

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Ethyl N-[2-(4-phenoxyphenoxy)ethyl]-carbamate

Jolanta Karpinska,^a Manuel Kuhs,^b Ake Rasmuson,^b Andrea Erxleben^a and Patrick McArdle^{a*}^aSchool of Chemistry, National University of Ireland, Galway, University road, Galway, Ireland, and ^bSchool of Chemistry, Limerick, Ireland
Correspondence e-mail: p.mcardle@nuigalway.ie

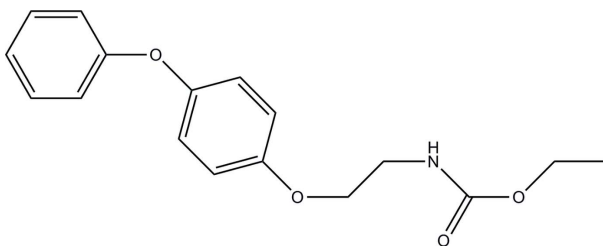
Received 14 August 2012; accepted 28 August 2012

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.063; wR factor = 0.126; data-to-parameter ratio = 14.4.

The title compound, $\text{C}_{17}\text{H}_{19}\text{NO}_4$, which is a non-toxic insect growth regulator with the common name fenoxycarb, contains two independent and conformationally different molecules in the asymmetric unit. Although the inter-ring dihedral angles are similar [62.21 (15) and 63.00 (14)°], the side-chain orientations differ. In the crystal, the molecules are linked through $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding associations, giving chains which extend along [110], while intra- and intermolecular aromatic $\text{C}-\text{H}\cdots\pi$ interactions give sheet structures parallel to [110].

Related literature

For studies on the role of fenoxycarb as an insect growth regulator, see: Paya *et al.* (2009); Sullivan (2010); Kavallieratos *et al.* (2012); Goncu & Parlak (2012). For related structures containing the phenyl ether motif, see: Ammon *et al.* (1983); Clayden *et al.* (1990); Glidewell *et al.* (2005). For hydrogen-bond data, see Lifson *et al.* (1979).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{19}\text{NO}_4$
 $M_r = 301.33$
 Triclinic, $P\bar{1}$
 $a = 5.9035$ (3) Å
 $b = 7.5712$ (4) Å

 $c = 35.373$ (2) Å
 $\alpha = 85.618$ (5)°
 $\beta = 86.213$ (5)°
 $\gamma = 89.046$ (4)°
 $V = 1572.88$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 150$ K
 $0.40 \times 0.30 \times 0.15$ mm

Data collection

 Oxford Diffraction Xcalibur,
 Sapphire3 CCD-detector
 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Oxford)

 Diffraction, 2010)
 $T_{\min} = 0.894$, $T_{\max} = 1.000$
 25379 measured reflections
 5745 independent reflections
 2937 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.074$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.126$
 $S = 0.97$
 5745 reflections

 399 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$\text{Cg}1$, $\text{Cg}2$, $\text{Cg}3$ and $\text{Cg}4$ are the centroids of the $\text{C}1-\text{C}6$, $\text{C}7-\text{C}12$, $\text{C}18-\text{C}23$ and $\text{C}24-\text{C}29$ rings, respectively.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}1-\text{H}1\text{N}1\cdots\text{O}7^{\text{i}}$	0.86	1.96	2.815 (4)	178
$\text{N}2-\text{H}2\text{N}2\cdots\text{O}3$	0.86	1.94	2.795 (4)	173
$\text{C}2-\text{H}2\cdots\text{Cg}3^{\text{ii}}$	0.93	2.85	3.562 (3)	134
$\text{C}5-\text{H}5\cdots\text{Cg}3^{\text{iii}}$	0.93	2.85	3.562 (3)	134
$\text{C}9-\text{H}9\cdots\text{Cg}4$	0.93	2.86	3.622 (3)	139
$\text{C}12-\text{H}12\cdots\text{Cg}4^{\text{i}}$	0.93	2.84	3.590 (3)	138
$\text{C}19-\text{H}19\cdots\text{Cg}1$	0.93	2.92	3.657 (3)	137
$\text{C}22-\text{H}22\cdots\text{Cg}1^{\text{iv}}$	0.93	2.84	3.583 (3)	137
$\text{C}26-\text{H}26\cdots\text{Cg}2^{\text{v}}$	0.93	2.87	3.579 (3)	134
$\text{C}29-\text{H}29\cdots\text{Cg}2^{\text{vi}}$	0.93	2.80	3.527 (3)	135

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SORTX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

The authors thank Science Foundation Ireland (SFI) for funding for the Solid State Pharmaceuticals Cluster (SSPC).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2230).

