	Kobusone	C ₁₄ H ₂₁ O ₂	CDCl ₃	[87]
143	10-Hydroxy-6,10-epoxy-7(14)-isodaucane	C ₁₅ H ₂₄ O ₂	CDCl ₃	[88]
144	Clovane-2 β ,9 α -diol	C ₁₅ H ₂₆ O ₂	CDCl ₃	[89]
145	Caryolane-1,9 β -diol	C ₁₅ H ₂₆ O ₂	CDCl ₃	[89]

6.1. NMR Data of Other Sesquiterpenoids 136–145

The NMR spectra of ¹H and ¹³C are summarized in Tables 28 and 29. This paper also compiles data regarding the nuclear magnetic resonance testing instruments used for the compounds numbered from 136 to 145. The ¹H and ¹³C data of compound 136 were recorded using a Bruker instrument operating at 300 MHz. For compound 137, NMR data collection was performed with the Bruker-Avance-III-400 instrument (Bruker, Switzerland). For compound 140, the NMR data were conducted on a Bruker AMX 400 instrument (Bruker, Zurich, Switzerland). The NMR data for compound 141 were taken with a Bruker Avance-500 instrument (Bruker, Switzerland). Both the ¹H and ¹³C data of compound 143 were measured using JEOL Lamda 400 (JEOL, Japan) and Lamda 600 instruments (JEOL, Japan). Finally, compounds 144 and 145 had their respective ¹H and ¹³C data collected via the JEOL JNM-GX400 instrument (JEOL, Japan). The ¹H data for compounds 138, 139, and 142 were measured at frequencies of 600 MHz, 300 MHz, and 400 MHz, respectively. The ¹³C NMR of compounds 138, 139, and 142 were tested under 150 MHz, 75 MHz, and 100 MHz, respectively.

Table 28. ¹H-NMR data of compounds 136–145.

