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(*E*)-4-Hydroxy-6-methyl-3-[1-(2-phenylhydrazinylidene)ethyl]-2*H*-pyran-2-one

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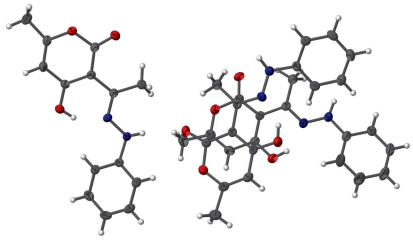
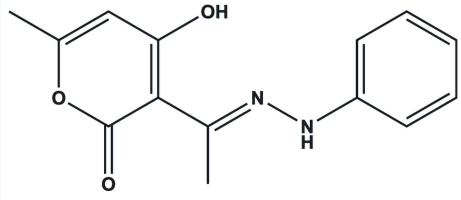
The title compound, $C_{14}H_{14}N_2O_3$, crystallized with three crystallographically independent molecules (*A*, *B* and *C*) in the asymmetric unit. The three molecules each have an *E* conformation about the C=N bond but differ in the orientation of the phenyl and pyran rings. The dihedral angles between the phenyl and pyran ring planes are 14.30 (1), 28.38 (1) and 25.58 (1)° in molecules *A*, *B* and *C*, respectively. There is an intramolecular O—H···N hydrogen bond in each molecule with an *S*(6) ring motif. In the crystal, molecules are linked by N—H···O and C—H···O hydrogen bonds, forming layers parallel to (001), enclosing $R_2^2(8)$ and $R_3^3(21)$ ring motifs. The layers are linked *via* C—H···π interactions, forming bilayers, which are joined by a further C—H···π interaction, forming a three-dimensional structure.

3D view



Chemical scheme



Structure description

Hydrazones have received much attention recently due to their biological activities (Ajani *et al.*, 2010). The principle aim of investigating the structural chemistry of hydrazones is to study their coordination properties (Garcia-Herbosa *et al.* 1994). In the present paper, we describe the synthesis and crystal structure of a new hydrazone ligand.

The title compound, Fig. 1, crystallized with three independent molecules (*A*, *B* and *C*) in the asymmetric unit. The three molecules exist in a *trans* or *E* conformation with

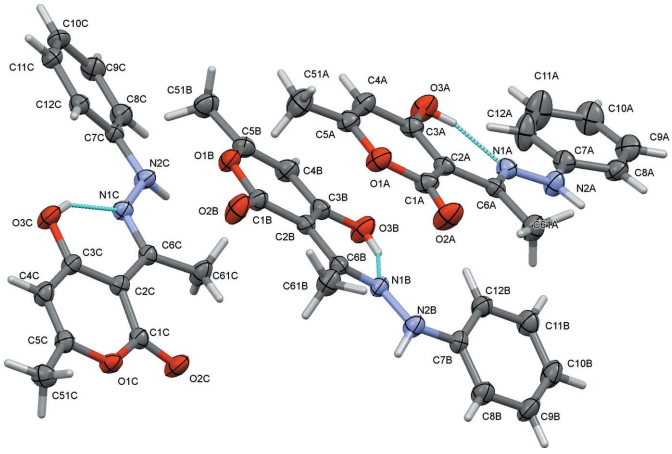


Figure 1
The molecular structure of the three independent molecules (*A*, *B* and *C*) of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

respect to the C═N bond but differ in the orientation of the phenyl ring with respect the pyran-2-one ring. The dihedral angles between the phenyl and pyran ring planes are 14.30 (1), 28.38 (1) and 25.58 (1)°, in molecules *A*, *B* and *C*, respectively. The N—N distances [1.356 (2)-1.377 (2) Å] are rather long compared to those observed in related compounds, *viz*. ethyl 4-chloro-3-oxo-2-(phenylhydrazono)butyrate [1.300 (2) Å; Alpaslan *et al.*, 2005*a*], (*E*)-ethyl 4-chloro-3-[2-(2-fluorophenyl)hydrazono]butanoate [1.306 (2) Å; Alpaslan *et al.*, 2005*b*] and (*Z*)-ethyl 4-chloro-2-[2-(2-methoxyphenyl) hydrazono]-3-oxobutanoate [1.300 (2) Å; Alpaslan *et al.*, 2005*c*]. This elongation may be the result of the intramolecular O—H···N hydrogen bonds that occur in each molecule (Fig. 1 and Table 1), and which form an *S*(6) ring motif. The C—$N_{iminium}$

