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Ethyl [5-(2-hydroxyphenyl)-3-methyl-4,5-dihydro-1*H*-pyrazol-1-yl]oxoacetate

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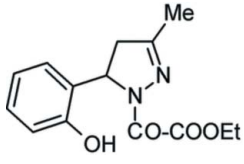
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.012 Å; R factor = 0.067; wR factor = 0.186; data-to-parameter ratio = 9.2.

The title compound, $C_{14}H_{16}N_2O_4$, crystallizes with four molecules in the asymmetric unit. The independent molecules differ from each other in the conformation of the chiral site of the molecule and in the conformation of the ethoxy chain. The ester group is inclined approximately at right angles with respect to the mean plane of the carbonylpyrazoline unit. Each molecule is involved in two intermolecular hydrogen bonds, in one acting as a donor and in the other as a hydrogen-bond acceptor.

Related literature

The present structure can be compared with six compounds incorporating the 1-carbonyl-5-phenylpyrazoline substructure as retrieved from the Cambridge Structural Database (Version 5.27; Allen, 2002). In all seven structures the carbonyl function is, due to its conjugation with the neighbouring N atom, coplanar with the mean plane of the pyrazoline ring. By contrast, the conformation on the bond linking the benzene and pyrazoline rings varies between cases: (1) the conformation in which the plane of the benzene ring bisects the heterocycle (the N2=C3 double bond) as observed for the title derivative and (2) the conformation where the benzene plane eclipses the N1—C5 bond as reported for 1-acetyl-5-(2-methoxyphenyl)-3-(2-methoxystyryl)-2-pyrazoline (Krishnakumar *et al.*, 2004).

For related literature, see: Allen *et al.* (1987); Burke-Laing & Laing (1976); Kalinowski *et al.* (1984); Lambert *et al.* (1998); Shmueli *et al.* (1973); Světlík *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{16}N_2O_4$
M_r = 276.29
Monoclinic, *Cc*
a = 17.133 (3) Å
b = 17.062 (3) Å
c = 20.130 (4) Å
β = 93.02 (4)°
V = 5876.3 (19) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Siemens *P*4 diffractometer
Absorption correction: none
7430 measured reflections
6753 independent reflections
3465 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.186
S = 1.04
6753 reflections
733 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4A—H4A···O1C^{i}	0.82	1.86	2.678 (7)	177
O4B—H4B···O1D^{ii}	0.82	1.88	2.665 (7)	160
O4C—H4C···O1A	0.82	1.89	2.700 (6)	171
O4D—H4D···O1B	0.82	1.87	2.664 (7)	164

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

Data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2337).

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supplementary materials

Acta Cryst. (2007). E**63**, o2607-o2608 [**doi:10.1107/S1600536807019083**]

Ethyl [5-(2-hydroxyphenyl)-3-methyl-4,5-dihydro-1*H*-pyrazol-1-yl]oxoacetate

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Comment

As a continuation of our project aimed at developing new therapeutic agents, we attempted to prepare, by the reaction of 4,5-dihydro-3-methyl-5-(2-hydroxyphenyl)-1*H*-pyrazole (I) (Světlík *et al.*, 2005) with diethyl oxalate, the tricyclic compound (II) possessing 1,4-oxazepine core. However, ^{1}H and ^{13}C NMR spectra were consistent with the structure (III) or (IV), but it was difficult to distinguish between these two alternatives from the NMR spectral data (Kalinowski *et al.*, 1984); on the other hand, the IR spectrum matches better the structure (III) (Lambert *et al.*, 1998). To confirm our presumption and to establish conformational features of the molecule, an X-ray analysis of the reaction product was undertaken.

The structure determination has shown that the four independent molecules (denoted as A - D) are, except for the terminal ethyl groups, identical to within 5/s as far as bond distances and angles are concerned. Thus, only one molecule (A) is shown in Fig. 2. A s can be seen, the compound has the structure (III).

The sp^2-hybridized N1 atom is involved in conjugation with the adjacent carbonyl function, as found for amides (Allen *et al.*, 1987), rather than the pyrazoline double bond (Burke-Laing & Laing, 1976). In all four molecules the C6—C7 bond lengths are close to the value of 1.487 (5) Å reported for a Csp^2—Csp^2 single bond (Shmueli *et al.*, 1973). Other bond distances are close to those generally expected.

As mentioned above, the conformational properties of the molecule is of prime interest here. The conformation of the hydroxyphenylpyrazoline portion in all independent molecules is the same: the pyrazoline ring is approximately planar with slight but significant distortion towards the C5-envelope; the phenyl ring occupies the pseudoaxial position and bisects the heterocycle. However, relative configuration on C5 in molecules B and C is opposite relative to molecules A and D. Another source of asymmetry is imposed by the approximatelly perpendicular orientation of the ethyl ester group relative to the mean plane of the carbonylpyrazoline moiety. As shown by the torsion angle N1—C6—C7—O2, both 'enantiomeric' values (ca. ±90°) are equally populated. The third difference among the independent molecules concerns the rotation around the O3—C8 bond.

The crystal packing is dominated by hydrogen bonding. As shown in Table 1, the hydroxy group of all independent molecules is involved in an H-bond interaction with the carbonyl atom adjacent to the heterocycle of a neighbouring molecule.

Experimental

The title compound was prepared by refluxing pyrazoline (I) (352 mg, 2 mmol) in diethyl oxalate (15 ml) for 2.5 h. After evaporation of the solvent, the resultant oil was dissolved in ether and left to crystallize to give colourless crystals (46% yield; m.p. 415-417 K). IR (KBr): 3165 (OH), 1746 (ester CO), 1641 (amide CO), 1600 (C=C/C=N) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.39 (3*H*, t, ester Me), 2.07 (3*H*, s, Me), 2.78 (1*H*, dd, J = 3.3 and 18.6 Hz, H of CH_2), 3.39 (1*H*, dd, J = 11.1 and 18.6 Hz, H of CH_2), 4.42 (2*H*, q, ester CH_2), 5.76 (1*H*, dd, J = 3.3 and 11.1 Hz, CH), 6.50 (1*H*, t, H_{ar}), 6.77 (1*H*, t, H_{ar}), 6.91

(2*H*, m, H_{ar}), 8.00 (1*H*, brs, OH). ^{13}C NMR ($CDCl_3$): δ 14.0 (ester Me), 16.0 (Me), 45.2 (CH_2), 54.2 (CH), 62.4 (ester CH_2), 116.9 (CH-3'), 120.2 (CH-5'), 124.6 (CH-6'), 125.0 (C-1'), 129.3 (CH-4'), 153.9 (C-2'), 159.2 (C=N), 161.6 and 162.2 (C=O ester/amide).

Refinement

H atoms were visible in difference maps and were subsequently treated as riding atoms with distances C—H = 0.93 (C_{arom}), 0.98 ($C_{tertiary}$), 0.97 (CH_2), 0.96 (CH_3) and 0.82 Å (OH); U_{iso} of the H atoms were set to 1.2 (1.5 for the methyl H atoms) times U_{eq} of the parent atom. Reflection 002, affected by secondary extinction, was deleted and the Friedel pairs merged before the least-squares refinement.

Figures

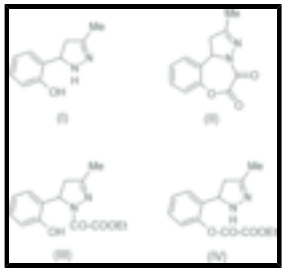


Fig. 1. Compounds (i)–(IV).

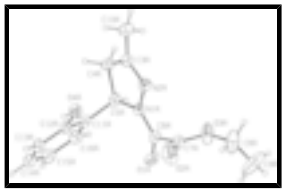


Fig. 2. Displacement ellipsoid plot of (III) with the labelling scheme for the non-H atoms, which are drawn as 25% probability ellipsoids. Only molecule A is shown for clarity.

