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12-Methoxy-15-(pyridin-3-ylmethyl-amino)podocarpa-8,11,13-trien-15-one

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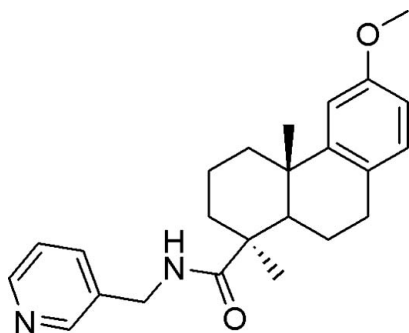
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.088; data-to-parameter ratio = 12.3.

The benzylic C–N bond of the title compound [systematic name: *N*-(3-pyridylmethyl)-6-methoxy-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene-1-carboxamide], $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_2$, is almost coplanar with the amide carbonyl linkage; the C–N–C=O angle is $6.8(4)^\circ$. N–H···N hydrogen bonds link the molecules into chains in the *a* direction. In addition, these chains are linked by weak intermolecular Ar–H···O interactions, *viz.* an aromatic C–H and carbonyl O atom of adjacent molecules.

Related literature

For similar structures see: Bakare *et al.* (2005); Couldwell *et al.* (1985); Mondal *et al.* (2003).



Experimental

Crystal data

$\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_2$
 $M_r = 378.50$
 Monoclinic, $P2_1$
 $a = 7.0164(4)$ Å
 $b = 7.9267(4)$ Å
 $c = 18.5843(11)$ Å
 $\beta = 90.871(6)^\circ$

$V = 1033.48(10)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293(2)$ K
 $0.55 \times 0.45 \times 0.22$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.900$, $T_{\max} = 1.000$
 (expected range = 0.884–0.983)

11646 measured reflections
 3150 independent reflections
 3081 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.088$
 $S = 1.04$
 3150 reflections
 256 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1C···N2 ⁱ	0.86	2.22	3.0311 (14)	157
C23–H23A···O1 ⁱⁱ	0.93	2.45	3.2386 (16)	143

Symmetry codes: (i) $x - 1, y, z$; (ii) $x, y + 1, z$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2167).

References

- Bakare, O., John, N., Butcher, R. J. & Zalkow, L. H. (2005). *Acta Cryst.* **E61**, o3791–o3793.
 Bruker (2000). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2004). *SADABS*. Version 2004/1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2005). *SAINT*. Version 7.34A. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2006). *APEX2*. Version 2.1-0. Bruker AXS Inc., Madison, Wisconsin, USA.
 Couldwell, M. C., Smith, R. A. J. & Simpson, J. (1985). *Acta Cryst.* **C41**, 983–985.
 Mondal, S., Mukherjee, M., Roy, A. & Mukherjee, D. (2003). *Acta Cryst.* **C59**, o132–o134.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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12-Methoxy-15-(pyridin-3-ylmethylamino)podocarpa-8,11,13-trien-15-one

C. Mouamba, R. J. Butcher and O. Bakare

Comment

As part of our anti-inflammatory and anti-cancer discovery program, we are exploring the derivatization of the C-4 carboxyl group of podocarpic acid (Bakare *et al.*, 2005) in order to design new molecules that can modulate the lipoxygenase and cyclooxygenase pathways. Consequently, the title compound, (I), was synthesized as one of a series of amide derivatives under investigation.

The geometric conformation parameters of the three fused six-membered rings in (I), Fig. 1 and Table 1, are similar to those observed previously (Couldwell *et al.*, 1985; Bakare *et al.*, 2005; Mondal *et al.*, 2003). Since this compound was made from natural podocarpic acid and the stereocenters were intact during reactions, the stereochemistry of the compound is as shown. As expected, Ring A adopts the usual chair conformation, while Ring B is observed in the half-chair as a result of being fused to the planar aromatic ring C. The benzylic carbon-C19 to amide-N1 bond is almost coplanar with the amide carbonyl C15—O1 bond, with a dihedral angle at 6.8 (4)°. This near co-planarity is probably due to the partial double bond character of the C15—N1 bond (C15—N1, 1.348 (2) Å). Interestingly, the fused three ring system of the podocarpic acid scaffold appears perpendicular to this plane giving the molecule a bowl-like appearance. N1—H···N2 hydrogen bonds link the molecules into chains in the *a* direction. In addition, these chains are linked by weak intermolecular Ar—H···O interactions comprising the carbonyl O1 atom of one molecule and the aromatic C23—H23 of another. (Fig. 2 and Table 2).