NO.	136 [82]	137 [83]	138 [67]	139 [84]	140 [85]	141 [86]	142 [87]	143 [88]	144 [89]	145 [89]
	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
1 α	—	—	5.90, dd, 17.8, 11.2	5.78, dd, 17.6, 10.6	3.85–4.00, m	3.52, m	—	—	—	—
1 β	—	1.66–1.63, m	—	—	—	3.44, m	1.93, ddd, 10.4, 8.8, 1.2	—	—	—
2 α	5.86, m	1.76–1.63, m	5.34, dd, 17.8, 1.1	4.74–4.99, m	1.34–1.45, m	1.32, m	1.67, m	1.82	3.79, dd, 10.5, 5.5	2.22, ddd, 12, 10, 8.5
2 β	—	—	5.25, dd, 18.0, 1.2	—	1.21–1.31, m	1.13, m	1.54, m	1.37	—	—
3 α	—	2.53–2.48, m	4.90, m	4.74–4.99, m	1.21–1.31, m	1.32, m	2.15, dt, 13.2, 3.6	1.87	1.51, dd, 11.5, 10	1.49, dd, 10, 9
3 β	—	—	—	—	0.97–1.08, m	1.00, m	0.94, td, 13.2, 4.4	1.27	1.71, dd, 11.5, 5.5	1.54, t, 10
4	2.32, m	—	—	—	2.60–2.76, m	2.70, m	—	1.58	—	—
5 α	1.92, m	1.05–1.03, m	2.79, br s	3.00, s	—	—	—	1.56	—	1.89, ddd, 12, 9, 6
5 β	1.78, m	—	—	—	—	—	2.69, dd, 10.0, 5.2	—	1.42, m	—
6 α	2.15, m	0.72–0.67, m	—	—	5.19, d, 1.7	5.23, d, 1.7	2.40, m	3.98	1.32, m	1.39, m
6 β	—	—	4.64, br s	—	—	—	1.44, m	—	1.35, m	1.53, m
7 α	2.33, m	—	2.02, m	—	3.41–3.49, m	3.50, m	2.55, dd, 6.4, 2.0	—	1.11, m	1.15, m
7 β	—	—	—	—	—	—	2.53, d, 6.4	—	1.50, m	1.42, m
8 α	1.26, m	2.05, dd, 8.8, 3.2	1.96, m	—	4.90–4.98, m	5.01, m	—	2.49	—	—
8 β	—	—	1.43, m	—	—	—	—	2.35	—	—
9 α	1.34, m	2.14–2.11, m	1.86, m	2.83, AB system, 17.6	2.60–2.76, m	2.75, dd, 16.2, 2.1	3.05, td, 8.8, 8.8	2.02	—	3.44, t, 3
9 β	—	2.02–1.99, m	—	—	2.45–2.56, m	2.51, dd, 16.2, 2.3	—	1.76	3.32, br s	—
10 α	1.39, m	1.88–1.84, m	—	—	—	—	2.06, dd, 10.4, 8.8	—	1.64, m	1.77, ddt, 15, 5, 3
10 β	1.12, m	—	—	—	—	—	1.66, d, 10.4	—	1.99, m	2.04, dddd, 15, 12.5, 5.5, 3
11 α	1.62, m	1.80–1.77, m	—	—	—	—	—	1.51	1.07, m	1.51, m
11 β	—	—	2.36, m	—	—	—	—	—	1.66, m	1.64, td, 12.5, 5
12 α	3.50, dd, 10.6, 5.8	0.92, d, 6.8	—	7.08, br s	—	—	1.30, s	0.89, d, 6.6	0.91, br d, 12.5	1.42, d
12 β	3.42, dd, 10.6, 5.8	—	—	—	—	—	—	—	1.56, d, 12.5	1.47, d
13 α	0.91, d, 6.7	0.92, d, 6.8	1.24, d, 7.0	2.16, br s	6.37, d, 2.9	6.36, d, 2.6	1.02, s	0.92, d, 6.6	0.86, s	1.00, s
13 β	—	—	—	—	5.94, d, 2.3	6.02, d, 2.3	—	—	—	—
14	0.79, d, 6.8	2.18, s	—	1.17, s	1.80, s	1.82, s	1.02, s	1.15, s	1.04, s	1.02, s
15 α	1.93, br s	—	1.67, s	1.83, br s	0.86, d, 6.9	0.90, d, 7.0	—	4.76, br s	0.96, s	0.93, s
15 β	—	—	—	—	—	—	—	4.70, br s	—	—
2' α	—	—	—	—	2.45–2.56, m	2.32, m	—	—	—	—
2' β	—	—	—	—	—	2.11, m	—	—	—	—
3'	—	—	—	—	1.15, d, 6.9	1.87, m	—	—	—	—
4' α	—	—	—	—	1.15, d, 6.9	1.37, m	—	—	—	—
4' β	—	—	—	—	—	1.26, m	—	—	—	—
5'	—	—	—	—	—	0.92, t, 7.5	—	—	—	—
6'	—	—	—	—	—	0.94, d, 7.0	—	—	—	—

Note: The ¹H-NMR data of 2'' of compound 140 were 2.04, s.

Table 29. ¹³C-NMR data of compounds 136–145.