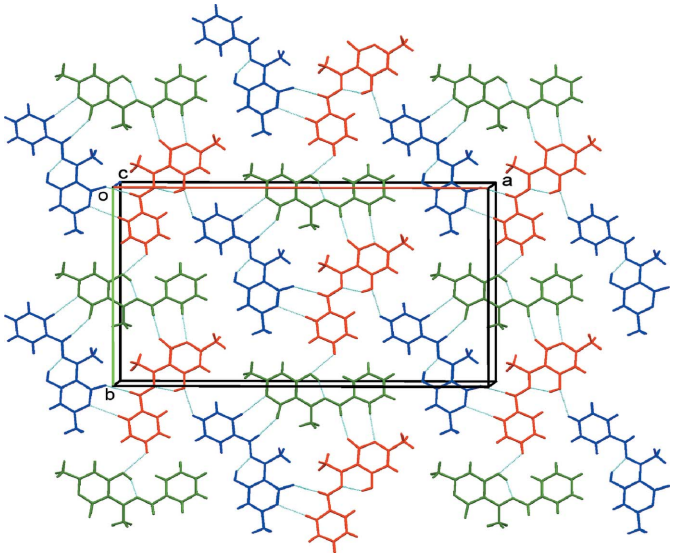


Figure 2
A view along the *c* axis of the hydrogen-bonded (dashed lines; see Table 1) layer in the crystal of the title compound (molecule *A* blue, molecule *B* red, molecule *C* green).

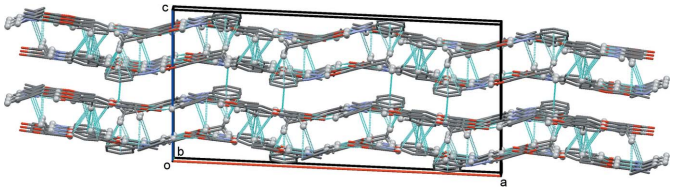


Figure 3
A view along the *b* axis of the crystal packing of the title compound. The hydrogen bonds and C—H···π interactions are shown as dashed lines (see Table 1). H atoms are shown as grey balls and those H atoms not involved in these interactions have been omitted for clarity.

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

*Cg*2, *Cg*4 ad *Cg*6 are the centroids of rings C7*A*–C12*A*, C7*B*–C12*B* and C7*C*–C12*C*, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3*A*—H3*A*···N1*A*	0.82	1.74	2.469 (2)	147
O3*B*—H3*B*···N1*B*	0.82	1.71	2.448 (2)	148
N2*A*—H2*A*···O2*C*^{i}	0.91	2.20	3.099 (2)	170
N2*B*—H2*B*···O2*A*^{i}	0.89	2.13	2.994 (2)	165
O3*C*—H3*C*···N1*C*	0.82	1.73	2.462 (2)	148
N2*C*—H2*C*···O2*B*	0.88	2.19	3.028 (2)	157
C8*B*—H8*B*···O1*A*^{i}	0.93	2.53	3.438 (2)	165
C8*C*—H8*C*···O1*B*	0.93	2.58	3.473 (2)	162
C10*A*—H10*A*···O3*B*^{ii}	0.93	2.58	3.294 (3)	134
C51*A*—H51*B*···*Cg*4^{iii}	0.96	2.84	3.729 (3)	154
C51*B*—H51*F*···*Cg*2^{iii}	0.96	2.62	3.436 (3)	143
C51*C*—H51*G*···*Cg*6^{iv}	0.96	2.95	3.729 (2)	139
C51*C*—H51*H*···*Cg*6^{v}	0.96	2.62	3.516 (2)	156

Symmetry codes: (i) $-x+2, -y, -z+2$; (ii) $-x+\frac{3}{2}, -y-\frac{1}{2}, -z+2$; (iii) $x, -y, z-\frac{1}{2}$; (iv) $-x+2, -y+1, -z+2$; (v) $-x+2, y, -z+\frac{5}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{14}N_2O_3$
M_r	258.27
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	30.1064 (12), 17.5911 (7), 13.7937 (8)
β (°)	92.613 (4)
V (Å^3)	7297.6 (6)
Z	24
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.1 × 0.1 × 0.1
Data collection	
Diffractometer	Agilent Xcalibur Sapphire 1
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min}, T_{max}	0.725, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37468, 7444, 4849
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.048, 0.125, 1.02
No. of reflections	7444
No. of parameters	524
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.21

Computer programs: *CrysAlis PRO* (Agilent, 2011), *SIR97* (Altomare *et al.*, 1999), *Mercury* (Macrae *et al.*, 2008), *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

bond lengths [1.294 (2)–1.301 (2) Å] are comparable to that observed in 1-dimethylamino-3-dimethyliminio-2-(*p*-methoxyphenyl) prop-1-ene perchlorate [1.307 (3) Å; Girija *et al.*, 2004].

In the crystal, the three molecules are linked *via* N—H···O and C—H···O hydrogen bonds forming layers parallel to the *ab* plane (Table 1 and Fig. 2). The layers are linked *via* C—H···π interactions forming bilayers, which in turn are joined by a further C—H···π interaction, forming a three-dimensional structure (Table 1 and Fig. 3).

