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THREE NEW TRITERPENOIDS FROM *ANTRODIA CINNAMOMEA*

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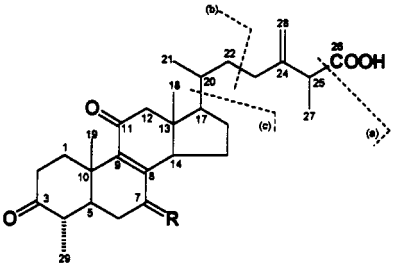
ABSTRACT.—Three new ergostane-type triterpenoids, antcins A [**1**], B [**2**], and C [**3**], and two known lanostane-type triterpenoids were isolated from a new species, *Antrodia cinnamomea*. These three new compounds were identified as 4α-methylergosta-8,24(28)-dien-3,11-dion-26-oic acid [**1**], 4α-methylergosta-8,24(28)-dien-3,7,11-trion-26-oic acid [**2**], and 4α-methylergosta-8,24(28)-dien-3,11-dion-7β-ol-26-oic acid [**3**], by spectroscopic analysis. The structure of **1** was confirmed by X-ray crystallography.

A new basidiomycete, *Antrodia cinnamomea* Chang & Chou, sp. nov. (family Polyporaceae, Aphyllophorales), the cause of brown heart rot of *Cinnamomum kanahirai* Hay. in Taiwan, was first identified in 1994 as a new species of the genus *Antrodia* (1). This organism is well-known in Taiwan by the name "niu chang ku" or "jang-jy" and is also a popular and very expensive medicinal material. The species is used traditionally as an antidote, an anticancer agent, and an anticnesmatic (anti-itching) drug, but no biological testing has yet been reported. In this paper we wish to report the isolation and structural elucidation of three new compounds from *A. cinnamomea*, namely, antcin A [**1**] (4α-methylergosta-8,24(28)-dien-3,11-dion-26-oic acid), antcin B [**2**] (4α-methylergosta-8,24(28)-dien-3,7,11-trion-26-oic acid), and antcin C [**3**] (4α-methylergosta-8,24(28)-dien-3,11-trion-7β-26-oic acid). Two known lanostanoids were also isolated from *A. cinnamomea*, namely, 24-methylenelanosta-7,9(11)-dien-3β-ol-21-oic acid [**4**] (2) and 24-methylenedihydrolanosterol [**5**] (3). The structure of **1** has been confirmed by X-ray crystallographic analysis.

RESULTS AND DISCUSSION

Antcin A [**1**] showed a positive Liebermann-Burchard test, and its molecular formula of $C_{29}H_{42}O_4$, was established by hrms. It exhibited a uv absorption band at 251.5 nm (log ε 3.85), which is similar to that of methyl ganoderate H (4), and characteristic of an ergostane-type triterpenoid with an α,β-unsaturated carbonyl group at $\Delta^{8(9)}$ and an 11-C=O. Its ir signals showed bands attributable to hydroxyl (3400 cm^{-1}), carbonyl (1710, 1734, 1653 cm^{-1}), and terminal methylene (890 cm^{-1}) groups. The mass spectrum showed prominent peaks at *m/z* 410 $[M^+-CO_2]$ (a), 341 $[M^+-C_6H_9O_2]$ (b) and 299 $[M^+-C_9H_{15}O_2]$ (c). These ion peaks (5) are characteristic fragments of triterpenoids with a 24-exo-methylene-26-oic acid side-chain. The ^{13}C-nmr spectrum of **1** revealed the presence of one carboxylic acid group (C-26, δ 179.9) and two six-membered cyclic ketones (C-3, δ 213.5; C-11, δ 200.1), with the more upfield signal being due to an α,β-unsaturated ketone. The ^1H-nmr spectrum of **1** showed signals for two tertiary methyl groups and three secondary methyl groups, as required by a compound bearing the 4-methylergostane skeleton. The methyl singlet signal at δ 0.73, which showed long-range coupling with the δ 2.33 (H-12α) resonance in the ^1H-^1H shift-correlated nmr spectrum, was assigned as Me-18 and the other singlet methyl signal (δ 1.33) as Me-19 (6). The other three doublet methyl sets were confirmed by their chemical shifts and by decoupling as follows: δ 1.31 (3H, d, J=6.8 Hz, Me-27) coupling with δ 3.15 (H-25β, m), δ 1.05 (3H, d, J=6.3 Hz, Me-29) coupling with δ 2.40 (H-4β, m), and δ 0.93 (3H, d, J=5.4 Hz, Me-21) coupling with δ 1.45 (H-20β, m). These



