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(*R**)-(−)-3-[4-(Benzyloxy)benzoylmethyl] 5-ethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

Xiao-Yun Wu,[a] Ai-Xi Hu[b]* and Gao Cao[c]

[a]School of Pharmaceutical Sciences, Southern Medical University, Guangzhou 510515, People's Republic of China, [b]College of Chemistry and Chemical Engineering, Hunan University, Changsha 410082, People's Republic of China, and [c]School of Chemical and Energy Engineering, South China University of Technology, Guangzhou 510640, People's Republic of China
Correspondence e-mail: axhu0731@yahoo.com.cn

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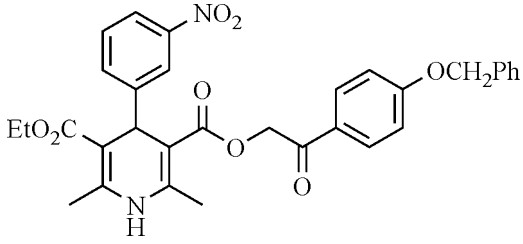
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.009 Å; R factor = 0.045; wR factor = 0.144; data-to-parameter ratio = 7.4.

In the optically active title compound, $C_{32}H_{30}N_2O_8$, the substituted 1,4-dihydropyridine ring adopts a flattened boat conformation. The crystal structure is stabilized by intermolecular N—H···O hydrogen bonding.

Related literature

The importance of 1,4-dihydropyridine derivatives was summarized by Goldmann & Stoltefuss (1991). A series of new 1,4-dihydropyridine compounds has been designed and synthesized by us (Wu *et al.*, 2006).



Experimental

Crystal data

$C_{32}H_{30}N_2O_8$
M_r = 570.58
Triclinic, $P\overline{1}$
a = 7.3646 (7) Å
b = 8.1772 (8) Å
c = 13.329 (1) Å
α = 97.905 (2)°
β = 100.352 (2)°
γ = 109.296 (2)°
V = 728.33 (12) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
0.46 × 0.28 × 0.16 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: none
5566 measured reflections
2808 independent reflections
1747 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.144
S = 1.02
2808 reflections
382 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.11	2.940 (5)	163

Symmetry code: (i) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: XU2347).

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supplementary materials

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(*R**)-(-)-3-[4-(Benzyloxy)benzoylmethyl] 5-ethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

X.-Y. Wu, A.-X. Hu and G. Cao

Comment

1,4-dihydropyridine derivatives have been widely investigated from the pharmacological point of view since Nifedipine was found to be highly effective calcium antagonist in 1975, and many compounds similar to Nifedipine in structure have already been widely used as therapeutic agents for the treatment of cerebra circulatory disorder, hypertension and so on (Goldmann & Stoltefuss 1991). According to the structure–activity relationship of 1,4-dihydropyridine calcium antagonists, a serials of new compounds were designed and synthesized (Wu *et al.*, 2006). The optical active title compound has been synthesized by the reaction of optical active 1,4-dihydropyridine monoester with a\-bromoalkylaryl ketone under mild conditions.

The 1,4-dihydropyridne (DHP) ring has a flattened boat conformation, N1 and C3 deviating from the mean plane by −0.185 (4) Å and −0.247 (5) Å, respectively. The dihedral angle found between plane C1/C2/C4/C5 and C2/C3/C4, C1/N1/N5 are 31.1° and 18.4°, respectively. Both the 3-nitrophenyl ring (C27–C32) and the DHP ring (N1, C1, C2, C3, C4, C5) are almost perpendicular to each other, the dihedral angle found between them is 80.8 (3)°. The phenyl rings C14/C15/C16/C17/C18/C19 and C21/C22/C23/C24/C25/C26 are almost perpendicular to each other, too, the dihedral angle found between them is 87.5 (3)°. The structure has intermolecular hydrogen bonds of the type N—H···O between the amine of one molecule and the carbonyl oxygen of neighbouring molecule.