Synthesis and crystallization

The title compound was prepared by reacting equimolar amounts of dehydroacetic acid and phenylhydrazine (1:1 *M* ratio), in absolute ethanol. The mixture was refluxed for 1 h, then the yellow solid which precipitated was filtered and recrystallized from 75% ethanol and 25% distilled water, giving colourless prismatic crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160729 [doi:10.1107/S241431461600729X]

(*E*)-4-Hydroxy-6-methyl-3-[1-(2-phenylhydrazinylidene)ethyl]-2*H*-pyran-2-one

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Crystal data

$C_{14}H_{14}N_2O_3$
M_r = 258.27
Monoclinic, *C*2/*c*
a = 30.1064 (12) Å
b = 17.5911 (7) Å
c = 13.7937 (8) Å
β = 92.613 (4)°
V = 7297.6 (6) Å^3
Z = 24
F(000) = 3264
D_x = 1.410 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 13642 reflections
θ = 3.0–28.5°
μ = 0.10 mm^{-1}
T = 293 K
Prism, colourless
0.1 × 0.1 × 0.1 mm

Data collection

Agilent Xcalibur Sapphire 1 (long nozzle) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.2632 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.725, T_{max} = 1.000
37468 measured reflections
7444 independent reflections
4849 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.4°, θ_{min} = 3.0°
h = −37→37
k = −21→21
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.125
S = 1.02
7444 reflections
524 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 1.4052P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2014), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00019 (4)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.92018 (4)	0.11844 (8)	0.83644 (11)	0.0362 (4)
O2A	0.96328 (5)	0.01943 (8)	0.82982 (13)	0.0463 (4)
O3A	0.80749 (4)	−0.00288 (8)	0.85760 (12)	0.0404 (4)
H3A	0.8117	−0.0489	0.8558	0.061*
N1A	0.85018 (5)	−0.12222 (9)	0.84702 (11)	0.0272 (4)
N2A	0.84735 (5)	−0.19903 (9)	0.84125 (13)	0.0335 (4)
H2A	0.8705	−0.2298	0.8283	0.050*
C1A	0.92495 (6)	0.04004 (12)	0.83610 (15)	0.0306 (5)
C2A	0.88587 (6)	−0.00496 (11)	0.84325 (13)	0.0244 (4)
C3A	0.84554 (6)	0.03292 (11)	0.85052 (15)	0.0284 (4)
C4A	0.84330 (7)	0.11340 (12)	0.84907 (15)	0.0335 (5)
H4A	0.8160	0.1378	0.8528	0.040*
C5A	0.88017 (7)	0.15357 (12)	0.84241 (15)	0.0328 (5)
C6A	0.88806 (6)	−0.08819 (11)	0.84065 (13)	0.0254 (4)
C7A	0.80575 (6)	−0.23277 (11)	0.84855 (15)	0.0303 (5)
C8A	0.80153 (7)	−0.30963 (12)	0.82736 (16)	0.0346 (5)
H8A	0.8261	−0.3375	0.8096	0.042*
C9A	0.76071 (7)	−0.34457 (13)	0.83280 (17)	0.0415 (6)
H9A	0.7581	−0.3962	0.8190	0.050*
C10A	0.72387 (8)	−0.30471 (14)	0.85816 (19)	0.0490 (6)
H10A	0.6964	−0.3287	0.8612	0.059*
C11A	0.72826 (8)	−0.22918 (15)	0.8789 (2)	0.0592 (8)
H11A	0.7034	−0.2016	0.8960	0.071*
C12A	0.76869 (7)	−0.19293 (13)	0.87504 (19)	0.0492 (7)
H12A	0.7710	−0.1415	0.8903	0.059*
C51A	0.88479 (8)	0.23714 (12)	0.84069 (18)	0.0468 (6)
H51A	0.8558	0.2601	0.8393	0.070*
H51B	0.8999	0.2520	0.7840	0.070*
H51C	0.9016	0.2536	0.8977	0.070*
C61A	0.93004 (6)	−0.13205 (12)	0.82913 (17)	0.0383 (5)
H61A	0.9371	−0.1603	0.8873	0.057*
H61B	0.9539	−0.0975	0.8172	0.057*
H61C	0.9261	−0.1665	0.7754	0.057*
O1B	0.82066 (4)	0.19617 (7)	1.10144 (11)	0.0335 (3)
O2B	0.89226 (5)	0.21154 (8)	1.08964 (12)	0.0435 (4)
O3B	0.82796 (4)	−0.03206 (8)	1.11672 (12)	0.0388 (4)
H3B	0.8535	−0.0477	1.1134	0.058*
N1B	0.90859 (5)	−0.02693 (9)	1.10124 (12)	0.0265 (4)
N2B	0.94645 (5)	−0.07052 (9)	1.09691 (12)	0.0306 (4)
H2B	0.9707	−0.0538	1.1291	0.046*
C1B	0.86300 (6)	0.16466 (11)	1.09636 (15)	0.0280 (5)
C2B	0.86675 (6)	0.08392 (10)	1.09932 (13)	0.0241 (4)
C3B	0.82784 (6)	0.04126 (11)	1.11247 (14)	0.0273 (4)
C4B	0.78639 (6)	0.07876 (12)	1.12250 (15)	0.0330 (5)
H4B	0.7610	0.0505	1.1337	0.040*

