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Ethyl (2*E*)-[1-(4-methoxyphenyl)-pyrrolidin-2-ylidene]acetate

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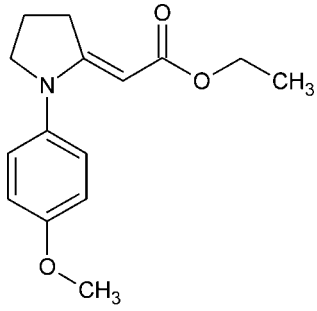
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.120; data-to-parameter ratio = 19.2.

The title compound, $C_{15}H_{19}NO_3$, crystallizes with two conformationally similar molecules, *A* and *B*, in the asymmetric unit. The structure contains several C—H···O interactions, two of which link molecules *A* and *B* in a pseudo-centrosymmetric fashion to form dimers described by the graph set $R_2^2(24)$. In addition, a C—H···π interaction between a benzene H atom of a *B* molecule and the benzene ring of an *A* molecule further link these dimers to form a ribbon of molecules running parallel to the [100] direction. The remaining C—H···O interactions between molecules *A* and *B*, *A* and *A*, and *B* and *B* provide additional stabilization for the structure.

Related literature

For details of the preparation and use of the title compound, see: Michael *et al.* (1988, 2001). For hydrogen-bonding motifs, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For discussion of C—H···O interactions, see: Desiraju (1996); Steiner & Desiraju (1998). For definition of ring-puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{15}H_{19}NO_3$
M_r = 261.31
Triclinic, $P\overline{1}$
a = 9.7056 (4) Å
b = 9.7763 (3) Å
c = 15.0413 (5) Å
α = 93.126 (2)°
β = 92.987 (2)°
γ = 103.301 (2)°
V = 1383.77 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.54 × 0.21 × 0.14 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: none
11703 measured reflections
6666 independent reflections
3779 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.120
S = 0.94
6666 reflections
347 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected torsion angles (°).

C10*A*—C9*A*—N1*A*—C3*A*	76.2 (2)	C10*B*—C9*B*—N1*B*—C3*B*	51.5 (2)
C13*A*—C12*A*—O3*A*—C15*A*	1.2 (2)	C13*B*—C12*B*—O3*B*—C15*B*	−176.74 (16)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4*A*—H4*A*···O1*B*^{i}	0.99	2.64	3.530 (2)	150
C7*A*—H7*B*···O1*A*^{ii}	0.99	2.60	3.546 (2)	160
C15*A*—H15*B*···O2*B*^{iii}	0.98	2.60	3.501 (2)	153
C5*B*—H5*D*···O1*B*^{iv}	0.99	2.55	3.463 (2)	153
C7*B*—H7*C*···O1*B*^{v}	0.99	2.71	3.490 (2)	137
C15*B*—H15*E*···O2*A*^{vi}	0.98	2.64	3.438 (2)	139
C13*B*—H13*B*···*Cg*	0.95	2.83	3.531 (2)	131

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x, -y+1, -z+2$; (iii) $x-1, y, z$; (iv) $-x+1, -y, -z$; (v) $-x+2, -y, -z$; (vi) $x+1, y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2016).

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supplementary materials

Acta Cryst. (2007). E**63**, o3139-o3140 [**doi:10.1107/S1600536807023689**]

Ethyl (2*E*)-[1-(4-methoxyphenyl)pyrrolidin-2-ylidene]acetate

M. A. Fernandes, J. P. Michael and C. B. Perry

Comment

N-Arylenaminones such as (I) can be prepared in various ways, including by the Eschenmoser sulfide contraction and by reaction of suitable anilines with ethyl 6-chloro-3-oxohexanoate (Michael *et al.*, 1988). Our interest in these compounds stems from their ready cyclization in hot polyphosphoric acid (a variant of the classic Conrad-Limpach reaction) to give tricyclic analogues of the quinolone antibacterial agents (Michael *et al.*, 2001).

Compound (I) crystallizes in the space group P-1 with two molecules in the asymmetric unit (Z' = 2, Fig. 1). The two molecules are conformationally similar, with the major differences between the two being the degree of rotation around the C9—N1 and C12—O3 bonds (Table 1). In both cases, the closest pucker descriptor of the pyrrolidine rings is twisted on C5A—C6A and C5B—C6B for molecules A and B, respectively. The Cremer & Pople puckering parameters (Cremer & Pople, 1975) for the pyrrolidine ring of molecule A are q_2 = 0.2204 (19) Å and φ_2 = 118.9 (5)°, and for molecule B are q_2 = 0.2935 (19) Å and φ_2 = 118.3 (3)°.

