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Crystal structure of the cytotoxic macrocyclic trichothecene Isororidin A

Mostafa A. Asmaey,[a,b] Dimitris A. Kalofolias,[c] Maria-Despoina Charavgi,[c] Ismail R. Abdel-Rahim,[d] Evangelia D. Chrysina[c]* and Dennis Abatis[a]*

[a]Division of Pharmacognosy and Natural Products Chemistry, Department of Pharmacy, School of Health Sciences, National and Kapodistrian University of Athens, Athens 15771, Greece, [b]Department of Chemistry, Faculty of Science, Al-Azhar University, Assiut branch, Assiut 71524, Egypt, [c]Institute of Chemical Biology, National Hellenic Research Foundation, 48 Vassileos Constantinou Avenue, Athens 11635, Greece, and [d]Botany and Microbiology Department, Faculty of Science, Assiut University, Egypt. *Correspondence e-mail: echrysina@eie.gr, abatis@pharm.uoa.gr

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The highly cytotoxic macrocyclic trichothecene Isororidin A ($C_{29}H_{40}O_9$) was isolated from the fungus *Myrothesium verrucaria* endophytic on the wild medicinal plant 'Datura' (*Datura stramonium* L.) and was characterized by one- (1D) and two-dimensional (2D) NMR spectroscopy. The three-dimensional structure of Isororidin A has been confirmed by X-ray crystallography at 0.81 Å resolution from crystals grown in the orthorhombic space group $P2_12_12_1$, with one molecule per asymmetric unit. Isororidin A is the epimer of previously described (by X-ray crystallography) Roridin A at position C-13′ of the macrocyclic ring.

1. Introduction

Macrocyclic trichothecenes (MTs) constitute the second major group (the other being the simple trichothecenes) of a class of highly functionalized sesquiterpenoid secondary metabolites, mainly of fungal origin, which are well known for their severe toxicity to both animals and humans (Grove, 2007; Shank *et al.*, 2011; Wu *et al.*, 2017). Most trichothecenes are at least tetracyclic, as they contain a spiro-epoxide group in the 12–13 position of the 'trichothecane' sesquiterpene skeleton. They also usually comprise a double bond at C9—C10; thus, they are considered as 12–13 epoxy-trichothec-9-ene derivatives [see (*a*) in Scheme 1]. In MTs, an extra cyclic diester or triester ring is connected to the trichothecene core skeleton at C-4 and C-15, making them pentacyclic macrolides. The presence of the spiro-epoxy group, the $\Delta^{9,10}$ bond and the macrocyclic ring in the molecule appear to be crucial for their biological properties, which include antifungal, antimalarial, antivirus and anticancer activity (de Carvalho *et al.*, 2015; Jarvis & Mazzola, 1982; McCormick *et al.*, 2011; Wu *et al.*, 2017). The MTs are further classified into the subgroups of the Roridoids, to which Roridin A and Isororidin A belong [see (*b*) and (*c*) in Scheme 1, respectively], the Baccharinoids, the Verrucaroids and the Trichoverroids, which are considered the biosynthetic precursors of the three former subgroups of MTs (Bräse *et al.*, 2009).

There has been a series of articles since the 1980s concerning the elucidation of the configuration of the stereogenic centres of the macrocyclic ring of the MTs, especially C-6′ and C-13′ in the Roridoids (Jarvis *et al.*, 1982, 1987, 1996; Jarvis & Wang, 1999). The task was based mainly on NMR spectroscopy (despite the technical limitations of the method at that time), as well as chemical manipulations when there were adequate quantities available, aided – in rare cases – by

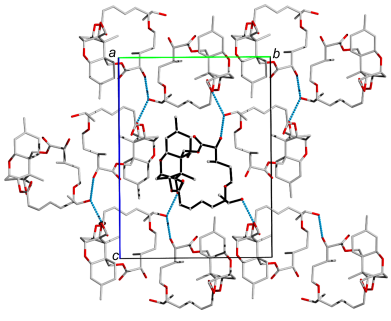


Table 1
NMR spectroscopic data for Isororidin A [400 (^{1}H) and 100 MHz (^{13}C), δ ppm][a].

Position (Scheme 1)	^{1}H NMR (*J* in Hz)	^{13}C NMR	COSY	HMBC	NOESY
2	3.74 (*d*, 5.1)	80.4	3b	4, 5, 12	3′, 13a
3	b: 2.14 (overlap by H-3′)	35.7	2, 4	2, 4	
	a: 2.47 (*dd*, 8.2, 15.2)			2, 5, 12	
4	5.84 (*dd*, 4.5, 8.2)	76.0	3	2, 3, 5, 6, 12, 11′	11
5	Cq	50.5			
6	Cq	45.0			
7	1.87 (*m*, 2H)	21.3	8	6, 8, 9, 11	13, 14
8	a: 1.93 (*d*, 8.0)	28.7	7	6,7, 9, 10	
	b: 1.98 (*m*)				
9	Cq	141.7			
10	5.41 (*d*, 5.4)	119.7	11, 16	6, 8, 11, 16	
11	3.72 (*br d*, 5.4)	68.5	10	7, 10, 15	4
12	Cq	66.4			
13	2.86 (*d*, 4.0)	48.5		2, 5, 12	14
	3.05 (*d*, 4.0)				
14	0.81 (*s*)	8.0	,	4, 5, 6, 12	2′, 3′, 15, 12′
15	4.32 (*d*, 12.2)	64.8	15	5, 6, 7, 1′	14
	4.46 (*d*, 12.2)			5, 6, 7, 11, 1′	
16	1.72 (*s*)	23.3	10	8, 9, 10	
1′	CO	175.6			
2′	4.04 (*d*, 4.0)	76.7	3′	1′, 4′, 12′	14, 3′, 12′
3′	2.08 (*m*	37.7	2′, 12′	1′, 2′	14, 2′
4′	1.58 (*m*)	34.9	4′, 5′	3′, 5′, 12′	
	1.73 (*m*)			2′, 3′, 5′	
5′	3.50 (*ddd*, 5.2, 8.7, 9.1)	70.9	4′, 5′	3′, 4′, 6′	
	3.58 (*ddd*, 5.2, 9.6, 9.8)				
6′	3.82 (*m*)	84.6	7′, 13′	5′, 7′, 8′, 14′	8′, 14′
7′	6.17 (*dd*, 3.0, 15.4)	142.3	6′, 8′	6′, 8′, 9′	13′, 14′
8′	7.60 (*ddt*, 11.4, 15.4, 1.1)	126.8	7′, 9′	6′, 9′, 10′	14, 3′, 10′, 12′
9′	6.75 (*t*, 11.4)	145.5	8′, 10′	7′, 8′, 11′	7′
10′	5.76 (*d*, 11.2)	117.9	9′	8′, 9′, 11′	14
11′	CO	168.1			
12′	1.09 (*d*, 6.8)	15.1	3′	2′, 3′, 4′	14, 2′, 3′, 8′
13′	3.69 (*m*)	71.0	6′, 14′	6′, 14′	7′, 8′, 14′
14′	1.16 (*d*, 6.4)	18.4	13′	6′, 13′	6′, 7′, 8′,13′

Note: (*a*) the assignments were based on ^{1}H–^{1}H COSY, HSQC–DEPT and HMBC experiments, and recorded in MeOD-d_4.

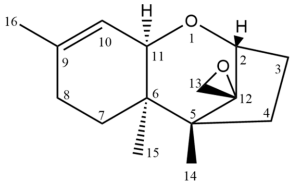
stereoselective synthesis and X-ray diffraction analyses. In 1982, Jarvis and co-workers isolated Roridin A and Isororidin A from a large-scale fermentation of *Myrothesium verrucaria* and resolved the relative configuration of Roridin A by X-ray crystallography. The absolute configuration of Roridin A was confirmed after oxidative cleavage of its hydroxyethyl moiety, which produced Verrucarin A, an MT whose absolute configuration had already been established (Jarvis *et al.*, 1982). The ^{1}H and ^{13}C NMR spectra of Roridin A and Isororidin A in $CDCl_3$ were almost identical, except for carbon C-6′, which differed in the ^{13}C NMR spectra by 1.1 ppm. The epimeric relation of the two fungal metabolites at C-13′ was deduced indirectly by the selective hydrogenation of Roridin A and Isororidin A to their respective tetrahydro derivatives, and then oxidation of the C-6′ hydroxyethyl group of these tetrahydro derivatives to an identical (in the ^{1}H NMR spectrum) corresponding methyl ketone (Jarvis *et al.*, 1982). Even though Isororidin A was re-isolated a few times in subsequent years from different fungal strains and by different research groups, verification of its structure was performed only by comparison of the NMR data in $CDCl_3$ with those reported in 1982, but without submitting the NMR data. Isororidin A is one of the most cytotoxic metabolites among all compounds containing C, H and O, and was on the shortlist of the National Cancer Institute (NCI) for the most promising anticancer agents in the 2000s (Amagata *et al.*, 2003; de Carvalho *et al.*, 2015; Sy-Cordero *et al.*, 2010). The mechanism of action of the macrocyclic trichothecenes is still underexplored, possibly due to their severe general cytotoxicity. However, there is evidence that MTs show large variations in both activity and selectivity against different cancer cell lines induced by alterations in their molecular structure. These findings indicate that MTs may still be considered as highly promising antitumour agents, as long as more detailed structure–activity relationship (SAR) and quantitative structure–activity relationship (QSAR) studies have been performed. For these studies, knowledge of the configuration and conformation of the MTs is undoubtedly critical (Wu *et al.*, 2017; Zhu *et al.*, 2020). In the current article, Isororidin A was isolated from the fungus *Myrothesium verrucaria* endophytic on the wild medicinal plant 'Datura' (*Datura stramonium* L.) and was characterized by 1D and 2D NMR spectroscopy. Its crystal structure is presented for the first time at 0.81 Å resolution.