C5B	0.78377 (6)	0.15347 (12)	1.11605 (15)	0.0307 (5)
C6B	0.90956 (6)	0.04645 (11)	1.09164 (13)	0.0246 (4)
C7B	0.94002 (6)	−0.14901 (11)	1.09955 (14)	0.0263 (4)
C8B	0.97515 (7)	−0.19505 (12)	1.13130 (16)	0.0351 (5)
H8B	1.0020	−0.1734	1.1527	0.042*
C9B	0.97026 (7)	−0.27271 (12)	1.13106 (17)	0.0408 (6)
H9B	0.9939	−0.3033	1.1526	0.049*
C10B	0.93070 (7)	−0.30586 (12)	1.09932 (17)	0.0401 (6)
H10B	0.9275	−0.3584	1.0997	0.048*
C11B	0.89621 (7)	−0.26032 (12)	1.06725 (16)	0.0366 (5)
H11B	0.8696	−0.2824	1.0451	0.044*
C12B	0.90023 (6)	−0.18240 (11)	1.06722 (15)	0.0316 (5)
H12B	0.8764	−0.1522	1.0457	0.038*
C51B	0.74299 (7)	0.20061 (13)	1.12286 (18)	0.0430 (6)
H51D	0.7191	0.1695	1.1440	0.064*
H51E	0.7350	0.2218	1.0603	0.064*
H51F	0.7486	0.2410	1.1686	0.064*
C61B	0.95219 (6)	0.08598 (12)	1.07389 (17)	0.0367 (5)
H61D	0.9677	0.0964	1.1348	0.055*
H61E	0.9461	0.1329	1.0402	0.055*
H61F	0.9703	0.0541	1.0352	0.055*
O1C	1.10738 (4)	0.43283 (7)	1.19270 (10)	0.0281 (3)
O2C	1.07871 (4)	0.31901 (8)	1.18408 (11)	0.0386 (4)
O3C	0.99149 (4)	0.53864 (7)	1.12150 (12)	0.0360 (4)
H3C	0.9710	0.5086	1.1105	0.054*
N1C	0.95353 (5)	0.41541 (9)	1.10040 (11)	0.0250 (4)
N2C	0.91419 (5)	0.37916 (9)	1.07685 (12)	0.0275 (4)
H2C	0.9105	0.3322	1.0977	0.041*
C1C	1.07080 (6)	0.38597 (11)	1.17251 (14)	0.0253 (4)
C2C	1.02955 (6)	0.42148 (10)	1.14246 (13)	0.0230 (4)
C3C	1.02809 (6)	0.50082 (11)	1.14407 (14)	0.0253 (4)
C4C	1.06635 (6)	0.54419 (11)	1.16983 (15)	0.0304 (5)
H4C	1.0646	0.5969	1.1715	0.036*
C5C	1.10493 (6)	0.50989 (11)	1.19177 (14)	0.0270 (4)
C6C	0.98991 (6)	0.37651 (10)	1.11594 (13)	0.0232 (4)
C7C	0.87613 (6)	0.42396 (11)	1.06602 (14)	0.0245 (4)
C8C	0.83493 (6)	0.39141 (12)	1.08257 (15)	0.0320 (5)
H8C	0.8333	0.3411	1.1028	0.038*
C9C	0.79655 (6)	0.43373 (12)	1.06902 (16)	0.0358 (5)
H9C	0.7693	0.4118	1.0811	0.043*
C10C	0.79808 (7)	0.50790 (12)	1.03792 (15)	0.0351 (5)
H10C	0.7721	0.5359	1.0280	0.042*
C11C	0.83893 (7)	0.54007 (12)	1.02172 (15)	0.0324 (5)
H11C	0.8403	0.5903	1.0010	0.039*
C12C	0.87773 (6)	0.49917 (11)	1.03562 (14)	0.0272 (4)
H12C	0.9049	0.5219	1.0247	0.033*
C51C	1.14880 (6)	0.54544 (12)	1.21459 (16)	0.0354 (5)
H51G	1.1679	0.5373	1.1616	0.053*