The structure contains several C—H···O interactions (Table 2). Two of these, C15A—H15B···O2B and C15B—H15E···O2A, link molecules A and B in a pseudo-centrosymmetric fashion, forming dimers described by the graph set $R^2_2(24)$ (Etter *et al.*, 1990; Bernstein *et al.*, 1995). In addition, a C—H···π interaction between the H13B atom of a B molecule and the phenyl ring of an A molecule (Fig. 1) further links these dimers to form a ribbon of molecules running parallel to the [100] direction (Fig. 2). The remaining C—H···O interactions between molecules A and B, A and A, and B and B provide additional stabilization for the structure (Table 2). Three of these C—H···O interactions are flagged by *PLATON* (Spek, 2003) as slightly long, but have C···O distances and other geometric characteristics well within the limits defined by Desiraju *et al.* (Desiraju, 1996; Steiner & Desiraju, 1998).

Experimental

Ethyl (2E)-[1-(4-methoxyphenyl)pyrrolidin-2-ylidene]acetate (I) was prepared in 65% yield from 4-methoxyaniline and ethyl 6-chloro-3-oxohexanoate, as described previously (Michael *et al.*, 1988). Crystals suitable for X-ray crystallography were obtained as colourless needles by recrystallization from hexane–ethyl acetate (*ca* 1:2).

Refinement

All H atoms were positioned geometrically, and allowed to ride on their parent atoms, with C—H bond lengths of 0.99 Å (CH_2), 0.98 Å (CH_3), or 0.95 Å (aromatic CH). Isotropic displacement parameters for these atoms were set equal to 1.2 (CH_2 and aromatic CH), or 1.5 (CH_3) times U_{eq} of the parent atom.

Figures

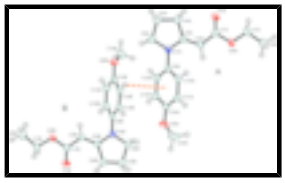


Fig. 1. The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intermolecular C—H···π interaction is shown with a dashed bond.

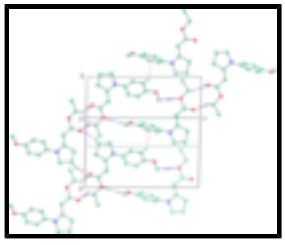


Fig. 2. Part of the crystal structure of (I) showing the intermolecular C—H···O (dashed purple lines), and C—H···π interactions (dashed orange lines). All hydrogen atoms not involved in these interactions have been omitted for clarity.

Ethyl (2E)-[1-(4-methoxyphenyl)pyrrolidin-2-ylidene]acetate

Crystal data

$C_{15}H_{19}NO_3$	$Z = 4$
$M_r = 261.31$	$F_{000} = 560$
	$D_x = 1.254$ Mg m^{-3}
Triclinic, $P\overline{1}$	$D_m = 1.254$ Mg m^{-3}
	D_m measured by not measured
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.7056$ (4) Å	Cell parameters from 2637 reflections
$b = 9.7763$ (3) Å	$\theta = 0.00$–0.00°
$c = 15.0413$ (5) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 93.126$ (2)°	$T = 173$ (2) K
$\beta = 92.987$ (2)°	Needle, colourless
$\gamma = 103.301$ (2)°	0.54 × 0.21 × 0.14 mm
$V = 1383.77$ (9) Å^3	

Data collection

Bruker APEX II CCD area-detector diffractometer	3779 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.041$
Monochromator: graphite	$\theta_{max} = 28.0^\circ$
$T = 173(2)$ K	$\theta_{min} = 1.4^\circ$
phi and ω scans	$h = -12 \rightarrow 12$
Absorption correction: none	$k = -12 \rightarrow 12$
11703 measured reflections	$l = -19 \rightarrow 18$
6666 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
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.120$	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.94$	$(\Delta/\sigma)_{max} < 0.001$
6666 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
347 parameters	$\Delta\rho_{min} = -0.22$ 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}
C1A	0.17022 (17)	0.52626 (17)	0.85839 (11)	0.0295 (4)
C2A	0.23671 (16)	0.46291 (16)	0.78914 (10)	0.0287 (4)
H2A	0.1999	0.4615	0.7292	0.034*
C3A	0.34824 (17)	0.40551 (16)	0.80472 (11)	0.0279 (4)
C4A	0.42096 (18)	0.39177 (19)	0.89320 (11)	0.0372 (4)
H4A	0.3577	0.3240	0.9283	0.045*
H4B	0.4483	0.4840	0.9279	0.045*
C5A	0.5506 (2)	0.3393 (2)	0.87207 (12)	0.0500 (5)
H5A	0.5653	0.2677	0.9133	0.060*
H5B	0.6362	0.4179	0.8769	0.060*
C6A	0.52023 (19)	0.2757 (2)	0.77750 (12)	0.0414 (5)
H6B	0.6069	0.2959	0.7439	0.050*
H6A	0.4829	0.1725	0.7763	0.050*
C7A	−0.02277 (19)	0.62950 (19)	0.88806 (12)	0.0406 (5)
H7A	0.0422	0.7115	0.9212	0.049*
H7B	−0.0634	0.5612	0.9314	0.049*
C8A	−0.1382 (2)	0.6755 (2)	0.83749 (15)	0.0582 (6)
H8A	−0.0967	0.7464	0.7968	0.087*

