Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetramethyl-1-(4-chlorobenzyl)-3a,7a-dihydro-1*H*-indole-2,3,3a,4-tetracarboxylate

Chang Kiu Lee,[a] Youngjin Kang,[a] Kiuook Jeon,[a] Suk-Hee Moon[b] and Ki-Min Park[c]*

[a]Division of Science Education and Department of Chemistry, Kangwon National University, Chuncheon 200-701, Republic of Korea, [b]Subdivision of Food Science, Kyungnam College of Information and Technology, Busan 616-701, Republic of Korea, and [c]Research Institute of Natural Science, Gyeongsang National University, Jinju 660-701, Republic of Korea
Correspondence e-mail: kmpark@gnu.ac.kr

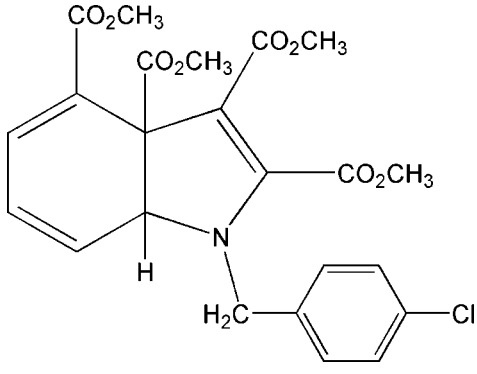
Received 28 August 2007; accepted 2 September 2007

Key indicators: single-crystal X-ray study; T = 446 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 17.2.

In the title compound, $C_{23}H_{22}ClNO_8$, the core structure, which consists of fused cyclohexadiene and pyrrole rings, is bent with a dihedral angle of 64.11 (5)° between the ring planes owing to a lack of π conjugation within the heterobicycle. Molecules are linked by an extensive network of intermolecular C—H···O hydrogen bonds.

Related literature

For details of the preparations of the title compound and related compounds, see: Acheson & Vernon (1962); Jones & Bean (1977); Lee *et al.* (1978). For related structures, see: Roychowdhury & Basak (1975); Preut *et al.* (1991).



Experimental

Crystal data

$C_{23}H_{22}ClNO_8$
M_r = 475.87
Monoclinic, $P2_1/n$
a = 12.8844 (8) Å
b = 12.3350 (7) Å
c = 15.3485 (9) Å
β = 112.471 (1)°
V = 2254.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 446 (2) K
0.50 × 0.35 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
13936 measured reflections
5132 independent reflections
3989 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.06
5132 reflections
298 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O5^{i}	0.93	2.56	3.441 (2)	159
C3—H3···O3^{ii}	0.93	2.53	3.220 (2)	132
C16—H16A···O5^{iii}	0.96	2.51	3.269 (2)	136
C17—H17B···O5^{i}	0.97	2.53	3.390 (2)	148
C19—H19···O7^{i}	0.93	2.56	3.485 (2)	177

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This research was supported by a grant (F0004021) from Information Display R&D Center, one of the 21st Century Frontier R&D Programs funded by the Ministry of Commerce, Industry and Energy of the Korean government.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2346).

References

Acheson, R. M. & Vernon, J. M. (1962). *J. Chem. Soc.* pp. 1148–1157.
Bruker. (2000). *SMART* (Version 5.625), *SAINT-Plus* (Version 6.22) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Jones, R. A. & Bean, G. P. (1977). *The Chemistry of Pyrroles*, pp. 146–256. New York: Academic Press.
Lee, C. K., Hahn, C. S. & Noland, W. E. (1978). *J. Org. Chem.* **43**, 3727–3729.
Preut, H., Götte, H. & Kreher, R. P. (1991). *Acta Cryst.* C**47**, 2242–2245.
Roychowdhury, P. & Basak, B. S. (1975). *Acta Cryst.* B**31**, 1559–1563.

 doi:10.1107/S1600536807042894

supplementary materials

Acta Cryst. (2007). E**63**, o4011 [**doi:10.1107/S1600536807042894**]