NO.	136 [82]	137 [83]	138 [67]	139 [84]	140 [85]	141 [86]	142 [87]	143 [88]	144 [89]	145 [89]	NO.	140 [85]
	CDCl ₃	CDCl ₃	CD ₃ OD	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃		CDCl ₃
1	201.4	34.4	135.8	145.5	64.2	61.7	51.1	54.5	44.6	70.7	1'	176.9
2	127.2	24.8	117.9	115.7	26.5	30.7	26.2	26.4	80.8	38	2'	34.6
3	161.4	41.7	115.5	113	31	30.3	38.8	30.6	47.5	34	3'	18.7
4	30.6	208.7	144.4	141.1	33	32.9	58.6	57.6	37.1	35	4'	18.8
5	22.6	29.9	61.5	64.1	132.1	131.9	61.3	58.9	50.6	43.8	1''	171.2
6	50.1	46.4	82.2	194	69	68.8	24.5	86	20.7	20.3	2''	21
7	30.9	214.2	43.3	120.2	42.9	42.7	37.4	144.8	33.2	35.3		
8	34.9	32.8	25	165.6	74.9	75.6	213.8	33.2	34.7	39.3	NO.	141 [86]
9	24.9	22.9	34.7	33.6	34.1	34.1	52.3	35.9	75.1	72.1		CDCl ₃
10	33.3	27.9	52.3	42.9	133.6	133.3	35	105.1	26	28.1	1'	173.4
11	35.8	30.9	43.8	119.3	136.3	136	34.2	34.1	26.4	33.3	2'	41.3
12	68.4	19.5	179.2	139.6	169.5	170.5	16	21.5	35.6	42.4	3'	31.6
13	16.7	19.2	15.4	9.1	124.9	125.1	29.1	20.4	25.4	20.8	4'	28.8
14	15.8	29.9	180	24.9	20.5	19.9	22	21.6	31.4	30.5	5'	10.6
15	24.2	—	21.9	25	18.6	18.1	—	108.4	28.4	26.7	6'	18.7

6.2. Bioactivity of Other Sesquiterpenoids

At the concentration of 10 $\mu\text{Mol/L}$, ainsliaea acid A (**138**) significantly inhibited nuclear factor kappa B (NF- κ B) in lipopolysaccharides-induced 293-NF- κ B-luciferase reporter cell lines with an inhibitory rate of 17.5%. Further experiments showed that ainsliaea acid A (**138**) exerted anti-inflammatory effects by inhibiting the production of tumor necrosis factor- α (TNF- α), interleukin-1 β (IL-1 β), IL-6, and IL-10 in RAW264.7 macrophages induced by LPS [67].

7. Conclusions

Sesquiterpenoids derived from plants of the genus *Ainsliaea* exhibit a wealth of pharmacological activities, demonstrating significant antitumor, anti-inflammatory, antibacterial, and antiobesity effects. Currently, the existing literature primarily focuses on the chemical composition and pharmacological effects of these plants; however, comprehensive NMR data summarizing the related components remain scarce. This paper consolidates the ^1H - and/or ^{13}C -NMR data for sesquiterpenes extracted from *Ainsliaea* species, and the pharmacological activities of sesquiterpenes are summarized, thereby providing a valuable reference for discovering novel sesquiterpenes and differentiating between various types. It also offers essential data support for structural analysis and compound identification. Additionally, exploring new sesquiterpene constituents is crucial for investigating their pharmacodynamic material basis, as well as their structure–activity relationships and mechanisms of action, and enriching the diversity of natural products.

The sesquiterpenoids found in the plants of *Ainsliaea* are mostly guaiane and eudesmane, and the active ingredients found are mostly guaiane and polymer sesquiterpene lactones. Guaiacanolactone containing a α -methylene- γ -lactone moiety and guaiac lactones containing hydroxyl groups with three outer-ring double bonds have a significant inhibitory effect on NO production. Trimeric sesquiterpene lactones with a β -configuration hydroxyl at the C-10 position and without a hydroxyl group at C-15 have high cytotoxic activity. Therefore, it is of great significance to find compounds containing these components in natural products or to obtain these compounds through synthesis and structural modification to improve the anti-inflammatory activity of guaiane sesquiterpenes and the cytotoxic activity of polymer sesquiterpene lactones. The limited amount of germacrane and other sesquiterpenes reported within this genus renders the data support for structural analysis and identification of these compounds insufficiently convincing. It is essential to conduct further investigations into their chemical constituents, identify additional sesquiterpenes, and enhance the diversity of sesquiterpene types within the genus. Moreover, a comprehensive summary of the NMR spectral data pertaining to triterpenoids, steroids and their derivatives, phenolic acids, flavonoids, anthraquinones, coumarins, lignans, and other components remains lacking and needs to be further summarized.

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