H8B	−0.1944	0.7163	0.8793	0.087*
H8C	−0.1996	0.5942	0.8030	0.087*
C9A	0.35464 (16)	0.30970 (17)	0.65066 (11)	0.0276 (4)
C10A	0.35809 (18)	0.41127 (17)	0.58982 (11)	0.0339 (4)
H10A	0.3991	0.5073	0.6078	0.041*
C11A	0.30246 (18)	0.37437 (17)	0.50308 (11)	0.0346 (4)
H11A	0.3043	0.4451	0.4620	0.041*
C12A	0.24384 (17)	0.23381 (17)	0.47590 (11)	0.0294 (4)
C13A	0.23974 (17)	0.13206 (17)	0.53626 (11)	0.0332 (4)
H13A	0.1994	0.0359	0.5183	0.040*
C14A	0.29465 (17)	0.17055 (17)	0.62314 (11)	0.0323 (4)
H14A	0.2911	0.1001	0.6646	0.039*
C15A	0.1331 (2)	0.06410 (19)	0.35860 (12)	0.0476 (5)
H15A	0.2034	0.0076	0.3677	0.071*
H15B	0.1027	0.0582	0.2951	0.071*
H15C	0.0508	0.0281	0.3929	0.071*
N1A	0.41348 (14)	0.34435 (14)	0.74039 (9)	0.0315 (3)
O1A	0.20722 (13)	0.54574 (13)	0.93772 (8)	0.0433 (3)
O2A	0.05333 (12)	0.56466 (11)	0.82451 (7)	0.0327 (3)
O3A	0.19482 (13)	0.20770 (12)	0.38803 (8)	0.0403 (3)
C1B	0.83658 (17)	−0.02700 (16)	0.12391 (11)	0.0290 (4)
C2B	0.79144 (16)	0.05642 (16)	0.19314 (10)	0.0267 (4)
H2B	0.8452	0.0749	0.2489	0.032*
C3B	0.67510 (16)	0.10985 (16)	0.18201 (10)	0.0264 (4)
C4B	0.58101 (17)	0.09749 (18)	0.09807 (11)	0.0335 (4)
H4C	0.5236	−0.0001	0.0859	0.040*
H4D	0.6381	0.1243	0.0464	0.040*
C5B	0.48628 (19)	0.19869 (19)	0.11569 (12)	0.0390 (5)
H5C	0.5274	0.2925	0.0944	0.047*
H5D	0.3902	0.1620	0.0862	0.047*
C6B	0.48175 (17)	0.2060 (2)	0.21622 (12)	0.0387 (5)
H6C	0.4716	0.2996	0.2395	0.046*
H6D	0.4026	0.1325	0.2353	0.046*
C7B	1.00681 (18)	−0.15775 (18)	0.08914 (12)	0.0392 (5)
H7C	1.0163	−0.1161	0.0306	0.047*
H7D	0.9389	−0.2507	0.0808	0.047*
C8B	1.1469 (2)	−0.1744 (2)	0.12535 (14)	0.0581 (6)
H8D	1.2157	−0.0833	0.1282	0.087*
H8E	1.1794	−0.2425	0.0863	0.087*
H8F	1.1380	−0.2084	0.1854	0.087*
C9B	0.66910 (16)	0.20555 (16)	0.33754 (10)	0.0254 (4)
C10B	0.81081 (16)	0.26330 (16)	0.36328 (11)	0.0274 (4)
H10B	0.8771	0.2863	0.3190	0.033*
C11B	0.85713 (16)	0.28796 (16)	0.45265 (10)	0.0276 (4)
H11B	0.9549	0.3255	0.4693	0.033*
C12B	0.76065 (17)	0.25783 (16)	0.51795 (11)	0.0294 (4)
C13B	0.61861 (17)	0.20185 (17)	0.49247 (11)	0.0340 (4)
H13B	0.5518	0.1817	0.5367	0.041*
C14B	0.57365 (17)	0.17523 (17)	0.40377 (11)	0.0319 (4)