2. Experimental

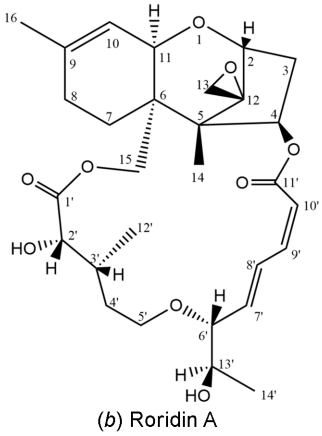
2.1. Isolation and crystallization

Isororidin A was isolated as a colourless solid (19.5 mg) after high-performance liquid chromatography (HPLC) using

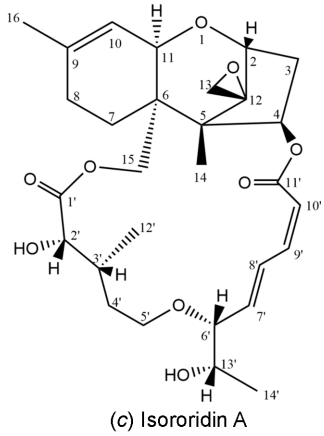
a semipreparative C18 column eluted with a linear gradient mixture of water and methanol. The gross structure of the compound was elucidated on the basis of a detailed analysis of its 1D/2D NMR and high-resolution mass spectroscopic (HRMS) data. The full 1D and 2D NMR data recorded in CD_3OD are reported for the first time (see the *Analytical data* section in the supporting information and Table 1). The relative configuration of its chiral centres was deduced from a combined study of nuclear Overhauser effect (NOE) correlations and $^3J_{HH}$ coupling constants, and by comparison with the NMR data of other Roridoids having similar structures (Amagata *et al.*, 2003; Jarvis & Wang, 1999). The absolute configuration of all its chiral centres was confirmed by the X-ray crystallographic analysis of its colourless needle-like crystals that were obtained after the slow evaporation of a solution in methanol from an NMR tube. Most of the Isororidin A crystals had morphological defects that may have led to twinned spots on the diffraction pattern and potential issues at the stage of processing and deconvolution. Therefore, a small fragment of an Isororidin A crystal, with the least morphological defects, was isolated and mounted on a litho loop to minimize the background contribution when exposed to X-rays. The loop was placed on the goniometer head and diffraction data were collected at 0.81 Å resolution.



(*a*) 12-13 epoxytrichothec-9-ene core



(*b*) Roridin A



(*c*) Isororidin A

Scheme 1

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Details of the geometry of the Isororidin A crystal structure regarding bond lengths (Å), bond angles (°), torsion angles (°) and the geometry of the hydrogen bonds [distances (Å) and angles (°)] are presented in the supporting information (Tables S1–S5).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{29}H_{40}O_9$
M_r	532.61
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	9.2707 (4), 15.2236 (6), 20.0806 (8)
V (Å^3)	2834.0 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.76
Crystal size (mm)	0.08 × 0.06 × 0.04
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2021)
T_{min}, T_{max}	0.673, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	101717, 5548, 5338
R_{int}	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.109, 1.07
No. of reflections	5548
No. of parameters	349
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.22
Absolute structure	Flack x determined using 2264 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.02 (3)

Computer programs: *APEX2* (Bruker, 2021), *SAINT* (Bruker, 2021), *SORTAV* (Blessing, 1995), *SHELXT* (Sheldrick, 2015*a*) and *SHELXL2018* (Sheldrick, 2015*b*).

3. Results and discussion

3.1. Structural commentary

The crystal structure of Isororidin A, isolated from the ethyl acetate extract of the culture broth of the endophytic fungus *M. verrucaria*, after a series of chromatographic separations, is presented at 0.81 Å resolution and confirms the configuration at position C13′. Isororidin A crystallized in the orthorhombic space group $P2_12_12_1$ (No. 19). A data set was initially collected at room temperature at 0.81 Å resolution (Table S1 in the supporting information) and the calculated Flack parameter (Flack, 1983; Parsons *et al.*, 2013) was 0.4 (4), which was not sufficient to assess the absolute configuration of Isororidin A. Therefore, a new data set was collected at 100 K. The crystal lattice and space group remained $P2_12_12_1$, with unit-cell dimensions a = 9.2707 (4), b = 15.2236 (6), c = 20.0806 (8) Å and $\alpha = \beta = \gamma = 90°$, and the Flack parameter calculated for this structure was −0.02 (3), confirming the absolute configuration of Isororidin A. The experimental details are summarized in Table 2 and Tables S2–S6 of the supporting information. The two experiments reveal no temperature-dependent phase change, as the unit-cell parameters are almost identical (Table 2 and Table S1). The measurement at 100 K resulted in an overall better data set with an improved R parameter and a higher precision Flack parameter. Therefore, the structure analysis that follows focuses on the structure determined at 100 K.

Table 3
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O8^{i}	0.84	1.94	2.761 (3)	167
O8—H8···O2^{ii}	0.84	2.10	2.895 (3)	158
C4—H4···O1^{iii}	1.00	2.55	3.467 (3)	153
C13—H13B···O9^{iv}	0.99	2.65	3.490 (3)	143
C7′—H7′···O6^{v}	0.95	2.62	3.473 (3)	150

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iv) $x+1, y, z$; (v) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.

The packing of the molecules is stabilized by two intermolecular hydrogen-bond interactions between atom O7, which acts as a donor to symmetry-related O8^{i} and O8, which acts as a donor to symmetry-related O2^{ii}, as well as intermolecular C—H···O interactions between C4 and O1^{iii}, C13 and O9^{iv}, and C7′ and O6^{v} (see Table 3 for symmetry codes). A schematic representation of the crystal structure, showing the stereoconfiguration of Isororidin A and its packing within the unit cell, is presented in Fig. 1.

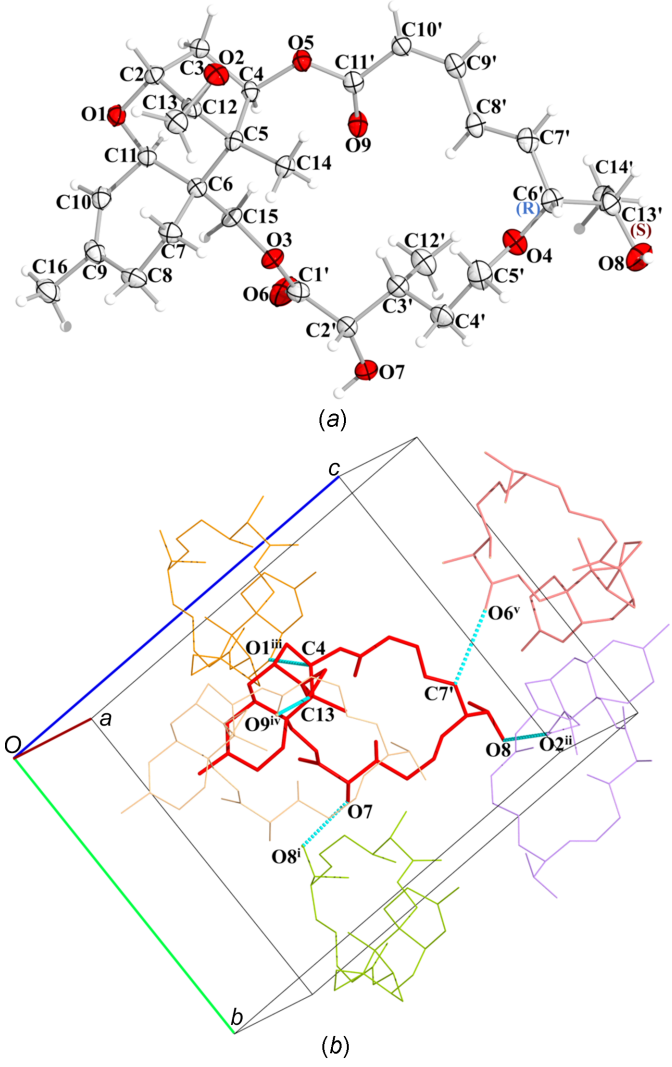


Figure 1
(*a*) Schematic representation of the Isororidin A X-ray diffraction solution, drawn with 50% probability displacement ellipsoids. O atoms are shown in red, C atoms in light grey and H atoms in pale pink. The absolute configurations of C6′ (*R*) and C13′ (*S*) shown in Scheme 1 are indicated. (*b*) A view of the intermolecular hydrogen-bond interactions formed between Isororidin A (shown in red) and its symmetry-related molecules [colour code for symmetry codes: $(x+\frac{1}{2}, -y+\frac{3}{2}, -z+1)$ in lime, $(-x+1, y+\frac{1}{2}, -z+\frac{3}{2})$ in lavender, $(x-\frac{1}{2}, -y+\frac{1}{2}, -z+1)$ in orange, $(x+1, y, z)$ in tan and $(-x+\frac{1}{2}, -y+1, z+\frac{1}{2})$ in salmon], while the hydrogen bonding is indicated with blue dashed lines.