Experimental

Cinchonidine (15 mmol) and racemic acid 1,4-dihydropyridine monoester, ethyl 1,4-dihydro-2,6-dimethyl-4-(3-nitrophenyl)-3,5-pyridinedicarboxylate (15 mmol) were stirred in reflux ethanol (35 ml) until the dissolution was complete, and then kept at room temperature for 24 h. The crystals formed were collected by filtration to give Cinchonidine salt (3.1 g). The 1,4-dihydropyridine monoester was obtained by dissolving of the Cinchonidine salt and sodium hydroxide in water. The solution were acidified with HCl, filtered, washed with water to give (-)-ethyl 1,4-dihydro-2,6-dimethyl-4-(3-nitrophenyl)-3,5-pyridinedicarboxylate (1.54 g, yield 31%, $[\alpha]_D^{20} = -4.4°$). The absolute configuration of the enantiomer has not been assigned.

Optical active 1,4-dihydropyridine monoester (5 mmol), 2-bromo-1-(4-benzyloxyphenyl)ethan-1-one (5 mmol) and K_2CO_3 (5 mmol) in DMF (5 ml) were stirred overnight at room temperature. The mixture was extracted with ethyl acetate, washed successively with water and brine, and then dried and the solvent was removed. The residue was purified by crystallization to give target compound (yield 75%, $[\alpha]_D^{20} = -93.1°$).

Crystals suitable for X-ray analysis were obtained by slow evaporation from saturated methanol solution.

Refinement

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined, with U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in geometrically idealized positions and refined as riding model, with N—H = 0.86 A°, C—H = 0.98 (methine), 0.93 (aromatic) and 0.97 Å (methylene). The constraint U_{iso}(H) = 1.2U_{eq}(carrier) was applied. In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Figures

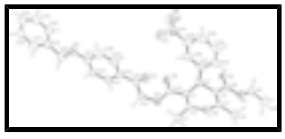

Fig. 1. Molecular structure of (I) showing 30% probability displacement ellipsoids.

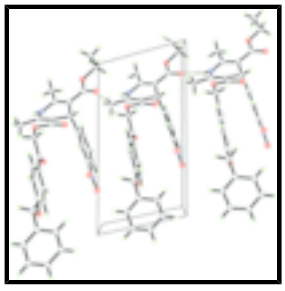

Fig. 2. Packing diagram.

(*R**)-(-)-3-[4-(Benzyloxy)benzoylmethyl] 5-ethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

Crystal data

$C_{32}H_{30}N_2O_8$	Z = 1
M_r = 570.58	F_{000} = 300
Triclinic, $P\overline{1}$	D_x = 1.301 Mg m^{-3}
Hall symbol: P 1	Melting point: 122 K
a = 7.3646 (7) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 8.1772 (8) Å	Cell parameters from 1853 reflections
c = 13.329 (1) Å	μ = 0.09 mm^{-1}
α = 97.905 (2)º	T = 295 (2) K
β = 100.352 (2)º	Block, yellow
γ = 109.296 (2)º	0.46 × 0.28 × 0.16 mm
V = 728.33 (12) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	1747 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.030

Monochromator: graphite
T = 295(2) K
ω scans
Absorption correction: none
5566 measured reflections
2808 independent reflections

θ_{max} = 26.0º
θ_{min} = 1.6º
h = −9→9
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.144
S = 1.02
2808 reflections
382 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: none

Special details

Experimental. ^{1}H NMR ($CDCl_3$, 400 MHz) (p.p.m.): 1.22 (t, J = 7.2 Hz, 3H, CH_3),2.39 (s, 3H, CH_3), 2.42(s, 3H, CH_3), 3.96–4.13 (m, 2H, CH_2), 5.13 (s, 2H, CH_2), 5.20 (s, 1H, DHP 4-H), 5.24, 5.29 (dd, J =16.4 Hz, J =16.4 Hz, 2H, CO_2CH_2CO), 5.98 (s, 1H, NH), 6.97–8.13 (m, 13H, Ar—H).