1 R=H, H
2 R=O
3 R=β-OH, H

data suggested that **1** was 4α-methylergosta-8,24(28)-dien-3,11-dion-26-oic acid, and its stereochemistry was confirmed by X-ray crystallography. An ORTEP drawing of the molecule of **1** is shown in Figure 1.

Antcin B [**2**] gave a positive Liebermann-Burchard test. Its ir spectrum showed hydroxyl (3440 cm^{-1}), ketone and acid (1707, 1734 cm^{-1}), conjugated ketone (1676 cm^{-1}), and terminal methylene (900 cm^{-1}) absorptions. The uv-vis spectrum of **2** was similar to that of antcin A [**1**], which indicated the presence of a 7,11-dion-8(9)-ene moiety. Hrms of **2** showed a molecular ion peak at *m/z* 468.2837, and the elemental formula was assigned as $C_{29}H_{40}O_5$. Compound **2** had the same side-chain as **1**, as shown by fragmentation ions at *m/z* 424 $[M^+-CO_2]$ (a), 354 $[M^+-C_6H_{10}O_2]$ (b), and 313 $[M^+-C_9H_{15}O_2]$ (c). The ^{13}C-nmr and DEPT spectra of **2** showed two signals for a terminal methylene of the side-chain at δ 148.0 (C-24) and 111.2 (C-28), and the conjugated system of 7,11-dion-8(9)-ene represented by the signals at δ 200.7 (C-7), 145.3 (C-8), 151.8 (C-9), and 202.5 (C-11). The lowest-field signal (δ 210.8) assigned to C-3 was the characteristic resonance for a six-membered cyclic ketone. The ^{1}H-nmr spectrum gave two signals for the two singlet methyl groups of a triterpenoid (Me-18, δ 0.71 and Me-19, δ 1.54). The other three doublet methyl groups could be confirmed by the same method as used for **1**. From these spectral data, **2** was established as 4α-methylergosta-8,24(28)-dien-3,7,11-trion-26-oic acid.

Antcin C [**3**] also gave a positive Liebermann-Burchard test. Its hrms showed a molecular ion peak at *m/z* 470.3051, which analyzed for $C_{29}H_{42}O_5$. The prominent peaks of the eims spectrum at *m/z* 452 $[M^+-OH]$, 426 $[M^+-CO_2]$ (a), 356 $[M^+-C_6H_9O_2]$ (b), and 316 $[M^+-C_9H_{15}O_2]$ (c) resembled those of **1** and **2**, indicating that the side-chain of all these molecules was identical. The ir spectrum of **3** showed the presence of a hydroxyl (3200 cm^{-1}, br), a carboxylic acid and six-membered cyclic ketone (1728,

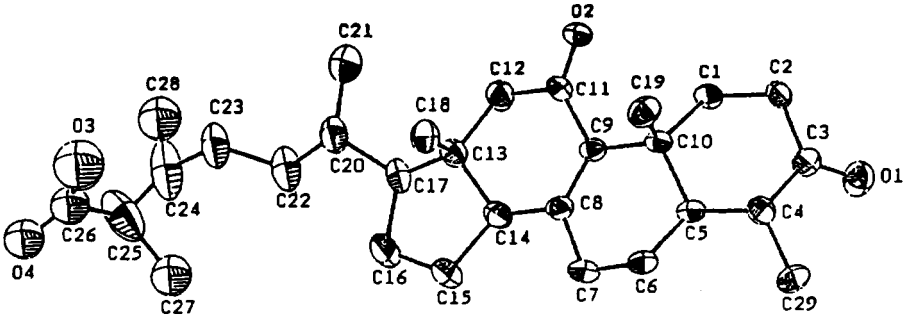


FIGURE 1. ORTEP drawing of **1**.