Experimental

To sodium hydride (NaH, 100 mg, 4.16 mmol) was added a solution of 12-methoxy-podocarpa-8,11,13-trien-15-oic acid (500 mg, 1.74 mmol) in dry benzene (15 ml). The resulting mixture was stirred for 30 minutes. Oxalyl chloride (2 ml) was added slowly and stirring continued for a further 1 h. The mixture was then filtered and the solvent removed *in vacuo* to give the the acid chloride, 12-methoxypodocarpa-8, 11,13-trienoyl chloride, as a yellow oil. The acid chloride, in benzene (10 ml), was added slowly to a stirred solution of 3-(aminomethyl)pyridine (468 mg, 4.33 mmol) in dry benzene (30 ml) at 0 °C. The reaction mixture was stirred at room temperature overnight, filtered under suction and the filtrate concentrated *in vacuo* to obtain a a yellow oil. The oily residue was treated with a hexane:ethyl acetate (75:25) to precipitate a white solid (344 mg, 53.0%). The white solid obtained was recrystallized from hexane:ethyl acetate (75:25).

Refinement

The methyl H atoms were constrained to an ideal geometry with C—H distances of 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but each group was allowed to rotate freely about its C—C bond. The position of the amine H atom was refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. While crystallizing in a chiral space group the molecule does not contain any atoms heavier than Si and the absolute configuration could not be determined by X-ray methods. Hence the refinement was carried out with the Friedel pairs averaged.

Figures

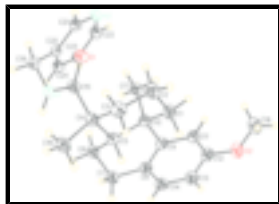


Fig. 1. View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level.

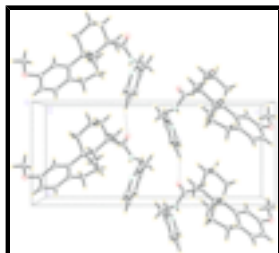


Fig. 2. View of the packing viewed down the *a* axis. Dashed bonds show weak C—H...O interactions.

6-methoxy-1,4a-dimethyl-*N*-(3-pyridylmethyl)-1,2,3,4,4a,9,10,10a-octahydrophenanthrene-1-carboxamide

Crystal data

$C_{24}H_{30}N_2O_2$

$M_r = 378.50$

Monoclinic, $P2_1$

$a = 7.0164$ (4) Å

$b = 7.9267$ (4) Å

$c = 18.5843$ (11) Å

$\beta = 90.871$ (6)°

$V = 1033.48$ (10) Å³

$Z = 2$

$F_{000} = 408$

$D_x = 1.216$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9074 reflections

$\theta = 2.2$ – 30.7 °

$\mu = 0.08$ mm⁻¹

$T = 293$ (2) K

Plate, colourless

$0.55 \times 0.45 \times 0.22$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