Ethyl [5-(2-hydroxyphenyl)-3-methyl-4,5-dihydro-1*H*-pyrazol-1-yl]oxoacetate

Crystal data

$C_{14}H_{16}N_2O_4$	F_{000} = 2336
M_r = 276.29	D_x = 1.249 Mg m^{-3}
Monoclinic, *Cc*	Melting point: 416 K
Hall symbol: C -2yc	Mo *K*α radiation λ = 0.71073 Å
a = 17.133 (3) Å	Cell parameters from 20 reflections
b = 17.062 (3) Å	θ = 7–18º
c = 20.130 (4) Å	μ = 0.09 mm^{-1}
β = 93.02 (4)º	*T* = 296 (2) K
V = 5876.3 (19) Å^3	Prism, colourless
Z = 16	0.30 × 0.20 × 0.15 mm

Data collection

Siemens P4 diffractometer	$R_{int} = 0.027$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.5°$
Monochromator: graphite	$\theta_{min} = 1.7°$
$T = 296(2)$ K	$h = 0 \rightarrow 22$
ω/2θ scans	$k = -1 \rightarrow 22$
Absorption correction: none	$l = -26 \rightarrow 26$
7430 measured reflections	3 standard reflections
6753 independent reflections	every 97 reflections
3465 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.067$	$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 4.0502P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.186$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.04$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
6753 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}
733 parameters	Extinction correction: none
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1A	0.6208 (3)	0.5326 (3)	0.3151 (2)	0.0548 (13)
N2A	0.6596 (3)	0.5108 (3)	0.2578 (3)	0.0632 (14)
C3A	0.7331 (4)	0.5083 (4)	0.2753 (4)	0.0702 (19)

C4A	0.7541 (4)	0.5291 (5)	0.3457 (4)	0.075 (2)
H4A1	0.7861	0.5760	0.3485	0.090*
H4A2	0.7819	0.4866	0.3685	0.090*
C5A	0.6731 (4)	0.5435 (4)	0.3758 (3)	0.0601 (16)
H5A	0.6699	0.5977	0.3913	0.072*
C6A	0.5445 (4)	0.5430 (4)	0.3117 (4)	0.0636 (17)
O1A	0.5076 (3)	0.5656 (3)	0.3592 (2)	0.0799 (14)
C7A	0.5008 (4)	0.5283 (5)	0.2465 (4)	0.073 (2)
O2A	0.4738 (5)	0.4655 (4)	0.2317 (3)	0.129 (3)
O3A	0.4921 (4)	0.5881 (4)	0.2090 (3)	0.114 (2)
C8A	0.4415 (8)	0.5814 (8)	0.1472 (5)	0.147 (5)
H8A1	0.4705	0.5984	0.1097	0.177*
H8A2	0.4271	0.5269	0.1401	0.177*
C9A	0.3776 (7)	0.6240 (12)	0.1503 (7)	0.218 (9)
H9A1	0.3569	0.6182	0.1933	0.327*
H9A2	0.3396	0.6067	0.1167	0.327*
H9A3	0.3900	0.6781	0.1429	0.327*
C10A	0.7932 (5)	0.4856 (6)	0.2279 (5)	0.101 (3)
H10A	0.7690	0.4811	0.1839	0.152*
H10B	0.8158	0.4361	0.2410	0.152*
H10C	0.8333	0.5248	0.2281	0.152*
C11A	0.6556 (3)	0.4882 (4)	0.4315 (3)	0.0557 (16)
C12A	0.6539 (4)	0.4063 (4)	0.4235 (3)	0.0540 (15)
C13A	0.6403 (4)	0.3569 (4)	0.4755 (4)	0.0688 (19)
H13A	0.6405	0.3029	0.4690	0.083*
C14A	0.6265 (4)	0.3866 (5)	0.5364 (4)	0.081 (2)
H14A	0.6162	0.3531	0.5714	0.098*
C15A	0.6279 (4)	0.4667 (6)	0.5461 (4)	0.082 (2)
H15A	0.6195	0.4869	0.5881	0.099*
C16A	0.6417 (4)	0.5175 (4)	0.4941 (4)	0.0707 (19)
H16A	0.6416	0.5713	0.5013	0.085*
O4A	0.6686 (3)	0.3799 (3)	0.3611 (2)	0.0742 (14)
H4A	0.6640	0.3321	0.3598	0.111*
N1B	0.0299 (3)	0.2709 (3)	0.3752 (2)	0.0547 (13)
N2B	−0.0143 (3)	0.2907 (3)	0.4300 (3)	0.0655 (14)
C3B	−0.0860 (4)	0.2896 (5)	0.4081 (4)	0.0701 (19)
C4B	−0.0990 (4)	0.2713 (5)	0.3368 (4)	0.075 (2)
H4B1	−0.1266	0.3137	0.3137	0.090*
H4B2	−0.1290	0.2235	0.3307	0.090*
C5B	−0.0172 (4)	0.2614 (4)	0.3116 (3)	0.0588 (16)
H5B	−0.0110	0.2080	0.2947	0.071*
C6B	0.1060 (4)	0.2630 (4)	0.3823 (3)	0.0610 (17)
O1B	0.1473 (3)	0.2434 (3)	0.3372 (2)	0.0810 (15)
C7B	0.1422 (4)	0.2772 (5)	0.4508 (4)	0.072 (2)
O2B	0.1578 (4)	0.2268 (3)	0.4906 (3)	0.0984 (18)
O3B	0.1592 (3)	0.3520 (3)	0.4600 (3)	0.0934 (17)
C8B	0.1969 (7)	0.3777 (6)	0.5239 (5)	0.125 (4)
H8B1	0.2250	0.3345	0.5454	0.150*
H8B2	0.2335	0.4198	0.5167	0.150*

C9B	0.1352 (10)	0.4046 (7)	0.5649 (6)	0.170 (6)
H9B1	0.1158	0.4541	0.5485	0.255*
H9B2	0.1554	0.4105	0.6100	0.255*
H9B3	0.0934	0.3670	0.5633	0.255*
C10B	−0.1490 (5)	0.3111 (6)	0.4520 (4)	0.100 (3)
H10D	−0.1267	0.3242	0.4953	0.150*
H10E	−0.1841	0.2676	0.4555	0.150*
H10F	−0.1771	0.3554	0.4337	0.150*
C11B	0.0054 (4)	0.3198 (4)	0.2594 (3)	0.0632 (17)
C12B	−0.0021 (4)	0.4012 (4)	0.2695 (4)	0.0672 (19)
C13B	0.0167 (5)	0.4515 (5)	0.2185 (4)	0.081 (2)
H13B	0.0122	0.5053	0.2242	0.097*
C14B	0.0417 (5)	0.4224 (6)	0.1598 (4)	0.093 (3)
H14B	0.0537	0.4571	0.1262	0.112*
C15B	0.0494 (5)	0.3438 (7)	0.1495 (4)	0.097 (3)
H15B	0.0661	0.3248	0.1093	0.116*
C16B	0.0321 (4)	0.2936 (5)	0.1996 (4)	0.076 (2)
H16B	0.0385	0.2400	0.1933	0.091*
O4B	−0.0273 (3)	0.4245 (3)	0.3280 (3)	0.0868 (16)
H4B	−0.0137	0.4700	0.3351	0.130*
N1C	0.1945 (3)	0.6096 (3)	0.3077 (3)	0.0547 (12)
N2C	0.2132 (3)	0.5688 (3)	0.2509 (3)	0.0612 (14)
C3C	0.2186 (4)	0.4959 (4)	0.2688 (4)	0.0672 (18)
C4C	0.2005 (4)	0.4792 (4)	0.3373 (4)	0.076 (2)
H4C1	0.2438	0.4524	0.3605	0.091*
H4C2	0.1542	0.4466	0.3386	0.091*
C5C	0.1867 (4)	0.5588 (4)	0.3684 (3)	0.0594 (16)
H5C	0.1329	0.5616	0.3827	0.071*
C6C	0.1797 (3)	0.6850 (4)	0.3058 (3)	0.0599 (17)
O1C	0.1572 (3)	0.7236 (3)	0.3536 (2)	0.0717 (13)
C7C	0.1940 (4)	0.7306 (4)	0.2412 (3)	0.0616 (17)
O2C	0.2570 (3)	0.7490 (4)	0.2253 (3)	0.0956 (18)
O3C	0.1275 (3)	0.7527 (3)	0.2119 (2)	0.0760 (13)
C8C	0.1321 (5)	0.8098 (5)	0.1566 (4)	0.087 (2)
H8C1	0.1551	0.8585	0.1731	0.105*
H8C2	0.1647	0.7890	0.1228	0.105*
C9C	0.0530 (5)	0.8240 (6)	0.1283 (5)	0.109 (3)
H9C1	0.0365	0.7802	0.1011	0.164*
H9C2	0.0527	0.8706	0.1016	0.164*
H9C3	0.0180	0.8304	0.1636	0.164*
C10C	0.2412 (6)	0.4351 (6)	0.2200 (5)	0.114 (3)
H10G	0.2381	0.4570	0.1760	0.171*
H10H	0.2062	0.3913	0.2217	0.171*
H10I	0.2936	0.4180	0.2309	0.171*
C11C	0.2427 (4)	0.5834 (4)	0.4250 (3)	0.0590 (16)
C12C	0.3232 (4)	0.5804 (4)	0.4209 (3)	0.0552 (15)
C13C	0.3722 (4)	0.5998 (4)	0.4749 (4)	0.074 (2)
H13C	0.4261	0.5974	0.4715	0.089*
C14C	0.3421 (5)	0.6226 (5)	0.5332 (4)	0.089 (2)