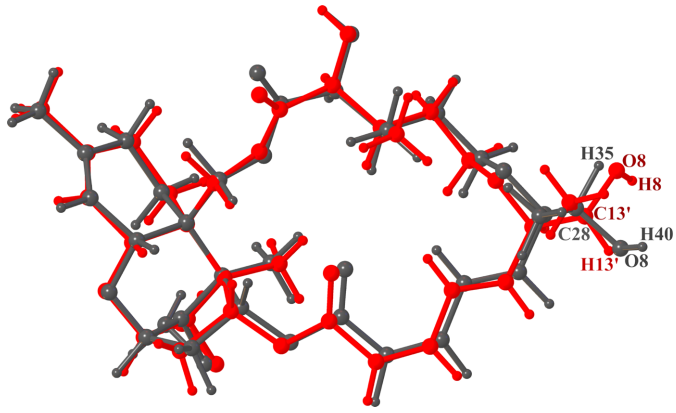


Figure 2
Superposition of the three-dimensional structures of determined Isororidin A (with an *S* configuration at C13′) and its stereoisomer (epimeric at C28 with an *R* configuration) Roridin A. Isororidin A is shown in red and Roridin A in grey.

Superposition of the crystal structure of Isororidin A with the only available previously determined structure of Roridin A (CCDC deposition No. 1110357, CSD refcode BIDPIN10; Jarvis *et al.*, 1982) showed that the overall structure is the same; more pronounced differences are observed in the macrocyclic ring, more specifically, in the vicinity of the C13′ atom [Figs. 1(*a*) and 2]. Both saturated pyran rings adopt distorted chair conformations, with a torsion angle C5—C6—

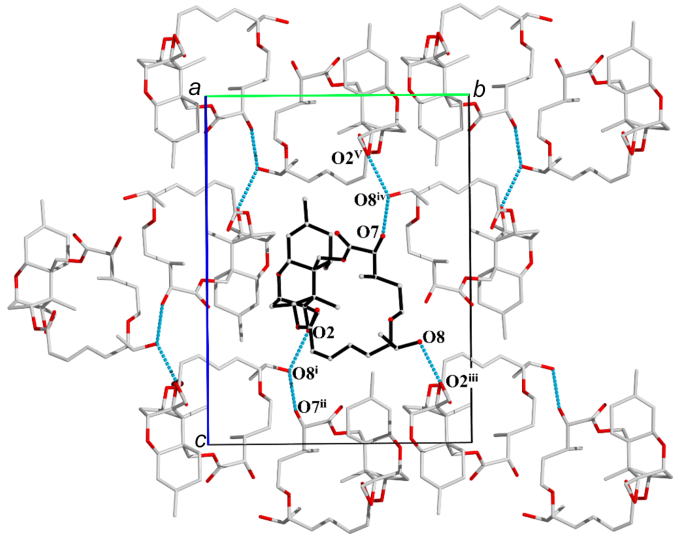


Figure 3
Schematic representation of the supramolecular structure of Isororidin A. The asymmetric unit is highlighted in black and the hydrogen bonds are indicated in blue. [Symmetry codes: (i) $-x+$, $y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (v) $-x+\frac{3}{2}, -y+1, z-\frac{1}{2}$.]

C11—O1 of −46.7 (2)° in Isororidin A *versus* −41.5° in Roridin A. The unsaturated cyclohexene rings adopt flattened half-chair conformations, while the five-membered rings in both structures adopt envelope conformations, with the C atom at position C12 (C11 for the Roridin A structure) pointing out of the plane. The differences observed between the two structures relate to the hydroxyethyl group and neighbouring atoms that include a significant rotation of the torsion angles O4—C6′—C13′—O8 and C7′—C6′—C13′—O8 by 106.7 and 104.6°, respectively. Additional differences are observed for torsion angles O7—C2′—C3′—C4′ by 18.4°, O7—C2′—C3′—C12′ by 17.2°, O9—C11′—C10′—C9′ by 17.7°, O5—C11′—C10′—C9′ by 16.2°, C7′—C6′—C13′—C14′ by 10.9°, O4—C6′—C13′—C14′ by 9.2° and O6—C1′—C2′—C3′ by 7.3°. The rest of the differences in the torsion angles observed in the 18-membered macrocyclic ring are less profound and in the range of 5°; for example, torsion angle O6—C1′—C2′—O7′ by 4.2° (Table S7 in the supporting information). These changes may be attributed to the intermolecular interactions formed in Isororidin A compared to Roridin A [Fig. 1(*b*)].

3.2. Supramolecular features

A schematic representation of the structure of Isororidin A and its packing with symmetry-related molecules within the crystal is shown in Fig. 3. Isororidin A crystallized in the orthorhombic space group $P2_12_12_1$. The difference observed in the epimeric C atom seems to foster the intermolecular interactions within the unit cell. Atom O8 is hydrogen bonded to O2 of a symmetry-related molecule within the unit cell, while in the case of Roridin A, the same atom interacts with O1.

3.3. Database survey

One entry is available in the Cambridge Structural Database (CSD; Groom *et al.*, 2016) for the structure of Roridin A (CCDC deposition No. 1110357, CSD refcode BIDPIN10; Jarvis *et al.*, 1982) determined in the space group $P2_1$ with unit-cell dimensions a = 10.197 (3), b = 14.079 (4), c = 9.606 (2) Å, $\alpha = \gamma = 90°$ and β = 94.6 (1)°.

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supporting information

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Crystal structure of the cytotoxic macrocyclic trichothecene Isororidin A

Mostafa A. Asmaey, Dimitris A. Kalofolias, Maria-Despoina Charavgi, Ismail R. Abdel-Rahim, Evangelia D. Chrysina and Dennis Abatis

Computing details

(I)

Crystal data

$C_{29}H_{40}O_9$
M_r = 532.61
Orthorhombic, $P2_12_12_1$
a = 9.2707 (4) Å
b = 15.2236 (6) Å
c = 20.0806 (8) Å
V = 2834.0 (2) Å^3
Z = 4
$F(000)$ = 1144
D_x = 1.248 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9959 reflections
θ = 4.4–74.9°
μ = 0.76 mm^{-1}
T = 100 K
Irregular, colourless
0.08 × 0.06 × 0.04 mm

Data collection

Bruker APEXII diffractometer
Radiation source: sealed x-ray tube
Graphite monochromator
φ or ω oscillation scans
Absorption correction: multi-scan (SADABS; Bruker, 2021)
T_{min} = 0.673, T_{max} = 0.754
101717 measured reflections
5548 independent reflections
5338 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 72.1°, θ_{min} = 3.6°
h = −11→11
k = −18→18
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.07
5548 reflections
349 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 1.0952P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack x determined using 2264 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.02 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.88908 (18)	0.27189 (12)	0.51273 (8)	0.0240 (4)
O2	0.9025 (2)	0.38349 (12)	0.67471 (9)	0.0272 (4)
O3	0.55743 (19)	0.51939 (11)	0.47181 (9)	0.0247 (4)
O4	0.1743 (2)	0.69785 (13)	0.64506 (9)	0.0308 (4)
O5	0.55825 (19)	0.34501 (11)	0.66449 (8)	0.0220 (4)
O6	0.3838 (2)	0.49608 (15)	0.39552 (11)	0.0424 (5)
O7	0.2988 (2)	0.66973 (14)	0.40501 (10)	0.0397 (5)
H7	0.337967	0.671580	0.367303	0.060*
O8	−0.0389 (2)	0.80771 (13)	0.70903 (10)	0.0361 (5)
H8	0.003475	0.841615	0.735599	0.054*
O9	0.37443 (19)	0.41830 (12)	0.61579 (9)	0.0269 (4)
C2	0.8665 (3)	0.27417 (17)	0.58343 (12)	0.0239 (5)
H2	0.941895	0.239374	0.607361	0.029*
C3	0.7140 (3)	0.24545 (15)	0.60435 (12)	0.0237 (5)
H3A	0.714192	0.222712	0.650543	0.028*
H3B	0.677134	0.199050	0.574293	0.028*
C4	0.6207 (3)	0.32912 (15)	0.59909 (11)	0.0198 (5)
H4	0.543332	0.321485	0.564896	0.024*
C5	0.7257 (2)	0.40513 (16)	0.57878 (12)	0.0189 (5)
C6	0.7370 (3)	0.40627 (15)	0.50005 (11)	0.0194 (5)
C7	0.8563 (3)	0.46869 (17)	0.47578 (13)	0.0255 (5)
H7A	0.835163	0.528884	0.491673	0.031*
H7B	0.949524	0.450303	0.495403	0.031*
C8	0.8701 (3)	0.46984 (18)	0.39997 (13)	0.0301 (6)
H8A	0.790742	0.505592	0.381129	0.036*
H8B	0.962284	0.498322	0.387684	0.036*
C9	0.8652 (3)	0.37965 (19)	0.36967 (12)	0.0280 (6)
C10	0.8205 (3)	0.31031 (17)	0.40345 (12)	0.0250 (5)
H10	0.818015	0.255442	0.380946	0.030*
C11	0.7731 (3)	0.31230 (16)	0.47532 (11)	0.0209 (5)
H11	0.684913	0.275005	0.480016	0.025*
C12	0.8689 (3)	0.36850 (17)	0.60468 (12)	0.0226 (5)
C13	0.9983 (3)	0.4175 (2)	0.62296 (13)	0.0289 (6)
H13A	0.998626	0.481667	0.614993	0.035*
H13B	1.092774	0.388245	0.616854	0.035*
C14	0.6885 (3)	0.49472 (15)	0.60854 (13)	0.0228 (5)
H14A	0.758586	0.538439	0.593183	0.034*
H14B	0.691533	0.491081	0.657245	0.034*
H14C	0.591501	0.512065	0.594273	0.034*

