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Ethyl 1-benzoyl-4-chloro-2-phenylprolinate

Rafael Tamazyan,[a]* Armen Ayvazyan,[a] Ashot Martirosyan,[b] Vahan Martirosyan[b] and Raymond Schinazi[c]

[a]Molecule Structure Research Centre, National Academy of Sciences RA, Azatutyan Avenue 26, 375014 Yerevan, Republic of Armenia, [b]Institute of Fine Organic Chemistry, National Academy of Sciences RA, Azatutyan Avenue 26, 375014 Yerevan, Republic of Armenia, and [c]Emory University School of Medicine, Veterans' Affairs, Medical Center 1670, Clairmont Road, 151-H Decatur, GA 30033-4004, USA
Correspondence e-mail: rafael@msrc.am

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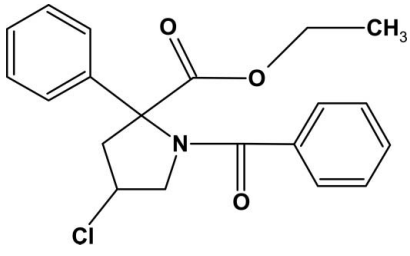
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.060; wR factor = 0.141; data-to-parameter ratio = 20.3.

The title compound, $C_{20}H_{20}ClNO_3$, belongs to a family of non-nucleoside reverse transcriptase inhibitors (NNRTI). It crystallizes with two independent molecules, *A* and *B*, in the asymmetric unit. In molecule *A*, the pyrrolidine ring is disordered between two positions with occupancies of 0.650 (2) and 0.350 (2), corresponding to two isomers, *viz*. 2*R*,4*R* and 2*R*,4*S*, respectively. The disorder of the pyrrolidine ring in molecule *B* [occupancies 0.512 (3) and 0.488 (3)] corresponds to the other two isomers, *viz*. 2*S*,4*R* and 2*S*,4*S*, respectively, showing that all four possible isomers are present in the crystal structure.

Related literature

For details of the synthesis, see: Martirosyan *et al.* (2000, 2004). For details of the pharmacological properties of compounds of this family, see: De Clercq (1996). For crystal structures of related compounds, see: Karapetyan *et al.* (2002); Tamazyan *et al.* (2002).



Experimental

Crystal data

$C_{20}H_{20}ClNO_3$
M_r = 357.82
Monoclinic, $P2_1$
a = 9.4241 (19) Å
b = 10.073 (2) Å
c = 19.849 (4) Å
β = 102.52 (3)°
V = 1839.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 (2) K
0.36 × 0.32 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.875, T_{max} = 0.927
11300 measured reflections
10705 independent reflections
5925 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
3 standard reflections
frequency: 180 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.141
S = 1.03
10705 reflections
527 parameters
478 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), with 1140 Friedel pairs
Flack parameter: 0.06 (6)

Data collection: *CAD-4 Manual* (Enraf–Nonius, 1988); cell refinement: *CAD-4 Manual*; data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *ORTEPII* (Johnson, 1976); 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: CV2292).

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supplementary materials

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Ethyl 1-benzoyl-4-chloro-2-phenylprolinate

R. Tamazyan, A. Ayvazyan, A. Martirosyan, V. Martirosyan and R. Schinazi

Comment

The interest to X-ray structural investigation of the title compound is stipulated by its potentially HIV-1 RT inhibition properties. This compound belongs to a family of non-nucleoside reverse transcriptase inhibitors (NNRTIs) (De Clercq, 1996) with butterfly-like conformation (Karapetyan *et al.*, 2002; Tamazyan *et al.*, 2002).

All four (2*S*,4*R*), (2*S*,4S), (2*R*,4*R*) and (2*R*,4 s) isomers of the title compound are present in crystal structure. The molecules crystallized in the monoclinic space group $P2_1$ with two independent molecules (Fig. 1). Each of two symmetry independent molecules corresponds to mixture of pairs of two isomers (2*R*,4*R*), (2*R*,4S) and (2*S*,4*R*), (2*S*,4S) respectively. In pair (2*R*,4*R*) (2*R*,4S) isomers are in ratio ~7:3, while in pair (2*S*,4*R*) (2*S*,4S) the isomers are in ratio ~1:1.

Experimental

The title compound was synthesized by cycloalkylation of ethyl-2-[2,3-dichloropropyl(phenyl)carboxamido]-2-phenylacetate in phase-transfer catalysis condition as it is described by (Martirosyan *et al.*, 2000, 2004). The crystals were grown from ethanol solution.

Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C). SADI and SIMI restrains were applied to positional and thermal parameters of disordered atoms in pyrrolidine rings.

Figures

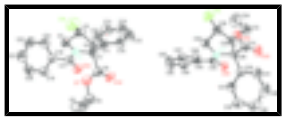


Fig. 1. Two independent molecules of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Only major parts of disordered pyrrolidine rings are shown corresponding to (2*S*,4*R*) (left) and (2*R*,4*R*) (right) isomers. H atoms omitted for clarity.