Tetramethyl-1-(4-chlorobenzyl)-3a,7a-dihydro-1*H*-indole-2,3,3a,4-tetracarboxylate

C. K. Lee, Y. Kang, K. Jeon, S.-H. Moon and K.-M. Park

Comment

The reaction of *N*-(4-chlorobenzyl)-1*H*-pyrrole with dimethyl acetylenedicarboxylate (DMAD) leads to the formation of the title compound, (I), (Fig. 1), *via* initial Diels-Alder addition to form a 7-azanorbornadiene intermediate and subsequent Michael-type addition of the second molecule of DMAD (Acheson & Vernon, 1962; Jones & Bean, 1977; Lee *et al.*, 1978).

The title compound, (I), crystallizes as a racemic mixture in the centrosymmetric space group $P2_1/c$ and the C7-*S* and C8-*R* isomer is shown in Fig. 1. Owing to a lack of π-conjugation within the hetero-bicycle of (I), the core structure is bent. The dihedral angle between the cyclohexadiene and pyrrole rings is 64.11 (5)° compared to the indole compound which was planar (Roychowdhury & Basak, 1975). The torsion angles C4–C7–C8–C1 and C5–C7–C8–N1 are 31.1 (2) Å and 26.2 (1) Å, respectively, indicating a *syn*-configuration at chiral centers of the moiety hetero-bicycle, similar to that of the related compound reported by Preut *et al.* (1991). In pyrrole unit, the N1–C6 distance (1.351 (2) Å) is similar with that of C6–C5 (1.356 (2) Å) and the sum of the bond angle around N1 is 359.4°. These facts indicate that a lone pair of nitrogen atom (N1) is delocalized. The dihedral angle between the plane of the benzene ring and the plane of the ester group attached on C7 is 7.37 (9)°, indicating an almost face-to-face orientation.

The crystal packing of (I) is stabilized by weak intermolecular C–H···O hydrogen bonds, Table 1. The dihedral angle between the pyrrole and benzene rings is 81.19 (6)° and there is no evidence of π–π intermolecular interactions between benzene rings in the crystal packing.

Experimental

Compound (I) was prepared by literature methods (Lee *et al.*, 1978). Crystals of (I) were obtained by slow evaporation of a methanol solution in a refrigerator.

Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2 and 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms.

Figures

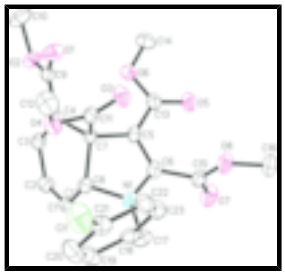


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. All H atoms except hydrogen atom at the chiral center are omitted for clarity.

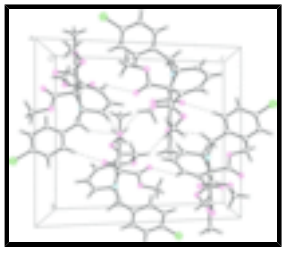


Fig. 2. The crystal packing in the title compound, viewed down the *a* axis. Dashed lines indicate C–H···O hydrogen bonds.

Tetramethyl-1-(4-chlorobenzyl)-3a,7a-dihydro-1*H*-indole-2,3,3a,4- tetracarboxylate