C15	0.5923 (3)	0.42707 (16)	0.46636 (12)	0.0221 (5)
H15A	0.597356	0.410455	0.418753	0.027*
H15B	0.515051	0.391867	0.487463	0.027*
C16	0.9125 (3)	0.3731 (2)	0.29815 (14)	0.0380 (7)
H16A	1.014581	0.389273	0.294656	0.057*
H16B	0.854565	0.413067	0.270815	0.057*
H16C	0.899321	0.312700	0.282417	0.057*
C1′	0.4428 (3)	0.54367 (19)	0.43490 (13)	0.0296 (6)
C2′	0.3983 (3)	0.63724 (18)	0.45204 (13)	0.0287 (6)
H2′	0.485817	0.675635	0.452173	0.034*
C3′	0.3294 (3)	0.63843 (18)	0.52177 (14)	0.0291 (6)
H3′	0.395944	0.606240	0.552451	0.035*
C4′	0.3149 (4)	0.7322 (2)	0.54813 (16)	0.0407 (7)
H4′A	0.399591	0.766598	0.533323	0.049*
H4′B	0.228023	0.759272	0.528101	0.049*
C5′	0.3034 (4)	0.7386 (2)	0.62343 (17)	0.0407 (7)
H5′A	0.387367	0.709419	0.644329	0.049*
H5′B	0.303563	0.801101	0.637090	0.049*
C6′	0.1495 (3)	0.69731 (18)	0.71470 (13)	0.0274 (5)
H6′	0.209471	0.744725	0.735249	0.033*
C7′	0.1896 (3)	0.61153 (17)	0.74684 (13)	0.0277 (5)
H7′	0.166965	0.605138	0.792725	0.033*
C8′	0.2536 (3)	0.54364 (17)	0.71731 (13)	0.0265 (5)
H8′	0.270664	0.545400	0.670677	0.032*
C9′	0.2979 (3)	0.46714 (17)	0.75504 (13)	0.0281 (5)
H9′	0.264277	0.463987	0.799677	0.034*
C10′	0.3807 (3)	0.40016 (17)	0.73433 (12)	0.0267 (5)
H10′	0.406954	0.356344	0.765766	0.032*
C11′	0.4333 (3)	0.39102 (15)	0.66526 (12)	0.0231 (5)
C12′	0.1877 (3)	0.5878 (2)	0.51995 (15)	0.0372 (6)
H12A	0.122060	0.615490	0.487947	0.056*
H12B	0.143561	0.588379	0.564304	0.056*
H12C	0.206171	0.526987	0.506428	0.056*
C13′	−0.0095 (3)	0.71915 (18)	0.72682 (13)	0.0298 (6)
H13′	−0.031032	0.711192	0.775237	0.036*
C14′	−0.1081 (3)	0.65997 (19)	0.68701 (15)	0.0334 (6)
H14D	−0.208572	0.677363	0.694432	0.050*
H14E	−0.094561	0.598944	0.701241	0.050*
H14F	−0.085000	0.665237	0.639549	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (8)	0.0305 (9)	0.0202 (8)	0.0080 (7)	0.0008 (7)	0.0009 (7)
O2	0.0247 (9)	0.0368 (10)	0.0201 (8)	−0.0014 (8)	−0.0056 (7)	0.0005 (7)
O3	0.0238 (8)	0.0235 (8)	0.0268 (9)	0.0042 (7)	−0.0031 (7)	0.0025 (7)
O4	0.0297 (10)	0.0358 (10)	0.0268 (9)	−0.0051 (8)	0.0036 (8)	−0.0023 (8)
O5	0.0215 (8)	0.0251 (8)	0.0194 (8)	0.0011 (7)	0.0014 (7)	−0.0021 (7)

O6	0.0451 (12)	0.0461 (12)	0.0359 (11)	0.0143 (10)	−0.0180 (10)	−0.0071 (9)
O7	0.0404 (11)	0.0464 (12)	0.0324 (10)	0.0195 (10)	0.0088 (9)	0.0164 (9)
O8	0.0454 (12)	0.0303 (9)	0.0326 (10)	0.0084 (9)	−0.0132 (9)	−0.0085 (8)
O9	0.0213 (8)	0.0327 (9)	0.0268 (9)	0.0046 (7)	−0.0003 (8)	−0.0053 (8)
C2	0.0220 (12)	0.0292 (13)	0.0205 (11)	0.0070 (10)	−0.0013 (9)	0.0027 (10)
C3	0.0282 (12)	0.0215 (12)	0.0216 (11)	0.0034 (10)	0.0038 (10)	0.0021 (9)
C4	0.0208 (11)	0.0216 (11)	0.0171 (11)	0.0007 (9)	0.0011 (9)	−0.0025 (8)
C5	0.0165 (10)	0.0210 (11)	0.0192 (11)	0.0011 (9)	−0.0027 (9)	−0.0016 (9)
C6	0.0180 (11)	0.0217 (11)	0.0184 (11)	−0.0007 (9)	−0.0013 (9)	−0.0004 (9)
C7	0.0232 (12)	0.0268 (12)	0.0265 (13)	−0.0043 (10)	−0.0015 (10)	0.0020 (10)
C8	0.0286 (13)	0.0345 (14)	0.0272 (13)	−0.0061 (11)	0.0013 (11)	0.0074 (11)
C9	0.0228 (12)	0.0406 (15)	0.0206 (12)	0.0018 (11)	−0.0005 (10)	0.0004 (10)
C10	0.0237 (12)	0.0300 (12)	0.0212 (12)	0.0055 (11)	−0.0008 (10)	−0.0033 (10)
C11	0.0194 (11)	0.0237 (11)	0.0196 (11)	0.0023 (9)	0.0002 (9)	−0.0009 (9)
C12	0.0197 (11)	0.0314 (13)	0.0167 (11)	0.0022 (10)	−0.0012 (9)	0.0019 (9)
C13	0.0196 (11)	0.0413 (15)	0.0258 (12)	−0.0022 (11)	−0.0042 (10)	0.0028 (11)
C14	0.0221 (11)	0.0212 (11)	0.0252 (12)	−0.0005 (9)	−0.0014 (10)	−0.0034 (9)
C15	0.0211 (12)	0.0213 (11)	0.0240 (12)	0.0006 (9)	−0.0042 (9)	−0.0003 (9)
C16	0.0398 (17)	0.0503 (17)	0.0239 (13)	0.0020 (14)	0.0043 (12)	0.0043 (12)
C1′	0.0300 (13)	0.0345 (14)	0.0243 (13)	0.0043 (12)	0.0010 (11)	0.0051 (11)
C2′	0.0276 (13)	0.0294 (13)	0.0289 (13)	0.0047 (11)	0.0065 (10)	0.0065 (10)
C3′	0.0295 (13)	0.0306 (13)	0.0272 (13)	0.0022 (11)	0.0054 (11)	0.0037 (10)
C4′	0.0431 (17)	0.0292 (14)	0.0498 (18)	−0.0002 (14)	0.0197 (15)	0.0049 (13)
C5′	0.0394 (16)	0.0319 (15)	0.0506 (18)	−0.0101 (13)	0.0124 (14)	−0.0109 (13)
C6′	0.0306 (13)	0.0275 (12)	0.0242 (12)	−0.0018 (11)	−0.0019 (10)	−0.0061 (10)
C7′	0.0275 (13)	0.0318 (13)	0.0238 (12)	0.0003 (11)	−0.0022 (10)	−0.0052 (10)
C8′	0.0253 (12)	0.0294 (13)	0.0247 (12)	−0.0017 (10)	0.0008 (10)	−0.0056 (10)
C9′	0.0261 (12)	0.0326 (13)	0.0256 (12)	−0.0030 (11)	0.0057 (10)	−0.0040 (11)
C10′	0.0283 (12)	0.0273 (12)	0.0245 (12)	−0.0005 (11)	0.0048 (10)	−0.0003 (10)
C11′	0.0222 (12)	0.0211 (11)	0.0259 (12)	−0.0040 (10)	0.0024 (10)	−0.0039 (10)
C12′	0.0342 (15)	0.0449 (16)	0.0325 (14)	−0.0029 (14)	0.0073 (12)	0.0003 (12)
C13′	0.0366 (14)	0.0313 (14)	0.0215 (12)	0.0046 (12)	0.0012 (11)	−0.0049 (10)
C14′	0.0300 (14)	0.0328 (14)	0.0373 (15)	−0.0024 (12)	0.0009 (11)	−0.0014 (11)

Geometric parameters (Å, º)

O1—C2	1.436 (3)	C12—C13	1.460 (4)
O1—C11	1.449 (3)	C13—H13A	0.9900
O2—C12	1.458 (3)	C13—H13B	0.9900
O2—C13	1.462 (3)	C14—H14A	0.9800
O3—C1′	1.347 (3)	C14—H14B	0.9800
O3—C15	1.446 (3)	C14—H14C	0.9800
O4—C5′	1.416 (4)	C15—H15A	0.9900
O4—C6′	1.417 (3)	C15—H15B	0.9900
O5—C11′	1.354 (3)	C16—H16A	0.9800
O5—C4	1.456 (3)	C16—H16B	0.9800
O6—C1′	1.204 (4)	C16—H16C	0.9800
O7—C2′	1.409 (3)	C1′—C2′	1.523 (4)