H14C	0.3757	0.6353	0.5694	0.107*
C15C	0.2625 (5)	0.6271 (5)	0.5392 (3)	0.085 (2)
H15C	0.2420	0.6426	0.5790	0.102*
C16C	0.2142 (4)	0.6082 (5)	0.4853 (4)	0.074 (2)
H16C	0.1605	0.6121	0.4889	0.088*
O4C	0.3502 (2)	0.5602 (3)	0.3606 (2)	0.0671 (12)
H4C	0.3976	0.5668	0.3612	0.101*
N1D	0.4548 (3)	0.2007 (3)	0.3763 (3)	0.0614 (14)
N2D	0.4413 (3)	0.2486 (4)	0.4311 (3)	0.0675 (15)
C3D	0.4410 (4)	0.3186 (5)	0.4079 (4)	0.079 (2)
C4D	0.4574 (4)	0.3284 (4)	0.3361 (4)	0.080 (2)
H4D1	0.5064	0.3555	0.3309	0.096*
H4D2	0.4156	0.3567	0.3122	0.096*
C5D	0.4616 (4)	0.2432 (4)	0.3125 (3)	0.0632 (18)
H5D	0.5138	0.2336	0.2964	0.076*
C6D	0.4642 (4)	0.1239 (4)	0.3856 (4)	0.0611 (17)
O1D	0.4816 (3)	0.0799 (3)	0.3401 (3)	0.0841 (15)
C7D	0.4557 (4)	0.0937 (4)	0.4561 (4)	0.0666 (18)
O2D	0.5106 (3)	0.0790 (4)	0.4930 (3)	0.1012 (19)
O3D	0.3816 (3)	0.0847 (4)	0.4680 (3)	0.0953 (18)
C8D	0.3637 (6)	0.0641 (7)	0.5346 (5)	0.115 (3)
H8D1	0.3203	0.0276	0.5338	0.138*
H8D2	0.4085	0.0393	0.5573	0.138*
C9D	0.3438 (8)	0.1338 (11)	0.5686 (6)	0.175 (7)
H9D1	0.3899	0.1650	0.5770	0.262*
H9D2	0.3218	0.1205	0.6100	0.262*
H9D3	0.3063	0.1631	0.5415	0.262*
C10D	0.4278 (6)	0.3889 (5)	0.4516 (4)	0.104 (3)
H10J	0.3784	0.4124	0.4389	0.156*
H10K	0.4688	0.4264	0.4465	0.156*
H10L	0.4279	0.3725	0.4973	0.156*
C11D	0.4019 (4)	0.2188 (4)	0.2593 (3)	0.0635 (18)
C12D	0.3230 (4)	0.2271 (4)	0.2667 (4)	0.0708 (19)
C13D	0.2697 (5)	0.2077 (5)	0.2147 (5)	0.090 (2)
H13D	0.2165	0.2153	0.2189	0.107*
C14D	0.2965 (6)	0.1771 (6)	0.1570 (4)	0.103 (3)
H14D	0.2608	0.1641	0.1223	0.123*
C15D	0.3757 (6)	0.1653 (6)	0.1492 (4)	0.106 (3)
H15D	0.3933	0.1438	0.1102	0.127*
C16D	0.4278 (5)	0.1863 (5)	0.2012 (4)	0.087 (2)
H16D	0.4810	0.1786	0.1971	0.104*
O4D	0.3009 (3)	0.2567 (4)	0.3255 (3)	0.0879 (16)
H4D	0.2556	0.2439	0.3312	0.132*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.055 (3)	0.050 (3)	0.059 (3)	0.010 (2)	−0.003 (2)	0.004 (2)