1710 cm^{-1}), a terminal methylene (893 cm^{-1}), and a conjugated ketone (1676 cm^{-1}). The uv absorption band at λ max 253 nm (log ε 3.60) indicated that the conjugated system was 11-on-8(9)-en-7-ol. The ^{1}H- and ^{13}C-nmr spectra of **3** closely resembled those of **2**, suggesting that their structures are similar except for the OH-7 in **3**, as opposed to the C-7 carbonyl in **2**. The ^{1}H-nmr chemical shift of H-7α of **3** appeared at δ 4.3 as a doublet of doublets (J=7.6 and 8.8 Hz) and the chemical shifts of H_2-6 shifted to δ 1.53 and 2.5 as compared to δ 2.46 and 2.52 in the ^{1}H-nmr spectrum of **2**. From their ^{13}C-nmr spectra, the principal difference between **1** and **3** was the appearance of a signal at δ 70.2 (7-*C*H-OH) in the latter compound.

The identities of compounds **4** and **5** were confirmed by comparison with previously reported data (2,3).

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—All mps were determined on a Mel-temp apparatus and are uncorrected. Uv-vis spectra were obtained on a Jasco 7850 instrument, optical rotations were measured on a Jasco DIP-360 polarimeter, and ir spectra were obtained on a Bio-Rad FTS-40FT-IR spectrometer. ^{1}H- and ^{13}C-nmr spectra were taken on a JEOL EX400 nmr spectrometer at 400 MHz in $CDCl_3$ with TMS as internal standard and are recorded in δ (ppm) units. ^{1}H-Nmr assignments were based on spin-spin decoupling experiments and ^{1}H-^{1}H shift-correlated spectra. ^{13}C-Nmr assignments were based on ^{1}H-^{13}C shift-correlated spectra. Lrms and hrms were obtained with JEOL JMS-D300 and JEOL JMS-HX 110 spectrometers, respectively. The X-ray data were acquired on a CAD 4 Kappa Axis single-crystal diffractometer. Hplc (Jasco 887-PU instrument) was performed with *n*-hexane/EtOAc on a Si gel 60 (Merck, 10 μm, 10 mm i.d.×250 mm) column employing a ri detector (Waters R401) at a flow rate of 2.0 ml/min.

FUNGAL MATERIAL.—*Antrodia cinnamomea*, growing in Ping-Tung, Taiwan, was collected in 1987 by Ju-Chen Wang, Ling-Chih Co., Taipei, and identified by Prof. Chiu-Yuan Chien of the Institute of Biological Science, National Taiwan Normal University. A voucher specimen has been deposited with Dr. T.T. Chang, Divison of Forest Protection, Taiwan Forestry Research Institute.

EXTRACTION AND ISOLATION.—The dry fruiting bodies of *A. cinnamomea* (200 g) were cut into small

TABLE 1. ^{1}H-Nmr Data of Compounds **1–3**.[a]