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}
C1	0.6718 (6)	0.3591 (6)	0.7572 (4)	0.0533 (11)
C2	0.5034 (6)	0.3903 (6)	0.7313 (4)	0.0513 (10)
C3	0.3081 (6)	0.2467 (6)	0.7268 (4)	0.0536 (11)
H3	0.2150	0.3049	0.7396	0.064*
C4	0.3348 (6)	0.1501 (5)	0.8135 (3)	0.0480 (10)
C5	0.5090 (6)	0.1267 (6)	0.8418 (4)	0.0556 (11)

C6	0.5611 (8)	0.0160 (8)	0.9149 (5)	0.0800 (16)
H6A	0.5869	0.0777	0.9856	0.120*
H6B	0.6771	−0.0053	0.9034	0.120*
H6C	0.4526	−0.0950	0.9023	0.120*
C7	0.8763 (7)	0.4639 (7)	0.7483 (5)	0.0768 (15)
H7A	0.8718	0.4784	0.6777	0.115*
H7B	0.9635	0.4022	0.7671	0.115*
H7C	0.9243	0.5783	0.7943	0.115*
C8	0.1677 (6)	0.0884 (5)	0.8604 (4)	0.0500 (10)
C9	0.0365 (8)	−0.0586 (8)	0.9877 (5)	0.0772 (15)
H9A	−0.0854	−0.1386	0.9389	0.093*
H9B	0.0125	0.0431	1.0207	0.093*
C10	0.1013 (11)	−0.1492 (11)	1.0668 (6)	0.111 (2)
H10A	0.1136	−0.2552	1.0329	0.167*
H10B	0.0055	−0.1795	1.1079	0.167*
H10C	0.2273	−0.0721	1.1111	0.167*
C11	0.4911 (7)	0.5559 (6)	0.7029 (4)	0.0564 (11)
C12	0.6556 (7)	0.8139 (6)	0.6503 (4)	0.0625 (13)
H12A	0.7818	0.9123	0.6706	0.075*
H12B	0.5550	0.8575	0.6674	0.075*
C13	0.6058 (6)	0.7412 (6)	0.5350 (4)	0.0586 (12)
C14	0.5909 (6)	0.8605 (6)	0.4637 (4)	0.0572 (11)
C15	0.6281 (7)	1.0397 (6)	0.4971 (4)	0.0591 (12)
H15	0.6646	1.0887	0.5683	0.071*
C16	0.6120 (7)	1.1463 (6)	0.4269 (4)	0.0618 (12)
H16	0.6336	1.2650	0.4510	0.074*
C17	0.5640 (7)	1.0775 (7)	0.3213 (4)	0.0659 (13)
C18	0.5269 (10)	0.8964 (8)	0.2864 (5)	0.0866 (17)
H18	0.4928	0.8473	0.2154	0.104*
C19	0.5412 (9)	0.7935 (7)	0.3575 (4)	0.0814 (17)
H19	0.5167	0.6741	0.3336	0.098*
C20	0.5785 (11)	1.3511 (7)	0.2736 (5)	0.0891 (17)
H20A	0.7176	1.4170	0.3065	0.107*
H20B	0.5006	1.3707	0.3222	0.107*
C21	0.5186 (10)	1.4100 (7)	0.1744 (5)	0.0808 (17)
C22	0.6499 (12)	1.4713 (10)	0.1165 (6)	0.111 (2)
H22	0.7793	1.4753	0.1362	0.134*
C23	0.5874 (18)	1.5295 (11)	0.0251 (7)	0.134 (3)
H23	0.6765	1.5753	−0.0144	0.161*
C24	0.3984 (18)	1.5174 (9)	−0.0035 (7)	0.116 (3)
H24	0.3568	1.5530	−0.0640	0.140*
C25	0.2676 (13)	1.4550 (9)	0.0537 (6)	0.106 (2)
H25	0.1375	1.4486	0.0330	0.127*
C26	0.3276 (12)	1.4010 (8)	0.1424 (5)	0.0929 (19)
H26	0.2370	1.3574	0.1816	0.111*
C27	0.2206 (6)	0.1232 (7)	0.6193 (4)	0.0579 (12)
C28	0.1756 (7)	0.1925 (9)	0.5333 (4)	0.0755 (15)
H28	0.1954	0.3125	0.5424	0.091*
C29	0.1031 (8)	0.0886 (13)	0.4355 (5)	0.093 (2)