H51H	1.1620	0.5230	1.2725	0.053*
H51I	1.1450	0.5990	1.2244	0.053*
C61C	0.98990 (7)	0.29251 (11)	1.10723 (18)	0.0379 (5)
H61G	0.9784	0.2705	1.1646	0.057*
H61H	1.0197	0.2749	1.0998	0.057*
H61I	0.9716	0.2777	1.0516	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0283 (7)	0.0266 (8)	0.0535 (10)	−0.0037 (6)	−0.0020 (7)	0.0060 (7)
O2A	0.0202 (7)	0.0350 (9)	0.0836 (13)	−0.0030 (7)	0.0003 (7)	0.0046 (8)
O3A	0.0231 (7)	0.0312 (8)	0.0674 (11)	−0.0024 (6)	0.0066 (7)	0.0023 (8)
N1A	0.0252 (8)	0.0251 (9)	0.0311 (10)	−0.0036 (7)	−0.0018 (7)	0.0001 (7)
N2A	0.0251 (9)	0.0242 (9)	0.0511 (12)	−0.0035 (7)	−0.0001 (8)	−0.0045 (8)
C1A	0.0261 (10)	0.0288 (11)	0.0365 (12)	−0.0021 (9)	−0.0030 (9)	0.0044 (9)
C2A	0.0226 (9)	0.0260 (10)	0.0243 (10)	−0.0031 (8)	−0.0011 (8)	0.0010 (8)
C3A	0.0230 (10)	0.0307 (11)	0.0315 (12)	−0.0026 (9)	0.0000 (8)	0.0020 (9)
C4A	0.0288 (11)	0.0305 (11)	0.0413 (13)	0.0043 (9)	0.0036 (9)	0.0031 (10)
C5A	0.0344 (11)	0.0286 (11)	0.0354 (12)	0.0024 (9)	−0.0003 (9)	0.0042 (9)
C6A	0.0234 (9)	0.0287 (11)	0.0239 (10)	−0.0019 (8)	−0.0020 (8)	−0.0012 (8)
C7A	0.0284 (10)	0.0290 (11)	0.0335 (12)	−0.0052 (9)	0.0006 (9)	0.0002 (9)
C8A	0.0313 (11)	0.0265 (11)	0.0455 (14)	0.0012 (9)	−0.0039 (9)	0.0030 (10)
C9A	0.0443 (13)	0.0261 (11)	0.0536 (15)	−0.0092 (10)	−0.0031 (11)	0.0038 (11)
C10A	0.0395 (13)	0.0414 (14)	0.0671 (18)	−0.0181 (11)	0.0125 (12)	−0.0008 (12)
C11A	0.0386 (13)	0.0454 (15)	0.096 (2)	−0.0115 (12)	0.0291 (14)	−0.0160 (15)
C12A	0.0416 (13)	0.0324 (13)	0.0755 (19)	−0.0122 (10)	0.0235 (12)	−0.0170 (12)
C51A	0.0501 (14)	0.0290 (12)	0.0616 (17)	0.0000 (11)	0.0062 (12)	0.0069 (12)
C61A	0.0279 (11)	0.0291 (12)	0.0579 (15)	−0.0019 (9)	0.0020 (10)	−0.0034 (10)
O1B	0.0268 (7)	0.0237 (7)	0.0497 (10)	0.0018 (6)	−0.0016 (6)	−0.0010 (7)
O2B	0.0315 (8)	0.0209 (8)	0.0785 (12)	−0.0044 (7)	0.0052 (8)	−0.0001 (8)
O3B	0.0274 (7)	0.0222 (8)	0.0666 (11)	−0.0028 (6)	0.0006 (7)	0.0018 (7)
N1B	0.0257 (8)	0.0221 (9)	0.0315 (10)	0.0030 (7)	0.0003 (7)	−0.0012 (7)
N2B	0.0240 (8)	0.0231 (9)	0.0445 (11)	0.0003 (7)	−0.0003 (7)	−0.0015 (8)
C1B	0.0241 (10)	0.0247 (10)	0.0351 (12)	0.0008 (9)	−0.0009 (8)	−0.0010 (9)
C2B	0.0255 (9)	0.0220 (10)	0.0244 (10)	−0.0014 (8)	−0.0027 (8)	−0.0012 (8)
C3B	0.0264 (10)	0.0230 (10)	0.0319 (12)	−0.0048 (8)	−0.0053 (8)	−0.0012 (9)
C4B	0.0232 (10)	0.0306 (12)	0.0449 (13)	−0.0048 (9)	−0.0026 (9)	−0.0002 (10)
C5B	0.0238 (10)	0.0328 (12)	0.0351 (12)	−0.0010 (9)	−0.0030 (8)	−0.0028 (9)
C6B	0.0270 (10)	0.0232 (10)	0.0233 (10)	−0.0017 (8)	−0.0019 (8)	−0.0006 (8)
C7B	0.0286 (10)	0.0211 (10)	0.0293 (11)	0.0002 (8)	0.0035 (8)	−0.0016 (8)
C8B	0.0253 (10)	0.0292 (11)	0.0504 (14)	0.0001 (9)	−0.0021 (9)	−0.0034 (10)
C9B	0.0356 (12)	0.0261 (11)	0.0602 (16)	0.0096 (10)	−0.0031 (11)	−0.0015 (11)
C10B	0.0410 (13)	0.0215 (11)	0.0578 (16)	−0.0008 (10)	0.0028 (11)	−0.0039 (10)
C11B	0.0318 (11)	0.0290 (11)	0.0488 (14)	−0.0056 (9)	−0.0012 (10)	−0.0046 (10)
C12B	0.0270 (10)	0.0282 (11)	0.0391 (13)	0.0030 (9)	−0.0026 (9)	−0.0018 (9)
C51B	0.0309 (11)	0.0402 (14)	0.0573 (16)	0.0049 (10)	−0.0037 (10)	−0.0030 (11)
C61B	0.0278 (11)	0.0282 (11)	0.0546 (15)	−0.0013 (9)	0.0060 (10)	−0.0007 (10)