Proton	Compound		
	1	**2**	**3**
1	1.37, 3.15	1.47, 3.07	1.25, 2.90
2	2.50, 2.37	2.41, 2.55	2.50, 2.35
4	2.40	2.48	2.35
5	1.40	1.90	1.43
6	1.78, 1.43	2.46, 2.52	1.53, 2.50
7	2.30, 2.22	—	4.40 dd (J=7.6, 8.8)
12α	2.33 d (J=14)	2.45 d (J=14)	2.30 d (J=14)
12β	2.80 d (J=14)	2.94 d (J=14)	2.85 d (J=14)
14	2.63 dd (J=11.5, 7.6)	2.67 dd (J=12, 7.1)	2.70 dd (J=11.4, 6.6)
15	1.52, 1.81	1.40, 2.53	1.90, 2.10
16	1.42, 1.97	1.31, 1.97	1.43, 1.90
17	1.50	1.45	1.40
18	0.73 s	0.71 s	0.79 s
19	1.33 s	1.54 s	1.46 s
20	1.45	1.42	1.40
21	0.93 d (J=5.4)	0.96 d (J=4.9)	0.94 d (J=5.6)
22	1.22, 1.67	1.25, 1.59	1.25, 1.58
23	1.99, 2.17	1.98, 2.15	1.95, 2.15
25	3.15 q (J=6.8)	3.15 q (J=6.9)	3.10 q (J=7.2)
27	1.30 d (J=6.8)	1.30 d (J=6.9)	1.31 d (J=7.2)
28	4.97, 4.92	4.97, 4.92	4.99, 4.93
29	1.05 d (J=6.3)	1.05 d (J=6.4)	1.04 d (J=6.4)

[a]Recorded as ppm in $CDCl_3$. Coupling constants (in Hz) in parentheses.

TABLE 2. ^{13}C-Nmr Data of Compounds **1–3**.[a]

Carbon	Compound		
	1	**2**	**3**
1	35.0 t	34.5 t	35.7 t
2	37.8 t	37.3 t	37.8 t
3	217.3 s	210.8 s	212.4 s
4	44.3 d	43.7 d	43.9 d
5	50.5 d	48.6 d	48.2 d
6	20.8 t	38.7 t	32.5 t
7	30.2 t	200.7 s	69.9 d
8	157.5 s	145.3 s	153.0 s
9	138.6 s	151.8 s	141.0 s
10	38.6 s	38.1 s	37.1 s
11	200.2 s	202.5 s	201.0 s
12	57.6 t	57.1 t	57.9 t
13	47.2 s	46.8 s	47.6 s
14	53.0 d	49.1 d	53.1 d
15	23.6 t	24.6 t	24.8 t
16	27.4 t	27.6 t	27.9 t
17	55.2 d	53.8 d	54.4 d
18	12.0 q	11.7 q	12.1 q
19	17.4 q	16.1 q	17.5 q
20	35.7 d	35.4 d	35.8 d
21	18.3 q	18.2 q	18.5 q
22	33.7 t	33.6 t	33.8 t
23	31.3 t	31.2 t	31.4 t
24	148.2 s	148.0 s	148.0 s
25	45.3 d	45.1 d	45.3 d
26	179.7 s	179.7 s	179.0 s
27	16.1 q	16.0 q	16.1 q
28	111.3 t	111.2 t	111.0 t
29	11.8 q	11.2 q	11.5 q

[a]Recorded as ppm in $CDCl_3$.

pieces and refluxed six times with MeOH (2 liters) for 5 h. The concentrated MeOH extract was partitioned between H_2O and $CHCl_3$, and the $CHCl_3$ fraction (40 g) was chromatographed on a Si gel column (800 g) by stepwise elution with 10% EtOAc/*n*-hexane, 20% EtOAc/*n*-hexane, and 50% EtOAc/*n*-hexane. The 10% EtOAc/*n*-hexane elution was rechromatographed on a Si gel column using 30% $CHCl_3$/*n*-hexane as solvent system to give **5** (30 mg). The 20% EtOAc/*n*-hexane eluate was separated repeatedly by Si gel cc (50% $CHCl_3$/*n*-hexane) and then separated by hplc (30% EtOAc/*n*-hexane) to afford **1** (60 mg), **2** (85 mg), and **3** (12 mg), respectively. The 50% EtOAc/*n*-hexane elution was chromatographed on a Si gel column repeatedly with 50% $CHCl_3$/*n*-hexane to give a white precipitate that was recrystallized from 50% EtOAc/$CHCl_3$ to afford **4** (140 mg).