N2A	0.061 (3)	0.051 (3)	0.078 (4)	−0.005 (3)	0.003 (3)	−0.004 (3)
C3A	0.064 (4)	0.065 (5)	0.082 (5)	0.000 (4)	0.004 (4)	0.014 (4)
C4A	0.045 (4)	0.083 (5)	0.095 (6)	−0.016 (3)	−0.002 (3)	0.011 (4)
C5A	0.059 (4)	0.051 (4)	0.069 (4)	0.004 (3)	−0.009 (3)	−0.001 (3)
C6A	0.053 (4)	0.069 (4)	0.068 (5)	0.011 (3)	−0.005 (3)	0.007 (4)
O1A	0.050 (3)	0.113 (4)	0.076 (3)	0.015 (3)	−0.004 (2)	−0.009 (3)
C7A	0.070 (5)	0.064 (5)	0.085 (5)	0.008 (4)	−0.002 (4)	0.012 (4)
O2A	0.163 (6)	0.101 (5)	0.119 (5)	−0.048 (4)	−0.047 (4)	0.023 (4)
O3A	0.161 (6)	0.088 (4)	0.086 (4)	0.008 (4)	−0.059 (4)	0.004 (3)
C8A	0.181 (12)	0.164 (11)	0.088 (7)	0.029 (9)	−0.069 (7)	0.010 (7)
C9A	0.114 (9)	0.38 (3)	0.151 (11)	0.120 (13)	−0.047 (8)	−0.041 (14)
C10A	0.072 (5)	0.120 (7)	0.113 (7)	0.020 (5)	0.028 (5)	0.012 (6)
C11A	0.046 (3)	0.062 (4)	0.058 (4)	0.015 (3)	−0.009 (3)	−0.006 (3)
C12A	0.054 (4)	0.058 (4)	0.049 (4)	0.009 (3)	−0.002 (3)	−0.004 (3)
C13A	0.071 (5)	0.058 (4)	0.077 (5)	0.014 (3)	0.003 (4)	0.010 (4)
C14A	0.070 (5)	0.096 (7)	0.078 (6)	0.020 (4)	0.006 (4)	0.011 (5)
C15A	0.078 (5)	0.115 (7)	0.053 (4)	0.020 (5)	0.006 (4)	−0.010 (4)
C16A	0.070 (4)	0.066 (4)	0.075 (5)	0.005 (3)	−0.007 (3)	−0.009 (4)
O4A	0.092 (4)	0.063 (3)	0.069 (3)	0.011 (3)	0.011 (3)	0.005 (3)
N1B	0.049 (3)	0.055 (3)	0.059 (3)	0.002 (2)	−0.009 (2)	0.007 (2)
N2B	0.065 (4)	0.072 (4)	0.060 (3)	0.008 (3)	0.002 (3)	0.006 (3)
C3B	0.049 (4)	0.082 (5)	0.080 (5)	0.008 (3)	0.009 (3)	0.024 (4)
C4B	0.058 (4)	0.086 (5)	0.080 (5)	−0.015 (4)	−0.003 (4)	0.013 (4)
C5B	0.062 (4)	0.066 (4)	0.046 (3)	−0.013 (3)	−0.018 (3)	−0.007 (3)
C6B	0.050 (4)	0.077 (5)	0.055 (4)	0.001 (3)	−0.003 (3)	0.004 (4)
O1B	0.062 (3)	0.109 (4)	0.072 (3)	0.008 (3)	0.004 (3)	−0.011 (3)
C7B	0.070 (5)	0.074 (5)	0.072 (5)	0.010 (4)	−0.009 (4)	0.006 (4)
O2B	0.114 (4)	0.084 (4)	0.093 (4)	0.004 (3)	−0.033 (3)	0.016 (3)
O3B	0.116 (4)	0.067 (3)	0.093 (4)	−0.015 (3)	−0.039 (3)	0.003 (3)
C8B	0.174 (10)	0.079 (6)	0.114 (8)	−0.029 (6)	−0.070 (7)	−0.005 (6)
C9B	0.266 (18)	0.110 (9)	0.130 (10)	0.045 (10)	−0.030 (11)	−0.031 (8)
C10B	0.084 (6)	0.122 (8)	0.097 (6)	0.015 (5)	0.024 (5)	0.021 (5)
C11B	0.060 (4)	0.072 (5)	0.056 (4)	−0.011 (3)	−0.012 (3)	0.001 (4)
C12B	0.063 (4)	0.068 (5)	0.069 (5)	−0.009 (4)	−0.015 (4)	0.006 (4)
C13B	0.090 (6)	0.077 (5)	0.075 (5)	−0.011 (4)	−0.013 (4)	0.014 (4)
C14B	0.098 (6)	0.117 (8)	0.064 (5)	−0.027 (5)	−0.004 (4)	0.022 (5)
C15B	0.104 (6)	0.127 (9)	0.061 (5)	−0.023 (6)	0.013 (4)	−0.006 (5)
C16B	0.090 (5)	0.066 (5)	0.071 (5)	−0.018 (4)	−0.010 (4)	−0.007 (4)
O4B	0.108 (4)	0.074 (4)	0.079 (4)	−0.004 (3)	0.008 (3)	−0.008 (3)
N1C	0.050 (3)	0.056 (3)	0.058 (3)	−0.006 (2)	−0.001 (2)	0.000 (3)
N2C	0.065 (3)	0.059 (3)	0.059 (3)	0.008 (3)	−0.011 (3)	−0.007 (3)
C3C	0.059 (4)	0.058 (4)	0.083 (5)	0.013 (3)	−0.011 (3)	−0.003 (4)
C4C	0.072 (4)	0.069 (5)	0.084 (5)	−0.022 (4)	−0.019 (4)	−0.006 (4)
C5C	0.059 (4)	0.057 (4)	0.062 (4)	−0.009 (3)	−0.005 (3)	0.009 (3)
C6C	0.048 (3)	0.071 (5)	0.059 (4)	0.014 (3)	−0.010 (3)	0.000 (4)
O1C	0.086 (3)	0.066 (3)	0.063 (3)	0.016 (3)	0.004 (2)	−0.002 (3)
C7C	0.051 (4)	0.066 (4)	0.066 (4)	0.010 (3)	−0.011 (3)	0.003 (3)
O2C	0.056 (3)	0.128 (5)	0.102 (4)	−0.013 (3)	−0.008 (3)	0.034 (4)
O3C	0.062 (3)	0.085 (3)	0.080 (3)	0.004 (2)	0.003 (2)	0.031 (3)

C8C	0.101 (6)	0.098 (6)	0.062 (4)	−0.021 (5)	−0.003 (4)	0.032 (4)
C9C	0.090 (6)	0.113 (7)	0.122 (7)	0.019 (5)	−0.015 (5)	0.056 (6)
C10C	0.120 (7)	0.095 (7)	0.122 (7)	0.025 (6)	−0.033 (6)	−0.044 (6)
C11C	0.059 (4)	0.054 (4)	0.064 (4)	0.006 (3)	0.001 (3)	0.007 (3)
C12C	0.057 (4)	0.051 (4)	0.057 (4)	0.007 (3)	0.002 (3)	0.001 (3)
C13C	0.065 (4)	0.066 (4)	0.089 (6)	0.007 (4)	−0.014 (4)	−0.004 (4)
C14C	0.095 (6)	0.104 (6)	0.066 (5)	0.017 (5)	−0.019 (4)	−0.017 (5)
C15C	0.105 (6)	0.108 (6)	0.043 (4)	0.025 (5)	0.003 (4)	0.002 (4)
C16C	0.072 (4)	0.091 (5)	0.059 (4)	0.009 (4)	0.016 (4)	0.012 (4)
O4C	0.052 (2)	0.077 (3)	0.072 (3)	0.007 (2)	−0.003 (2)	−0.007 (3)
N1D	0.061 (3)	0.070 (4)	0.053 (3)	0.004 (3)	−0.001 (3)	0.010 (3)
N2D	0.065 (3)	0.080 (4)	0.057 (3)	0.004 (3)	−0.007 (3)	0.001 (3)
C3D	0.073 (5)	0.091 (6)	0.073 (5)	0.007 (4)	−0.020 (4)	0.007 (5)
C4D	0.077 (5)	0.058 (4)	0.103 (6)	−0.020 (4)	−0.025 (4)	0.006 (4)
C5D	0.047 (3)	0.089 (5)	0.053 (4)	−0.006 (3)	0.001 (3)	0.021 (4)
C6D	0.047 (3)	0.065 (4)	0.071 (5)	−0.010 (3)	−0.002 (3)	0.017 (4)
O1D	0.103 (4)	0.075 (4)	0.074 (3)	0.012 (3)	0.007 (3)	−0.002 (3)
C7D	0.069 (5)	0.065 (4)	0.065 (4)	0.003 (3)	−0.005 (4)	0.011 (3)
O2D	0.074 (3)	0.138 (5)	0.089 (4)	0.008 (3)	−0.023 (3)	0.032 (4)
O3D	0.071 (3)	0.139 (5)	0.075 (3)	−0.024 (3)	−0.007 (3)	0.035 (3)
C8D	0.100 (7)	0.158 (10)	0.089 (7)	−0.009 (7)	0.016 (5)	0.047 (7)
C9D	0.135 (10)	0.29 (2)	0.100 (9)	0.023 (12)	0.034 (7)	0.054 (11)
C10D	0.121 (7)	0.085 (6)	0.105 (7)	0.024 (5)	−0.023 (5)	−0.032 (5)
C11D	0.051 (4)	0.079 (5)	0.060 (4)	−0.015 (3)	−0.004 (3)	0.025 (4)
C12D	0.064 (5)	0.076 (5)	0.071 (5)	0.003 (4)	−0.013 (4)	0.012 (4)
C13D	0.075 (5)	0.096 (6)	0.094 (6)	−0.006 (4)	−0.023 (5)	0.005 (5)
C14D	0.114 (8)	0.124 (8)	0.066 (6)	−0.018 (6)	−0.039 (5)	0.007 (5)
C15D	0.108 (8)	0.142 (9)	0.066 (5)	−0.022 (6)	−0.009 (5)	0.005 (5)
C16D	0.097 (6)	0.096 (6)	0.068 (5)	−0.014 (5)	0.001 (4)	0.013 (4)
O4D	0.053 (3)	0.114 (5)	0.095 (4)	0.000 (3)	−0.003 (3)	−0.010 (3)