$T_{\min} = 0.900$, $T_{\max} = 1.000$

11646 measured reflections

3150 independent reflections

3081 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\text{max}} = 30.7$ °

$\theta_{\text{min}} = 2.2$ °

$h = -10 \rightarrow 10$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 25$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.0836P]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} = 0.002$
3150 reflections	$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$
256 parameters	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.69388 (13)	0.33391 (13)	0.38936 (5)	0.0233 (2)
O2	0.86838 (16)	0.73577 (15)	-0.03721 (5)	0.0299 (2)
N1	0.48902 (13)	0.54988 (14)	0.40643 (5)	0.0184 (2)
H1C	0.3837	0.5984	0.3944	0.022*
N2	1.10503 (14)	0.72422 (15)	0.41192 (6)	0.0201 (2)
C1	0.69900 (18)	0.33687 (17)	0.17249 (6)	0.0218 (2)
H1A	0.6116	0.3002	0.1347	0.026*
H1B	0.8251	0.3443	0.1520	0.026*
C2	0.70219 (19)	0.20383 (17)	0.23270 (7)	0.0232 (2)
H2A	0.7330	0.0945	0.2125	0.028*
H2B	0.8007	0.2324	0.2678	0.028*
C3	0.50940 (19)	0.19362 (17)	0.27011 (7)	0.0222 (2)
H3A	0.5203	0.1147	0.3099	0.027*
H3B	0.4157	0.1488	0.2363	0.027*
C4	0.43549 (15)	0.36460 (16)	0.29934 (6)	0.0169 (2)
C5	0.44139 (15)	0.49801 (15)	0.23627 (6)	0.0161 (2)

supplementary materials

H5A	0.3558	0.4519	0.1990	0.019*
C6	0.35943 (17)	0.67454 (16)	0.25265 (6)	0.0184 (2)
H6A	0.2462	0.6636	0.2816	0.022*
H6B	0.4526	0.7404	0.2796	0.022*
C7	0.30917 (17)	0.76442 (17)	0.18135 (7)	0.0211 (2)
H7A	0.1955	0.7133	0.1605	0.025*
H7B	0.2807	0.8818	0.1912	0.025*
C8	0.46809 (17)	0.75558 (17)	0.12738 (6)	0.0189 (2)
C9	0.61628 (16)	0.63630 (16)	0.13341 (6)	0.0173 (2)
C10	0.63800 (16)	0.51509 (16)	0.19860 (6)	0.0161 (2)
C11	0.75564 (17)	0.63164 (18)	0.07919 (6)	0.0203 (2)
H11A	0.8564	0.5558	0.0832	0.024*
C12	0.74398 (18)	0.73953 (18)	0.01969 (7)	0.0229 (2)
C13	0.59769 (18)	0.8604 (2)	0.01412 (7)	0.0255 (3)
H13A	0.5912	0.9341	-0.0248	0.031*
C14	0.46337 (18)	0.86725 (19)	0.06800 (7)	0.0248 (3)
H14A	0.3671	0.9477	0.0650	0.030*
C15	0.55164 (16)	0.41437 (16)	0.36840 (6)	0.0172 (2)
C16	0.22613 (16)	0.33573 (18)	0.32146 (7)	0.0231 (2)
H16A	0.2221	0.2535	0.3593	0.035*
H16B	0.1536	0.2957	0.2807	0.035*
H16C	0.1729	0.4400	0.3380	0.035*
C17	0.79681 (16)	0.59029 (18)	0.24879 (6)	0.0202 (2)
H17A	0.9125	0.6023	0.2223	0.030*
H17B	0.8185	0.5160	0.2889	0.030*
H17C	0.7571	0.6987	0.2660	0.030*
C18	1.0220 (2)	0.6164 (2)	-0.03192 (8)	0.0342 (3)
H18A	1.0931	0.6178	-0.0757	0.051*
H18B	0.9712	0.5055	-0.0244	0.051*
H18C	1.1044	0.6462	0.0078	0.051*
C19	0.60253 (16)	0.61336 (18)	0.46844 (6)	0.0192 (2)
H19A	0.6479	0.5188	0.4970	0.023*
H19B	0.5223	0.6826	0.4986	0.023*
C20	0.77156 (16)	0.71681 (16)	0.44370 (6)	0.0166 (2)
C21	0.95294 (16)	0.64312 (17)	0.43840 (6)	0.0185 (2)
H21A	0.9687	0.5325	0.4540	0.022*
C22	1.08051 (17)	0.88544 (18)	0.39003 (6)	0.0207 (2)
H22A	1.1844	0.9420	0.3710	0.025*
C23	0.90734 (18)	0.97176 (17)	0.39450 (6)	0.0214 (2)
H23A	0.8965	1.0835	0.3797	0.026*
C24	0.75155 (17)	0.88529 (18)	0.42179 (6)	0.0200 (2)
H24B	0.6344	0.9392	0.4255	0.024*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0218 (4)	0.0242 (5)	0.0238 (4)	0.0078 (4)	0.0000 (3)	0.0023 (4)
O2	0.0338 (5)	0.0307 (6)	0.0254 (4)	0.0018 (4)	0.0094 (4)	0.0072 (4)