Antcin A [**1**].—Colorless prisms, mp 173–175°; $[\alpha]_D$ +152° (*c*=0.25, $CHCl_3$); ir ν max (KBr) 3400, 3064, 2960, 2872, 1734, 1710, 1653, 1610, 1589, 1458, 1379, 1172, 890 cm^{-1}; uv λ max (MeOH) (log ϵ) 251.5 nm (3.85); ^{1}H-nmr data, see Table 1; ^{13}C-nmr data, see Table 2; eims (30 eV) *m/z* 454 (M^+, 63), 410 (M^+-CO_2, 36), 341 ($M^+-C_6H_9O_2$, 5), 299 (M^+−side-chain, 12), 296 (18), 271 (19), 260 (70), 205 (100), 121 (20); hrms, *m/z* 454.3094 (calcd for $C_{29}H_{42}O_4$, 454.3085).

Antcin B [**2**].—Yellow needles, mp 136–138°; $[\alpha]_D$ +78.7° (*c*=0.61, $CHCl_3$); ir ν max (KBr) 3440, 3082, 2978, 2937, 2879, 1734, 1707, 1676, 1645, 1458, 1415, 1379, 1234, 900 cm^{-1}; uv λ max (MeOH) (log ϵ) 251 nm (3.27); ^{1}H-nmr data, see Table 1; ^{13}C-nmr data, see Table 2; eims (30 eV) *m/z* 468 (M^+, 16), 424 (M^+-CO_2, 20), 354 ($M^+-C_6H_{10}O_2$, 13), 313 (M^+−side-chain, 5), 286 (6), 91 (6), 28 (100); hrms, *m/z* 468.2873 (calcd for $C_{29}H_{40}O_5$, 468.2876).

Antcin C [**3**].—White needles, mp 187–189°; $[\alpha]_D$ +60.0° (*c*=0.1, $CHCl_3$); ir ν max (KBr) 3100, 1728, 1710, 1676, 1653, 1639, 1458, 1377, 1197, 893 cm^{-1}; uv λ max (MeOH) (log ϵ) 253 (3.60); ^{1}H-nmr data, see Table 1; ^{13}C-nmr data, see Table 2; eims (30 eV) *m/z* 470 (M^+, 55), 452 (M^+−OH, 24), 426

TABLE 3. Crystal Data and Conditions for Crystallographic Data Collection and Structure Refinement for Antcin A [**1**].

Formula	$C_{29}O_4H_{42}$
Formula weight	454.7
Diffractometer used	Nonius, CAD4
Space group	Monoclinic $P2_1$
a (Å)	9.8330 (24)
b (Å)	7.6482 (22)
c (Å)	18.055 (3)
β (deg)	102.180 (18)
V (Å^3)	1327.3 (6)
Z	2
Dcalc (g·cm^{-3})	1.138
λ (M_oK_α) (Å)	0.7107
F(000)	496
Unit cell detn #; (2 θ range)	24; (18.86–22.64 deg.)
Scan type	θ/2 θ
2 θ scan width (deg)	2(0.95+0.35 tan θ)
Scan speed (deg/min)	2.06–8.24
2 θ (max)	45.0
h k l ranges	(−10;10) (0;8) (0;19)
μ (M_oK_α) (cm^{-1})	0.689
Crystal size (mm)	0.20×0.25×0.35
Transmission	1.000; 1.000
Temperature	298.00
No. of meas. reflns	1897
No. of obsd. reflns [I>2.0 sig (I)]	1590
No. of unique reflns	1897
RF; Rw[a]	0.074; 0.073
GoF[a]	2.50
Refinement program	NRCVAX (10)
No. of atoms	70
No. of refined params	293 (1590 out of 1897 reflns.)
Minimize function	SUM(w|F0-F_c|^2)
g(2nd. ext. coeff.)×10^4	0.475 (17)
(Δ/σ) max	0.1937
(Δρ) max.min eÅ^{-3}	−0.280; 0.320

[a]RF=Sum(Fo-Fc)/Sum(Fo); R_w=Sqrt[Sum(w(Fo-Fc)^2)/Sum(wFo^2)]; GoF=Sqrt[Sum(w(Fo-Fc)^2/No. of reflns-No. of params.)]