O1C	0.0209 (6)	0.0242 (7)	0.0389 (8)	−0.0009 (6)	−0.0027 (6)	0.0024 (6)
O2C	0.0285 (8)	0.0227 (8)	0.0638 (11)	0.0028 (6)	−0.0082 (7)	0.0031 (7)
O3C	0.0238 (7)	0.0225 (7)	0.0613 (11)	0.0027 (6)	−0.0035 (7)	0.0011 (7)
N1C	0.0187 (8)	0.0240 (9)	0.0322 (9)	−0.0014 (7)	−0.0013 (7)	−0.0010 (7)
N2C	0.0195 (8)	0.0198 (8)	0.0428 (11)	−0.0017 (6)	−0.0043 (7)	0.0020 (7)
C1C	0.0235 (9)	0.0231 (10)	0.0292 (11)	−0.0026 (8)	0.0012 (8)	−0.0006 (8)
C2C	0.0204 (9)	0.0234 (10)	0.0254 (10)	−0.0008 (8)	0.0031 (7)	−0.0001 (8)
C3C	0.0223 (10)	0.0222 (10)	0.0314 (11)	0.0024 (8)	0.0016 (8)	0.0017 (8)
C4C	0.0290 (11)	0.0211 (10)	0.0412 (13)	−0.0032 (8)	0.0020 (9)	−0.0015 (9)
C5C	0.0290 (10)	0.0234 (10)	0.0286 (11)	−0.0040 (8)	0.0024 (8)	−0.0004 (9)
C6C	0.0225 (9)	0.0222 (10)	0.0251 (10)	−0.0010 (8)	0.0016 (8)	−0.0001 (8)
C7C	0.0213 (9)	0.0236 (10)	0.0282 (11)	0.0010 (8)	−0.0033 (8)	−0.0042 (8)
C8C	0.0262 (10)	0.0278 (11)	0.0415 (13)	−0.0039 (9)	−0.0018 (9)	−0.0008 (9)
C9C	0.0217 (10)	0.0383 (13)	0.0470 (14)	−0.0044 (9)	−0.0026 (9)	−0.0048 (10)
C10C	0.0259 (10)	0.0370 (12)	0.0414 (13)	0.0090 (9)	−0.0083 (9)	−0.0072 (10)
C11C	0.0350 (11)	0.0257 (11)	0.0359 (12)	0.0053 (9)	−0.0065 (9)	−0.0012 (9)
C12C	0.0246 (10)	0.0245 (10)	0.0323 (11)	−0.0026 (8)	−0.0024 (8)	−0.0012 (9)
C51C	0.0286 (11)	0.0349 (12)	0.0421 (13)	−0.0081 (9)	−0.0042 (9)	0.0015 (10)
C61C	0.0264 (11)	0.0239 (11)	0.0630 (16)	0.0001 (9)	−0.0030 (10)	−0.0057 (10)

Geometric parameters (Å, º)

O1A—C5A	1.360 (2)	C7B—C8B	1.387 (3)
O1A—C1A	1.387 (2)	C7B—C12B	1.389 (3)
O2A—C1A	1.216 (2)	C8B—C9B	1.374 (3)
O3A—C3A	1.315 (2)	C8B—H8B	0.9300
O3A—H3A	0.8200	C9B—C10B	1.379 (3)
N1A—C6A	1.294 (2)	C9B—H9B	0.9300
N1A—N2A	1.356 (2)	C10B—C11B	1.369 (3)
N2A—C7A	1.394 (2)	C10B—H10B	0.9300
N2A—H2A	0.9064	C11B—C12B	1.376 (3)
C1A—C2A	1.425 (3)	C11B—H11B	0.9300
C2A—C3A	1.393 (3)	C12B—H12B	0.9300
C2A—C6A	1.466 (3)	C51B—H51D	0.9600
C3A—C4A	1.417 (3)	C51B—H51E	0.9600
C4A—C5A	1.323 (3)	C51B—H51F	0.9600
C4A—H4A	0.9300	C61B—H61D	0.9600
C5A—C51A	1.477 (3)	C61B—H61E	0.9600
C6A—C61A	1.495 (3)	C61B—H61F	0.9600
C7A—C12A	1.381 (3)	O1C—C5C	1.358 (2)
C7A—C8A	1.388 (3)	O1C—C1C	1.393 (2)
C8A—C9A	1.379 (3)	O2C—C1C	1.211 (2)
C8A—H8A	0.9300	O3C—C3C	1.312 (2)
C9A—C10A	1.371 (3)	O3C—H3C	0.8200
C9A—H9A	0.9300	N1C—C6C	1.301 (2)
C10A—C11A	1.364 (3)	N1C—N2C	1.371 (2)
C10A—H10A	0.9300	N2C—C7C	1.393 (2)
C11A—C12A	1.377 (3)	N2C—H2C	0.8836