References

- Ammon, H. L., Bhattacharjee, S. K., Ravi, P. S. & Potlock, S. J. (1983). *Acta Cryst.* **C39**, 304–306.
 Clayden, N. J., Williams, D. & O'Mahoney, C. A. (1990). *J. Chem. Soc. Perkin Trans. 2*, pp. 729–733.
 Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2005). *Acta Cryst.* **C61**, o185–o187.
 Goncu, E. & Parlak, O. (2012). *Fol. Histochem. Cytohiol. Pol. Acad. Sci. Pol. Histochem. Cytochem. Soc.* **50**, 52–57.
 Kavallieratos, N. G., Athanassiou, C. G., Vayias, B. J. & Tomanovic, Z. (2012). *J. Food Prot.* **75**, 942–950.
 Lifson, S., Hagler, A. T. & Dauber, P. (1979). *J. Am. Chem. Soc.* **101**, 5111–5121.

- McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
- Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
- Paya, P., Oliva, J., Zafrilla, P., Camara, M. A. & Barba, A. (2009). *Ecotoxicology*, **18**, 1137–1142.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sullivan, J. J. (2010). *Reviews of Environmental Contamination and Toxicology*, Vol 202, edited by D. M. Whitacre, pp. 155–184. New York: Springer.

supporting information

Acta Cryst. (2012). E68, o2834–o2835 [https://doi.org/10.1107/S1600536812037105]

Ethyl *N*-[2-(4-phenoxyphenoxy)ethyl]carbamate

Jolanta Karpinska, Manuel Kuhs, Ake Rasmuson, Andrea Erxleben and Patrick McArdle

S1. Comment

The title compound fenoxycarb, C₁₇H₁₉NO₄ is an insect growth regulator with juvenile hormone activity (Paya *et al.*, 2009; Sullivan *et al.*, 2010; Kavallieratos *et al.*, 2012; Goncu & Parlak, 2012). As part of our studies on the crystallization behaviour of this compound the crystal structure of fenocarb has been determined.

There are two chemically identical but conformationally different molecules in the asymmetric unit (Fig. 1). Although the two benzene rings adopt similar orientations [inter-ring dihedral angles, 62.21 (15) and 63.00 (14)°], the side chains differ in the region associated with the intermolecular N—H···O hydrogen-bonding interactions between the two molecules, with torsion angles O2—C13—C14—N1 and C13—C14—N1—C15 [−64.6 (4) and −85.2 (4)° in molecule 1] and O6—C30—C31—N2 and C30—C31—N2—C32 [57.4 (3) and −151.2 (3)° in molecule 2] (Fig. 2). The N2—H···O3 and N1—H···O3ⁱ hydrogen bonds (Table 1) generate a one-dimensional motif that is parallel to [110] (Fig. 3). The hydrogen bonds are estimated to be of moderate strength at 8 kcal/mol (Lifson *et al.*, 1979) and they are supported by weaker C—H···π contacts. With the aromatic ring centroids numbered in order of carbon number the distances for the interactions H26···Cg2, H29···Cg2, H9···Cg4 and H12···Cg4 are 2.87, 2.80, 2.86 and 2.86 Å respectively. The one-dimensional chains are built into two-dimensional sheets by further C—H···π contacts involving H19···Cg1, H22···Cg1, H2···Cg3 and H5···Cg3 are 2.92, 2.84, 2.85 and 2.85 Å respectively (Fig. 4). There are no π-π contacts involving the aromatic rings as adjacent rings are non-coplanar. The unit cell packing is shown in Fig.5.

S2. Experimental

The title compound of high purity (>98.8%) was obtained from Syngenta, Switzerland. Colourless, lath shaped crystals were grown from a solution in ethanol by slow evaporation at room temperature.

S3. Refinement

All H atoms were included in the refinement in calculated positions [N—H = 0.88 Å, C—H(aromatic) = 0.93 Å, C—H(methylene) = 0.97 Å or C—H(methyl) = 0.96 Å] and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{C methyl})$.