C30	0.0708 (9)	−0.0890 (15)	0.4179 (6)	0.111 (3)
H30	0.0221	−0.1584	0.3506	0.133*
C31	0.1123 (9)	−0.1625 (10)	0.5025 (6)	0.098 (2)
H31	0.0912	−0.2828	0.4921	0.117*
C32	0.1851 (7)	−0.0585 (7)	0.6024 (5)	0.0731 (14)
H32	0.2106	−0.1098	0.6588	0.088*
N1	0.6607 (5)	0.2105 (5)	0.7986 (3)	0.0565 (10)
H1	0.7535	0.1688	0.7972	0.068*
N2	0.0568 (10)	0.1652 (16)	0.3455 (6)	0.128 (3)
O1	0.0167 (4)	0.1171 (4)	0.8359 (2)	0.0587 (8)
O2	0.1918 (5)	−0.0024 (5)	0.9338 (3)	0.0745 (10)
O3	0.3392 (5)	0.5796 (4)	0.6805 (3)	0.0800 (11)
O4	0.6662 (4)	0.6790 (4)	0.7062 (3)	0.0647 (9)
O5	0.5795 (7)	0.5867 (5)	0.5045 (3)	0.0899 (12)
O6	0.5427 (6)	1.1658 (5)	0.2441 (3)	0.0836 (11)
O7	0.0752 (11)	0.3189 (14)	0.3632 (6)	0.162 (3)
O8	0.0010 (11)	0.0749 (14)	0.2595 (5)	0.183 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.046 (2)	0.050 (3)	0.072 (3)	0.026 (2)	0.020 (2)	0.013 (2)
C2	0.044 (2)	0.052 (3)	0.069 (3)	0.026 (2)	0.018 (2)	0.018 (2)
C3	0.038 (2)	0.064 (3)	0.075 (3)	0.034 (2)	0.020 (2)	0.021 (2)
C4	0.039 (2)	0.047 (2)	0.065 (3)	0.0227 (18)	0.0138 (19)	0.011 (2)
C5	0.048 (2)	0.055 (3)	0.077 (3)	0.030 (2)	0.021 (2)	0.020 (2)
C6	0.063 (3)	0.087 (4)	0.119 (5)	0.049 (3)	0.030 (3)	0.049 (3)
C7	0.052 (3)	0.068 (3)	0.124 (5)	0.030 (2)	0.032 (3)	0.031 (3)
C8	0.043 (2)	0.046 (2)	0.063 (3)	0.0201 (19)	0.009 (2)	0.012 (2)
C9	0.067 (3)	0.088 (4)	0.100 (4)	0.040 (3)	0.033 (3)	0.047 (3)
C10	0.115 (5)	0.124 (6)	0.121 (6)	0.058 (5)	0.035 (4)	0.065 (5)
C11	0.053 (3)	0.057 (3)	0.071 (3)	0.031 (2)	0.021 (2)	0.016 (2)
C12	0.055 (3)	0.040 (2)	0.093 (4)	0.019 (2)	0.015 (2)	0.017 (2)
C13	0.053 (3)	0.045 (3)	0.075 (3)	0.018 (2)	0.017 (2)	0.005 (2)
C14	0.056 (3)	0.041 (2)	0.077 (3)	0.022 (2)	0.018 (2)	0.009 (2)
C15	0.059 (3)	0.046 (3)	0.071 (3)	0.016 (2)	0.019 (2)	0.009 (2)
C16	0.069 (3)	0.042 (2)	0.075 (4)	0.021 (2)	0.019 (3)	0.010 (3)
C17	0.074 (3)	0.061 (3)	0.070 (4)	0.034 (2)	0.016 (3)	0.015 (3)
C18	0.125 (5)	0.067 (3)	0.071 (4)	0.052 (3)	0.011 (3)	−0.001 (3)
C19	0.116 (5)	0.053 (3)	0.076 (4)	0.046 (3)	0.005 (3)	0.000 (3)
C20	0.122 (5)	0.058 (3)	0.087 (4)	0.034 (3)	0.021 (3)	0.014 (3)
C21	0.115 (5)	0.047 (3)	0.070 (4)	0.020 (3)	0.019 (4)	0.009 (3)
C22	0.119 (5)	0.112 (6)	0.093 (5)	0.023 (4)	0.037 (4)	0.022 (4)
C23	0.178 (9)	0.099 (6)	0.083 (6)	−0.012 (6)	0.047 (6)	0.019 (4)
C24	0.174 (9)	0.066 (4)	0.089 (5)	0.023 (5)	0.021 (6)	0.017 (4)
C25	0.142 (6)	0.078 (4)	0.102 (5)	0.056 (4)	0.014 (5)	0.008 (4)
C26	0.133 (6)	0.073 (4)	0.088 (5)	0.051 (4)	0.035 (4)	0.024 (3)
C27	0.032 (2)	0.078 (3)	0.064 (3)	0.020 (2)	0.0151 (19)	0.010 (3)