Ethyl 1-benzoyl-4-chloro-2-phenylprolinate

Crystal data

$C_{20}H_{20}ClNO_3$	F_{000} = 752
M_r = 357.82	D_x = 1.292 Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 471 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation λ = 0.71073 Å

a = 9.4241 (19) Å	Cell parameters from 22 reflections
b = 10.073 (2) Å	θ = 13–15º
c = 19.849 (4) Å	μ = 0.23 mm^{-1}
β = 102.52 (3)º	T = 293 (2) K
V = 1839.5 (7) $Å^3$	Irregular, colourless
Z = 4	0.36 × 0.32 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.025
Radiation source: fine-focus sealed tube	θ_{max} = 30.0º
Monochromator: graphite	θ_{min} = 1.1º
T = 293(2) K	h = 0→13
θ/2θ scans	k = −14→14
Absorption correction: ψ scan (North *et al.*, 1968)	l = −27→27
T_{min} = 0.875, T_{max} = 0.927	3 standard reflections
11300 measured reflections	every 180 min
10705 independent reflections	intensity decay: none
5925 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.060	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.141	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.03	$\Delta\rho_{max}$ = 0.26 e $Å^{-3}$
10705 reflections	$\Delta\rho_{min}$ = −0.18 e $Å^{-3}$
527 parameters	Extinction correction: none
478 restraints	Absolute structure: Flack (1983), with how many Friedel pairs?
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.06 (6)
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 calculat-

ing *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}	Occ. (<1)
ClA	0.2307 (2)	−0.24427 (15)	0.64892 (11)	0.0765 (6)	0.488 (3)
C5A	0.1090 (5)	−0.0052 (3)	0.67783 (16)	0.0522 (8)	0.488 (3)
H5AB	0.2033	0.0129	0.7073	0.063*	0.488 (3)
H5AA	0.0433	−0.0366	0.7058	0.063*	0.488 (3)
C4A	0.1206 (3)	−0.10374 (18)	0.62151 (12)	0.0519 (13)	0.488 (3)
H4A	0.0230	−0.1330	0.5984	0.062*	0.488 (3)
C3A	0.1874 (5)	−0.0246 (5)	0.5713 (2)	0.0491 (8)	0.488 (3)
H3AA	0.2913	−0.0132	0.5878	0.059*	0.488 (3)
H3AB	0.1681	−0.0646	0.5258	0.059*	0.488 (3)
ClB	0.2574 (2)	−0.23266 (13)	0.65602 (12)	0.0707 (5)	0.512 (3)
C5B	0.0877 (2)	−0.0178 (5)	0.6710 (2)	0.0522 (8)	0.512 (3)
H5BA	0.1107	−0.0082	0.7208	0.063*	0.512 (3)
H5BB	0.0089	−0.0811	0.6580	0.063*	0.512 (3)
C4B	0.2200 (3)	−0.06090 (19)	0.64475 (10)	0.0486 (12)	0.512 (3)
H4B	0.3051	−0.0091	0.6674	0.058*	0.512 (3)
C3B	0.1750 (6)	−0.0220 (5)	0.56936 (13)	0.0493 (8)	0.512 (3)
H3BA	0.2588	−0.0140	0.5486	0.059*	0.512 (3)
H3BB	0.1079	−0.0862	0.5435	0.059*	0.512 (3)
N2	0.1058 (2)	0.10411 (17)	0.57210 (9)	0.0444 (5)	
C1	0.0469 (2)	0.1191 (2)	0.63468 (11)	0.0465 (6)	
C6	0.1208 (3)	0.2370 (3)	0.67887 (12)	0.0558 (6)	
O7	0.26410 (18)	0.23518 (18)	0.68232 (9)	0.0657 (5)	
C8	0.3490 (3)	0.3431 (2)	0.71983 (13)	0.0839 (10)	
H8B	0.2967	0.3817	0.7521	0.101*	
H8A	0.4407	0.3090	0.7460	0.101*	
C9	0.3759 (4)	0.4439 (3)	0.67220 (17)	0.1180 (16)	
H9A	0.4264	0.4052	0.6399	0.177*	
H9B	0.4341	0.5135	0.6974	0.177*	
H9C	0.2850	0.4801	0.6477	0.177*	
O10	0.0596 (2)	0.3113 (2)	0.70958 (10)	0.0787 (6)	
C11	−0.1186 (3)	0.1324 (2)	0.61935 (13)	0.0535 (6)	
C12	−0.1998 (3)	0.1426 (3)	0.55303 (15)	0.0660 (8)	
H12	−0.1547	0.1417	0.5157	0.079*	
C13	−0.3520 (3)	0.1544 (3)	0.54218 (19)	0.0855 (10)	
H13	−0.4077	0.1615	0.4975	0.103*	
C14	−0.4184 (3)	0.1554 (3)	0.5970 (2)	0.0893 (11)	
H14	−0.5187	0.1651	0.5895	0.107*	
C15	−0.3374 (4)	0.1419 (3)	0.6633 (2)	0.0871 (10)	
H15	−0.3828	0.1403	0.7005	0.105*	
C16	−0.1891 (3)	0.1310 (3)	0.67392 (15)	0.0663 (8)	
H16	−0.1345	0.1224	0.7187	0.080*	
C17	0.1162 (2)	0.2116 (2)	0.53205 (12)	0.0496 (6)	