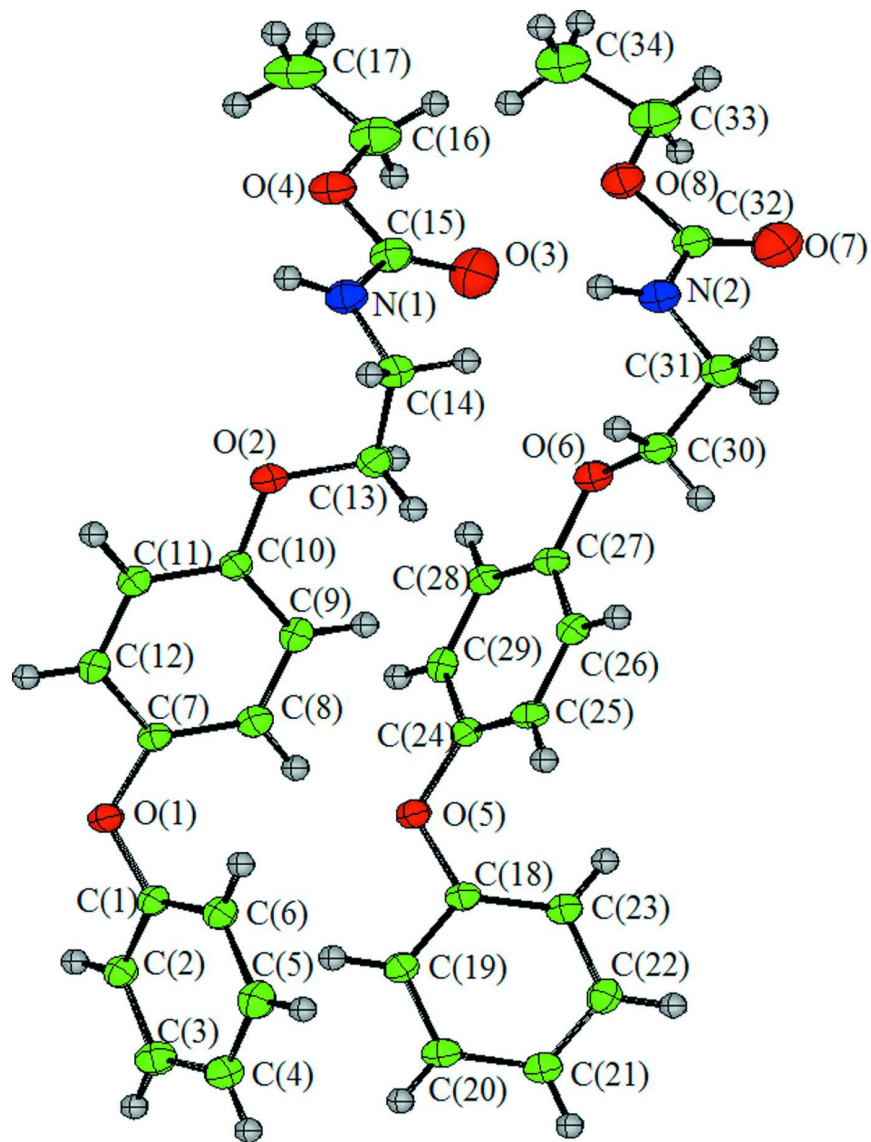


Figure 1

The atom numbering scheme for the two independent molecules in the asymmetric unit of fenoxycarb. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

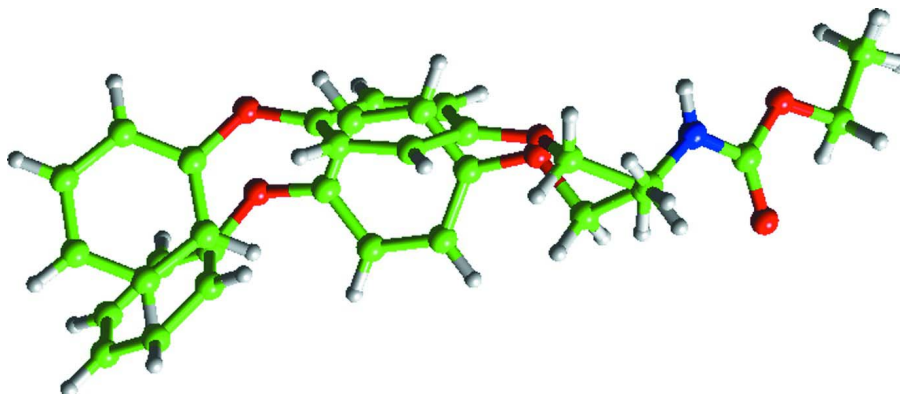


Figure 2

Molecular overlay of the two conformationally different fenoxycarb molecules.