C28	0.046 (3)	0.109 (4)	0.073 (4)	0.022 (3)	0.020 (3)	0.028 (3)
C29	0.054 (3)	0.142 (7)	0.070 (5)	0.019 (4)	0.019 (3)	0.019 (5)
C30	0.059 (4)	0.161 (9)	0.079 (5)	0.017 (5)	0.012 (3)	−0.018 (5)
C31	0.066 (4)	0.098 (5)	0.105 (6)	0.021 (3)	0.013 (4)	−0.024 (5)
C32	0.054 (3)	0.073 (4)	0.089 (4)	0.026 (3)	0.015 (3)	0.003 (3)
N1	0.044 (2)	0.053 (2)	0.088 (3)	0.0314 (17)	0.0222 (19)	0.021 (2)
N2	0.086 (4)	0.201 (9)	0.078 (5)	0.020 (5)	0.026 (4)	0.042 (6)
O1	0.0424 (17)	0.068 (2)	0.078 (2)	0.0295 (15)	0.0182 (14)	0.0267 (17)
O2	0.060 (2)	0.095 (3)	0.100 (3)	0.0494 (19)	0.0328 (18)	0.055 (2)
O3	0.065 (2)	0.071 (2)	0.134 (3)	0.0450 (19)	0.039 (2)	0.047 (2)
O4	0.0558 (19)	0.0481 (19)	0.090 (2)	0.0204 (15)	0.0090 (16)	0.0197 (17)
O5	0.134 (3)	0.043 (2)	0.095 (3)	0.036 (2)	0.029 (2)	0.0092 (19)
O6	0.122 (3)	0.056 (2)	0.076 (2)	0.041 (2)	0.017 (2)	0.0108 (19)
O7	0.133 (5)	0.211 (8)	0.117 (5)	0.023 (6)	0.004 (4)	0.086 (6)
O8	0.159 (6)	0.289 (10)	0.069 (4)	0.050 (6)	0.022 (3)	0.024 (5)

Geometric parameters (Å, °)

C1—C2	1.343 (5)	C15—H15	0.9300
C1—N1	1.386 (6)	C16—C17	1.376 (7)
C1—C7	1.502 (7)	C16—H16	0.9300
C2—C11	1.481 (6)	C17—O6	1.350 (6)
C2—C3	1.511 (6)	C17—C18	1.407 (7)
C3—C4	1.507 (6)	C18—C19	1.363 (8)
C3—C27	1.525 (7)	C18—H18	0.9300
C3—H3	0.9800	C19—H19	0.9300
C4—C5	1.354 (5)	C20—O6	1.434 (6)
C4—C8	1.454 (6)	C20—C21	1.510 (9)
C5—N1	1.372 (6)	C20—H20A	0.9700
C5—C6	1.501 (6)	C20—H20B	0.9700
C6—H6A	0.9600	C21—C22	1.355 (9)
C6—H6B	0.9600	C21—C26	1.369 (9)
C6—H6C	0.9600	C22—C23	1.423 (12)
C7—H7A	0.9600	C22—H22	0.9300
C7—H7B	0.9600	C23—C24	1.341 (13)
C7—H7C	0.9600	C23—H23	0.9300
C8—O1	1.209 (4)	C24—C25	1.347 (11)
C8—O2	1.328 (5)	C24—H24	0.9300
C9—O2	1.440 (6)	C25—C26	1.368 (10)
C9—C10	1.468 (8)	C25—H25	0.9300
C9—H9A	0.9700	C26—H26	0.9300
C9—H9B	0.9700	C27—C28	1.383 (7)
C10—H10A	0.9600	C27—C32	1.398 (7)
C10—H10B	0.9600	C28—C29	1.360 (9)
C10—H10C	0.9600	C28—H28	0.9300
C11—O3	1.191 (5)	C29—C30	1.370 (11)
C11—O4	1.337 (5)	C29—N2	1.466 (10)
C12—O4	1.429 (5)	C30—C31	1.382 (11)
C12—C13	1.501 (7)	C30—H30	0.9300