Crystal data

$C_{23}H_{22}ClNO_8$	F_{000} = 992
M_r = 475.87	D_x = 1.402 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6747 reflections
a = 12.8844 (8) Å	θ = 2.4–28.2º
b = 12.3350 (7) Å	μ = 0.22 mm^{-1}
c = 15.3485 (9) Å	T = 446 (2) K
β = 112.471 (1)º	Block, yellow
V = 2254.1 (2) Å^3	0.50 × 0.35 × 0.35 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3989 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.030
Monochromator: graphite	θ_{max} = 27.5º
T = 446(2) K	θ_{min} = 1.8º
φ and ω scans	h = −16→16
Absorption correction: none	k = −15→14
13936 measured reflections	l = −19→11
5132 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.7066P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = <0.001$
5132 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
298 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.81143 (4)	0.32249 (4)	0.20324 (3)	0.04669 (13)
O1	0.45128 (10)	0.85362 (10)	−0.04992 (8)	0.0398 (3)
O2	0.33449 (9)	0.91151 (9)	−0.19189 (8)	0.0325 (3)
O3	0.69018 (9)	0.76987 (9)	0.03583 (7)	0.0326 (3)
O4	0.55635 (9)	0.64221 (9)	−0.01694 (7)	0.0311 (2)
O5	0.77351 (9)	0.98920 (9)	−0.13061 (8)	0.0353 (3)
O6	0.59618 (9)	0.98517 (8)	−0.13705 (8)	0.0305 (2)
O7	0.89923 (10)	0.81442 (11)	−0.20986 (8)	0.0422 (3)
O8	0.95022 (9)	0.81130 (10)	−0.05262 (8)	0.0352 (3)
N1	0.74121 (10)	0.65145 (10)	−0.15598 (9)	0.0271 (3)
C1	0.55594 (13)	0.62580 (13)	−0.28022 (11)	0.0290 (3)
H1	0.5841	0.5799	−0.3140	0.035*
C2	0.46045 (13)	0.67819 (13)	−0.32558 (10)	0.0297 (3)
H2	0.4229	0.6680	−0.3901	0.036*
C3	0.41351 (12)	0.75158 (12)	−0.27551 (10)	0.0252 (3)
H3	0.3411	0.7780	−0.3066	0.030*
C4	0.47138 (11)	0.78200 (11)	−0.18640 (10)	0.0216 (3)
C5	0.68261 (11)	0.82092 (12)	−0.14121 (10)	0.0235 (3)

C6	0.76738 (12)	0.75793 (12)	−0.14440 (9)	0.0247 (3)
C7	0.59195 (11)	0.74492 (11)	−0.13385 (9)	0.0209 (3)
C8	0.61985 (11)	0.63855 (12)	−0.17588 (10)	0.0242 (3)
H8	0.6078	0.5753	−0.1423	0.029*
C9	0.42044 (12)	0.85190 (12)	−0.13475 (10)	0.0253 (3)
C10	0.27820 (17)	0.97607 (16)	−0.14419 (15)	0.0472 (5)
H10A	0.2180	1.0161	−0.1899	0.071*
H10B	0.3308	1.0256	−0.1017	0.071*
H10C	0.2485	0.9293	−0.1094	0.071*
C11	0.61854 (12)	0.72414 (12)	−0.02791 (10)	0.0239 (3)
C12	0.57395 (16)	0.61703 (15)	0.07972 (11)	0.0397 (4)
H12A	0.5260	0.5580	0.0810	0.060*
H12B	0.5565	0.6795	0.1089	0.060*
H12C	0.6510	0.5969	0.1135	0.060*
C13	0.69099 (12)	0.93752 (12)	−0.13481 (10)	0.0243 (3)
C14	0.59806 (16)	1.10186 (13)	−0.13296 (13)	0.0374 (4)
H14A	0.5269	1.1279	−0.1352	0.056*
H14B	0.6122	1.1303	−0.1856	0.056*
H14C	0.6564	1.1251	−0.0753	0.056*
C15	0.87881 (12)	0.79818 (13)	−0.14117 (10)	0.0275 (3)
C16	1.05624 (14)	0.86206 (17)	−0.04081 (14)	0.0451 (4)
H16A	1.1019	0.8681	0.0252	0.068*
H16B	1.0425	0.9330	−0.0687	0.068*
H16C	1.0946	0.8187	−0.0711	0.068*
C17	0.81662 (13)	0.55840 (13)	−0.13847 (11)	0.0309 (3)
H17A	0.8915	0.5832	−0.1293	0.037*
H17B	0.7909	0.5106	−0.1928	0.037*
C18	0.82047 (12)	0.49683 (12)	−0.05216 (10)	0.0273 (3)
C19	0.75483 (14)	0.40459 (13)	−0.06175 (12)	0.0367 (4)
H19	0.7134	0.3777	−0.1217	0.044*
C20	0.75027 (15)	0.35201 (14)	0.01665 (12)	0.0385 (4)
H20	0.7047	0.2915	0.0097	0.046*
C21	0.81471 (13)	0.39120 (13)	0.10511 (11)	0.0307 (3)
C22	0.88103 (13)	0.48238 (13)	0.11721 (11)	0.0298 (3)
H22	0.9239	0.5076	0.1774	0.036*
C23	0.88276 (12)	0.53567 (13)	0.03828 (11)	0.0286 (3)
H23	0.9259	0.5980	0.0457	0.034*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0551 (3)	0.0451 (3)	0.0408 (2)	−0.0104 (2)	0.0193 (2)	0.00712 (19)
O1	0.0433 (7)	0.0506 (7)	0.0282 (6)	0.0109 (6)	0.0167 (5)	−0.0020 (5)
O2	0.0314 (6)	0.0317 (6)	0.0380 (6)	0.0096 (5)	0.0173 (5)	0.0041 (5)
O3	0.0308 (6)	0.0401 (6)	0.0207 (5)	−0.0090 (5)	0.0029 (4)	−0.0025 (4)
O4	0.0368 (6)	0.0330 (6)	0.0227 (5)	−0.0092 (5)	0.0105 (5)	0.0017 (4)
O5	0.0313 (6)	0.0327 (6)	0.0439 (7)	−0.0088 (5)	0.0166 (5)	−0.0001 (5)
O6	0.0300 (5)	0.0242 (5)	0.0386 (6)	−0.0011 (4)	0.0147 (5)	−0.0027 (4)