O7—H7	0.8400	C2′—C3′	1.539 (4)
O8—C13′	1.421 (3)	C2′—H2′	1.0000
O8—H8	0.8400	C3′—C12′	1.524 (4)
O9—C11′	1.207 (3)	C3′—C4′	1.528 (4)
C2—C12	1.498 (4)	C3′—H3′	1.0000
C2—C3	1.538 (3)	C4′—C5′	1.519 (4)
C2—H2	1.0000	C4′—H4′A	0.9900
C3—C4	1.543 (3)	C4′—H4′B	0.9900
C3—H3A	0.9900	C5′—H5′A	0.9900
C3—H3B	0.9900	C5′—H5′B	0.9900
C4—C5	1.566 (3)	C6′—C7′	1.503 (4)
C4—H4	1.0000	C6′—C13′	1.530 (4)
C5—C14	1.528 (3)	C6′—H6′	1.0000
C5—C12	1.531 (3)	C7′—C8′	1.331 (4)
C5—C6	1.584 (3)	C7′—H7′	0.9500
C6—C15	1.535 (3)	C8′—C9′	1.449 (4)
C6—C7	1.538 (3)	C8′—H8′	0.9500
C6—C11	1.551 (3)	C9′—C10′	1.343 (4)
C7—C8	1.528 (4)	C9′—H9′	0.9500
C7—H7A	0.9900	C10′—C11′	1.477 (3)
C7—H7B	0.9900	C10′—H10′	0.9500
C8—C9	1.502 (4)	C12′—H12A	0.9800
C8—H8A	0.9900	C12′—H12B	0.9800
C8—H8B	0.9900	C12′—H12C	0.9800
C9—C10	1.321 (4)	C13′—C14′	1.512 (4)
C9—C16	1.505 (4)	C13′—H13′	1.0000
C10—C11	1.509 (3)	C14′—H14D	0.9800
C10—H10	0.9500	C14′—H14E	0.9800
C11—H11	1.0000	C14′—H14F	0.9800

C2—O1—C11	113.23 (17)	O3—C15—C6	111.26 (19)
C12—O2—C13	59.98 (16)	O3—C15—H15A	109.4
C1′—O3—C15	113.7 (2)	C6—C15—H15A	109.4
C5′—O4—C6′	116.2 (2)	O3—C15—H15B	109.4
C11′—O5—C4	115.90 (18)	C6—C15—H15B	109.4
C2′—O7—H7	109.5	H15A—C15—H15B	108.0
C13′—O8—H8	109.5	C9—C16—H16A	109.5
O1—C2—C12	107.62 (19)	C9—C16—H16B	109.5
O1—C2—C3	113.4 (2)	H16A—C16—H16B	109.5
C12—C2—C3	102.03 (19)	C9—C16—H16C	109.5
O1—C2—H2	111.1	H16A—C16—H16C	109.5
C12—C2—H2	111.1	H16B—C16—H16C	109.5
C3—C2—H2	111.1	O6—C1′—O3	123.7 (3)
C2—C3—C4	105.16 (19)	O6—C1′—C2′	126.0 (3)
C2—C3—H3A	110.7	O3—C1′—C2′	110.3 (2)
C4—C3—H3A	110.7	O7—C2′—C1′	110.7 (2)
C2—C3—H3B	110.7	O7—C2′—C3′	109.5 (2)
C4—C3—H3B	110.7	C1′—C2′—C3′	109.2 (2)

H3A—C3—H3B	108.8	O7—C2′—H2′	109.1
O5—C4—C3	107.36 (18)	C1′—C2′—H2′	109.1
O5—C4—C5	111.07 (18)	C3′—C2′—H2′	109.1
C3—C4—C5	106.24 (19)	C12′—C3′—C4′	113.9 (2)
O5—C4—H4	110.7	C12′—C3′—C2′	109.3 (2)
C3—C4—H4	110.7	C4′—C3′—C2′	111.3 (2)
C5—C4—H4	110.7	C12′—C3′—H3′	107.4
C14—C5—C12	112.8 (2)	C4′—C3′—H3′	107.4
C14—C5—C4	114.66 (19)	C2′—C3′—H3′	107.4
C12—C5—C4	100.44 (19)	C5′—C4′—C3′	114.3 (2)
C14—C5—C6	113.28 (19)	C5′—C4′—H4′A	108.7
C12—C5—C6	106.61 (18)	C3′—C4′—H4′A	108.7
C4—C5—C6	107.99 (18)	C5′—C4′—H4′B	108.7
C15—C6—C7	111.19 (19)	C3′—C4′—H4′B	108.7
C15—C6—C11	103.77 (18)	H4′A—C4′—H4′B	107.6
C7—C6—C11	108.23 (19)	O4—C5′—C4′	109.7 (3)
C15—C6—C5	112.62 (19)	O4—C5′—H5′A	109.7
C7—C6—C5	111.73 (19)	C4′—C5′—H5′A	109.7
C11—C6—C5	108.88 (18)	O4—C5′—H5′B	109.7
C8—C7—C6	112.5 (2)	C4′—C5′—H5′B	109.7
C8—C7—H7A	109.1	H5′A—C5′—H5′B	108.2
C6—C7—H7A	109.1	O4—C6′—C7′	112.9 (2)
C8—C7—H7B	109.1	O4—C6′—C13′	108.2 (2)
C6—C7—H7B	109.1	C7′—C6′—C13′	111.0 (2)
H7A—C7—H7B	107.8	O4—C6′—H6′	108.2
C9—C8—C7	113.0 (2)	C7′—C6′—H6′	108.2
C9—C8—H8A	109.0	C13′—C6′—H6′	108.2
C7—C8—H8A	109.0	C8′—C7′—C6′	126.4 (3)
C9—C8—H8B	109.0	C8′—C7′—H7′	116.8
C7—C8—H8B	109.0	C6′—C7′—H7′	116.8
H8A—C8—H8B	107.8	C7′—C8′—C9′	121.2 (2)
C10—C9—C8	122.1 (2)	C7′—C8′—H8′	119.4
C10—C9—C16	121.9 (3)	C9′—C8′—H8′	119.4
C8—C9—C16	116.0 (2)	C10′—C9′—C8′	127.6 (2)
C9—C10—C11	124.5 (2)	C10′—C9′—H9′	116.2
C9—C10—H10	117.8	C8′—C9′—H9′	116.2
C11—C10—H10	117.8	C9′—C10′—C11′	123.5 (2)
O1—C11—C10	105.75 (18)	C9′—C10′—H10′	118.3
O1—C11—C6	112.71 (18)	C11′—C10′—H10′	118.3
C10—C11—C6	112.8 (2)	O9—C11′—O5	123.7 (2)
O1—C11—H11	108.5	O9—C11′—C10′	126.3 (2)
C10—C11—H11	108.5	O5—C11′—C10′	110.0 (2)
C6—C11—H11	108.5	C3′—C12′—H12A	109.5
O2—C12—C13	60.13 (16)	C3′—C12′—H12B	109.5
O2—C12—C2	115.3 (2)	H12A—C12′—H12B	109.5
C13—C12—C2	125.0 (2)	C3′—C12′—H12C	109.5
O2—C12—C5	117.1 (2)	H12A—C12′—H12C	109.5
C13—C12—C5	127.7 (2)	H12B—C12′—H12C	109.5

C2—C12—C5	103.9 (2)	O8—C13′—C14′	108.4 (2)
C12—C13—O2	59.90 (15)	O8—C13′—C6′	110.6 (2)
C12—C13—H13A	117.8	C14′—C13′—C6′	111.6 (2)
O2—C13—H13A	117.8	O8—C13′—H13′	108.7
C12—C13—H13B	117.8	C14′—C13′—H13′	108.7
O2—C13—H13B	117.8	C6′—C13′—H13′	108.7
H13A—C13—H13B	114.9	C13′—C14′—H14D	109.5
C5—C14—H14A	109.5	C13′—C14′—H14E	109.5
C5—C14—H14B	109.5	H14D—C14′—H14E	109.5
H14A—C14—H14B	109.5	C13′—C14′—H14F	109.5
C5—C14—H14C	109.5	H14D—C14′—H14F	109.5
H14A—C14—H14C	109.5	H14E—C14′—H14F	109.5
H14B—C14—H14C	109.5		

C11—O1—C2—C12	−65.2 (2)	O1—C2—C12—C5	72.8 (2)
C11—O1—C2—C3	46.9 (3)	C3—C2—C12—C5	−46.8 (2)
O1—C2—C3—C4	−85.5 (2)	C14—C5—C12—O2	38.1 (3)
C12—C2—C3—C4	30.0 (2)	C4—C5—C12—O2	−84.4 (2)
C11′—O5—C4—C3	−154.6 (2)	C6—C5—C12—O2	163.10 (19)
C11′—O5—C4—C5	89.7 (2)	C14—C5—C12—C13	−33.7 (3)
C2—C3—C4—O5	−121.9 (2)	C4—C5—C12—C13	−156.3 (2)
C2—C3—C4—C5	−3.0 (2)	C6—C5—C12—C13	91.2 (3)
O5—C4—C5—C14	−29.1 (3)	C14—C5—C12—C2	166.5 (2)
C3—C4—C5—C14	−145.6 (2)	C4—C5—C12—C2	44.0 (2)
O5—C4—C5—C12	92.1 (2)	C6—C5—C12—C2	−68.5 (2)
C3—C4—C5—C12	−24.3 (2)	C2—C12—C13—O2	−101.5 (3)
O5—C4—C5—C6	−156.44 (18)	C5—C12—C13—O2	102.7 (3)
C3—C4—C5—C6	87.1 (2)	C1′—O3—C15—C6	170.8 (2)
C14—C5—C6—C15	−64.8 (3)	C7—C6—C15—O3	−51.0 (3)
C12—C5—C6—C15	170.5 (2)	C11—C6—C15—O3	−167.16 (18)
C4—C5—C6—C15	63.3 (2)	C5—C6—C15—O3	75.3 (2)
C14—C5—C6—C7	61.2 (3)	C15—O3—C1′—O6	−7.6 (4)
C12—C5—C6—C7	−63.5 (2)	C15—O3—C1′—C2′	170.7 (2)
C4—C5—C6—C7	−170.71 (19)	O6—C1′—C2′—O7	−13.8 (4)
C14—C5—C6—C11	−179.32 (19)	O3—C1′—C2′—O7	168.0 (2)
C12—C5—C6—C11	56.0 (2)	O6—C1′—C2′—C3′	106.9 (3)
C4—C5—C6—C11	−51.2 (2)	O3—C1′—C2′—C3′	−71.3 (3)
C15—C6—C7—C8	−53.9 (3)	O7—C2′—C3′—C12′	54.8 (3)
C11—C6—C7—C8	59.4 (3)	C1′—C2′—C3′—C12′	−66.6 (3)
C5—C6—C7—C8	179.3 (2)	O7—C2′—C3′—C4′	−71.8 (3)
C6—C7—C8—C9	−44.4 (3)	C1′—C2′—C3′—C4′	166.8 (3)
C7—C8—C9—C10	14.6 (4)	C12′—C3′—C4′—C5′	78.2 (4)
C7—C8—C9—C16	−166.1 (2)	C2′—C3′—C4′—C5′	−157.8 (3)
C8—C9—C10—C11	−1.4 (4)	C6′—O4—C5′—C4′	179.0 (2)
C16—C9—C10—C11	179.3 (2)	C3′—C4′—C5′—O4	−64.7 (4)
C2—O1—C11—C10	175.6 (2)	C5′—O4—C6′—C7′	−99.2 (3)
C2—O1—C11—C6	51.9 (3)	C5′—O4—C6′—C13′	137.6 (2)
C9—C10—C11—O1	−106.0 (3)	O4—C6′—C7′—C8′	5.8 (4)