Geometric parameters (Å, °)

N1A—C6A	1.318 (8)	N1C—C6C	1.311 (8)
N1A—N2A	1.413 (7)	N1C—N2C	1.390 (7)
N1A—C5A	1.488 (8)	N1C—C5C	1.510 (8)
N2A—C3A	1.290 (9)	N2C—C3C	1.297 (8)
C3A—C10A	1.492 (11)	C3C—C4C	1.456 (10)
C3A—C4A	1.487 (11)	C3C—C10C	1.493 (11)
C4A—C5A	1.561 (9)	C4C—C5C	1.520 (10)
C4A—H4A1	0.9700	C4C—H4C1	0.9700
C4A—H4A2	0.9700	C4C—H4C2	0.9700
C5A—C11A	1.507 (9)	C5C—C11C	1.509 (9)
C5A—H5A	0.9800	C5C—H5C	0.9800
C6A—O1A	1.236 (8)	C6C—O1C	1.245 (8)
C6A—C7A	1.497 (10)	C6C—C7C	1.546 (10)
C7A—O2A	1.198 (9)	C7C—O2C	1.183 (8)
C7A—O3A	1.273 (9)	C7C—O3C	1.311 (7)
O3A—C8A	1.482 (10)	O3C—C8C	1.484 (8)

C8A—C9A	1.318 (15)	C8C—C9C	1.462 (11)
C8A—H8A1	0.9700	C8C—H8C1	0.9700
C8A—H8A2	0.9700	C8C—H8C2	0.9700
C9A—H9A1	0.9600	C9C—H9C1	0.9600
C9A—H9A2	0.9600	C9C—H9C2	0.9600
C9A—H9A3	0.9600	C9C—H9C3	0.9600
C10A—H10A	0.9600	C10C—H10G	0.9600
C10A—H10B	0.9600	C10C—H10H	0.9600
C10A—H10C	0.9600	C10C—H10I	0.9600
C11A—C16A	1.389 (9)	C11C—C12C	1.386 (8)
C11A—C12A	1.407 (9)	C11C—C16C	1.398 (9)
C12A—O4A	1.370 (7)	C12C—O4C	1.366 (7)
C12A—C13A	1.373 (9)	C12C—C13C	1.378 (10)
C13A—C14A	1.359 (10)	C13C—C14C	1.364 (11)
C13A—H13A	0.9300	C13C—H13C	0.9300
C14A—C15A	1.382 (12)	C14C—C15C	1.378 (12)
C14A—H14A	0.9300	C14C—H14C	0.9300
C15A—C16A	1.389 (11)	C15C—C16C	1.368 (10)
C15A—H15A	0.9300	C15C—H15C	0.9300
C16A—H16A	0.9300	C16C—H16C	0.9300
O4A—H4A	0.8200	O4C—H4C	0.8200
N1B—C6B	1.312 (8)	N1D—C6D	1.333 (9)
N1B—N2B	1.412 (7)	N1D—N2D	1.401 (8)
N1B—C5B	1.486 (7)	N1D—C5D	1.485 (8)
N2B—C3B	1.284 (8)	N2D—C3D	1.283 (10)
C3B—C4B	1.473 (10)	C3D—C4D	1.497 (11)
C3B—C10B	1.477 (10)	C3D—C10D	1.511 (11)
C4B—C5B	1.526 (10)	C4D—C5D	1.532 (10)
C4B—H4B1	0.9700	C4D—H4D1	0.9700
C4B—H4B2	0.9700	C4D—H4D2	0.9700
C5B—C11B	1.512 (9)	C5D—C11D	1.500 (9)
C5B—H5B	0.9800	C5D—H5D	0.9800
C6B—O1B	1.226 (7)	C6D—O1D	1.233 (8)
C6B—C7B	1.502 (10)	C6D—C7D	1.524 (10)
C7B—O2B	1.195 (8)	C7D—O2D	1.194 (8)
C7B—O3B	1.320 (9)	C7D—O3D	1.314 (8)
O3B—C8B	1.475 (10)	O3D—C8D	1.435 (10)
C8B—C9B	1.449 (17)	C8D—C9D	1.422 (17)
C8B—H8B1	0.9700	C8D—H8D1	0.9700
C8B—H8B2	0.9700	C8D—H8D2	0.9700
C9B—H9B1	0.9600	C9D—H9D1	0.9600
C9B—H9B2	0.9600	C9D—H9D2	0.9600
C9B—H9B3	0.9600	C9D—H9D3	0.9600
C10B—H10D	0.9600	C10D—H10J	0.9600
C10B—H10E	0.9600	C10D—H10K	0.9600
C10B—H10F	0.9600	C10D—H10L	0.9600
C11B—C16B	1.385 (10)	C11D—C12D	1.374 (9)
C11B—C12B	1.410 (10)	C11D—C16D	1.388 (10)
C12B—O4B	1.335 (9)	C12D—O4D	1.358 (9)

C12B—C13B	1.389 (10)	C12D—C13D	1.393 (10)
C13B—C14B	1.371 (12)	C13D—C14D	1.374 (12)
C13B—H13B	0.9300	C13D—H13D	0.9300
C14B—C15B	1.366 (13)	C14D—C15D	1.388 (13)
C14B—H14B	0.9300	C14D—H14D	0.9300
C15B—C16B	1.367 (11)	C15D—C16D	1.387 (11)
C15B—H15B	0.9300	C15D—H15D	0.9300
C16B—H16B	0.9300	C16D—H16D	0.9300
O4B—H4B	0.8200	O4D—H4D	0.8200
C6A—N1A—N2A	120.1 (5)	C6C—N1C—N2C	121.3 (6)
C6A—N1A—C5A	125.4 (5)	C6C—N1C—C5C	124.2 (6)
N2A—N1A—C5A	114.5 (5)	N2C—N1C—C5C	114.4 (5)
C3A—N2A—N1A	106.2 (6)	C3C—N2C—N1C	105.5 (6)
N2A—C3A—C10A	122.0 (8)	N2C—C3C—C4C	115.8 (7)
N2A—C3A—C4A	115.9 (7)	N2C—C3C—C10C	120.0 (8)
C10A—C3A—C4A	122.2 (7)	C4C—C3C—C10C	124.2 (8)
C3A—C4A—C5A	103.4 (6)	C3C—C4C—C5C	105.1 (6)
C3A—C4A—H4A1	111.1	C3C—C4C—H4C1	110.7
C5A—C4A—H4A1	111.1	C5C—C4C—H4C1	110.7
C3A—C4A—H4A2	111.1	C3C—C4C—H4C2	110.7
C5A—C4A—H4A2	111.1	C5C—C4C—H4C2	110.7
H4A1—C4A—H4A2	109.0	H4C1—C4C—H4C2	108.8
N1A—C5A—C11A	113.4 (5)	N1C—C5C—C11C	111.7 (5)
N1A—C5A—C4A	99.8 (5)	N1C—C5C—C4C	99.0 (5)
C11A—C5A—C4A	114.0 (5)	C11C—C5C—C4C	116.9 (5)
N1A—C5A—H5A	109.7	N1C—C5C—H5C	109.6
C11A—C5A—H5A	109.7	C11C—C5C—H5C	109.6
C4A—C5A—H5A	109.7	C4C—C5C—H5C	109.6
O1A—C6A—N1A	123.2 (6)	O1C—C6C—N1C	124.3 (6)
O1A—C6A—C7A	118.6 (6)	O1C—C6C—C7C	116.9 (6)
N1A—C6A—C7A	118.2 (7)	N1C—C6C—C7C	118.7 (6)
O2A—C7A—O3A	122.5 (8)	O2C—C7C—O3C	125.8 (7)
O2A—C7A—C6A	122.5 (7)	O2C—C7C—C6C	123.2 (6)
O3A—C7A—C6A	115.0 (7)	O3C—C7C—C6C	110.6 (6)
C7A—O3A—C8A	118.7 (8)	C7C—O3C—C8C	116.5 (5)
C9A—C8A—O3A	111.7 (11)	C9C—C8C—O3C	108.4 (6)
C9A—C8A—H8A1	109.3	C9C—C8C—H8C1	110.0
O3A—C8A—H8A1	109.3	O3C—C8C—H8C1	110.0
C9A—C8A—H8A2	109.3	C9C—C8C—H8C2	110.0
O3A—C8A—H8A2	109.3	O3C—C8C—H8C2	110.0
H8A1—C8A—H8A2	107.9	H8C1—C8C—H8C2	108.4
C8A—C9A—H9A1	109.5	C8C—C9C—H9C1	109.5
C8A—C9A—H9A2	109.5	C8C—C9C—H9C2	109.5
H9A1—C9A—H9A2	109.5	H9C1—C9C—H9C2	109.5
C8A—C9A—H9A3	109.5	C8C—C9C—H9C3	109.5
H9A1—C9A—H9A3	109.5	H9C1—C9C—H9C3	109.5
H9A2—C9A—H9A3	109.5	H9C2—C9C—H9C3	109.5
C3A—C10A—H10A	109.5	C3C—C10C—H10G	109.5
C3A—C10A—H10B	109.5	C3C—C10C—H10H	109.5