H14B	0.4762	0.1356	0.3874	0.038*
C15B	0.9377 (2)	0.3441 (2)	0.63622 (12)	0.0549 (6)
H15D	0.9989	0.2822	0.6176	0.082*
H15E	0.9464	0.3598	0.7014	0.082*
H15F	0.9667	0.4345	0.6094	0.082*
N1B	0.61959 (13)	0.18097 (14)	0.24637 (9)	0.0281 (3)
O1B	0.77983 (13)	−0.06314 (13)	0.04944 (8)	0.0420 (3)
O2B	0.95603 (12)	−0.06652 (12)	0.15178 (7)	0.0351 (3)
O3B	0.79418 (12)	0.27977 (13)	0.60753 (8)	0.0434 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0285 (9)	0.0320 (9)	0.0274 (10)	0.0064 (7)	0.0024 (7)	−0.0002 (7)
C2A	0.0305 (9)	0.0343 (9)	0.0210 (9)	0.0087 (7)	−0.0010 (7)	−0.0025 (7)
C3A	0.0273 (9)	0.0290 (8)	0.0253 (9)	0.0032 (7)	0.0014 (7)	−0.0001 (7)
C4A	0.0380 (10)	0.0438 (10)	0.0301 (10)	0.0127 (9)	−0.0062 (8)	−0.0012 (8)
C5A	0.0446 (12)	0.0706 (14)	0.0398 (12)	0.0250 (11)	−0.0039 (9)	0.0040 (10)
C6A	0.0346 (10)	0.0516 (11)	0.0431 (12)	0.0215 (9)	−0.0014 (9)	0.0032 (9)
C7A	0.0415 (11)	0.0458 (11)	0.0392 (11)	0.0179 (9)	0.0167 (9)	−0.0022 (9)
C8A	0.0495 (13)	0.0694 (14)	0.0678 (15)	0.0337 (11)	0.0179 (11)	0.0119 (12)
C9A	0.0260 (9)	0.0332 (9)	0.0260 (9)	0.0118 (7)	0.0064 (7)	−0.0013 (7)
C10A	0.0431 (11)	0.0267 (8)	0.0332 (10)	0.0108 (8)	0.0056 (8)	−0.0015 (7)
C11A	0.0454 (11)	0.0329 (9)	0.0286 (10)	0.0144 (8)	0.0051 (8)	0.0046 (7)
C12A	0.0274 (9)	0.0378 (9)	0.0248 (9)	0.0110 (8)	0.0059 (7)	−0.0021 (7)
C13A	0.0329 (10)	0.0300 (9)	0.0347 (10)	0.0028 (8)	0.0069 (8)	−0.0017 (8)
C14A	0.0346 (10)	0.0325 (9)	0.0308 (10)	0.0078 (8)	0.0082 (8)	0.0065 (8)
C15A	0.0421 (11)	0.0533 (12)	0.0400 (12)	0.0010 (10)	−0.0034 (9)	−0.0130 (10)
N1A	0.0321 (8)	0.0394 (8)	0.0263 (8)	0.0158 (7)	0.0007 (6)	−0.0002 (6)
O1A	0.0464 (8)	0.0643 (9)	0.0223 (7)	0.0217 (7)	−0.0004 (6)	−0.0059 (6)
O2A	0.0317 (6)	0.0424 (7)	0.0272 (6)	0.0155 (5)	0.0046 (5)	−0.0025 (5)
O3A	0.0460 (8)	0.0445 (7)	0.0289 (7)	0.0099 (6)	−0.0019 (6)	−0.0031 (6)
C1B	0.0268 (9)	0.0292 (9)	0.0290 (10)	0.0029 (7)	0.0030 (7)	0.0002 (7)
C2B	0.0278 (9)	0.0290 (8)	0.0220 (8)	0.0049 (7)	0.0014 (7)	−0.0021 (7)
C3B	0.0260 (9)	0.0261 (8)	0.0240 (9)	0.0004 (7)	0.0023 (7)	−0.0016 (7)
C4B	0.0308 (9)	0.0388 (10)	0.0283 (9)	0.0050 (8)	−0.0044 (8)	−0.0006 (8)
C5B	0.0329 (10)	0.0479 (11)	0.0362 (11)	0.0133 (9)	−0.0083 (8)	−0.0041 (9)
C6B	0.0260 (9)	0.0524 (11)	0.0385 (11)	0.0145 (8)	−0.0052 (8)	−0.0060 (9)
C7B	0.0428 (11)	0.0367 (10)	0.0406 (11)	0.0143 (8)	0.0125 (9)	−0.0070 (8)
C8B	0.0571 (14)	0.0749 (15)	0.0543 (14)	0.0385 (12)	0.0123 (11)	0.0020 (12)
C9B	0.0261 (8)	0.0254 (8)	0.0256 (9)	0.0092 (7)	0.0015 (7)	−0.0033 (7)
C10B	0.0259 (8)	0.0288 (8)	0.0263 (9)	0.0037 (7)	0.0051 (7)	−0.0015 (7)
C11B	0.0226 (8)	0.0290 (8)	0.0282 (9)	0.0020 (7)	−0.0011 (7)	−0.0036 (7)
C12B	0.0322 (9)	0.0309 (9)	0.0238 (9)	0.0067 (7)	0.0018 (7)	−0.0048 (7)
C13B	0.0285 (9)	0.0421 (10)	0.0309 (10)	0.0065 (8)	0.0101 (8)	−0.0029 (8)
C14B	0.0221 (8)	0.0374 (9)	0.0345 (10)	0.0053 (7)	0.0037 (7)	−0.0068 (8)
C15B	0.0474 (12)	0.0733 (14)	0.0296 (11)	−0.0108 (11)	−0.0074 (9)	−0.0083 (10)
N1B	0.0225 (7)	0.0364 (8)	0.0256 (7)	0.0092 (6)	−0.0005 (6)	−0.0044 (6)