C18	0.1774 (3)	0.1935 (2)	0.46970 (12)	0.0495 (6)	
C19	0.2730 (3)	0.2881 (3)	0.45560 (13)	0.0612 (7)	
H19	0.2988	0.3595	0.4854	0.073*	
C20	0.3304 (3)	0.2773 (3)	0.39760 (15)	0.0777 (9)	
H20	0.3941	0.3419	0.3885	0.093*	
C21	0.2949 (4)	0.1739 (4)	0.35388 (15)	0.0849 (10)	
H21	0.3367	0.1660	0.3157	0.102*	
C22	0.1962 (4)	0.0794 (3)	0.36581 (15)	0.0826 (10)	
H22	0.1698	0.0095	0.3350	0.099*	
C23	0.1378 (3)	0.0891 (3)	0.42295 (13)	0.0635 (8)	
H23	0.0713	0.0259	0.4308	0.076*	
O24	0.07592 (19)	0.32159 (17)	0.54748 (9)	0.0632 (5)	
ClC	0.12063 (14)	0.02879 (11)	0.87321 (7)	0.0641 (4)	0.650 (2)
C30A	0.1593 (3)	0.2336 (4)	0.96650 (12)	0.0508 (7)	0.650 (2)
H30A	0.1815	0.1625	1.0001	0.061*	0.650 (2)
H30B	0.0588	0.2599	0.9626	0.061*	0.650 (2)
C29A	0.18275 (19)	0.18999 (18)	0.90027 (13)	0.0519 (10)	0.650 (2)
H29A	0.1332	0.2529	0.8653	0.062*	0.650 (2)
C28A	0.3450 (2)	0.2071 (4)	0.9068 (2)	0.0449 (6)	0.650 (2)
H28A	0.3986	0.1312	0.9292	0.054*	0.650 (2)
H28B	0.3686	0.2204	0.8621	0.054*	0.650 (2)
ClD	0.1432 (3)	0.0064 (2)	0.89083 (17)	0.0753 (8)	0.350 (2)
C30B	0.1468 (4)	0.2354 (5)	0.9680 (3)	0.0505 (9)	0.350 (2)
H30C	0.1132	0.2022	1.0077	0.061*	0.350 (2)
H30D	0.0639	0.2632	0.9328	0.061*	0.350 (2)
C29B	0.2338 (3)	0.1378 (3)	0.94182 (19)	0.0500 (18)	0.350 (2)
H29B	0.2945	0.0958	0.9826	0.060*	0.350 (2)
C28B	0.3383 (5)	0.2067 (8)	0.9050 (4)	0.0450 (9)	0.350 (2)
H28C	0.4235	0.1528	0.9047	0.054*	0.350 (2)
H28D	0.2918	0.2303	0.8580	0.054*	0.350 (2)
N27	0.37485 (18)	0.32630 (18)	0.94957 (9)	0.0409 (4)	
C26	0.2614 (2)	0.3526 (2)	0.98875 (11)	0.0411 (5)	
C31	0.1797 (2)	0.4832 (2)	0.96391 (13)	0.0490 (6)	
O32	0.16150 (18)	0.49700 (18)	0.89589 (9)	0.0601 (5)	
C33	0.0942 (3)	0.6224 (3)	0.86960 (12)	0.0808 (9)	
H33A	−0.0074	0.6227	0.8724	0.097*	
H33B	0.1426	0.6955	0.8971	0.097*	
C34	0.1062 (5)	0.6376 (4)	0.79866 (14)	0.1121 (13)	
H34A	0.2069	0.6434	0.7967	0.168*	
H34B	0.0568	0.7171	0.7799	0.168*	
H34C	0.0628	0.5624	0.7723	0.168*	
O35	0.1314 (2)	0.55831 (19)	0.99991 (10)	0.0706 (5)	
C36	0.3196 (2)	0.3580 (2)	1.06703 (11)	0.0443 (5)	
C37	0.4658 (3)	0.3550 (2)	1.09670 (12)	0.0518 (6)	
H37	0.5329	0.3481	1.0687	0.062*	
C38	0.5144 (3)	0.3623 (3)	1.16785 (13)	0.0673 (8)	
H38	0.6134	0.3614	1.1874	0.081*	
C39	0.4150 (4)	0.3707 (3)	1.20928 (14)	0.0748 (9)	
H39	0.4471	0.3758	1.2569	0.090*	

C40	0.2698 (4)	0.3717 (3)	1.18081 (14)	0.0741 (8)
H40	0.2030	0.3768	1.2090	0.089*
C41	0.2218 (3)	0.3650 (3)	1.10988 (13)	0.0578 (7)
H41	0.1225	0.3651	1.0907	0.069*
C42	0.4682 (2)	0.4255 (2)	0.94158 (11)	0.0423 (5)
C43	0.5815 (2)	0.3977 (2)	0.90132 (11)	0.0416 (5)
C44	0.6580 (2)	0.2789 (2)	0.90635 (12)	0.0510 (6)
H44	0.6377	0.2115	0.9349	0.061*
C45	0.7639 (3)	0.2612 (3)	0.86901 (14)	0.0619 (7)
H45	0.8160	0.1822	0.8731	0.074*
C46	0.7935 (3)	0.3590 (3)	0.82573 (14)	0.0662 (8)
H46	0.8629	0.3449	0.7995	0.079*
C47	0.7198 (3)	0.4781 (3)	0.82140 (14)	0.0611 (7)
H47	0.7406	0.5454	0.7930	0.073*
C48	0.6145 (3)	0.4970 (2)	0.85964 (12)	0.0526 (6)
H48	0.5657	0.5776	0.8571	0.063*
O49	0.45967 (17)	0.53440 (16)	0.96811 (8)	0.0525 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
ClA	0.1017 (12)	0.0510 (8)	0.0724 (10)	0.0228 (9)	0.0096 (9)	0.0096 (8)
C5A	0.0620 (16)	0.0495 (15)	0.0422 (15)	0.0035 (14)	0.0049 (14)	0.0008 (13)
C4A	0.065 (3)	0.039 (2)	0.047 (3)	0.004 (2)	0.001 (2)	−0.002 (2)
C3A	0.0587 (15)	0.0433 (14)	0.0461 (14)	0.0072 (13)	0.0130 (13)	0.0014 (13)
ClB	0.0894 (11)	0.0492 (8)	0.0690 (10)	0.0161 (8)	0.0074 (9)	0.0114 (8)
C5B	0.0619 (16)	0.0492 (15)	0.0427 (15)	0.0040 (14)	0.0049 (14)	0.0013 (13)
C4B	0.058 (3)	0.040 (2)	0.045 (3)	0.005 (2)	0.005 (2)	0.0037 (19)
C3B	0.0592 (15)	0.0434 (14)	0.0460 (14)	0.0071 (13)	0.0129 (13)	0.0013 (12)
N2	0.0533 (11)	0.0383 (9)	0.0418 (10)	0.0074 (8)	0.0106 (8)	−0.0008 (8)
C1	0.0525 (13)	0.0431 (12)	0.0456 (12)	0.0020 (10)	0.0146 (10)	0.0024 (10)
C6	0.0608 (14)	0.0561 (15)	0.0508 (13)	−0.0007 (12)	0.0124 (11)	−0.0097 (11)
O7	0.0565 (10)	0.0642 (11)	0.0733 (11)	−0.0020 (9)	0.0077 (9)	−0.0194 (9)
C8	0.0738 (18)	0.083 (2)	0.090 (2)	−0.0084 (17)	0.0052 (16)	−0.0283 (18)
C9	0.123 (3)	0.0574 (19)	0.150 (4)	−0.015 (2)	−0.022 (3)	0.005 (2)
O10	0.0799 (12)	0.0732 (12)	0.0892 (12)	−0.0046 (10)	0.0318 (10)	−0.0367 (10)
C11	0.0533 (13)	0.0443 (12)	0.0625 (15)	0.0039 (11)	0.0118 (12)	−0.0029 (11)
C12	0.0531 (15)	0.0670 (16)	0.0745 (18)	0.0062 (13)	0.0059 (13)	0.0021 (14)
C13	0.0629 (18)	0.086 (2)	0.100 (2)	0.0059 (16)	−0.0004 (17)	0.0073 (19)
C14	0.0501 (16)	0.082 (2)	0.137 (3)	0.0083 (15)	0.0226 (18)	−0.004 (2)
C15	0.0699 (18)	0.081 (2)	0.120 (3)	0.0031 (16)	0.0416 (18)	−0.0055 (19)
C16	0.0635 (16)	0.0672 (17)	0.0742 (17)	0.0044 (13)	0.0281 (13)	−0.0060 (14)
C17	0.0469 (12)	0.0443 (12)	0.0545 (14)	0.0005 (10)	0.0043 (11)	0.0028 (10)
C18	0.0543 (13)	0.0485 (12)	0.0437 (12)	0.0044 (11)	0.0064 (11)	0.0098 (10)
C19	0.0700 (16)	0.0573 (16)	0.0555 (14)	−0.0004 (12)	0.0120 (13)	0.0088 (11)
C20	0.0855 (19)	0.080 (2)	0.0714 (18)	−0.0111 (17)	0.0253 (15)	0.0161 (16)
C21	0.113 (2)	0.095 (2)	0.0530 (16)	−0.007 (2)	0.0319 (17)	0.0052 (16)
C22	0.122 (3)	0.079 (2)	0.0484 (16)	−0.0094 (19)	0.0223 (16)	−0.0092 (14)