N1	0.0136 (4)	0.0216 (5)	0.0198 (4)	0.0030 (4)	0.0005 (3)	-0.0002 (4)
N2	0.0153 (4)	0.0221 (5)	0.0228 (4)	0.0023 (4)	0.0015 (3)	-0.0002 (4)
C1	0.0267 (6)	0.0193 (6)	0.0194 (5)	0.0052 (5)	0.0040 (4)	-0.0005 (5)
C2	0.0269 (6)	0.0184 (6)	0.0243 (5)	0.0056 (5)	0.0043 (4)	0.0012 (5)
C3	0.0256 (5)	0.0148 (5)	0.0262 (6)	-0.0003 (5)	0.0010 (4)	0.0002 (5)
C4	0.0148 (4)	0.0150 (5)	0.0208 (5)	-0.0012 (4)	0.0011 (4)	0.0017 (4)
C5	0.0145 (4)	0.0152 (5)	0.0184 (4)	-0.0005 (4)	-0.0002 (4)	0.0005 (4)
C6	0.0185 (5)	0.0165 (5)	0.0204 (5)	0.0018 (4)	0.0014 (4)	0.0006 (4)
C7	0.0199 (5)	0.0208 (6)	0.0226 (5)	0.0039 (4)	0.0012 (4)	0.0039 (5)
C8	0.0182 (5)	0.0194 (5)	0.0191 (5)	-0.0012 (4)	-0.0022 (4)	0.0021 (4)
C9	0.0172 (5)	0.0182 (5)	0.0163 (4)	-0.0020 (4)	-0.0012 (4)	0.0005 (4)
C10	0.0152 (4)	0.0166 (5)	0.0164 (4)	0.0003 (4)	0.0004 (3)	0.0007 (4)
C11	0.0194 (5)	0.0224 (6)	0.0190 (5)	-0.0012 (5)	0.0006 (4)	0.0020 (4)
C12	0.0240 (5)	0.0249 (6)	0.0197 (5)	-0.0037 (5)	0.0012 (4)	0.0024 (5)
C13	0.0271 (6)	0.0274 (7)	0.0219 (5)	-0.0012 (5)	-0.0024 (4)	0.0087 (5)
C14	0.0230 (5)	0.0251 (6)	0.0261 (6)	0.0018 (5)	-0.0034 (4)	0.0080 (5)
C15	0.0151 (4)	0.0181 (5)	0.0185 (5)	-0.0002 (4)	0.0035 (4)	0.0035 (4)
C16	0.0158 (5)	0.0217 (6)	0.0319 (6)	-0.0044 (4)	0.0027 (4)	0.0023 (5)
C17	0.0150 (5)	0.0267 (6)	0.0190 (5)	-0.0019 (4)	-0.0013 (4)	0.0017 (4)
C18	0.0329 (7)	0.0359 (8)	0.0342 (7)	0.0028 (6)	0.0139 (6)	0.0095 (6)
C19	0.0156 (4)	0.0260 (6)	0.0159 (4)	0.0011 (4)	0.0032 (4)	0.0004 (4)
C20	0.0153 (5)	0.0214 (6)	0.0131 (4)	0.0023 (4)	0.0007 (3)	-0.0009 (4)
C21	0.0162 (5)	0.0209 (5)	0.0184 (5)	0.0034 (4)	0.0004 (4)	0.0015 (4)
C22	0.0181 (5)	0.0212 (6)	0.0229 (5)	-0.0013 (4)	0.0020 (4)	-0.0018 (5)
C23	0.0239 (5)	0.0169 (5)	0.0233 (5)	0.0031 (4)	0.0015 (4)	-0.0005 (4)
C24	0.0183 (5)	0.0215 (6)	0.0202 (5)	0.0058 (4)	0.0007 (4)	-0.0029 (5)