O7	0.0346 (6)	0.0659 (9)	0.0307 (6)	−0.0049 (6)	0.0176 (5)	0.0026 (6)
O8	0.0209 (5)	0.0550 (7)	0.0267 (6)	−0.0030 (5)	0.0059 (4)	−0.0013 (5)
N1	0.0206 (6)	0.0286 (7)	0.0306 (7)	0.0043 (5)	0.0079 (5)	0.0017 (5)
C1	0.0287 (8)	0.0320 (8)	0.0265 (8)	−0.0002 (6)	0.0107 (6)	−0.0079 (6)
C2	0.0291 (8)	0.0369 (8)	0.0195 (7)	−0.0016 (6)	0.0053 (6)	−0.0054 (6)
C3	0.0199 (6)	0.0299 (8)	0.0233 (7)	0.0017 (6)	0.0052 (5)	0.0020 (6)
C4	0.0194 (6)	0.0227 (7)	0.0221 (7)	−0.0011 (5)	0.0072 (5)	0.0020 (5)
C5	0.0202 (7)	0.0286 (7)	0.0204 (7)	−0.0015 (6)	0.0065 (5)	0.0021 (5)
C6	0.0217 (7)	0.0332 (8)	0.0175 (6)	0.0002 (6)	0.0056 (5)	0.0030 (6)
C7	0.0191 (6)	0.0228 (7)	0.0195 (6)	−0.0010 (5)	0.0058 (5)	−0.0003 (5)
C8	0.0213 (7)	0.0253 (7)	0.0245 (7)	0.0008 (5)	0.0071 (6)	0.0000 (6)
C9	0.0230 (7)	0.0260 (7)	0.0285 (8)	−0.0021 (6)	0.0117 (6)	0.0003 (6)
C10	0.0501 (11)	0.0436 (10)	0.0625 (12)	0.0198 (9)	0.0379 (10)	0.0090 (9)
C11	0.0216 (7)	0.0255 (7)	0.0232 (7)	0.0013 (6)	0.0070 (6)	0.0011 (5)
C12	0.0509 (10)	0.0430 (10)	0.0263 (8)	−0.0086 (8)	0.0159 (8)	0.0056 (7)
C13	0.0258 (7)	0.0288 (7)	0.0180 (6)	−0.0035 (6)	0.0080 (5)	0.0002 (5)
C14	0.0479 (10)	0.0251 (8)	0.0403 (9)	0.0016 (7)	0.0180 (8)	−0.0034 (7)
C15	0.0219 (7)	0.0350 (8)	0.0256 (7)	0.0023 (6)	0.0089 (6)	0.0020 (6)
C16	0.0218 (8)	0.0564 (12)	0.0529 (11)	−0.0061 (8)	0.0095 (8)	−0.0097 (9)
C17	0.0293 (8)	0.0363 (8)	0.0270 (8)	0.0114 (7)	0.0107 (6)	0.0015 (6)
C18	0.0256 (7)	0.0278 (8)	0.0265 (7)	0.0097 (6)	0.0078 (6)	−0.0001 (6)
C19	0.0400 (9)	0.0294 (8)	0.0291 (8)	0.0020 (7)	0.0002 (7)	−0.0047 (7)
C20	0.0395 (9)	0.0263 (8)	0.0392 (9)	−0.0054 (7)	0.0031 (7)	−0.0002 (7)
C21	0.0319 (8)	0.0282 (8)	0.0313 (8)	0.0028 (6)	0.0113 (7)	0.0026 (6)
C22	0.0300 (8)	0.0307 (8)	0.0257 (8)	−0.0011 (6)	0.0074 (6)	−0.0043 (6)
C23	0.0256 (7)	0.0279 (8)	0.0302 (8)	−0.0005 (6)	0.0085 (6)	−0.0017 (6)