C23	0.0758 (17)	0.0649 (16)	0.0465 (14)	−0.0104 (14)	0.0062 (13)	0.0035 (12)
O24	0.0763 (11)	0.0434 (9)	0.0706 (11)	0.0116 (9)	0.0176 (9)	0.0036 (8)
ClC	0.0604 (6)	0.0631 (6)	0.0698 (7)	−0.0207 (5)	0.0168 (5)	−0.0220 (5)
C30A	0.0422 (13)	0.0531 (14)	0.0588 (14)	−0.0083 (12)	0.0144 (12)	−0.0077 (12)
C29A	0.0456 (19)	0.055 (2)	0.054 (2)	−0.0075 (17)	0.0071 (17)	−0.0024 (17)
C28A	0.0437 (12)	0.0430 (12)	0.0497 (13)	−0.0070 (11)	0.0137 (11)	−0.0062 (11)
ClD	0.0652 (13)	0.0575 (12)	0.1014 (18)	−0.0216 (11)	0.0140 (12)	−0.0222 (12)
C30B	0.0420 (16)	0.0527 (17)	0.0582 (16)	−0.0079 (15)	0.0142 (15)	−0.0080 (15)
C29B	0.041 (3)	0.043 (3)	0.058 (4)	−0.012 (3)	−0.006 (3)	−0.004 (3)
C28B	0.0440 (15)	0.0429 (15)	0.0496 (16)	−0.0071 (14)	0.0136 (14)	−0.0064 (14)
N27	0.0388 (9)	0.0394 (9)	0.0458 (9)	−0.0011 (8)	0.0117 (8)	−0.0005 (8)
C26	0.0382 (10)	0.0408 (11)	0.0456 (11)	0.0008 (9)	0.0122 (9)	0.0001 (9)
C31	0.0420 (12)	0.0501 (13)	0.0558 (14)	−0.0004 (10)	0.0128 (11)	0.0011 (11)
O32	0.0611 (10)	0.0619 (10)	0.0561 (10)	0.0168 (9)	0.0098 (8)	0.0119 (8)
C33	0.082 (2)	0.0752 (19)	0.082 (2)	0.0255 (16)	0.0124 (16)	0.0243 (16)
C34	0.159 (3)	0.096 (3)	0.086 (2)	0.032 (3)	0.036 (2)	0.033 (2)
O35	0.0787 (11)	0.0618 (11)	0.0754 (12)	0.0242 (9)	0.0258 (10)	−0.0020 (9)
C36	0.0508 (12)	0.0368 (11)	0.0468 (12)	−0.0022 (10)	0.0140 (10)	−0.0007 (9)
C37	0.0508 (13)	0.0571 (14)	0.0484 (12)	−0.0038 (11)	0.0123 (11)	0.0038 (11)
C38	0.0686 (17)	0.0745 (18)	0.0527 (15)	−0.0119 (14)	−0.0006 (13)	0.0069 (14)
C39	0.101 (2)	0.0788 (19)	0.0420 (14)	−0.0110 (17)	0.0107 (15)	0.0008 (13)
C40	0.100 (2)	0.0754 (19)	0.0571 (15)	0.0012 (17)	0.0401 (15)	−0.0006 (13)
C41	0.0609 (14)	0.0604 (15)	0.0563 (14)	0.0036 (12)	0.0222 (12)	0.0001 (12)
C42	0.0404 (12)	0.0431 (11)	0.0420 (12)	0.0000 (9)	0.0060 (10)	0.0037 (10)
C43	0.0375 (11)	0.0430 (11)	0.0423 (12)	−0.0057 (9)	0.0043 (9)	−0.0037 (9)
C44	0.0458 (12)	0.0488 (13)	0.0605 (14)	−0.0018 (11)	0.0160 (11)	0.0045 (11)
C45	0.0434 (12)	0.0550 (13)	0.0896 (18)	0.0077 (12)	0.0197 (13)	−0.0027 (15)
C46	0.0548 (14)	0.0804 (19)	0.0701 (16)	−0.0030 (14)	0.0284 (12)	−0.0092 (15)
C47	0.0635 (15)	0.0665 (16)	0.0583 (15)	−0.0052 (13)	0.0241 (12)	0.0096 (12)
C48	0.0537 (13)	0.0471 (13)	0.0581 (14)	0.0008 (11)	0.0145 (12)	0.0046 (11)
O49	0.0587 (9)	0.0416 (8)	0.0604 (10)	−0.0070 (7)	0.0202 (8)	−0.0066 (8)