C12—H12A	0.9700	C31—C32	1.384 (9)
C12—H12B	0.9700	C31—H31	0.9300
C13—O5	1.211 (5)	C32—H32	0.9300
C13—C14	1.467 (7)	N1—H1	0.8600
C14—C19	1.381 (7)	N2—O8	1.195 (10)
C14—C15	1.390 (6)	N2—O7	1.203 (11)
C15—C16	1.380 (7)		
C2—C1—N1	117.2 (4)	C17—C16—C15	120.3 (4)
C2—C1—C7	129.1 (4)	C17—C16—H16	119.9
N1—C1—C7	113.8 (4)	C15—C16—H16	119.9
C1—C2—C11	124.9 (4)	O6—C17—C16	126.6 (4)
C1—C2—C3	119.1 (4)	O6—C17—C18	114.5 (5)
C11—C2—C3	115.9 (3)	C16—C17—C18	118.9 (5)
C4—C3—C2	109.4 (3)	C19—C18—C17	119.6 (5)
C4—C3—C27	113.0 (4)	C19—C18—H18	120.2
C2—C3—C27	111.7 (4)	C17—C18—H18	120.2
C4—C3—H3	107.5	C18—C19—C14	122.3 (5)
C2—C3—H3	107.5	C18—C19—H19	118.9
C27—C3—H3	107.5	C14—C19—H19	118.9
C5—C4—C8	125.0 (4)	O6—C20—C21	106.6 (5)
C5—C4—C3	118.7 (4)	O6—C20—H20A	110.4
C8—C4—C3	116.4 (3)	C21—C20—H20A	110.4
C4—C5—N1	118.0 (4)	O6—C20—H20B	110.4
C4—C5—C6	128.4 (4)	C21—C20—H20B	110.4
N1—C5—C6	113.6 (4)	H20A—C20—H20B	108.6
C5—C6—H6A	109.5	C22—C21—C26	119.4 (7)
C5—C6—H6B	109.5	C22—C21—C20	121.4 (7)
H6A—C6—H6B	109.5	C26—C21—C20	119.2 (6)
C5—C6—H6C	109.5	C21—C22—C23	119.2 (8)
H6A—C6—H6C	109.5	C21—C22—H22	120.4
H6B—C6—H6C	109.5	C23—C22—H22	120.4
C1—C7—H7A	109.5	C24—C23—C22	119.3 (8)
C1—C7—H7B	109.5	C24—C23—H23	120.4
H7A—C7—H7B	109.5	C22—C23—H23	120.4
C1—C7—H7C	109.5	C23—C24—C25	121.5 (8)
H7A—C7—H7C	109.5	C23—C24—H24	119.3
H7B—C7—H7C	109.5	C25—C24—H24	119.3
O1—C8—O2	121.5 (4)	C24—C25—C26	119.5 (8)
O1—C8—C4	123.3 (4)	C24—C25—H25	120.3
O2—C8—C4	115.3 (3)	C26—C25—H25	120.3
O2—C9—C10	107.6 (5)	C25—C26—C21	121.2 (7)
O2—C9—H9A	110.2	C25—C26—H26	119.4
C10—C9—H9A	110.2	C21—C26—H26	119.4
O2—C9—H9B	110.2	C28—C27—C32	117.5 (5)
C10—C9—H9B	110.2	C28—C27—C3	119.2 (5)
H9A—C9—H9B	108.5	C32—C27—C3	123.3 (4)
C9—C10—H10A	109.5	C29—C28—C27	121.3 (6)
C9—C10—H10B	109.5	C29—C28—H28	119.4
H10A—C10—H10B	109.5	C27—C28—H28	119.4