C9—C10—C11—C6	17.6 (3)	C13′—C6′—C7′—C8′	127.4 (3)
C15—C6—C11—O1	−166.89 (18)	C6′—C7′—C8′—C9′	174.7 (2)
C7—C6—C11—O1	74.9 (2)	C7′—C8′—C9′—C10′	−169.7 (3)
C5—C6—C11—O1	−46.7 (2)	C8′—C9′—C10′—C11′	−4.7 (4)
C15—C6—C11—C10	73.4 (2)	C4—O5—C11′—O9	−0.1 (3)
C7—C6—C11—C10	−44.8 (3)	C4—O5—C11′—C10′	178.57 (19)
C5—C6—C11—C10	−166.4 (2)	C9′—C10′—C11′—O9	−28.5 (4)
C13—O2—C12—C2	117.4 (3)	C9′—C10′—C11′—O5	152.8 (2)
C13—O2—C12—C5	−119.9 (3)	O4—C6′—C13′—O8	−68.1 (3)
O1—C2—C12—O2	−157.67 (19)	C7′—C6′—C13′—O8	167.6 (2)
C3—C2—C12—O2	82.7 (2)	O4—C6′—C13′—C14′	52.7 (3)
O1—C2—C12—C13	−87.6 (3)	C7′—C6′—C13′—C14′	−71.6 (3)
C3—C2—C12—C13	152.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7···O8^{i}	0.84	1.94	2.761 (3)	167
O8—H8···O2^{ii}	0.84	2.10	2.895 (3)	158
C4—H4···O1^{iii}	1.00	2.55	3.467 (3)	153
C13—H13*B*···O9^{iv}	0.99	2.65	3.490 (3)	143
C7′—H7′···O6^{v}	0.95	2.62	3.473 (3)	150

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) *x*−1/2, −*y*+1/2, −*z*+1; (iv) *x*+1, *y*, *z*; (v) −*x*+1/2, −*y*+1, *z*+1/2.

Isororidin A (II)

Crystal data

$C_{29}H_{40}O_9$
M_r = 532.61
Orthorhombic, $P2_12_12_1$
a = 9.302 (3) Å
b = 15.412 (6) Å
c = 20.191 (8) Å
V = 2894.6 (19) Å^3
Z = 4
F(000) = 1144
D_x = 1.222 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54180 Å
Cell parameters from 3458 reflections
θ = 4.3–72.7°
μ = 0.74 mm^{-1}
T = 293 K
Block, colourless
0.47 × 0.47 × 0.24 mm

Data collection

SuperNova (Cu) X-ray Source diffractometer
Radiation source: micro-focus sealed X-ray tube
Absorption correction: analytical CrysAlisPro 1.171.40.67a (Rigaku Oxford Diffraction, 2019) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by R.C. Clark & J.S. Reid. (Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min} = 0.775, T_{max} = 0.878
6984 measured reflections
5037 independent reflections
4285 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 72.9°, θ_{min} = 3.6°
h = −6→11
k = −16→18
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.086$
$wR(F^2) = 0.265$
$S = 1.05$
5037 reflections
349 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Absolute structure: Classical Flack method preferred over Parsons because s.u. lower.
Absolute structure parameter: 0.4 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1160 (4)	0.2734 (3)	0.4876 (2)	0.0489 (9)
O5	0.4444 (4)	0.3442 (2)	0.33576 (19)	0.0459 (8)
O9	0.6291 (4)	0.4150 (3)	0.3838 (2)	0.0552 (10)
O8	1.0280 (6)	0.8042 (3)	0.2920 (3)	0.0711 (13)
H40	0.996908	0.836900	0.263394	0.107*
O4	0.8192 (5)	0.6918 (3)	0.3550 (2)	0.0588 (11)
O6	0.6101 (7)	0.4992 (4)	0.6056 (3)	0.0914 (19)
O3	0.4451 (4)	0.5178 (2)	0.5267 (2)	0.0470 (8)
O7	0.6931 (6)	0.6700 (4)	0.5946 (3)	0.0780 (16)
H39	0.654210	0.670235	0.631038	0.117*
O2	0.1010 (4)	0.3816 (3)	0.3259 (2)	0.0552 (10)
C5	0.2667 (5)	0.4062 (3)	0.4991 (2)	0.0374 (10)
C10	0.2299 (5)	0.3139 (3)	0.5240 (3)	0.0410 (10)
H10	0.316100	0.277728	0.519881	0.049*
C9	0.1829 (6)	0.3130 (4)	0.5960 (3)	0.0489 (12)
H9	0.185959	0.260368	0.618428	0.059*
C8	0.1381 (6)	0.3814 (5)	0.6287 (3)	0.0545 (14)
C7	0.1345 (7)	0.4703 (5)	0.5984 (3)	0.0614 (16)
H7	0.212397	0.504623	0.616696	0.074*
H8	0.044767	0.498355	0.610164	0.074*
C6	0.1486 (6)	0.4680 (4)	0.5230 (3)	0.0510 (13)
H6	0.169475	0.526086	0.507177	0.061*
H5	0.057494	0.450213	0.504050	0.061*
C15	0.0923 (9)	0.3768 (6)	0.7001 (4)	0.078 (2)
H18	0.106251	0.318860	0.716334	0.117*
H19	−0.007506	0.392031	0.703596	0.117*
H20	0.148797	0.416494	0.725830	0.117*
C4	0.2781 (5)	0.4043 (3)	0.4206 (3)	0.0370 (9)

C3	0.3811 (5)	0.3291 (3)	0.4010 (3)	0.0410 (10)
H4	0.456412	0.321639	0.434432	0.049*
C2	0.2894 (6)	0.2480 (3)	0.3958 (3)	0.0495 (12)
H3	0.325979	0.203022	0.424943	0.059*
H2	0.289369	0.226140	0.350788	0.059*
C1	0.1377 (5)	0.2748 (4)	0.4166 (3)	0.0464 (12)
H1	0.064181	0.241008	0.393355	0.056*
C11	0.1364 (5)	0.3678 (4)	0.3957 (3)	0.0440 (11)
C12	0.0054 (6)	0.4163 (5)	0.3763 (3)	0.0589 (15)
H12	−0.086756	0.387974	0.382581	0.071*
H11	0.004807	0.478567	0.383133	0.071*
C13	0.3143 (6)	0.4929 (3)	0.3906 (3)	0.0467 (11)
H13	0.410265	0.509081	0.402971	0.070*
H14	0.247744	0.535526	0.406821	0.070*
H15	0.307528	0.489630	0.343241	0.070*
C26	0.5702 (6)	0.3883 (3)	0.3352 (3)	0.0471 (12)
C25	0.6222 (7)	0.3981 (4)	0.2658 (3)	0.0539 (13)
H31	0.598031	0.355482	0.235147	0.065*
C24	0.7018 (6)	0.4650 (4)	0.2455 (3)	0.0556 (13)
H30	0.734023	0.462248	0.201998	0.067*
C23	0.7443 (6)	0.5408 (4)	0.2820 (3)	0.0512 (12)
H29	0.727688	0.542171	0.327450	0.061*
C22	0.8056 (6)	0.6081 (4)	0.2540 (3)	0.0523 (12)
H28	0.828538	0.602330	0.209401	0.063*
C21	0.8430 (7)	0.6929 (4)	0.2853 (3)	0.0523 (13)
H27	0.782251	0.738141	0.265737	0.063*
C28	1.0009 (7)	0.7169 (4)	0.2733 (3)	0.0571 (14)
H35	1.022230	0.710209	0.226072	0.068*
C29	1.1011 (7)	0.6590 (5)	0.3124 (4)	0.0708 (19)
H36	1.197581	0.680668	0.308900	0.106*
H38	1.096988	0.601105	0.295030	0.106*
H37	1.072340	0.658622	0.358053	0.106*
C20	0.6981 (9)	0.7363 (5)	0.3781 (4)	0.077 (2)
H26	0.612231	0.712286	0.357938	0.092*
H25	0.704638	0.796976	0.365662	0.092*
C19	0.6872 (9)	0.7289 (4)	0.4530 (4)	0.073 (2)
H24	0.773092	0.753899	0.472527	0.088*
H23	0.605803	0.763021	0.467929	0.088*
C18	0.6703 (6)	0.6377 (4)	0.4784 (3)	0.0517 (13)
H22	0.606637	0.606921	0.447720	0.062*
C17	0.5983 (6)	0.6359 (4)	0.5475 (3)	0.0520 (13)
H21	0.511966	0.672257	0.546192	0.062*
C16	0.5547 (7)	0.5449 (4)	0.5645 (3)	0.0527 (13)
C14	0.4109 (5)	0.4275 (3)	0.5324 (3)	0.0414 (10)
H16	0.406303	0.411712	0.578824	0.050*
H17	0.486538	0.393422	0.511897	0.050*
C27	0.8125 (8)	0.5867 (6)	0.4818 (4)	0.078 (2)
H34	0.873613	0.611882	0.514942	0.117*