Geometric parameters (Å, °)

ClA—C4A	1.7701 (18)	ClC—C29A	1.7697 (17)
C5A—C4A	1.516 (2)	C30A—C29A	1.448 (2)
C5A—C1	1.559 (4)	C30A—C26	1.541 (4)
C5A—H5AB	0.9700	C30A—H30A	0.9700
C5A—H5AA	0.9700	C30A—H30B	0.9700
C4A—C3A	1.516 (2)	C29A—C28A	1.516 (2)
C4A—H4A	0.9800	C29A—H29A	0.9800
C3A—N2	1.510 (5)	C28A—N27	1.462 (5)
C3A—H3AA	0.9700	C28A—H28A	0.9700
C3A—H3AB	0.9700	C28A—H28B	0.9700
ClB—C4B	1.7702 (18)	ClD—C29B	1.769 (2)
C5B—C4B	1.516 (2)	C30B—C29B	1.447 (3)
C5B—C1	1.565 (5)	C30B—C26	1.593 (5)
C5B—H5BA	0.9700	C30B—H30C	0.9700
C5B—H5BB	0.9700	C30B—H30D	0.9700

C4B—C3B	1.516 (2)	C29B—C28B	1.516 (2)
C4B—H4B	0.9800	C29B—H29B	0.9800
C3B—N2	1.434 (6)	C28B—N27	1.490 (9)
C3B—H3BA	0.9700	C28B—H28C	0.9700
C3B—H3BB	0.9700	C28B—H28D	0.9700
N2—C17	1.359 (3)	N27—C42	1.363 (3)
N2—C1	1.474 (3)	N27—C26	1.476 (3)
C1—C11	1.528 (3)	C26—C36	1.532 (3)
C1—C6	1.549 (3)	C26—C31	1.549 (3)
C6—O10	1.191 (3)	C31—O35	1.196 (3)
C6—O7	1.338 (3)	C31—O32	1.331 (3)
O7—C8	1.455 (3)	O32—C33	1.458 (3)
C8—C9	1.447 (2)	C33—C34	1.445 (2)
C8—H8B	0.9700	C33—H33A	0.9700
C8—H8A	0.9700	C33—H33B	0.9700
C9—H9A	0.9600	C34—H34A	0.9600
C9—H9B	0.9600	C34—H34B	0.9600
C9—H9C	0.9600	C34—H34C	0.9600
C11—C12	1.376 (4)	C36—C37	1.376 (3)
C11—C16	1.388 (4)	C36—C41	1.385 (3)
C12—C13	1.408 (4)	C37—C38	1.389 (4)
C12—H12	0.9300	C37—H37	0.9300
C13—C14	1.368 (5)	C38—C39	1.377 (4)
C13—H13	0.9300	C38—H38	0.9300
C14—C15	1.377 (5)	C39—C40	1.362 (4)
C14—H14	0.9300	C39—H39	0.9300
C15—C16	1.372 (4)	C40—C41	1.384 (4)
C15—H15	0.9300	C40—H40	0.9300
C16—H16	0.9300	C41—H41	0.9300
C17—O24	1.231 (3)	C42—O49	1.227 (3)
C17—C18	1.486 (4)	C42—C43	1.493 (3)
C18—C19	1.381 (4)	C43—C48	1.376 (3)
C18—C23	1.399 (3)	C43—C44	1.389 (3)
C19—C20	1.378 (4)	C44—C45	1.378 (3)
C19—H19	0.9300	C44—H44	0.9300
C20—C21	1.351 (4)	C45—C46	1.375 (4)
C20—H20	0.9300	C45—H45	0.9300
C21—C22	1.387 (5)	C46—C47	1.380 (4)
C21—H21	0.9300	C46—H46	0.9300
C22—C23	1.367 (4)	C47—C48	1.386 (4)
C22—H22	0.9300	C47—H47	0.9300
C23—H23	0.9300	C48—H48	0.9300
C4A—C5A—C1	101.5 (2)	C29A—C30A—C26	106.7 (2)
C4A—C5A—H5AB	111.5	C29A—C30A—H30A	110.4
C1—C5A—H5AB	111.5	C26—C30A—H30A	110.4
C4A—C5A—H5AA	111.5	C29A—C30A—H30B	110.4
C1—C5A—H5AA	111.5	C26—C30A—H30B	110.4
H5AB—C5A—H5AA	109.3	H30A—C30A—H30B	108.6
C3A—C4A—C5A	104.3 (3)	C30A—C29A—C28A	103.8 (3)