O1B	0.0428 (7)	0.0528 (8)	0.0296 (7)	0.0144 (6)	−0.0030 (6)	−0.0137 (6)
O2B	0.0354 (7)	0.0399 (7)	0.0326 (7)	0.0156 (6)	0.0045 (6)	−0.0076 (5)
O3B	0.0385 (7)	0.0611 (8)	0.0242 (7)	0.0003 (6)	0.0025 (6)	−0.0050 (6)

Geometric parameters (Å, °)

C1A—O1A	1.2199 (18)	C1B—O1B	1.2189 (18)
C1A—O2A	1.358 (2)	C1B—O2B	1.357 (2)
C1A—C2A	1.438 (2)	C1B—C2B	1.436 (2)
C2A—C3A	1.345 (2)	C2B—C3B	1.355 (2)
C2A—H2A	0.9500	C2B—H2B	0.9500
C3A—N1A	1.3675 (19)	C3B—N1B	1.3653 (18)
C3A—C4A	1.501 (2)	C3B—C4B	1.502 (2)
C4A—C5A	1.506 (2)	C4B—C5B	1.520 (2)
C4A—H4A	0.9900	C4B—H4C	0.9900
C4A—H4B	0.9900	C4B—H4D	0.9900
C5A—C6A	1.507 (2)	C5B—C6B	1.513 (2)
C5A—H5A	0.9900	C5B—H5C	0.9900
C5A—H5B	0.9900	C5B—H5D	0.9900
C6A—N1A	1.461 (2)	C6B—N1B	1.468 (2)
C6A—H6B	0.9900	C6B—H6C	0.9900
C6A—H6A	0.9900	C6B—H6D	0.9900
C7A—O2A	1.4437 (18)	C7B—O2B	1.4440 (17)
C7A—C8A	1.489 (3)	C7B—C8B	1.485 (3)
C7A—H7A	0.9900	C7B—H7C	0.9900
C7A—H7B	0.9900	C7B—H7D	0.9900
C8A—H8A	0.9800	C8B—H8D	0.9800
C8A—H8B	0.9800	C8B—H8E	0.9800
C8A—H8C	0.9800	C8B—H8F	0.9800
C9A—C14A	1.382 (2)	C9B—C10B	1.387 (2)
C9A—C10A	1.383 (2)	C9B—C14B	1.393 (2)
C9A—N1A	1.429 (2)	C9B—N1B	1.4183 (19)
C10A—C11A	1.382 (2)	C10B—C11B	1.385 (2)
C10A—H10A	0.9500	C10B—H10B	0.9500
C11A—C12A	1.391 (2)	C11B—C12B	1.389 (2)
C11A—H11A	0.9500	C11B—H11B	0.9500
C12A—O3A	1.3712 (19)	C12B—O3B	1.3632 (18)
C12A—C13A	1.378 (2)	C12B—C13B	1.386 (2)
C13A—C14A	1.384 (2)	C13B—C14B	1.374 (2)
C13A—H13A	0.9500	C13B—H13B	0.9500
C14A—H14A	0.9500	C14B—H14B	0.9500
C15A—O3A	1.4295 (19)	C15B—O3B	1.424 (2)
C15A—H15A	0.9800	C15B—H15D	0.9800
C15A—H15B	0.9800	C15B—H15E	0.9800
C15A—H15C	0.9800	C15B—H15F	0.9800
O1A—C1A—O2A	121.46 (15)	O1B—C1B—O2B	121.40 (15)
O1A—C1A—C2A	127.81 (17)	O1B—C1B—C2B	127.75 (17)
O2A—C1A—C2A	110.73 (14)	O2B—C1B—C2B	110.84 (14)
C3A—C2A—C1A	123.46 (16)	C3B—C2B—C1B	122.52 (15)