Geometric parameters (Å, °)

Cl1—C21	1.7428 (16)	C7—C11	1.5495 (19)
O1—C9	1.2074 (18)	C7—C8	1.5634 (19)
O2—C9	1.3393 (18)	C8—H8	0.9800
O2—C10	1.4499 (19)	C10—H10A	0.9600
O3—C11	1.1988 (17)	C10—H10B	0.9600
O4—C11	1.3397 (18)	C10—H10C	0.9600
O4—C12	1.4464 (18)	C12—H12A	0.9600
O5—C13	1.2201 (17)	C12—H12B	0.9600
O6—C13	1.3441 (18)	C12—H12C	0.9600
O6—C14	1.4405 (19)	C14—H14A	0.9600
O7—C15	1.1971 (18)	C14—H14B	0.9600
O8—C15	1.3281 (18)	C14—H14C	0.9600
O8—C16	1.450 (2)	C16—H16A	0.9600
N1—C6	1.351 (2)	C16—H16B	0.9600
N1—C17	1.4608 (19)	C16—H16C	0.9600
N1—C8	1.4827 (18)	C17—C18	1.511 (2)
C1—C2	1.327 (2)	C17—H17A	0.9700
C1—C8	1.503 (2)	C17—H17B	0.9700
C1—H1	0.9300	C18—C19	1.392 (2)
C2—C3	1.460 (2)	C18—C23	1.396 (2)