H33	0.859594	0.589071	0.439554	0.117*
H32	0.792810	0.527350	0.493052	0.117*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0333 (16)	0.073 (2)	0.040 (2)	−0.0189 (16)	0.0039 (17)	−0.0017 (17)
O5	0.0357 (16)	0.0644 (17)	0.0375 (19)	−0.0007 (16)	0.0033 (16)	0.0029 (16)
O9	0.0357 (17)	0.081 (2)	0.049 (2)	−0.0155 (18)	−0.0011 (19)	0.0094 (19)
O8	0.080 (3)	0.070 (2)	0.063 (3)	−0.017 (2)	−0.023 (3)	0.018 (2)
O4	0.052 (2)	0.084 (2)	0.041 (2)	0.014 (2)	0.0078 (19)	0.0072 (19)
O6	0.094 (4)	0.101 (4)	0.079 (4)	−0.041 (3)	−0.053 (4)	0.024 (3)
O3	0.0348 (15)	0.0587 (17)	0.048 (2)	−0.0084 (15)	−0.0088 (18)	−0.0012 (16)
O7	0.070 (3)	0.106 (3)	0.058 (3)	−0.044 (3)	0.018 (3)	−0.033 (3)
O2	0.0410 (19)	0.087 (2)	0.038 (2)	−0.0024 (18)	−0.0140 (18)	0.0003 (19)
C5	0.0241 (18)	0.055 (2)	0.033 (2)	0.0005 (18)	−0.005 (2)	−0.0008 (18)
C10	0.030 (2)	0.057 (2)	0.036 (3)	−0.0048 (19)	0.003 (2)	0.002 (2)
C9	0.036 (2)	0.075 (3)	0.036 (3)	−0.011 (2)	0.000 (2)	0.007 (2)
C8	0.033 (2)	0.089 (4)	0.041 (3)	−0.003 (3)	0.005 (2)	0.000 (3)
C7	0.049 (3)	0.084 (4)	0.050 (3)	0.015 (3)	0.004 (3)	−0.016 (3)
C6	0.036 (2)	0.073 (3)	0.044 (3)	0.007 (2)	−0.002 (3)	−0.006 (2)
C15	0.073 (4)	0.119 (6)	0.042 (3)	−0.005 (4)	0.012 (4)	−0.005 (4)
C4	0.0248 (18)	0.053 (2)	0.033 (2)	−0.0032 (17)	−0.005 (2)	0.0018 (19)
C3	0.030 (2)	0.060 (2)	0.033 (2)	−0.0010 (19)	0.004 (2)	0.0060 (19)
C2	0.046 (3)	0.056 (2)	0.047 (3)	−0.005 (2)	0.010 (3)	−0.002 (2)
C1	0.031 (2)	0.069 (3)	0.040 (3)	−0.018 (2)	0.002 (2)	−0.004 (2)
C11	0.028 (2)	0.071 (3)	0.033 (2)	−0.005 (2)	−0.007 (2)	−0.003 (2)
C12	0.031 (2)	0.093 (4)	0.052 (3)	0.012 (3)	−0.014 (3)	−0.004 (3)
C13	0.039 (2)	0.056 (2)	0.046 (3)	0.000 (2)	−0.007 (2)	0.006 (2)
C26	0.035 (2)	0.057 (2)	0.049 (3)	0.001 (2)	0.009 (3)	0.003 (2)
C25	0.052 (3)	0.066 (2)	0.044 (3)	−0.005 (2)	0.014 (3)	0.000 (2)
C24	0.051 (3)	0.073 (3)	0.042 (3)	0.000 (3)	0.010 (3)	0.005 (3)
C23	0.041 (3)	0.069 (3)	0.043 (3)	−0.002 (2)	0.004 (3)	0.009 (2)
C22	0.046 (3)	0.071 (3)	0.040 (3)	−0.004 (2)	0.000 (3)	0.010 (2)
C21	0.050 (3)	0.068 (3)	0.039 (3)	0.002 (3)	0.002 (3)	0.010 (2)
C28	0.060 (3)	0.068 (3)	0.044 (3)	−0.014 (3)	0.007 (3)	0.010 (2)
C29	0.051 (3)	0.080 (4)	0.081 (5)	0.004 (3)	0.005 (4)	0.008 (4)
C20	0.073 (5)	0.080 (4)	0.078 (5)	0.015 (4)	0.025 (4)	0.017 (4)
C19	0.074 (4)	0.067 (3)	0.078 (5)	−0.003 (3)	0.038 (4)	−0.009 (3)
C18	0.041 (3)	0.065 (3)	0.049 (3)	−0.002 (2)	0.010 (3)	−0.006 (2)
C17	0.043 (3)	0.064 (3)	0.049 (3)	−0.013 (2)	0.009 (3)	−0.012 (2)
C16	0.047 (3)	0.072 (3)	0.039 (3)	−0.016 (3)	−0.006 (3)	−0.004 (2)
C14	0.028 (2)	0.056 (2)	0.040 (3)	0.0003 (18)	−0.008 (2)	0.002 (2)
C27	0.060 (4)	0.113 (5)	0.060 (4)	0.028 (4)	0.016 (4)	0.009 (4)

Geometric parameters (Å, º)

O1—C10	1.433 (6)	C2—H2	0.9700
O1—C1	1.447 (7)	C1—C11	1.494 (8)
O5—C26	1.354 (7)	C1—H1	0.9800
O5—C3	1.461 (6)	C11—C12	1.482 (7)
O9—C26	1.196 (7)	C12—H12	0.9700
O8—C28	1.420 (7)	C12—H11	0.9700
O8—H40	0.8200	C13—H13	0.9600
O4—C20	1.400 (9)	C13—H14	0.9600
O4—C21	1.424 (7)	C13—H15	0.9600
O6—C16	1.205 (8)	C26—C25	1.490 (8)
O3—C16	1.341 (7)	C25—C24	1.334 (9)
O3—C14	1.432 (6)	C25—H31	0.9300
O7—C17	1.399 (8)	C24—C23	1.436 (9)
O7—H39	0.8200	C24—H30	0.9300
O2—C12	1.454 (8)	C23—C22	1.313 (8)
O2—C11	1.463 (6)	C23—H29	0.9300
C5—C6	1.533 (7)	C22—C21	1.493 (9)
C5—C14	1.536 (6)	C22—H28	0.9300
C5—C10	1.549 (7)	C21—C28	1.534 (9)
C5—C4	1.587 (7)	C21—H27	0.9800
C10—C9	1.517 (7)	C28—C29	1.511 (10)
C10—H10	0.9800	C28—H35	0.9800
C9—C8	1.312 (9)	C29—H36	0.9600
C9—H9	0.9300	C29—H38	0.9600
C8—C7	1.500 (10)	C29—H37	0.9600
C8—C15	1.504 (9)	C20—C19	1.520 (11)
C7—C6	1.528 (9)	C20—H26	0.9700
C7—H7	0.9700	C20—H25	0.9700
C7—H8	0.9700	C19—C18	1.504 (9)
C6—H6	0.9700	C19—H24	0.9700
C6—H5	0.9700	C19—H23	0.9700
C15—H18	0.9600	C18—C27	1.540 (9)
C15—H19	0.9600	C18—C17	1.547 (9)
C15—H20	0.9600	C18—H22	0.9800
C4—C11	1.519 (6)	C17—C16	1.499 (8)
C4—C13	1.531 (6)	C17—H21	0.9800
C4—C3	1.556 (7)	C14—H16	0.9700
C3—C2	1.517 (7)	C14—H17	0.9700
C3—H4	0.9800	C27—H34	0.9600
C2—C1	1.529 (7)	C27—H33	0.9600
C2—H3	0.9700	C27—H32	0.9600
C10—O1—C1	113.5 (3)	C4—C13—H13	109.5
C26—O5—C3	115.8 (4)	C4—C13—H14	109.5
C28—O8—H40	109.5	H13—C13—H14	109.5
C20—O4—C21	116.7 (5)	C4—C13—H15	109.5