Geometric parameters (Å, °)

O1—C15	1.2420 (14)	C8—C14	1.4148 (17)
O2—C12	1.3817 (15)	C9—C11	1.4152 (15)
O2—C18	1.4365 (19)	C9—C10	1.5519 (16)
N1—C15	1.3623 (16)	C10—C17	1.5606 (16)
N1—C19	1.4790 (15)	C11—C12	1.3993 (17)
N1—H1C	0.8600	C11—H11A	0.9300
N2—C21	1.3455 (16)	C12—C13	1.407 (2)
N2—C22	1.3513 (18)	C13—C14	1.3864 (18)
C1—C2	1.5375 (18)	C13—H13A	0.9300
C1—C10	1.5558 (17)	C14—H14A	0.9300
C1—H1A	0.9700	C16—H16A	0.9600
C1—H1B	0.9700	C16—H16B	0.9600
C2—C3	1.5324 (17)	C16—H16C	0.9600
C2—H2A	0.9700	C17—H17A	0.9600
C2—H2B	0.9700	C17—H17B	0.9600
C3—C4	1.5522 (18)	C17—H17C	0.9600
C3—H3A	0.9700	C18—H18A	0.9600
C3—H3B	0.9700	C18—H18B	0.9600
C4—C16	1.5485 (15)	C18—H18C	0.9600
C4—C15	1.5606 (16)	C19—C20	1.5188 (17)

supplementary materials

C4—C5	1.5796 (16)	C19—H19A	0.9700
C5—C6	1.5449 (17)	C19—H19B	0.9700
C5—C10	1.5623 (15)	C20—C24	1.4026 (18)
C5—H5A	0.9800	C20—C21	1.4051 (15)
C6—C7	1.5405 (17)	C21—H21A	0.9300
C6—H6A	0.9700	C22—C23	1.3980 (17)
C6—H6B	0.9700	C22—H22A	0.9300
C7—C8	1.5127 (16)	C23—C24	1.3924 (18)
C7—H7A	0.9700	C23—H23A	0.9300
C7—H7B	0.9700	C24—H24B	0.9300
C8—C9	1.4085 (17)		
C12—O2—C18	116.36 (11)	C1—C10—C5	108.06 (10)
C15—N1—C19	119.89 (10)	C17—C10—C5	113.11 (9)
C15—N1—H1C	120.1	C12—C11—C9	120.93 (11)
C19—N1—H1C	120.1	C12—C11—H11A	119.5
C21—N2—C22	117.64 (11)	C9—C11—H11A	119.5
C2—C1—C10	113.38 (10)	O2—C12—C11	124.16 (12)
C2—C1—H1A	108.9	O2—C12—C13	115.28 (11)
C10—C1—H1A	108.9	C11—C12—C13	120.55 (11)
C2—C1—H1B	108.9	C14—C13—C12	118.46 (12)
C10—C1—H1B	108.9	C14—C13—H13A	120.8
H1A—C1—H1B	107.7	C12—C13—H13A	120.8
C3—C2—C1	111.30 (10)	C13—C14—C8	122.07 (12)
C3—C2—H2A	109.4	C13—C14—H14A	119.0
C1—C2—H2A	109.4	C8—C14—H14A	119.0
C3—C2—H2B	109.4	O1—C15—N1	120.41 (11)
C1—C2—H2B	109.4	O1—C15—C4	122.35 (11)
H2A—C2—H2B	108.0	N1—C15—C4	117.23 (10)
C2—C3—C4	114.49 (10)	C4—C16—H16A	109.5
C2—C3—H3A	108.6	C4—C16—H16B	109.5
C4—C3—H3A	108.6	H16A—C16—H16B	109.5
C2—C3—H3B	108.6	C4—C16—H16C	109.5
C4—C3—H3B	108.6	H16A—C16—H16C	109.5
H3A—C3—H3B	107.6	H16B—C16—H16C	109.5
C16—C4—C3	106.69 (10)	C10—C17—H17A	109.5
C16—C4—C15	107.74 (9)	C10—C17—H17B	109.5
C3—C4—C15	109.60 (10)	H17A—C17—H17B	109.5
C16—C4—C5	109.37 (9)	C10—C17—H17C	109.5
C3—C4—C5	108.20 (9)	H17A—C17—H17C	109.5
C15—C4—C5	114.95 (9)	H17B—C17—H17C	109.5
C6—C5—C10	110.10 (9)	O2—C18—H18A	109.5
C6—C5—C4	116.50 (9)	O2—C18—H18B	109.5
C10—C5—C4	115.08 (9)	H18A—C18—H18B	109.5
C6—C5—H5A	104.6	O2—C18—H18C	109.5
C10—C5—H5A	104.6	H18A—C18—H18C	109.5
C4—C5—H5A	104.6	H18B—C18—H18C	109.5
C7—C6—C5	109.30 (9)	N1—C19—C20	111.20 (9)
C7—C6—H6A	109.8	N1—C19—H19A	109.4
C5—C6—H6A	109.8	C20—C19—H19A	109.4