C2—H2	0.9300	C19—C20	1.388 (2)
C3—C4	1.337 (2)	C19—H19	0.9300
C3—H3	0.9300	C20—C21	1.380 (2)
C4—C9	1.483 (2)	C20—H20	0.9300
C4—C7	1.5216 (18)	C21—C22	1.381 (2)
C5—C6	1.356 (2)	C22—C23	1.386 (2)
C5—C13	1.443 (2)	C22—H22	0.9300
C5—C7	1.5352 (19)	C23—H23	0.9300
C6—C15	1.502 (2)		
C9—O2—C10	114.76 (13)	O4—C12—H12A	109.5
C11—O4—C12	115.10 (12)	O4—C12—H12B	109.5
C13—O6—C14	115.84 (12)	H12A—C12—H12B	109.5
C15—O8—C16	115.57 (13)	O4—C12—H12C	109.5
C6—N1—C17	128.67 (13)	H12A—C12—H12C	109.5
C6—N1—C8	108.78 (11)	H12B—C12—H12C	109.5
C17—N1—C8	121.90 (12)	O5—C13—O6	122.55 (14)
C2—C1—C8	121.90 (13)	O5—C13—C5	124.43 (14)
C2—C1—H1	119.0	O6—C13—C5	112.99 (12)
C8—C1—H1	119.0	O6—C14—H14A	109.5
C1—C2—C3	120.89 (13)	O6—C14—H14B	109.5
C1—C2—H2	119.6	H14A—C14—H14B	109.5
C3—C2—H2	119.6	O6—C14—H14C	109.5
C4—C3—C2	122.09 (13)	H14A—C14—H14C	109.5
C4—C3—H3	119.0	H14B—C14—H14C	109.5
C2—C3—H3	119.0	O7—C15—O8	125.55 (14)
C3—C4—C9	121.48 (13)	O7—C15—C6	123.73 (14)
C3—C4—C7	121.00 (12)	O8—C15—C6	110.71 (12)
C9—C4—C7	117.52 (12)	O8—C16—H16A	109.5
C6—C5—C13	122.22 (13)	O8—C16—H16B	109.5
C6—C5—C7	107.38 (12)	H16A—C16—H16B	109.5
C13—C5—C7	130.01 (12)	O8—C16—H16C	109.5
N1—C6—C5	113.44 (13)	H16A—C16—H16C	109.5
N1—C6—C15	120.87 (13)	H16B—C16—H16C	109.5
C5—C6—C15	125.60 (14)	N1—C17—C18	110.66 (12)
C4—C7—C5	115.39 (11)	N1—C17—H17A	109.5
C4—C7—C11	112.18 (11)	C18—C17—H17A	109.5
C5—C7—C11	107.99 (11)	N1—C17—H17B	109.5
C4—C7—C8	112.51 (11)	C18—C17—H17B	109.5
C5—C7—C8	100.41 (11)	H17A—C17—H17B	108.1
C11—C7—C8	107.50 (11)	C19—C18—C23	118.75 (14)
N1—C8—C1	109.62 (12)	C19—C18—C17	120.20 (14)
N1—C8—C7	102.46 (11)	C23—C18—C17	120.86 (14)
C1—C8—C7	114.12 (12)	C20—C19—C18	121.05 (15)
N1—C8—H8	110.1	C20—C19—H19	119.5
C1—C8—H8	110.1	C18—C19—H19	119.5
C7—C8—H8	110.1	C21—C20—C19	118.71 (16)
O1—C9—O2	123.05 (14)	C21—C20—H20	120.6
O1—C9—C4	123.78 (14)	C19—C20—H20	120.6
O2—C9—C4	113.15 (12)	C20—C21—C22	121.74 (15)