C11A—H11A	0.9300	C1C—C2C	1.434 (2)
C12A—H12A	0.9300	C2C—C3C	1.397 (3)
C51A—H51A	0.9600	C2C—C6C	1.464 (2)
C51A—H51B	0.9600	C3C—C4C	1.413 (3)
C51A—H51C	0.9600	C4C—C5C	1.332 (3)
C61A—H61A	0.9600	C4C—H4C	0.9300
C61A—H61B	0.9600	C5C—C51C	1.482 (3)
C61A—H61C	0.9600	C6C—C61C	1.483 (3)
O1B—C5B	1.364 (2)	C7C—C12C	1.389 (3)
O1B—C1B	1.395 (2)	C7C—C8C	1.395 (3)
O2B—C1B	1.213 (2)	C8C—C9C	1.380 (3)
O3B—C3B	1.291 (2)	C8C—H8C	0.9300
O3B—H3B	0.8200	C9C—C10C	1.375 (3)
N1B—C6B	1.298 (2)	C9C—H9C	0.9300
N1B—N2B	1.377 (2)	C10C—C11C	1.381 (3)
N2B—C7B	1.395 (2)	C10C—H10C	0.9300
N2B—H2B	0.8876	C11C—C12C	1.378 (3)
C1B—C2B	1.425 (3)	C11C—H11C	0.9300
C2B—C3B	1.410 (3)	C12C—H12C	0.9300
C2B—C6B	1.456 (3)	C51C—H51G	0.9600
C3B—C4B	1.424 (3)	C51C—H51H	0.9600
C4B—C5B	1.319 (3)	C51C—H51I	0.9600
C4B—H4B	0.9300	C61C—H61G	0.9600
C5B—C51B	1.488 (3)	C61C—H61H	0.9600
C6B—C61B	1.490 (3)	C61C—H61I	0.9600

C5A—O1A—C1A	122.99 (16)	C9B—C8B—C7B	119.98 (19)
C3A—O3A—H3A	109.5	C9B—C8B—H8B	120.0
C6A—N1A—N2A	120.67 (16)	C7B—C8B—H8B	120.0
N1A—N2A—C7A	118.33 (16)	C8B—C9B—C10B	120.8 (2)
N1A—N2A—H2A	124.1	C8B—C9B—H9B	119.6
C7A—N2A—H2A	117.4	C10B—C9B—H9B	119.6
O2A—C1A—O1A	113.30 (17)	C11B—C10B—C9B	119.1 (2)
O2A—C1A—C2A	128.92 (19)	C11B—C10B—H10B	120.4
O1A—C1A—C2A	117.78 (17)	C9B—C10B—H10B	120.4
C3A—C2A—C1A	117.67 (18)	C10B—C11B—C12B	121.2 (2)
C3A—C2A—C6A	121.33 (17)	C10B—C11B—H11B	119.4
C1A—C2A—C6A	120.98 (17)	C12B—C11B—H11B	119.4
O3A—C3A—C2A	122.79 (18)	C11B—C12B—C7B	119.71 (19)
O3A—C3A—C4A	116.01 (17)	C11B—C12B—H12B	120.1
C2A—C3A—C4A	121.20 (18)	C7B—C12B—H12B	120.1
C5A—C4A—C3A	119.67 (19)	C5B—C51B—H51D	109.5
C5A—C4A—H4A	120.2	C5B—C51B—H51E	109.5
C3A—C4A—H4A	120.2	H51D—C51B—H51E	109.5
C4A—C5A—O1A	120.67 (19)	C5B—C51B—H51F	109.5
C4A—C5A—C51A	127.8 (2)	H51D—C51B—H51F	109.5
O1A—C5A—C51A	111.53 (18)	H51E—C51B—H51F	109.5
N1A—C6A—C2A	114.80 (17)	C6B—C61B—H61D	109.5