C9—C10—H10C	109.5	C28—C29—C30	121.6 (7)
H10A—C10—H10C	109.5	C28—C29—N2	120.3 (8)
H10B—C10—H10C	109.5	C30—C29—N2	118.1 (8)
O3—C11—O4	122.4 (4)	C29—C30—C31	118.4 (7)
O3—C11—C2	123.3 (4)	C29—C30—H30	120.8
O4—C11—C2	114.3 (3)	C31—C30—H30	120.8
O4—C12—C13	110.2 (4)	C30—C31—C32	120.5 (7)
O4—C12—H12A	109.6	C30—C31—H31	119.7
C13—C12—H12A	109.6	C32—C31—H31	119.7
O4—C12—H12B	109.6	C31—C32—C27	120.6 (6)
C13—C12—H12B	109.6	C31—C32—H32	119.7
H12A—C12—H12B	108.1	C27—C32—H32	119.7
O5—C13—C14	122.7 (5)	C5—N1—C1	123.2 (3)
O5—C13—C12	118.8 (4)	C5—N1—H1	118.4
C14—C13—C12	118.5 (4)	C1—N1—H1	118.4
C19—C14—C15	117.6 (4)	O8—N2—O7	123.0 (10)
C19—C14—C13	118.8 (4)	O8—N2—C29	119.9 (11)
C15—C14—C13	123.6 (4)	O7—N2—C29	117.0 (9)
C16—C15—C14	121.3 (5)	C8—O2—C9	118.1 (3)
C16—C15—H15	119.3	C11—O4—C12	114.9 (3)
C14—C15—H15	119.3	C17—O6—C20	117.7 (4)
N1—C1—C2—C11	169.2 (5)	O6—C20—C21—C26	90.2 (8)
C7—C1—C2—C11	−10.9 (10)	C26—C21—C22—C23	1.6 (13)
N1—C1—C2—C3	−12.7 (8)	C20—C21—C22—C23	−178.5 (7)
C7—C1—C2—C3	167.3 (6)	C21—C22—C23—C24	−1.8 (15)
C1—C2—C3—C4	37.5 (7)	C22—C23—C24—C25	0.9 (15)
C11—C2—C3—C4	−144.2 (4)	C23—C24—C25—C26	0.1 (14)
C1—C2—C3—C27	−88.2 (6)	C22—C21—C26—C25	−0.6 (11)
C11—C2—C3—C27	90.2 (5)	C20—C21—C26—C25	179.6 (7)
C2—C3—C4—C5	−34.7 (7)	C24—C25—C26—C21	−0.3 (12)
C27—C3—C4—C5	90.4 (6)	C4—C3—C27—C28	175.7 (4)
C2—C3—C4—C8	145.3 (4)	C2—C3—C27—C28	−60.5 (5)
C27—C3—C4—C8	−89.6 (5)	C4—C3—C27—C32	−5.1 (6)
C8—C4—C5—N1	−172.5 (5)	C2—C3—C27—C32	118.6 (5)
C3—C4—C5—N1	7.5 (8)	C32—C27—C28—C29	−1.5 (8)
C8—C4—C5—C6	8.7 (9)	C3—C27—C28—C29	177.7 (5)
C3—C4—C5—C6	−171.3 (6)	C27—C28—C29—C30	0.7 (10)
C5—C4—C8—O1	177.0 (6)	C27—C28—C29—N2	179.9 (6)
C3—C4—C8—O1	−3.0 (7)	C28—C29—C30—C31	0.2 (11)
C5—C4—C8—O2	−2.5 (8)	N2—C29—C30—C31	−179.0 (7)
C3—C4—C8—O2	177.5 (5)	C29—C30—C31—C32	−0.1 (11)
C1—C2—C11—O3	−179.9 (6)	C30—C31—C32—C27	−0.7 (10)
C3—C2—C11—O3	1.9 (8)	C28—C27—C32—C31	1.5 (8)
C1—C2—C11—O4	−0.6 (8)	C3—C27—C32—C31	−177.6 (6)
C3—C2—C11—O4	−178.8 (5)	C4—C5—N1—C1	22.3 (8)
O4—C12—C13—O5	1.0 (7)	C6—C5—N1—C1	−158.7 (6)
O4—C12—C13—C14	−179.2 (4)	C2—C1—N1—C5	−19.6 (8)
O5—C13—C14—C19	1.0 (9)	C7—C1—N1—C5	160.5 (6)
C12—C13—C14—C19	−178.6 (5)	C28—C29—N2—O7	−3.1 (13)