O2—C10—H10A	109.5	C20—C21—Cl1	118.40 (13)
O2—C10—H10B	109.5	C22—C21—Cl1	119.85 (12)
H10A—C10—H10B	109.5	C21—C22—C23	118.97 (14)
O2—C10—H10C	109.5	C21—C22—H22	120.5
H10A—C10—H10C	109.5	C23—C22—H22	120.5
H10B—C10—H10C	109.5	C22—C23—C18	120.74 (15)
O3—C11—O4	124.28 (13)	C22—C23—H23	119.6
O3—C11—C7	124.82 (13)	C18—C23—H23	119.6
O4—C11—C7	110.71 (11)		
C8—C1—C2—C3	0.4 (2)	C3—C4—C9—O1	155.02 (15)
C1—C2—C3—C4	10.3 (2)	C7—C4—C9—O1	−24.3 (2)
C2—C3—C4—C9	−176.68 (13)	C3—C4—C9—O2	−23.48 (19)
C2—C3—C4—C7	2.6 (2)	C7—C4—C9—O2	157.18 (12)
C17—N1—C6—C5	−162.44 (14)	C12—O4—C11—O3	5.4 (2)
C8—N1—C6—C5	8.31 (16)	C12—O4—C11—C7	−179.31 (12)
C17—N1—C6—C15	20.7 (2)	C4—C7—C11—O3	−120.62 (15)
C8—N1—C6—C15	−168.60 (12)	C5—C7—C11—O3	7.64 (19)
C13—C5—C6—N1	−176.27 (13)	C8—C7—C11—O3	115.19 (16)
C7—C5—C6—N1	10.27 (16)	C4—C7—C11—O4	64.08 (15)
C13—C5—C6—C15	0.5 (2)	C5—C7—C11—O4	−167.66 (11)
C7—C5—C6—C15	−172.99 (13)	C8—C7—C11—O4	−60.11 (14)
C3—C4—C7—C5	91.32 (16)	C14—O6—C13—O5	−0.8 (2)
C9—C4—C7—C5	−89.34 (15)	C14—O6—C13—C5	−178.73 (12)
C3—C4—C7—C11	−144.44 (13)	C6—C5—C13—O5	−0.9 (2)
C9—C4—C7—C11	34.90 (17)	C7—C5—C13—O5	170.93 (14)
C3—C4—C7—C8	−23.09 (18)	C6—C5—C13—O6	177.00 (12)
C9—C4—C7—C8	156.26 (12)	C7—C5—C13—O6	−11.2 (2)
C6—C5—C7—C4	−144.05 (12)	C16—O8—C15—O7	7.9 (2)
C13—C5—C7—C4	43.2 (2)	C16—O8—C15—C6	−172.97 (14)
C6—C5—C7—C11	89.54 (13)	N1—C6—C15—O7	81.1 (2)
C13—C5—C7—C11	−83.23 (17)	C5—C6—C15—O7	−95.4 (2)
C6—C5—C7—C8	−22.86 (14)	N1—C6—C15—O8	−98.03 (16)
C13—C5—C7—C8	164.37 (14)	C5—C6—C15—O8	85.46 (17)
C6—N1—C8—C1	99.12 (14)	C6—N1—C17—C18	108.34 (17)
C17—N1—C8—C1	−89.38 (16)	C8—N1—C17—C18	−61.33 (18)
C6—N1—C8—C7	−22.42 (14)	N1—C17—C18—C19	97.88 (17)
C17—N1—C8—C7	149.08 (13)	N1—C17—C18—C23	−77.16 (17)
C2—C1—C8—N1	−135.62 (15)	C23—C18—C19—C20	0.4 (2)
C2—C1—C8—C7	−21.4 (2)	C17—C18—C19—C20	−174.75 (15)
C4—C7—C8—N1	149.46 (11)	C18—C19—C20—C21	−1.7 (3)
C5—C7—C8—N1	26.23 (13)	C19—C20—C21—C22	1.6 (3)
C11—C7—C8—N1	−86.55 (12)	C19—C20—C21—Cl1	−178.26 (13)
C4—C7—C8—C1	31.05 (16)	C20—C21—C22—C23	0.0 (2)
C5—C7—C8—C1	−92.18 (13)	Cl1—C21—C22—C23	179.79 (12)
C11—C7—C8—C1	155.04 (12)	C21—C22—C23—C18	−1.4 (2)
C10—O2—C9—O1	−2.0 (2)	C19—C18—C23—C22	1.2 (2)
C10—O2—C9—C4	176.49 (13)	C17—C18—C23—C22	176.28 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O5^{i}	0.93	2.56	3.441 (2)	159
C3—H3···O3^{ii}	0.93	2.53	3.220 (2)	132
C16—H16A···O5^{iii}	0.96	2.51	3.269 (2)	136
C17—H17B···O5^{i}	0.97	2.53	3.390 (2)	148
C19—H19···O7^{i}	0.93	2.56	3.485 (2)	177