H10A—C10A—H10B	109.5	H10G—C10C—H10H	109.5
C3A—C10A—H10C	109.5	C3C—C10C—H10I	109.5
H10A—C10A—H10C	109.5	H10G—C10C—H10I	109.5
H10B—C10A—H10C	109.5	H10H—C10C—H10I	109.5
C16A—C11A—C12A	117.2 (6)	C12C—C11C—C16C	117.1 (6)
C16A—C11A—C5A	120.0 (6)	C12C—C11C—C5C	122.7 (6)
C12A—C11A—C5A	122.7 (6)	C16C—C11C—C5C	120.1 (6)
O4A—C12A—C13A	122.9 (6)	O4C—C12C—C13C	122.7 (6)
O4A—C12A—C11A	115.3 (6)	O4C—C12C—C11C	116.5 (6)
C13A—C12A—C11A	121.8 (6)	C13C—C12C—C11C	120.8 (6)
C14A—C13A—C12A	120.2 (7)	C14C—C13C—C12C	120.3 (7)
C14A—C13A—H13A	119.9	C14C—C13C—H13C	119.9
C12A—C13A—H13A	119.9	C12C—C13C—H13C	119.9
C13A—C14A—C15A	119.6 (8)	C13C—C14C—C15C	120.8 (7)
C13A—C14A—H14A	120.2	C13C—C14C—H14C	119.6
C15A—C14A—H14A	120.2	C15C—C14C—H14C	119.6
C14A—C15A—C16A	120.8 (7)	C16C—C15C—C14C	118.5 (7)
C14A—C15A—H15A	119.6	C16C—C15C—H15C	120.7
C16A—C15A—H15A	119.6	C14C—C15C—H15C	120.7
C11A—C16A—C15A	120.3 (7)	C15C—C16C—C11C	122.4 (7)
C11A—C16A—H16A	119.9	C15C—C16C—H16C	118.8
C15A—C16A—H16A	119.9	C11C—C16C—H16C	118.8
C12A—O4A—H4A	109.5	C12C—O4C—H4C	109.5
C6B—N1B—N2B	120.7 (5)	C6D—N1D—N2D	119.2 (6)
C6B—N1B—C5B	125.0 (6)	C6D—N1D—C5D	125.8 (6)
N2B—N1B—C5B	114.3 (5)	N2D—N1D—C5D	114.9 (6)
C3B—N2B—N1B	105.7 (5)	C3D—N2D—N1D	104.7 (6)
N2B—C3B—C4B	115.5 (6)	N2D—C3D—C4D	117.2 (7)
N2B—C3B—C10B	120.4 (7)	N2D—C3D—C10D	121.7 (7)
C4B—C3B—C10B	123.9 (7)	C4D—C3D—C10D	121.0 (8)
C3B—C4B—C5B	104.7 (6)	C3D—C4D—C5D	102.0 (6)
C3B—C4B—H4B1	110.8	C3D—C4D—H4D1	111.4
C5B—C4B—H4B1	110.8	C5D—C4D—H4D1	111.4
C3B—C4B—H4B2	110.8	C3D—C4D—H4D2	111.4
C5B—C4B—H4B2	110.8	C5D—C4D—H4D2	111.4
H4B1—C4B—H4B2	108.9	H4D1—C4D—H4D2	109.2
N1B—C5B—C11B	112.3 (5)	N1D—C5D—C11D	113.6 (5)
N1B—C5B—C4B	99.6 (5)	N1D—C5D—C4D	100.8 (6)
C11B—C5B—C4B	115.5 (6)	C11D—C5D—C4D	116.2 (6)
N1B—C5B—H5B	109.7	N1D—C5D—H5D	108.6
C11B—C5B—H5B	109.7	C11D—C5D—H5D	108.6
C4B—C5B—H5B	109.7	C4D—C5D—H5D	108.6
O1B—C6B—N1B	123.8 (6)	O1D—C6D—N1D	121.9 (6)
O1B—C6B—C7B	119.8 (6)	O1D—C6D—C7D	121.6 (7)
N1B—C6B—C7B	116.4 (6)	N1D—C6D—C7D	116.5 (7)
O2B—C7B—O3B	124.2 (7)	O2D—C7D—O3D	126.7 (7)
O2B—C7B—C6B	124.5 (8)	O2D—C7D—C6D	122.7 (7)
O3B—C7B—C6B	111.2 (7)	O3D—C7D—C6D	110.6 (6)
C7B—O3B—C8B	119.5 (6)	C7D—O3D—C8D	117.2 (6)