C7—C6—H6B	109.8	N1—C19—H19B	109.4
C5—C6—H6B	109.8	C20—C19—H19B	109.4
H6A—C6—H6B	108.3	H19A—C19—H19B	108.0
C8—C7—C6	112.80 (10)	C24—C20—C21	117.53 (11)
C8—C7—H7A	109.0	C24—C20—C19	121.78 (11)
C6—C7—H7A	109.0	C21—C20—C19	120.60 (11)
C8—C7—H7B	109.0	N2—C21—C20	123.45 (12)
C6—C7—H7B	109.0	N2—C21—H21A	118.3
H7A—C7—H7B	107.8	C20—C21—H21A	118.3
C9—C8—C14	119.37 (11)	N2—C22—C23	123.49 (12)
C9—C8—C7	121.94 (10)	N2—C22—H22A	118.3
C14—C8—C7	118.65 (11)	C23—C22—H22A	118.3
C8—C9—C11	118.56 (11)	C24—C23—C22	117.94 (12)
C8—C9—C10	122.77 (10)	C24—C23—H23A	121.0
C11—C9—C10	118.62 (10)	C22—C23—H23A	121.0
C9—C10—C1	110.06 (9)	C23—C24—C20	119.92 (11)
C9—C10—C17	106.91 (10)	C23—C24—H24B	120.0
C1—C10—C17	109.63 (10)	C20—C24—H24B	120.0
C9—C10—C5	109.06 (9)		
C10—C1—C2—C3	55.32 (14)	C6—C5—C10—C17	67.25 (12)
C1—C2—C3—C4	-54.19 (14)	C4—C5—C10—C17	-66.84 (13)
C2—C3—C4—C16	169.37 (10)	C8—C9—C11—C12	-1.86 (18)
C2—C3—C4—C15	-74.25 (12)	C10—C9—C11—C12	-179.28 (11)
C2—C3—C4—C5	51.79 (13)	C18—O2—C12—C11	-2.5 (2)
C16—C4—C5—C6	59.87 (13)	C18—O2—C12—C13	178.42 (13)
C3—C4—C5—C6	175.71 (9)	C9—C11—C12—O2	-176.22 (12)
C15—C4—C5—C6	-61.44 (13)	C9—C11—C12—C13	2.9 (2)
C16—C4—C5—C10	-169.05 (10)	O2—C12—C13—C14	177.65 (12)
C3—C4—C5—C10	-53.20 (13)	C11—C12—C13—C14	-1.5 (2)
C15—C4—C5—C10	69.64 (13)	C12—C13—C14—C8	-0.8 (2)
C10—C5—C6—C7	66.96 (12)	C9—C8—C14—C13	1.7 (2)
C4—C5—C6—C7	-159.68 (9)	C7—C8—C14—C13	-176.25 (12)
C5—C6—C7—C8	-48.11 (13)	C19—N1—C15—O1	6.78 (17)
C6—C7—C8—C9	18.28 (17)	C19—N1—C15—C4	-174.29 (10)
C6—C7—C8—C14	-163.79 (12)	C16—C4—C15—O1	123.05 (12)
C14—C8—C9—C11	-0.39 (18)	C3—C4—C15—O1	7.33 (15)
C7—C8—C9—C11	177.52 (11)	C5—C4—C15—O1	-114.76 (13)
C14—C8—C9—C10	176.92 (11)	C16—C4—C15—N1	-55.86 (14)
C7—C8—C9—C10	-5.17 (18)	C3—C4—C15—N1	-171.58 (10)
C8—C9—C10—C1	140.03 (11)	C5—C4—C15—N1	66.34 (13)
C11—C9—C10—C1	-42.67 (14)	C15—N1—C19—C20	77.78 (14)
C8—C9—C10—C17	-100.98 (12)	N1—C19—C20—C24	79.61 (14)
C11—C9—C10—C17	76.33 (13)	N1—C19—C20—C21	-96.98 (13)
C8—C9—C10—C5	21.65 (15)	C22—N2—C21—C20	0.60 (18)
C11—C9—C10—C5	-161.05 (10)	C24—C20—C21—N2	-1.76 (17)
C2—C1—C10—C9	-173.60 (10)	C19—C20—C21—N2	174.97 (11)
C2—C1—C10—C17	69.08 (13)	C21—N2—C22—C23	0.94 (17)
C2—C1—C10—C5	-54.60 (13)	N2—C22—C23—C24	-1.21 (18)
C6—C5—C10—C9	-51.57 (12)	C22—C23—C24—C20	-0.04 (17)