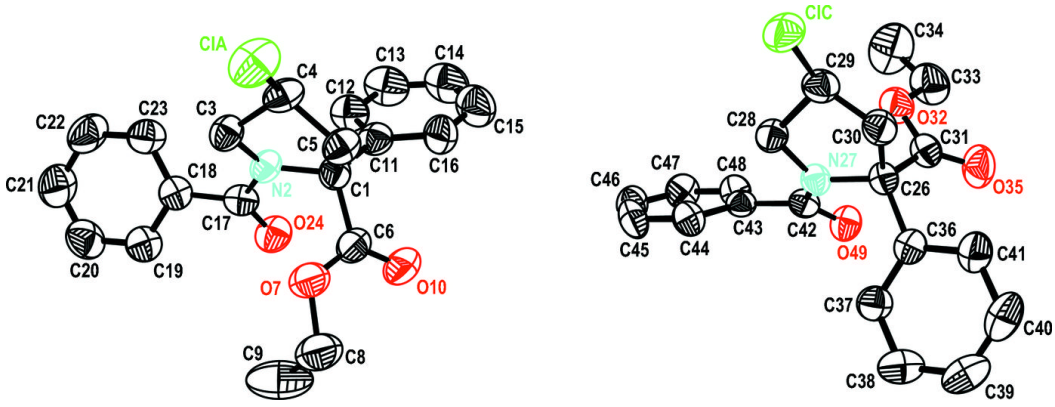
C3A—C4A—ClA	108.8 (2)	C30A—C29A—ClC	116.6 (2)
C5A—C4A—ClA	115.3 (2)	C28A—C29A—ClC	113.0 (2)
C3A—C4A—H4A	109.4	C30A—C29A—H29A	107.7
C5A—C4A—H4A	109.4	C28A—C29A—H29A	107.7
ClA—C4A—H4A	109.4	ClC—C29A—H29A	107.7
N2—C3A—C4A	99.2 (3)	N27—C28A—C29A	101.9 (2)
N2—C3A—H3AA	111.9	N27—C28A—H28A	111.4
C4A—C3A—H3AA	111.9	C29A—C28A—H28A	111.4
N2—C3A—H3AB	111.9	N27—C28A—H28B	111.4
C4A—C3A—H3AB	111.9	C29A—C28A—H28B	111.4
H3AA—C3A—H3AB	109.6	H28A—C28A—H28B	109.3
C4B—C5B—C1	103.4 (3)	C29B—C30B—C26	101.0 (2)
C4B—C5B—H5BA	111.1	C29B—C30B—H30C	111.6
C1—C5B—H5BA	111.1	C26—C30B—H30C	111.6
C4B—C5B—H5BB	111.1	C29B—C30B—H30D	111.6
C1—C5B—H5BB	111.1	C26—C30B—H30D	111.6
H5BA—C5B—H5BB	109.0	H30C—C30B—H30D	109.4
C3B—C4B—C5B	101.0 (3)	C30B—C29B—C28B	109.9 (4)
C3B—C4B—ClB	112.5 (3)	C30B—C29B—ClD	118.2 (3)
C5B—C4B—ClB	113.1 (3)	C28B—C29B—ClD	110.5 (4)
C3B—C4B—H4B	110.0	C30B—C29B—H29B	105.8
C5B—C4B—H4B	110.0	C28B—C29B—H29B	105.8
ClB—C4B—H4B	110.0	ClD—C29B—H29B	105.8
N2—C3B—C4B	102.9 (3)	N27—C28B—C29B	100.0 (4)
N2—C3B—H3BA	111.2	N27—C28B—H28C	111.8
C4B—C3B—H3BA	111.2	C29B—C28B—H28C	111.8
N2—C3B—H3BB	111.2	N27—C28B—H28D	111.8
C4B—C3B—H3BB	111.2	C29B—C28B—H28D	111.8
H3BA—C3B—H3BB	109.1	H28C—C28B—H28D	109.5
C17—N2—C3B	126.3 (2)	C42—N27—C28A	125.6 (2)
C17—N2—C1	119.94 (18)	C42—N27—C26	119.27 (18)
C3B—N2—C1	112.4 (2)	C28A—N27—C26	112.56 (17)
C17—N2—C3A	125.2 (2)	C42—N27—C28B	126.5 (2)
C1—N2—C3A	112.52 (19)	C26—N27—C28B	111.0 (2)
N2—C1—C11	113.30 (18)	N27—C26—C36	113.57 (17)
N2—C1—C6	110.52 (19)	N27—C26—C30A	100.82 (18)
C11—C1—C6	111.10 (19)	C36—C26—C30A	112.45 (19)
N2—C1—C5A	102.4 (2)	N27—C26—C31	110.59 (18)
C11—C1—C5A	115.2 (2)	C36—C26—C31	109.67 (18)
C6—C1—C5A	103.64 (19)	C30A—C26—C31	109.43 (19)
N2—C1—C5B	101.4 (2)	N27—C26—C30B	105.0 (2)
C11—C1—C5B	107.86 (19)	C36—C26—C30B	111.2 (3)
C6—C1—C5B	112.3 (2)	C31—C26—C30B	106.5 (2)
O10—C6—O7	125.3 (2)	O35—C31—O32	123.9 (2)
O10—C6—C1	124.3 (2)	O35—C31—C26	125.0 (2)
O7—C6—C1	110.1 (2)	O32—C31—C26	110.95 (19)
C6—O7—C8	116.9 (2)	C31—O32—C33	113.84 (19)
C9—C8—O7	110.2 (2)	C34—C33—O32	108.7 (2)
C9—C8—H8B	109.6	C34—C33—H33A	110.0