C9B—C8B—O3B	107.0 (10)	C9D—C8D—O3D	108.2 (9)
C9B—C8B—H8B1	110.3	C9D—C8D—H8D1	110.1
O3B—C8B—H8B1	110.3	O3D—C8D—H8D1	110.1
C9B—C8B—H8B2	110.3	C9D—C8D—H8D2	110.1
O3B—C8B—H8B2	110.3	O3D—C8D—H8D2	110.1
H8B1—C8B—H8B2	108.6	H8D1—C8D—H8D2	108.4
C8B—C9B—H9B1	109.5	C8D—C9D—H9D1	109.5
C8B—C9B—H9B2	109.5	C8D—C9D—H9D2	109.5
H9B1—C9B—H9B2	109.5	H9D1—C9D—H9D2	109.5
C8B—C9B—H9B3	109.5	C8D—C9D—H9D3	109.5
H9B1—C9B—H9B3	109.5	H9D1—C9D—H9D3	109.5
H9B2—C9B—H9B3	109.5	H9D2—C9D—H9D3	109.5
C3B—C10B—H10D	109.5	C3D—C10D—H10J	109.5
C3B—C10B—H10E	109.5	C3D—C10D—H10K	109.5
H10D—C10B—H10E	109.5	H10J—C10D—H10K	109.5
C3B—C10B—H10F	109.5	C3D—C10D—H10L	109.5
H10D—C10B—H10F	109.5	H10J—C10D—H10L	109.5
H10E—C10B—H10F	109.5	H10K—C10D—H10L	109.5
C16B—C11B—C12B	118.7 (7)	C12D—C11D—C16D	119.5 (7)
C16B—C11B—C5B	119.9 (7)	C12D—C11D—C5D	122.2 (7)
C12B—C11B—C5B	121.4 (6)	C16D—C11D—C5D	118.4 (6)
O4B—C12B—C13B	124.5 (7)	O4D—C12D—C11D	116.9 (6)
O4B—C12B—C11B	117.2 (7)	O4D—C12D—C13D	122.8 (7)
C13B—C12B—C11B	118.4 (8)	C11D—C12D—C13D	120.2 (8)
C14B—C13B—C12B	120.6 (8)	C14D—C13D—C12D	119.5 (8)
C14B—C13B—H13B	119.7	C14D—C13D—H13D	120.3
C12B—C13B—H13B	119.7	C12D—C13D—H13D	120.3
C15B—C14B—C13B	121.6 (8)	C13D—C14D—C15D	121.5 (8)
C15B—C14B—H14B	119.2	C13D—C14D—H14D	119.3
C13B—C14B—H14B	119.2	C15D—C14D—H14D	119.3
C14B—C15B—C16B	118.5 (8)	C16D—C15D—C14D	118.1 (9)
C14B—C15B—H15B	120.8	C16D—C15D—H15D	121.0
C16B—C15B—H15B	120.8	C14D—C15D—H15D	121.0
C15B—C16B—C11B	122.2 (8)	C11D—C16D—C15D	121.2 (9)
C15B—C16B—H16B	118.9	C11D—C16D—H16D	119.4
C11B—C16B—H16B	118.9	C15D—C16D—H16D	119.4
C12B—O4B—H4B	109.5	C12D—O4D—H4D	109.5
C6A—N1A—N2A—C3A	177.3 (6)	C6C—N1C—N2C—C3C	−175.5 (6)
C5A—N1A—N2A—C3A	−1.5 (7)	C5C—N1C—N2C—C3C	−0.3 (7)
N1A—N2A—C3A—C10A	178.8 (6)	N1C—N2C—C3C—C4C	3.1 (7)
N1A—N2A—C3A—C4A	−1.0 (8)	N1C—N2C—C3C—C10C	−177.8 (6)
N2A—C3A—C4A—C5A	2.9 (8)	N2C—C3C—C4C—C5C	−4.7 (8)
C10A—C3A—C4A—C5A	−176.9 (7)	C10C—C3C—C4C—C5C	176.2 (7)
C6A—N1A—C5A—C11A	62.7 (8)	C6C—N1C—C5C—C11C	−63.4 (8)
N2A—N1A—C5A—C11A	−118.6 (6)	N2C—N1C—C5C—C11C	121.5 (6)
C6A—N1A—C5A—C4A	−175.7 (6)	C6C—N1C—C5C—C4C	172.8 (6)
N2A—N1A—C5A—C4A	3.1 (7)	N2C—N1C—C5C—C4C	−2.3 (6)
C3A—C4A—C5A—N1A	−3.2 (7)	C3C—C4C—C5C—N1C	3.7 (6)
C3A—C4A—C5A—C11A	118.0 (6)	C3C—C4C—C5C—C11C	−116.3 (6)

N2A—N1A—C6A—O1A	−176.6 (6)	N2C—N1C—C6C—O1C	174.5 (6)
C5A—N1A—C6A—O1A	2.1 (11)	C5C—N1C—C6C—O1C	−0.3 (10)
N2A—N1A—C6A—C7A	2.1 (9)	N2C—N1C—C6C—C7C	−8.1 (8)
C5A—N1A—C6A—C7A	−179.1 (6)	C5C—N1C—C6C—C7C	177.1 (5)
O1A—C6A—C7A—O2A	−90.6 (11)	O1C—C6C—C7C—O2C	101.3 (8)
N1A—C6A—C7A—O2A	90.6 (10)	N1C—C6C—C7C—O2C	−76.3 (9)
O1A—C6A—C7A—O3A	87.0 (9)	O1C—C6C—C7C—O3C	−71.8 (8)
N1A—C6A—C7A—O3A	−91.7 (9)	N1C—C6C—C7C—O3C	110.6 (6)
O2A—C7A—O3A—C8A	4.9 (14)	O2C—C7C—O3C—C8C	−4.1 (11)
C6A—C7A—O3A—C8A	−172.7 (9)	C6C—C7C—O3C—C8C	168.7 (6)
C7A—O3A—C8A—C9A	112.3 (15)	C7C—O3C—C8C—C9C	177.1 (7)
N1A—C5A—C11A—C16A	−126.0 (6)	N1C—C5C—C11C—C12C	−62.8 (8)
C4A—C5A—C11A—C16A	120.7 (7)	C4C—C5C—C11C—C12C	50.3 (8)
N1A—C5A—C11A—C12A	55.3 (8)	N1C—C5C—C11C—C16C	119.3 (7)
C4A—C5A—C11A—C12A	−58.0 (8)	C4C—C5C—C11C—C16C	−127.7 (7)
C16A—C11A—C12A—O4A	−179.4 (6)	C16C—C11C—C12C—O4C	−176.5 (6)
C5A—C11A—C12A—O4A	−0.7 (8)	C5C—C11C—C12C—O4C	5.5 (9)
C16A—C11A—C12A—C13A	−0.9 (9)	C16C—C11C—C12C—C13C	1.2 (9)
C5A—C11A—C12A—C13A	177.8 (6)	C5C—C11C—C12C—C13C	−176.8 (6)
O4A—C12A—C13A—C14A	179.6 (6)	O4C—C12C—C13C—C14C	177.3 (7)
C11A—C12A—C13A—C14A	1.1 (10)	C11C—C12C—C13C—C14C	−0.2 (11)
C12A—C13A—C14A—C15A	−1.3 (11)	C12C—C13C—C14C—C15C	−0.4 (13)
C13A—C14A—C15A—C16A	1.3 (11)	C13C—C14C—C15C—C16C	0.0 (13)
C12A—C11A—C16A—C15A	0.8 (9)	C14C—C15C—C16C—C11C	1.1 (13)
C5A—C11A—C16A—C15A	−177.9 (6)	C12C—C11C—C16C—C15C	−1.7 (11)
C14A—C15A—C16A—C11A	−1.1 (11)	C5C—C11C—C16C—C15C	176.4 (7)
C6B—N1B—N2B—C3B	−176.4 (6)	C6D—N1D—N2D—C3D	174.9 (6)
C5B—N1B—N2B—C3B	4.2 (7)	C5D—N1D—N2D—C3D	−1.7 (7)
N1B—N2B—C3B—C4B	−1.5 (8)	N1D—N2D—C3D—C4D	−2.5 (8)
N1B—N2B—C3B—C10B	−177.4 (7)	N1D—N2D—C3D—C10D	179.7 (6)
N2B—C3B—C4B—C5B	−1.5 (9)	N2D—C3D—C4D—C5D	5.4 (8)
C10B—C3B—C4B—C5B	174.2 (7)	C10D—C3D—C4D—C5D	−176.9 (7)
C6B—N1B—C5B—C11B	−61.4 (8)	C6D—N1D—C5D—C11D	63.3 (8)
N2B—N1B—C5B—C11B	118.0 (6)	N2D—N1D—C5D—C11D	−120.3 (6)
C6B—N1B—C5B—C4B	175.8 (6)	C6D—N1D—C5D—C4D	−171.6 (6)
N2B—N1B—C5B—C4B	−4.8 (7)	N2D—N1D—C5D—C4D	4.7 (6)
C3B—C4B—C5B—N1B	3.5 (7)	C3D—C4D—C5D—N1D	−5.3 (6)
C3B—C4B—C5B—C11B	−117.0 (6)	C3D—C4D—C5D—C11D	117.9 (6)
N2B—N1B—C6B—O1B	177.5 (6)	N2D—N1D—C6D—O1D	−174.9 (6)
C5B—N1B—C6B—O1B	−3.2 (10)	C5D—N1D—C6D—O1D	1.4 (10)
N2B—N1B—C6B—C7B	−0.9 (9)	N2D—N1D—C6D—C7D	2.7 (8)
C5B—N1B—C6B—C7B	178.5 (6)	C5D—N1D—C6D—C7D	179.0 (5)
O1B—C6B—C7B—O2B	−82.0 (10)	O1D—C6D—C7D—O2D	77.0 (10)
N1B—C6B—C7B—O2B	96.4 (9)	N1D—C6D—C7D—O2D	−100.6 (8)
O1B—C6B—C7B—O3B	94.0 (8)	O1D—C6D—C7D—O3D	−101.2 (8)
N1B—C6B—C7B—O3B	−87.6 (8)	N1D—C6D—C7D—O3D	81.2 (8)
O2B—C7B—O3B—C8B	−2.7 (13)	O2D—C7D—O3D—C8D	8.0 (13)
C6B—C7B—O3B—C8B	−178.7 (8)	C6D—C7D—O3D—C8D	−173.9 (8)
C7B—O3B—C8B—C9B	−95.6 (10)	C7D—O3D—C8D—C9D	97.2 (11)