supplementary materials

C4—C5—C10—C9	174.34 (9)	C21—C20—C24—C23	1.42 (16)
C6—C5—C10—C1	-171.20 (9)	C19—C20—C24—C23	-175.27 (11)
C4—C5—C10—C1	54.71 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1C \cdots N2 ⁱ	0.86	2.22	3.0311 (14)	157
C23—H23A \cdots O1 ⁱⁱ	0.93	2.45	3.2386 (16)	143

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y+1, z$.

Fig. 1

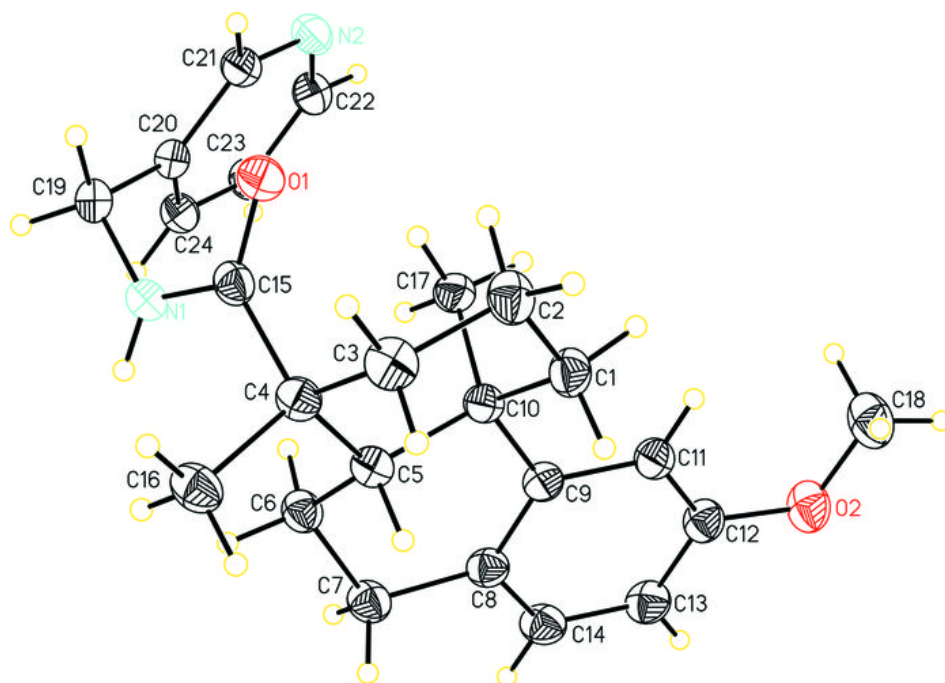


Fig. 2