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

Fig. 1

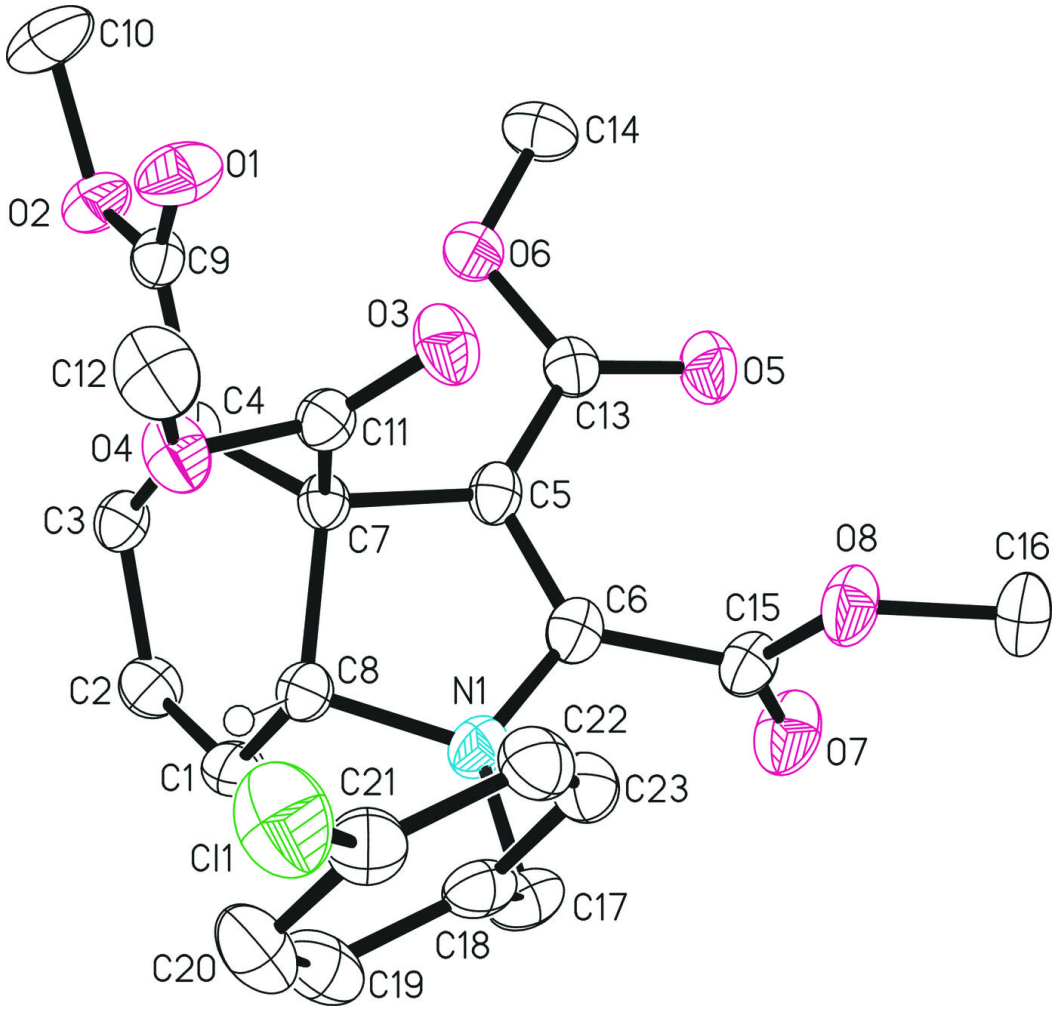

C10
O1
O2
C9
C14
O6
O3
C12
C4
C11
C13
O5
O4
C5
C3
C7
O8
C16
C6
C15
C2
C8
N1
C22
O7
C1
C21
C23
Cl1
C17
C18
C20
C19

Fig. 2