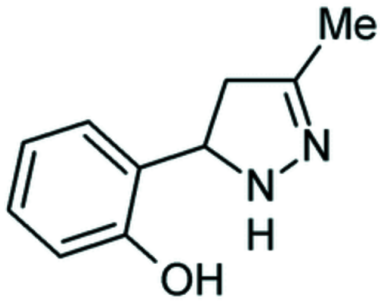
N1B—C5B—C11B—C16B	120.4 (7)	N1D—C5D—C11D—C12D	60.7 (9)
C4B—C5B—C11B—C16B	−126.3 (7)	C4D—C5D—C11D—C12D	−55.6 (9)
N1B—C5B—C11B—C12B	−61.2 (8)	N1D—C5D—C11D—C16D	−118.7 (7)
C4B—C5B—C11B—C12B	52.1 (8)	C4D—C5D—C11D—C16D	125.0 (8)
C16B—C11B—C12B—O4B	−179.3 (6)	C16D—C11D—C12D—O4D	177.8 (7)
C5B—C11B—C12B—O4B	2.3 (9)	C5D—C11D—C12D—O4D	−1.6 (10)
C16B—C11B—C12B—C13B	0.9 (10)	C16D—C11D—C12D—C13D	−4.1 (11)
C5B—C11B—C12B—C13B	−177.4 (6)	C5D—C11D—C12D—C13D	176.5 (7)
O4B—C12B—C13B—C14B	−179.6 (7)	O4D—C12D—C13D—C14D	−179.2 (8)
C11B—C12B—C13B—C14B	0.1 (11)	C11D—C12D—C13D—C14D	2.7 (12)
C12B—C13B—C14B—C15B	−0.3 (13)	C12D—C13D—C14D—C15D	−0.2 (14)
C13B—C14B—C15B—C16B	−0.5 (14)	C13D—C14D—C15D—C16D	−1.0 (15)
C14B—C15B—C16B—C11B	1.6 (13)	C12D—C11D—C16D—C15D	3.0 (12)
C12B—C11B—C16B—C15B	−1.8 (11)	C5D—C11D—C16D—C15D	−177.6 (8)
C5B—C11B—C16B—C15B	176.6 (7)	C14D—C15D—C16D—C11D	−0.4 (13)

Hydrogen-bond geometry (Å, °)

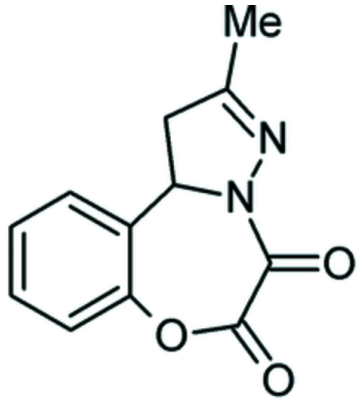
D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4A—H4A···O1C^{i}	0.82	1.86	2.678 (7)	177
O4B—H4B···O1D^{ii}	0.82	1.88	2.665 (7)	160
O4C—H4C···O1A	0.82	1.89	2.700 (6)	171
O4D—H4D···O1B	0.82	1.87	2.664 (7)	164

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

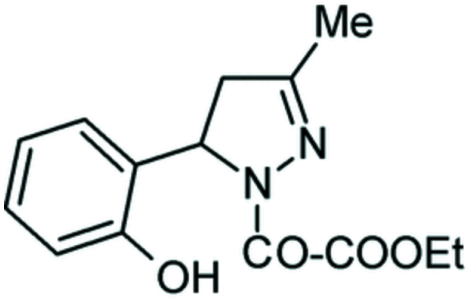
Fig. 1



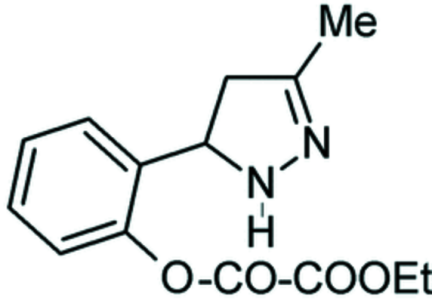
(I)



(II)

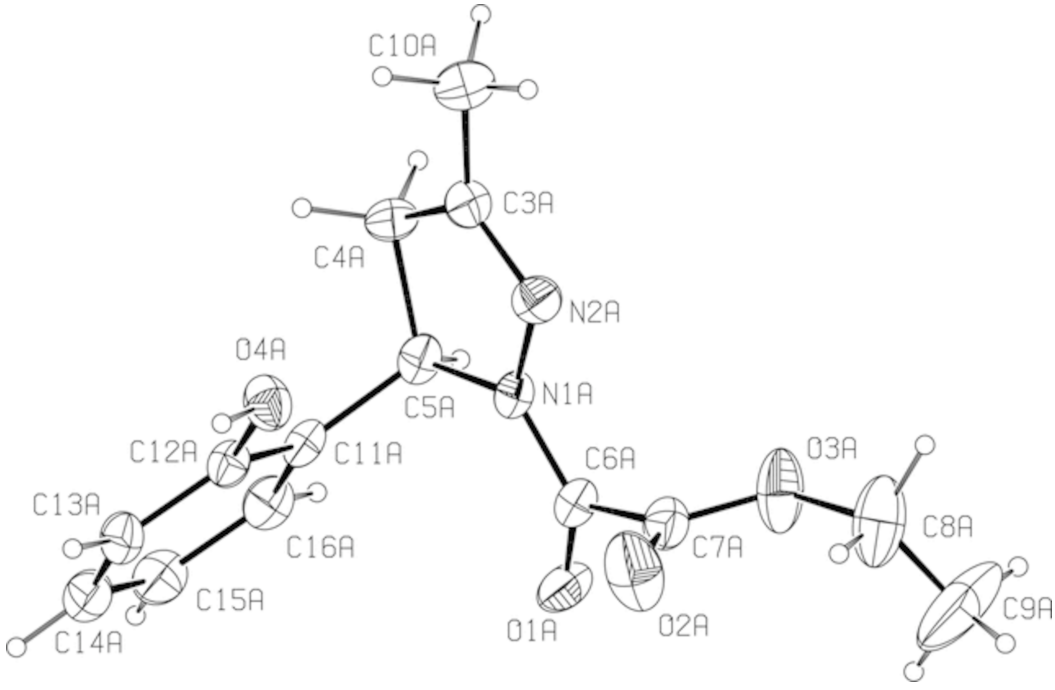


(III)



(IV)

Fig. 2


C10A
C3A
C4A
N2A
O4A
C5A
N1A
C12A
C11A
C6A
O3A
C13A
C16A
C7A
C8A
C15A
O1A
O2A
C9A
C14A