C3A—C2A—H2A	118.3	C3B—C2B—H2B	118.7
C1A—C2A—H2A	118.3	C1B—C2B—H2B	118.7
C2A—C3A—N1A	124.80 (15)	C2B—C3B—N1B	125.93 (15)
C2A—C3A—C4A	127.71 (15)	C2B—C3B—C4B	126.49 (14)
N1A—C3A—C4A	107.46 (15)	N1B—C3B—C4B	107.55 (14)
C3A—C4A—C5A	105.81 (14)	C3B—C4B—C5B	104.97 (13)
C3A—C4A—H4A	110.6	C3B—C4B—H4C	110.8
C5A—C4A—H4A	110.6	C5B—C4B—H4C	110.8
C3A—C4A—H4B	110.6	C3B—C4B—H4D	110.8
C5A—C4A—H4B	110.6	C5B—C4B—H4D	110.8
H4A—C4A—H4B	108.7	H4C—C4B—H4D	108.8
C4A—C5A—C6A	104.88 (15)	C6B—C5B—C4B	103.25 (15)
C4A—C5A—H5A	110.8	C6B—C5B—H5C	111.1
C6A—C5A—H5A	110.8	C4B—C5B—H5C	111.1
C4A—C5A—H5B	110.8	C6B—C5B—H5D	111.1
C6A—C5A—H5B	110.8	C4B—C5B—H5D	111.1
H5A—C5A—H5B	108.8	H5C—C5B—H5D	109.1
N1A—C6A—C5A	103.85 (14)	N1B—C6B—C5B	102.98 (13)
N1A—C6A—H6B	111.0	N1B—C6B—H6C	111.2
C5A—C6A—H6B	111.0	C5B—C6B—H6C	111.2
N1A—C6A—H6A	111.0	N1B—C6B—H6D	111.2
C5A—C6A—H6A	111.0	C5B—C6B—H6D	111.2
H6B—C6A—H6A	109.0	H6C—C6B—H6D	109.1
O2A—C7A—C8A	107.74 (15)	O2B—C7B—C8B	108.21 (14)
O2A—C7A—H7A	110.2	O2B—C7B—H7C	110.1
C8A—C7A—H7A	110.2	C8B—C7B—H7C	110.1
O2A—C7A—H7B	110.2	O2B—C7B—H7D	110.1
C8A—C7A—H7B	110.2	C8B—C7B—H7D	110.1
H7A—C7A—H7B	108.5	H7C—C7B—H7D	108.4
C7A—C8A—H8A	109.5	C7B—C8B—H8D	109.5
C7A—C8A—H8B	109.5	C7B—C8B—H8E	109.5
H8A—C8A—H8B	109.5	H8D—C8B—H8E	109.5
C7A—C8A—H8C	109.5	C7B—C8B—H8F	109.5
H8A—C8A—H8C	109.5	H8D—C8B—H8F	109.5
H8B—C8A—H8C	109.5	H8E—C8B—H8F	109.5
C14A—C9A—C10A	118.80 (15)	C10B—C9B—C14B	118.39 (15)
C14A—C9A—N1A	119.30 (15)	C10B—C9B—N1B	121.71 (14)
C10A—C9A—N1A	121.89 (14)	C14B—C9B—N1B	119.87 (14)
C11A—C10A—C9A	120.61 (15)	C11B—C10B—C9B	120.96 (15)
C11A—C10A—H10A	119.7	C11B—C10B—H10B	119.5
C9A—C10A—H10A	119.7	C9B—C10B—H10B	119.5
C10A—C11A—C12A	120.06 (17)	C10B—C11B—C12B	120.00 (15)
C10A—C11A—H11A	120.0	C10B—C11B—H11B	120.0
C12A—C11A—H11A	120.0	C12B—C11B—H11B	120.0
O3A—C12A—C13A	124.77 (14)	O3B—C12B—C13B	115.89 (14)
O3A—C12A—C11A	115.60 (16)	O3B—C12B—C11B	124.90 (14)
C13A—C12A—C11A	119.63 (16)	C13B—C12B—C11B	119.21 (15)
C12A—C13A—C14A	119.74 (15)	C14B—C13B—C12B	120.54 (15)
C12A—C13A—H13A	120.1	C14B—C13B—H13B	119.7