O7—C8—H8B	109.6	O32—C33—H33A	110.0
C9—C8—H8A	109.6	C34—C33—H33B	110.0
O7—C8—H8A	109.6	O32—C33—H33B	110.0
H8B—C8—H8A	108.1	H33A—C33—H33B	108.3
C8—C9—H9A	109.5	C33—C34—H34A	109.5
C8—C9—H9B	109.5	C33—C34—H34B	109.5
H9A—C9—H9B	109.5	H34A—C34—H34B	109.5
C8—C9—H9C	109.5	C33—C34—H34C	109.5
H9A—C9—H9C	109.5	H34A—C34—H34C	109.5
H9B—C9—H9C	109.5	H34B—C34—H34C	109.5
C12—C11—C16	119.1 (2)	C37—C36—C41	118.4 (2)
C12—C11—C1	122.0 (2)	C37—C36—C26	122.5 (2)
C16—C11—C1	119.0 (2)	C41—C36—C26	119.0 (2)
C11—C12—C13	119.3 (3)	C36—C37—C38	120.8 (2)
C11—C12—H12	120.3	C36—C37—H37	119.6
C13—C12—H12	120.3	C38—C37—H37	119.6
C14—C13—C12	120.3 (3)	C39—C38—C37	119.6 (3)
C14—C13—H13	119.8	C39—C38—H38	120.2
C12—C13—H13	119.8	C37—C38—H38	120.2
C13—C14—C15	120.4 (3)	C40—C39—C38	120.3 (3)
C13—C14—H14	119.8	C40—C39—H39	119.8
C15—C14—H14	119.8	C38—C39—H39	119.8
C16—C15—C14	119.3 (3)	C39—C40—C41	120.0 (3)
C16—C15—H15	120.3	C39—C40—H40	120.0
C14—C15—H15	120.3	C41—C40—H40	120.0
C15—C16—C11	121.6 (3)	C40—C41—C36	120.8 (3)
C15—C16—H16	119.2	C40—C41—H41	119.6
C11—C16—H16	119.2	C36—C41—H41	119.6
O24—C17—N2	120.4 (2)	O49—C42—N27	120.1 (2)
O24—C17—C18	120.9 (2)	O49—C42—C43	121.2 (2)
N2—C17—C18	118.7 (2)	N27—C42—C43	118.68 (19)
C19—C18—C23	118.5 (2)	C48—C43—C44	119.2 (2)
C19—C18—C17	118.2 (2)	C48—C43—C42	117.8 (2)
C23—C18—C17	123.3 (2)	C44—C43—C42	122.9 (2)
C20—C19—C18	120.4 (3)	C45—C44—C43	119.9 (2)
C20—C19—H19	119.8	C45—C44—H44	120.1
C18—C19—H19	119.8	C43—C44—H44	120.1
C21—C20—C19	120.7 (3)	C46—C45—C44	120.8 (3)
C21—C20—H20	119.7	C46—C45—H45	119.6
C19—C20—H20	119.7	C44—C45—H45	119.6
C20—C21—C22	120.1 (3)	C45—C46—C47	119.6 (3)
C20—C21—H21	119.9	C45—C46—H46	120.2
C22—C21—H21	119.9	C47—C46—H46	120.2
C23—C22—C21	119.8 (3)	C46—C47—C48	119.7 (2)
C23—C22—H22	120.1	C46—C47—H47	120.2
C21—C22—H22	120.1	C48—C47—H47	120.2
C22—C23—C18	120.5 (3)	C43—C48—C47	120.8 (2)
C22—C23—H23	119.8	C43—C48—H48	119.6
C18—C23—H23	119.8	C47—C48—H48	119.6