(M^+-CO_2, 22), 356 ($M^+-C_6H_9O_2$, 22), 316 (M^+−side-chain, 25), 297 (25), 245 (20), 221 (100), 175 (27), 121 (35), 95 (35); hrms, *m/z* 470.3051 (calcd for $C_{29}H_{42}O_5$, 470.3034).

24-Methylenelanosta-7,9(11)-dien-3β-ol-21-oic acid [**4**].—White powder, mp 258–260° (2); ^{13}C nmr (pyridine-d_6) δ 178.3 (C-21), 156.0 (C-24), 146.7 (C-9), 142.9 (C-8), 121.3 (C-7), 116.7 (C-11), 107.1 (C-28), 16.3 (C-18), 23.4 (C-19), 22.0 (C-26), 22.1 (C-27), 26.0 (C-29), 28.7 (C-30), 16.6 (C-31), 78.1 (C-3), 23.6, 28.5, 27.3, 31.7, 31.9, 32.8, 34.3, 36.1, 36.5, 37.9, 39.4, 44.4, 48.2, 49.9, 50.6.

24-Methylenedihydrolanosterol [**5**].—White powder, mp 158–159° (3).

X-RAY CRYSTALLOGRAPHIC ANALYSIS OF **1**.[1]—Single crystal X-ray diffraction was applied to antcin A [**1**]. Intensity measurements were made on a Kappa diffractometer with MoKα radiation. Essential details of the measurement and the result of refinements are given in Table 3. No absorption correction was applied and H atoms were mostly calculated with ideal geometries. The terminal carboxylated part is somewhat

[1]Hydrogen coordinates, thermal parameters, bond distances and angles, and observed and calculated structure factors have been deposited with the Cambridge Crystallographic Data Centre and can be obtained upon request from Dr. Olga Kennard, University Chemical Laboratory, 12 Union Road, Cambridge CB2 1EZ, UK.

TABLE 4. Positional Parameters and Their Estimated Standard Value Deviations for Antcin A [1].