O5—C13—C14—C15	−177.5 (6)	C30—C29—N2—O7	176.1 (10)
C12—C13—C14—C15	2.8 (8)	C28—C29—N2—O8	176.7 (8)
C19—C14—C15—C16	1.4 (8)	C30—C29—N2—O8	−4.1 (12)
C13—C14—C15—C16	180.0 (5)	O1—C8—O2—C9	−3.0 (8)
C14—C15—C16—C17	−2.1 (8)	C4—C8—O2—C9	176.5 (5)
C15—C16—C17—O6	179.8 (6)	C10—C9—O2—C8	−177.6 (7)
C15—C16—C17—C18	1.5 (9)	O3—C11—O4—C12	−19.1 (8)
O6—C17—C18—C19	−178.8 (7)	C2—C11—O4—C12	161.6 (5)
C16—C17—C18—C19	−0.3 (11)	C13—C12—O4—C11	−73.8 (6)
C17—C18—C19—C14	−0.4 (12)	C16—C17—O6—C20	1.9 (10)
C15—C14—C19—C18	−0.2 (10)	C18—C17—O6—C20	−179.8 (7)
C13—C14—C19—C18	−178.8 (7)	C21—C20—O6—C17	−173.0 (6)
O6—C20—C21—C22	−89.7 (9)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.11	2.940 (5)	163

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

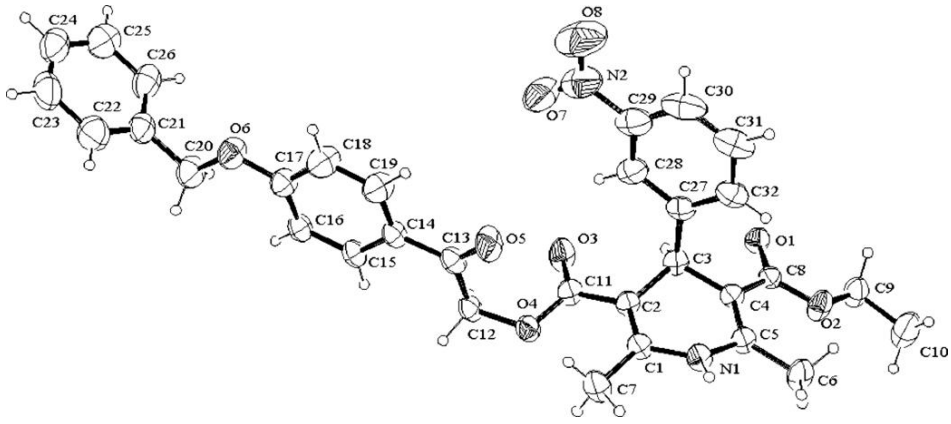
O8
N2
O7
C29
C30
C31
C28
C27
C32
O1
C3
C8
C9
O2
C10
C4
C5
C1
N1
C6
C7
C2
C11
O3
O4
C12
C13
O5
C14
C15
C16
C17
C18
C19
O6
C20
C21
C22
C23
C24
C25
C26

Fig. 2