C1—C5A—C4A—C3A	44.9 (3)	C26—C30A—C29A—C28A	37.6 (3)
C1—C5A—C4A—ClA	164.2 (2)	C26—C30A—C29A—ClC	162.62 (17)
C5A—C4A—C3A—N2	−40.5 (3)	C30A—C29A—C28A—N27	−35.1 (3)
ClA—C4A—C3A—N2	−164.10 (19)	ClC—C29A—C28A—N27	−162.4 (2)
C1—C5B—C4B—C3B	−41.5 (3)	C26—C30B—C29B—C28B	−35.2 (5)
C1—C5B—C4B—ClB	−161.96 (19)	C26—C30B—C29B—ClD	−163.2 (3)
C5B—C4B—C3B—N2	40.8 (4)	C30B—C29B—C28B—N27	36.4 (5)
ClB—C4B—C3B—N2	161.7 (3)	ClD—C29B—C28B—N27	168.6 (3)
C4B—C3B—N2—C17	141.0 (3)	C29A—C28A—N27—C42	−140.9 (2)
C4B—C3B—N2—C1	−25.4 (4)	C29A—C28A—N27—C26	20.6 (3)
C4B—C3B—N2—C3A	68 (3)	C29A—C28A—N27—C28B	−26 (7)
C4A—C3A—N2—C17	−175.8 (2)	C29B—C28B—N27—C42	179.0 (2)
C4A—C3A—N2—C3B	−66 (3)	C29B—C28B—N27—C28A	112 (7)
C4A—C3A—N2—C1	21.7 (3)	C29B—C28B—N27—C26	−21.7 (4)
C17—N2—C1—C11	76.7 (2)	C42—N27—C26—C36	−75.4 (2)
C3B—N2—C1—C11	−116.0 (3)	C28A—N27—C26—C36	121.8 (2)
C3A—N2—C1—C11	−119.8 (3)	C28B—N27—C26—C36	123.6 (3)
C17—N2—C1—C6	−48.8 (3)	C42—N27—C26—C30A	164.10 (19)
C3B—N2—C1—C6	118.6 (3)	C28A—N27—C26—C30A	1.3 (3)
C3A—N2—C1—C6	114.8 (3)	C28B—N27—C26—C30A	3.1 (3)
C17—N2—C1—C5A	−158.7 (2)	C42—N27—C26—C31	48.4 (2)
C3B—N2—C1—C5A	8.7 (3)	C28A—N27—C26—C31	−114.4 (2)
C3A—N2—C1—C5A	4.9 (3)	C28B—N27—C26—C31	−112.6 (3)
C17—N2—C1—C5B	−168.0 (2)	C42—N27—C26—C30B	162.9 (3)
C3B—N2—C1—C5B	−0.7 (3)	C28A—N27—C26—C30B	0.1 (3)
C3A—N2—C1—C5B	−4.5 (3)	C28B—N27—C26—C30B	1.9 (4)
C4A—C5A—C1—N2	−29.6 (3)	C29A—C30A—C26—N27	−24.3 (3)
C4A—C5A—C1—C11	93.8 (3)	C29A—C30A—C26—C36	−145.6 (2)
C4A—C5A—C1—C6	−144.7 (3)	C29A—C30A—C26—C31	92.3 (3)
C4A—C5A—C1—C5B	55.1 (11)	C29A—C30A—C26—C30B	140 (4)
C4B—C5B—C1—N2	26.5 (3)	C29B—C30B—C26—N27	19.8 (4)
C4B—C5B—C1—C11	145.8 (2)	C29B—C30B—C26—C36	−103.5 (4)
C4B—C5B—C1—C6	−91.5 (3)	C29B—C30B—C26—C30A	4(4)
C4B—C5B—C1—C5A	−70.7 (11)	C29B—C30B—C26—C31	137.1 (3)
N2—C1—C6—O10	142.2 (2)	N27—C26—C31—O35	−146.5 (2)
C11—C1—C6—O10	15.5 (3)	C36—C26—C31—O35	−20.5 (3)
C5A—C1—C6—O10	−108.7 (3)	C30A—C26—C31—O35	103.3 (3)
C5B—C1—C6—O10	−105.4 (3)	C30B—C26—C31—O35	99.9 (3)
N2—C1—C6—O7	−43.4 (3)	N27—C26—C31—O32	37.6 (2)
C11—C1—C6—O7	−170.0 (2)	C36—C26—C31—O32	163.61 (18)
C5A—C1—C6—O7	65.7 (3)	C30A—C26—C31—O32	−72.6 (2)
C5B—C1—C6—O7	69.1 (3)	C30B—C26—C31—O32	−75.9 (3)
O10—C6—O7—C8	−8.1 (4)	O35—C31—O32—C33	8.1 (3)
C1—C6—O7—C8	177.55 (19)	C26—C31—O32—C33	−176.0 (2)
C6—O7—C8—C9	−98.2 (3)	C31—O32—C33—C34	168.8 (3)
N2—C1—C11—C12	−6.2 (3)	N27—C26—C36—C37	7.7 (3)
C6—C1—C11—C12	118.9 (3)	C30A—C26—C36—C37	121.3 (2)
C5A—C1—C11—C12	−123.6 (3)	C31—C26—C36—C37	−116.6 (2)
C5B—C1—C11—C12	−117.6 (3)	C30B—C26—C36—C37	125.8 (2)

N2—C1—C11—C16	172.3 (2)	N27—C26—C36—C41	−171.8 (2)
C6—C1—C11—C16	−62.6 (3)	C30A—C26—C36—C41	−58.1 (3)
C5A—C1—C11—C16	54.8 (3)	C31—C26—C36—C41	63.9 (3)
C5B—C1—C11—C16	60.8 (3)	C30B—C26—C36—C41	−53.6 (3)
C16—C11—C12—C13	1.4 (4)	C41—C36—C37—C38	−1.6 (4)
C1—C11—C12—C13	179.9 (2)	C26—C36—C37—C38	178.9 (2)
C11—C12—C13—C14	−0.2 (5)	C36—C37—C38—C39	0.9 (4)
C12—C13—C14—C15	−1.4 (5)	C37—C38—C39—C40	0.2 (4)
C13—C14—C15—C16	1.6 (5)	C38—C39—C40—C41	−0.4 (5)
C14—C15—C16—C11	−0.3 (5)	C39—C40—C41—C36	−0.3 (4)
C12—C11—C16—C15	−1.2 (4)	C37—C36—C41—C40	1.4 (4)
C1—C11—C16—C15	−179.7 (3)	C26—C36—C41—C40	−179.2 (2)
C3B—N2—C17—O24	−165.9 (3)	C28A—N27—C42—O49	162.0 (2)
C1—N2—C17—O24	−0.4 (3)	C26—N27—C42—O49	1.6 (3)
C3A—N2—C17—O24	−161.7 (3)	C28B—N27—C42—O49	159.4 (4)
C3B—N2—C17—C18	13.9 (4)	C28A—N27—C42—C43	−19.1 (3)
C1—N2—C17—C18	179.39 (19)	C26—N27—C42—C43	−179.48 (18)
C3A—N2—C17—C18	18.1 (4)	C28B—N27—C42—C43	−21.7 (4)
O24—C17—C18—C19	41.8 (3)	O49—C42—C43—C48	−38.8 (3)
N2—C17—C18—C19	−138.0 (2)	N27—C42—C43—C48	142.4 (2)
O24—C17—C18—C23	−135.0 (3)	O49—C42—C43—C44	138.7 (2)
N2—C17—C18—C23	45.2 (3)	N27—C42—C43—C44	−40.2 (3)
C23—C18—C19—C20	−1.6 (4)	C48—C43—C44—C45	−0.9 (3)
C17—C18—C19—C20	−178.6 (2)	C42—C43—C44—C45	−178.3 (2)
C18—C19—C20—C21	−0.4 (4)	C43—C44—C45—C46	−1.1 (4)
C19—C20—C21—C22	2.1 (5)	C44—C45—C46—C47	2.2 (4)
C20—C21—C22—C23	−1.8 (5)	C45—C46—C47—C48	−1.3 (4)
C21—C22—C23—C18	−0.3 (5)	C44—C43—C48—C47	1.8 (3)
C19—C18—C23—C22	1.9 (4)	C42—C43—C48—C47	179.4 (2)
C17—C18—C23—C22	178.8 (3)	C46—C47—C48—C43	−0.7 (4)

Fig. 1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