N1A—C6A—C61A	121.34 (18)	C6B—C61B—H61E	109.5
C2A—C6A—C61A	123.85 (17)	H61D—C61B—H61E	109.5
C12A—C7A—C8A	118.87 (19)	C6B—C61B—H61F	109.5
C12A—C7A—N2A	122.85 (19)	H61D—C61B—H61F	109.5
C8A—C7A—N2A	118.28 (18)	H61E—C61B—H61F	109.5
C9A—C8A—C7A	119.7 (2)	C5C—O1C—C1C	123.14 (14)
C9A—C8A—H8A	120.1	C3C—O3C—H3C	109.5
C7A—C8A—H8A	120.1	C6C—N1C—N2C	120.41 (15)
C10A—C9A—C8A	121.3 (2)	N1C—N2C—C7C	117.36 (15)
C10A—C9A—H9A	119.4	N1C—N2C—H2C	118.6
C8A—C9A—H9A	119.4	C7C—N2C—H2C	116.7
C11A—C10A—C9A	118.7 (2)	O2C—C1C—O1C	113.59 (16)
C11A—C10A—H10A	120.7	O2C—C1C—C2C	128.63 (17)
C9A—C10A—H10A	120.7	O1C—C1C—C2C	117.78 (16)
C10A—C11A—C12A	121.4 (2)	C3C—C2C—C1C	117.27 (16)
C10A—C11A—H11A	119.3	C3C—C2C—C6C	121.20 (16)
C12A—C11A—H11A	119.3	C1C—C2C—C6C	121.46 (17)
C11A—C12A—C7A	120.1 (2)	O3C—C3C—C2C	121.98 (17)
C11A—C12A—H12A	120.0	O3C—C3C—C4C	116.86 (17)
C7A—C12A—H12A	120.0	C2C—C3C—C4C	121.16 (17)
C5A—C51A—H51A	109.5	C5C—C4C—C3C	120.34 (18)
C5A—C51A—H51B	109.5	C5C—C4C—H4C	119.8
H51A—C51A—H51B	109.5	C3C—C4C—H4C	119.8
C5A—C51A—H51C	109.5	C4C—C5C—O1C	120.07 (17)
H51A—C51A—H51C	109.5	C4C—C5C—C51C	128.09 (19)
H51B—C51A—H51C	109.5	O1C—C5C—C51C	111.83 (16)
C6A—C61A—H61A	109.5	N1C—C6C—C2C	115.30 (16)
C6A—C61A—H61B	109.5	N1C—C6C—C61C	120.92 (16)
H61A—C61A—H61B	109.5	C2C—C6C—C61C	123.78 (16)
C6A—C61A—H61C	109.5	C12C—C7C—N2C	122.11 (17)
H61A—C61A—H61C	109.5	C12C—C7C—C8C	118.92 (17)
H61B—C61A—H61C	109.5	N2C—C7C—C8C	118.91 (17)
C5B—O1B—C1B	122.75 (15)	C9C—C8C—C7C	120.1 (2)
C3B—O3B—H3B	109.5	C9C—C8C—H8C	119.9
C6B—N1B—N2B	121.79 (16)	C7C—C8C—H8C	119.9
N1B—N2B—C7B	115.71 (15)	C10C—C9C—C8C	120.96 (19)
N1B—N2B—H2B	117.2	C10C—C9C—H9C	119.5
C7B—N2B—H2B	115.3	C8C—C9C—H9C	119.5
O2B—C1B—O1B	113.71 (17)	C9C—C10C—C11C	118.81 (19)
O2B—C1B—C2B	128.52 (18)	C9C—C10C—H10C	120.6
O1B—C1B—C2B	117.77 (16)	C11C—C10C—H10C	120.6
C3B—C2B—C1B	117.97 (17)	C12C—C11C—C10C	121.3 (2)
C3B—C2B—C6B	120.79 (17)	C12C—C11C—H11C	119.4
C1B—C2B—C6B	121.21 (17)	C10C—C11C—H11C	119.4
O3B—C3B—C2B	122.47 (18)	C11C—C12C—C7C	119.90 (18)
O3B—C3B—C4B	117.34 (17)	C11C—C12C—H12C	120.1
C2B—C3B—C4B	120.19 (18)	C7C—C12C—H12C	120.1
C5B—C4B—C3B	120.34 (19)	C5C—C51C—H51G	109.5

C5B—C4B—H4B	119.8	C5C—C51C—H51H	109.5
C3B—C4B—H4B	119.8	H51G—C51C—H51H	109.5
C4B—C5B—O1B	120.80 (18)	C5C—C51C—H51I	109.5
C4B—C5B—C51B	126.72 (19)	H51G—C51C—H51I	109.5
O1B—C5B—C51B	112.47 (18)	H51H—C51C—H51I	109.5
N1B—C6B—C2B	114.76 (17)	C6C—C61C—H61G	109.5
N1B—C6B—C61B	120.29 (17)	C6C—C61C—H61H	109.5
C2B—C6B—C61B	124.95 (17)	H61G—C61C—H61H	109.5
C8B—C7B—C12B	119.23 (18)	C6C—C61C—H61I	109.5
C8B—C7B—N2B	118.78 (17)	H61G—C61C—H61I	109.5
C12B—C7B—N2B	121.92 (17)	H61H—C61C—H61I	109.5

Hydrogen-bond geometry (Å, º)

Cg2, Cg4 ad Cg6 are the centroids of rings C7*A*–C12*A*, C7*B*–C12*B* and C7*C*–C12*C*, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3*A*—H3*A*···N1*A*	0.82	1.74	2.469 (2)	147
O3*B*—H3*B*···N1*B*	0.82	1.71	2.448 (2)	148
N2*A*—H2*A*···O2*C*^{i}	0.91	2.20	3.099 (2)	170
N2*B*—H2*B*···O2*A*^{i}	0.89	2.13	2.994 (2)	165
O3*C*—H3*C*···N1*C*	0.82	1.73	2.462 (2)	148
N2*C*—H2*C*···O2*B*	0.88	2.19	3.028 (2)	157
C8*B*—H8*B*···O1*A*^{i}	0.93	2.53	3.438 (2)	165
C8*C*—H8*C*···O1*B*	0.93	2.58	3.473 (2)	162
C10*A*—H10*A*···O3*B*^{ii}	0.93	2.58	3.294 (3)	134
C51*A*—H51*B*···*Cg*4^{iii}	0.96	2.84	3.729 (3)	154
C51*B*—H51*F*···*Cg*2^{iii}	0.96	2.62	3.436 (3)	143
C51*C*—H51*G*···*Cg*6^{iv}	0.96	2.95	3.729 (2)	139
C51*C*—H51*H*···*Cg*6^{v}	0.96	2.62	3.516 (2)	156

Symmetry codes: (i) −*x*+2, −*y*, −*z*+2; (ii) −*x*+3/2, −*y*−1/2, −*z*+2; (iii) *x*, −*y*, *z*−1/2; (iv) −*x*+2, −*y*+1, −*z*+2; (v) −*x*+2, *y*, −*z*+5/2.