	x	y	z	Beq.[a]
C-1	0.7744 (9)	0.4215 (10)	0.5042 (4)	4.3 (4)
C-2	0.7719 (9)	0.3932 (11)	0.5878 (4)	4.8 (4)
C-3	0.8392 (7)	0.2275 (12)	0.6164 (4)	4.2 (4)
C-4	0.7828 (7)	0.0666 (12)	0.5709 (4)	4.2 (4)
C-5	0.7925 (8)	0.1038 (10)	0.4877 (4)	3.9 (4)
C-6	0.7562 (9)	−0.0528 (11)	0.4354 (5)	5.1 (4)
C-7	0.7971 (9)	−0.0212 (11)	0.3609 (5)	5.2 (4)
C-8	0.7744 (7)	0.1628 (10)	0.3322 (4)	3.9 (3)
C-9	0.7340 (7)	0.2900 (10)	0.3723 (4)	3.5 (3)
C-10	0.7119 (7)	0.2656 (11)	0.4538 (4)	3.7 (3)
C-11	0.6948 (8)	0.4606 (11)	0.3337 (4)	4.4 (4)
C-12	0.7385 (9)	0.4997 (12)	0.2598 (4)	5.0 (4)
C-13	0.7180 (8)	0.3379 (11)	0.2089 (4)	4.3 (4)
C-14	0.8066 (7)	0.1956 (11)	0.2560 (4)	4.3 (4)
C-15	0.8010 (9)	0.0432 (14)	0.1985 (5)	6.0 (5)
C-16	0.8006 (10)	0.1398 (15)	0.1230 (5)	6.8 (5)
C-17	0.7818 (9)	0.3418 (13)	0.1375 (4)	5.1 (4)
C-18	0.5636 (9)	0.2898 (14)	0.1901 (5)	5.7 (5)
C-19	0.5552 (8)	0.2489 (14)	0.4507 (5)	5.4 (4)
C-20	0.7029 (9)	0.4366 (16)	0.0666 (5)	6.2 (5)
C-21	0.6747 (14)	0.6259 (19)	0.0799 (6)	9.4 (8)
C-22	0.7852 (10)	0.4187 (19)	0.0017 (5)	7.7 (7)
C-23	0.7018 (12)	0.4639 (20)	−0.0757 (5)	9.1 (8)
C-24	0.7683 (12)	0.4656 (24)	−0.1399 (6)	11.2 (10)
C-25	0.7791 (13)	0.3563 (24)	−0.1907 (7)	12.3 (10)
C-26	0.6210 (19)	0.303 (3)	−0.2391 (10)	8.0 (5)
C-26′	0.661 (4)	0.194 (7)	−0.2104 (22)	13.8 (13)
C-27	0.9041 (19)	0.216 (3)	−0.1342 (10)	8.8 (5)
C-27′	0.827 (3)	0.160 (4)	−0.1428 (15)	8.2 (7)
C-28	0.8090 (21)	0.649 (3)	−0.1546 (12)	9.9 (6)
C-28′	0.783 (3)	0.614 (5)	−0.2060 (17)	9.3 (8)
C-29	0.8548 (11)	−0.1026 (12)	0.6050 (5)	6.2 (5)
O-1	0.9344 (6)	0.2191 (9)	0.6696 (3)	5.9 (3)
O-2	0.6256 (6)	0.5693 (0)	0.3598 (3)	5.5 (3)
O-3	0.5171 (18)	0.341 (3)	−0.2187 (9)	13.5 (5)
O-4	0.6058 (12)	0.2102 (19)	−0.2891 (7)	8.6 (3)
O-3′	0.570 (3)	0.336 (5)	−0.2727 (18)	18.1 (11)
O-4′	0.666 (3)	0.098 (5)	−0.2534 (17)	17.9 (11)
H-1A	0.722	0.533	0.485	5.1
H-1B	0.872	0.442	0.498	5.1
H-2A	0.825	0.493	0.617	5.7
H-2B	0.675	0.407	0.595	5.7
H-4	0.680	0.062	0.570	5.1
H-5	0.893	0.130	0.490	4.6
H-6A	0.653	−0.066	0.428	5.5
H-6B	0.799	−0.161	0.463	5.5
H-7A	0.898	−0.046	0.368	5.9
H-7B	0.745	−0.102	0.321	5.9
H-12A	0.838	0.538	0.269	5.4
H-12B	0.687	0.605	0.234	5.4
H-14	0.904	0.241	0.265	5.1
H-15A	0.715	−0.027	0.197	7.1
H-15B	0.881	−0.039	0.214	7.1
H-16A	0.725	0.092	0.083	7.2
H-16B	0.887	0.110	0.105	7.2
H-17	0.874	0.399	0.150	6.1
H-18A	0.527	0.291	0.237	6.5
H-18B	0.554	0.172	0.167	6.5
H-18C	0.512	0.379	0.154	6.5
H-19A	0.515	0.147	0.418	6.5
H-19B	0.504	0.356	0.427	6.5
H-19C	0.533	0.236	0.501	6.5
H-20	0.609	0.378	0.049	7.0
H-21A	0.617	0.651	0.118	10.1
H-21B	0.628	0.689	0.033	10.1
H-21C	0.768	0.694	0.097	10.1
H-22A	0.875	0.485	0.012	8.7
H-22B	0.817	0.294	−0.001	8.7
H-23A	0.659	0.580	−0.067	10.4
H-23B	0.617	0.384	−0.084	10.4

[a]Beq is the mean of the principal axes of the thermal ellipsoid.

disordered with large thermal parameters. The atomic coordinates are given in Table 4. The high values RF and Rw are attributed to the disorder on the side-chain (eq. C-26, C-27, C-28, O-3, and O-4).

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