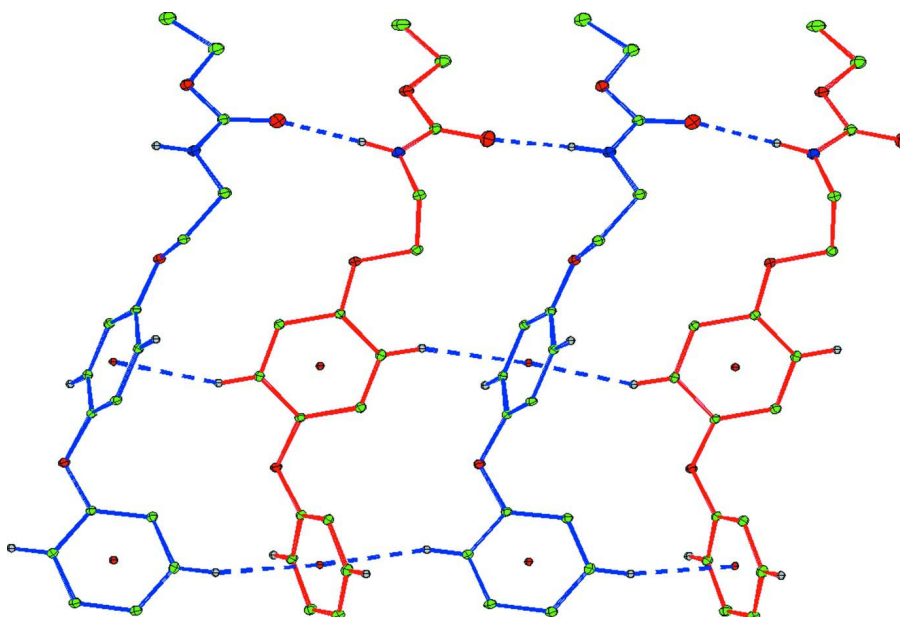


Figure 3

The one-dimensional hydrogen-bonded chains in the crystal structure of fenoxycarb.

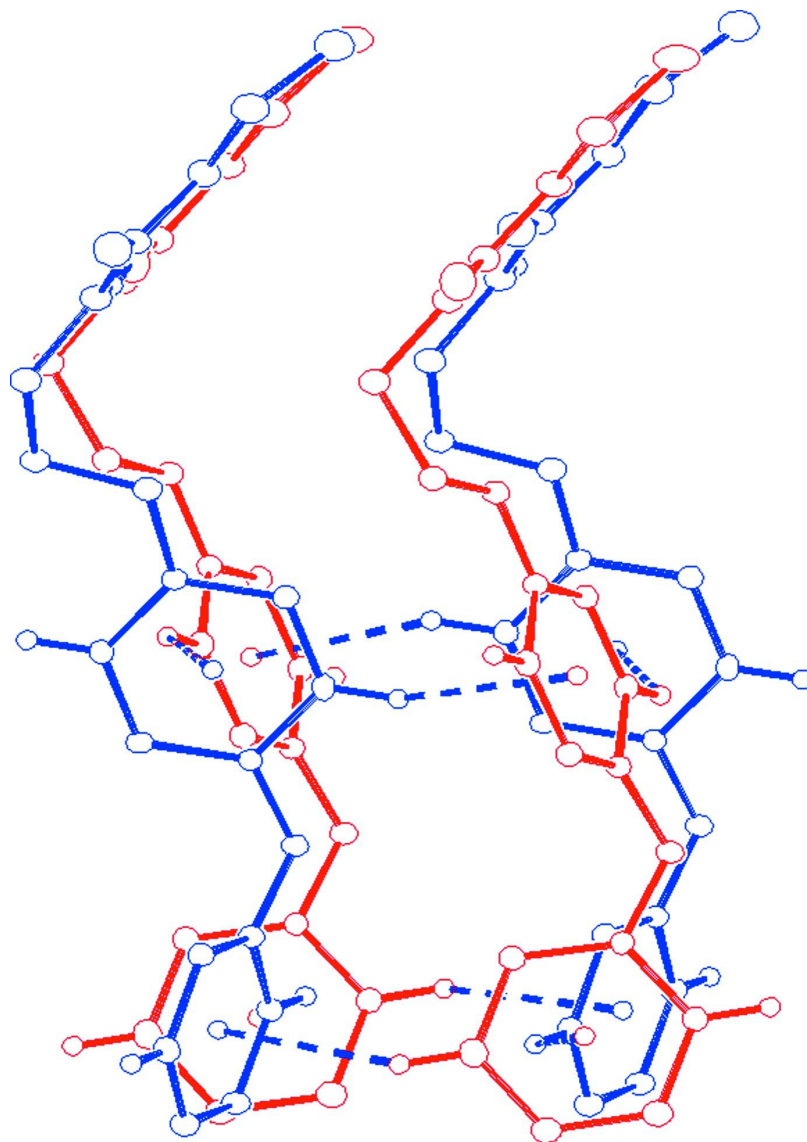


Figure 4

A view down the H-bonded chains showing C—H... π interactions.