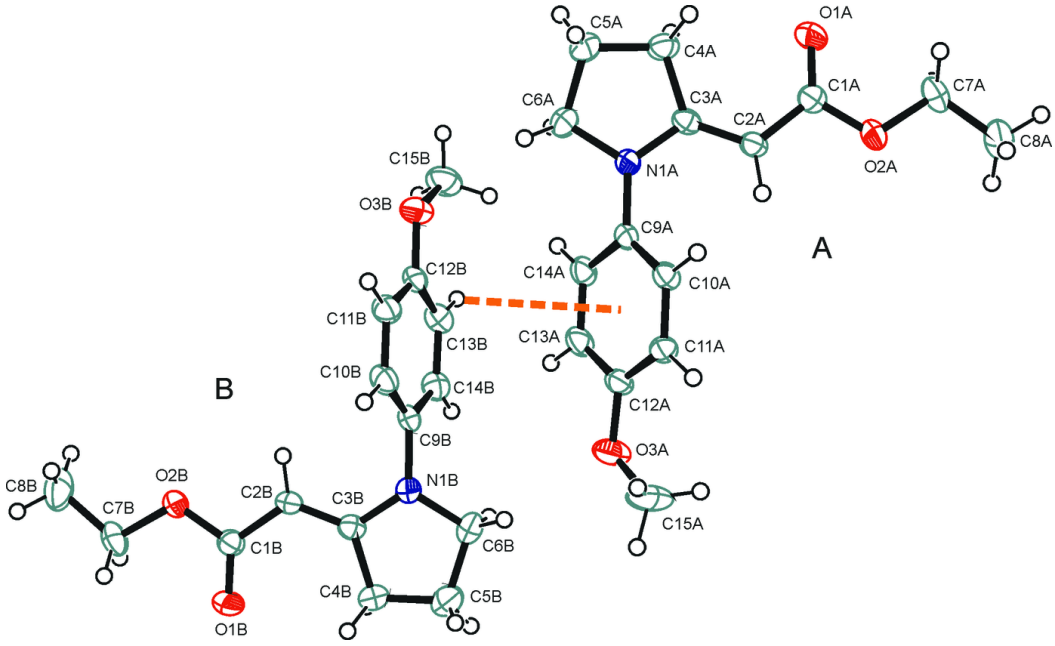
C14A—C13A—H13A	120.1	C12B—C13B—H13B	119.7
C9A—C14A—C13A	121.15 (17)	C13B—C14B—C9B	120.88 (15)
C9A—C14A—H14A	119.4	C13B—C14B—H14B	119.6
C13A—C14A—H14A	119.4	C9B—C14B—H14B	119.6
O3A—C15A—H15A	109.5	O3B—C15B—H15D	109.5
O3A—C15A—H15B	109.5	O3B—C15B—H15E	109.5
H15A—C15A—H15B	109.5	H15D—C15B—H15E	109.5
O3A—C15A—H15C	109.5	O3B—C15B—H15F	109.5
H15A—C15A—H15C	109.5	H15D—C15B—H15F	109.5
H15B—C15A—H15C	109.5	H15E—C15B—H15F	109.5
C3A—N1A—C9A	123.25 (14)	C3B—N1B—C9B	126.00 (14)
C3A—N1A—C6A	112.78 (14)	C3B—N1B—C6B	112.13 (13)
C9A—N1A—C6A	120.97 (13)	C9B—N1B—C6B	120.75 (13)
C1A—O2A—C7A	115.79 (13)	C1B—O2B—C7B	115.68 (13)
C12A—O3A—C15A	116.68 (14)	C12B—O3B—C15B	117.54 (13)
O1A—C1A—C2A—C3A	4.9 (3)	O1B—C1B—C2B—C3B	−1.4 (3)
O2A—C1A—C2A—C3A	−174.83 (14)	O2B—C1B—C2B—C3B	179.57 (14)
C1A—C2A—C3A—N1A	179.86 (14)	C1B—C2B—C3B—N1B	174.85 (14)
C1A—C2A—C3A—C4A	1.9 (3)	C1B—C2B—C3B—C4B	−2.8 (2)
C2A—C3A—C4A—C5A	−172.15 (17)	C2B—C3B—C4B—C5B	−169.03 (16)
N1A—C3A—C4A—C5A	9.64 (18)	N1B—C3B—C4B—C5B	12.94 (17)
C3A—C4A—C5A—C6A	−20.0 (2)	C3B—C4B—C5B—C6B	−26.37 (17)
C4A—C5A—C6A—N1A	22.6 (2)	C4B—C5B—C6B—N1B	29.56 (17)
C14A—C9A—C10A—C11A	0.1 (3)	C14B—C9B—C10B—C11B	1.2 (2)
N1A—C9A—C10A—C11A	178.92 (15)	N1B—C9B—C10B—C11B	179.33 (14)
C9A—C10A—C11A—C12A	−0.8 (3)	C9B—C10B—C11B—C12B	−1.5 (2)
C10A—C11A—C12A—O3A	−178.39 (15)	C10B—C11B—C12B—O3B	−179.01 (15)
C10A—C11A—C12A—C13A	1.0 (3)	C10B—C11B—C12B—C13B	0.6 (2)
O3A—C12A—C13A—C14A	178.91 (15)	O3B—C12B—C13B—C14B	−179.77 (15)
C11A—C12A—C13A—C14A	−0.4 (3)	C11B—C12B—C13B—C14B	0.6 (3)
C10A—C9A—C14A—C13A	0.5 (2)	C12B—C13B—C14B—C9B	−0.9 (3)