C16—O3—C14	115.2 (4)	H13—C13—H15	109.5
C17—O7—H39	109.5	H14—C13—H15	109.5
C12—O2—C11	61.1 (3)	O9—C26—O5	124.2 (5)
C6—C5—C14	110.8 (4)	O9—C26—C25	126.0 (5)
C6—C5—C10	108.0 (4)	O5—C26—C25	109.8 (5)
C14—C5—C10	104.3 (4)	C24—C25—C26	123.1 (6)
C6—C5—C4	112.0 (4)	C24—C25—H31	118.4
C14—C5—C4	112.5 (4)	C26—C25—H31	118.4
C10—C5—C4	108.8 (4)	C25—C24—C23	128.6 (6)
O1—C10—C9	106.0 (4)	C25—C24—H30	115.7
O1—C10—C5	113.4 (4)	C23—C24—H30	115.7
C9—C10—C5	112.6 (4)	C22—C23—C24	122.8 (6)
O1—C10—H10	108.3	C22—C23—H29	118.6
C9—C10—H10	108.3	C24—C23—H29	118.6
C5—C10—H10	108.3	C23—C22—C21	127.7 (6)
C8—C9—C10	124.5 (5)	C23—C22—H28	116.2
C8—C9—H9	117.7	C21—C22—H28	116.2
C10—C9—H9	117.7	O4—C21—C22	111.8 (5)
C9—C8—C7	122.4 (5)	O4—C21—C28	107.9 (5)
C9—C8—C15	122.3 (7)	C22—C21—C28	111.5 (5)
C7—C8—C15	115.3 (6)	O4—C21—H27	108.5
C8—C7—C6	112.6 (5)	C22—C21—H27	108.5
C8—C7—H7	109.1	C28—C21—H27	108.5
C6—C7—H7	109.1	O8—C28—C29	108.1 (6)
C8—C7—H8	109.1	O8—C28—C21	110.8 (6)
C6—C7—H8	109.1	C29—C28—C21	111.5 (5)
H7—C7—H8	107.8	O8—C28—H35	108.8
C7—C6—C5	113.0 (5)	C29—C28—H35	108.8
C7—C6—H6	109.0	C21—C28—H35	108.8
C5—C6—H6	109.0	C28—C29—H36	109.5
C7—C6—H5	109.0	C28—C29—H38	109.5
C5—C6—H5	109.0	H36—C29—H38	109.5
H6—C6—H5	107.8	C28—C29—H37	109.5
C8—C15—H18	109.5	H36—C29—H37	109.5
C8—C15—H19	109.5	H38—C29—H37	109.5
H18—C15—H19	109.5	O4—C20—C19	110.4 (7)
C8—C15—H20	109.5	O4—C20—H26	109.6
H18—C15—H20	109.5	C19—C20—H26	109.6
H19—C15—H20	109.5	O4—C20—H25	109.6
C11—C4—C13	112.9 (4)	C19—C20—H25	109.6
C11—C4—C3	100.0 (4)	H26—C20—H25	108.1
C13—C4—C3	115.3 (4)	C18—C19—C20	114.6 (6)
C11—C4—C5	106.3 (4)	C18—C19—H24	108.6
C13—C4—C5	113.2 (4)	C20—C19—H24	108.6
C3—C4—C5	108.0 (4)	C18—C19—H23	108.6
O5—C3—C2	107.2 (4)	C20—C19—H23	108.6
O5—C3—C4	111.1 (4)	H24—C19—H23	107.6
C2—C3—C4	106.6 (4)	C19—C18—C27	113.7 (6)

O5—C3—H4	110.6	C19—C18—C17	111.7 (5)
C2—C3—H4	110.6	C27—C18—C17	108.8 (5)
C4—C3—H4	110.6	C19—C18—H22	107.5
C3—C2—C1	106.1 (4)	C27—C18—H22	107.5
C3—C2—H3	110.5	C17—C18—H22	107.5
C1—C2—H3	110.5	O7—C17—C16	111.5 (6)
C3—C2—H2	110.5	O7—C17—C18	109.4 (5)
C1—C2—H2	110.5	C16—C17—C18	109.9 (5)
H3—C2—H2	108.7	O7—C17—H21	108.7
O1—C1—C11	107.1 (4)	C16—C17—H21	108.7
O1—C1—C2	113.3 (5)	C18—C17—H21	108.7
C11—C1—C2	100.9 (4)	O6—C16—O3	122.4 (6)
O1—C1—H1	111.6	O6—C16—C17	126.1 (5)
C11—C1—H1	111.6	O3—C16—C17	111.5 (5)
C2—C1—H1	111.6	O3—C14—C5	111.5 (4)
O2—C11—C12	59.2 (4)	O3—C14—H16	109.3
O2—C11—C1	114.5 (4)	C5—C14—H16	109.3
C12—C11—C1	124.4 (5)	O3—C14—H17	109.3
O2—C11—C4	117.5 (4)	C5—C14—H17	109.3
C12—C11—C4	127.9 (5)	H16—C14—H17	108.0
C1—C11—C4	104.7 (4)	C18—C27—H34	109.5
O2—C12—C11	59.8 (3)	C18—C27—H33	109.5
O2—C12—H12	117.8	H34—C27—H33	109.5
C11—C12—H12	117.8	C18—C27—H32	109.5
O2—C12—H11	117.8	H34—C27—H32	109.5
C11—C12—H11	117.8	H33—C27—H32	109.5
H12—C12—H11	114.9		

C1—O1—C10—C9	175.3 (4)	O1—C1—C11—C4	72.6 (5)
C1—O1—C10—C5	51.3 (6)	C2—C1—C11—C4	−46.2 (5)
C6—C5—C10—O1	75.4 (5)	C13—C4—C11—O2	38.3 (6)
C14—C5—C10—O1	−166.6 (4)	C3—C4—C11—O2	−84.8 (5)
C4—C5—C10—O1	−46.4 (5)	C5—C4—C11—O2	162.9 (4)
C6—C5—C10—C9	−44.8 (5)	C13—C4—C11—C12	−32.7 (8)
C14—C5—C10—C9	73.1 (5)	C3—C4—C11—C12	−155.8 (6)
C4—C5—C10—C9	−166.6 (4)	C5—C4—C11—C12	92.0 (6)
O1—C10—C9—C8	−106.0 (6)	C13—C4—C11—C1	166.6 (4)
C5—C10—C9—C8	18.4 (7)	C3—C4—C11—C1	43.5 (5)
C10—C9—C8—C7	−2.6 (9)	C5—C4—C11—C1	−68.8 (5)
C10—C9—C8—C15	179.8 (6)	C1—C11—C12—O2	−100.1 (6)
C9—C8—C7—C6	15.4 (8)	C4—C11—C12—O2	102.5 (6)
C15—C8—C7—C6	−166.9 (5)	C3—O5—C26—O9	−0.5 (7)
C8—C7—C6—C5	−44.7 (7)	C3—O5—C26—C25	179.5 (4)
C14—C5—C6—C7	−54.1 (6)	O9—C26—C25—C24	−29.4 (9)
C10—C5—C6—C7	59.5 (6)	O5—C26—C25—C24	150.5 (6)
C4—C5—C6—C7	179.4 (5)	C26—C25—C24—C23	−4.5 (10)
C6—C5—C4—C11	−63.9 (5)	C25—C24—C23—C22	−170.0 (7)
C14—C5—C4—C11	170.5 (4)	C24—C23—C22—C21	174.3 (6)

C10—C5—C4—C11	55.5 (5)	C20—O4—C21—C22	−105.1 (6)
C6—C5—C4—C13	60.6 (5)	C20—O4—C21—C28	131.9 (6)
C14—C5—C4—C13	−65.0 (5)	C23—C22—C21—O4	6.8 (9)
C10—C5—C4—C13	180.0 (4)	C23—C22—C21—C28	127.7 (6)
C6—C5—C4—C3	−170.5 (4)	O4—C21—C28—O8	−68.0 (6)
C14—C5—C4—C3	64.0 (5)	C22—C21—C28—O8	168.8 (5)
C10—C5—C4—C3	−51.1 (5)	O4—C21—C28—C29	52.4 (7)
C26—O5—C3—C2	−153.7 (4)	C22—C21—C28—C29	−70.7 (7)
C26—O5—C3—C4	90.3 (5)	C21—O4—C20—C19	179.8 (6)
C11—C4—C3—O5	92.9 (5)	O4—C20—C19—C18	−61.6 (9)
C13—C4—C3—O5	−28.5 (6)	C20—C19—C18—C27	80.2 (9)
C5—C4—C3—O5	−156.2 (4)	C20—C19—C18—C17	−156.2 (6)
C11—C4—C3—C2	−23.6 (5)	C19—C18—C17—O7	−69.4 (8)
C13—C4—C3—C2	−145.0 (5)	C27—C18—C17—O7	57.0 (7)
C5—C4—C3—C2	87.3 (5)	C19—C18—C17—C16	167.9 (6)
O5—C3—C2—C1	−122.6 (5)	C27—C18—C17—C16	−65.8 (7)
C4—C3—C2—C1	−3.5 (6)	C14—O3—C16—O6	−7.9 (9)
C10—O1—C1—C11	−64.0 (5)	C14—O3—C16—C17	170.8 (5)
C10—O1—C1—C2	46.4 (6)	O7—C17—C16—O6	−12.6 (9)
C3—C2—C1—O1	−84.4 (5)	C18—C17—C16—O6	109.0 (8)
C3—C2—C1—C11	29.8 (6)	O7—C17—C16—O3	168.7 (5)
C12—O2—C11—C1	116.8 (6)	C18—C17—C16—O3	−69.7 (6)
C12—O2—C11—C4	−119.7 (6)	C16—O3—C14—C5	168.2 (4)
O1—C1—C11—O2	−157.3 (4)	C6—C5—C14—O3	−50.8 (6)
C2—C1—C11—O2	83.9 (5)	C10—C5—C14—O3	−166.8 (4)
O1—C1—C11—C12	−89.1 (6)	C4—C5—C14—O3	75.4 (5)
C2—C1—C11—C12	152.1 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H40···O2^{i}	0.82	2.13	2.921 (6)	161
O7—H39···O8^{ii}	0.82	1.99	2.785 (7)	164

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+3/2, −*z*+1.