N1A—C9A—C14A—C13A	−178.36 (15)	C10B—C9B—C14B—C13B	0.0 (2)
C12A—C13A—C14A—C9A	−0.4 (3)	N1B—C9B—C14B—C13B	−178.17 (15)
C2A—C3A—N1A—C9A	−12.4 (2)	C2B—C3B—N1B—C9B	−3.5 (2)
C4A—C3A—N1A—C9A	165.84 (14)	C4B—C3B—N1B—C9B	174.58 (14)
C2A—C3A—N1A—C6A	−172.96 (16)	C2B—C3B—N1B—C6B	−171.40 (15)
C4A—C3A—N1A—C6A	5.32 (18)	C4B—C3B—N1B—C6B	6.66 (18)
C14A—C9A—N1A—C3A	−104.94 (18)	C10B—C9B—N1B—C3B	51.5 (2)
C10A—C9A—N1A—C3A	76.2 (2)	C14B—C9B—N1B—C3B	−130.39 (16)
C14A—C9A—N1A—C6A	54.0 (2)	C10B—C9B—N1B—C6B	−141.55 (16)
C10A—C9A—N1A—C6A	−124.82 (18)	C14B—C9B—N1B—C6B	36.6 (2)
C5A—C6A—N1A—C3A	−17.92 (19)	C5B—C6B—N1B—C3B	−23.41 (18)
C5A—C6A—N1A—C9A	−178.93 (14)	C5B—C6B—N1B—C9B	167.95 (14)
O1A—C1A—O2A—C7A	0.1 (2)	O1B—C1B—O2B—C7B	−2.4 (2)
C2A—C1A—O2A—C7A	179.90 (13)	C2B—C1B—O2B—C7B	176.66 (13)
C8A—C7A—O2A—C1A	174.90 (15)	C8B—C7B—O2B—C1B	172.56 (15)
C13A—C12A—O3A—C15A	1.2 (2)	C13B—C12B—O3B—C15B	−176.74 (16)
C11A—C12A—O3A—C15A	−179.54 (15)	C11B—C12B—O3B—C15B	2.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4A—H4A···O1B^{i}	0.99	2.64	3.530 (2)	150
C7A—H7B···O1A^{ii}	0.99	2.60	3.546 (2)	160
C15A—H15B···O2B^{iii}	0.98	2.60	3.501 (2)	153
C5B—H5D···O1B^{iv}	0.99	2.55	3.463 (2)	153
C7B—H7C···O1B^{v}	0.99	2.71	3.490 (2)	137
C15B—H15E···O2A^{vi}	0.98	2.64	3.438 (2)	139
C13B—H13B···Cg	0.95	2.83	3.531 (2)	131

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

Fig. 1


C5A
C4A
O1A
C6A
C3A
C1A
C7A
C2A
C8A
O2A
N1A
C15B
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C9A
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C12B
C14A
C10A
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C12A
C9B
O3A
N1B
O2B
C2B
C8B
C3B
C15A
C7B
C6B
C1B
C4B
C5B
O1B

Fig. 2