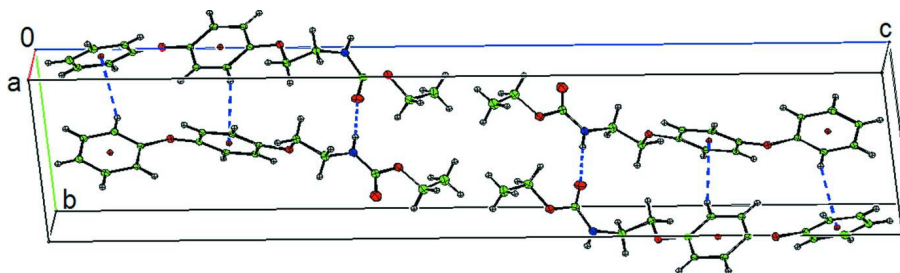


Figure 5

The unit cell packing of fenoxycarb viewed down *a*.

Ethyl *N*-[2-(4-phenoxyphenoxy)ethyl]carbamate

Crystal data

$C_{17}H_{19}NO_4$
 $M_r = 301.33$
 Triclinic, $P\bar{1}$
 Hall symbol: -P 1
 $a = 5.9035$ (3) Å
 $b = 7.5712$ (4) Å
 $c = 35.373$ (2) Å
 $\alpha = 85.618$ (5)°
 $\beta = 86.213$ (5)°
 $\gamma = 89.046$ (4)°
 $V = 1572.88$ (15) Å³

$Z = 4$
 $F(000) = 640$
 $D_x = 1.273$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
 Cell parameters from 2643 reflections
 $\theta = 2.8$ – 29.2 °
 $\mu = 0.09$ mm⁻¹
 $T = 150$ K
 Parallelepiped, colourless
 $0.40 \times 0.30 \times 0.15$ mm

Data collection

Oxford Diffraction Xcalibur, Sapphire3 CCD-detector
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 Graphite monochromator
 Detector resolution: 16.1048 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Oxford Diffraction, 2010)

$T_{\min} = 0.894$, $T_{\max} = 1.000$
 25379 measured reflections
 5745 independent reflections
 2937 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.074$
 $\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.8$ °
 $h = -6 \rightarrow 7$
 $k = -9 \rightarrow 9$
 $l = -42 \rightarrow 36$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.126$
 $S = 0.97$
 5745 reflections
 399 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0202P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.0999 (3)	0.0094 (3)	0.14727 (6)	0.0292 (6)
O2	0.2377 (3)	-0.0627 (3)	0.28990 (6)	0.0284 (6)
O4	0.2198 (4)	0.1319 (3)	0.41170 (6)	0.0397 (7)

N1	0.3603 (5)	-0.0324 (4)	0.36637 (8)	0.0341 (7)
H1N1	0.2510	-0.1030	0.3739	0.044*
O3	0.5364 (4)	0.2199 (3)	0.37656 (8)	0.0543 (8)
C1	0.0310 (5)	0.0155 (4)	0.11300 (9)	0.0229 (8)
C2	-0.0716 (5)	0.1035 (4)	0.08305 (9)	0.0260 (8)
H2	-0.2109	0.1606	0.0871	0.034*
C3	0.0335 (6)	0.1065 (4)	0.04699 (10)	0.0329 (9)
H3	-0.0349	0.1651	0.0266	0.043*
C4	0.2414 (6)	0.0216 (4)	0.04128 (10)	0.0330 (9)
H4	0.3129	0.0235	0.0170	0.043*
C5	0.3429 (6)	-0.0661 (4)	0.07175 (10)	0.0336 (9)
H5	0.4828	-0.1224	0.0679	0.044*
C6	0.2356 (5)	-0.0698 (4)	0.10793 (9)	0.0266 (8)
H6	0.3019	-0.1295	0.1284	0.035*
C7	0.0047 (5)	-0.0086 (4)	0.18145 (9)	0.0222 (8)
C8	0.1958 (5)	0.0809 (4)	0.18832 (9)	0.0268 (8)
H8	0.2701	0.1513	0.1688	0.035*
C9	0.2801 (5)	0.0672 (4)	0.22452 (9)	0.0270 (8)
H9	0.4104	0.1276	0.2290	0.035*
C10	0.1681 (5)	-0.0371 (4)	0.25359 (9)	0.0239 (8)
C11	-0.0258 (5)	-0.1266 (4)	0.24658 (9)	0.0241 (8)
H11	-0.1013	-0.1966	0.2660	0.031*
C12	-0.1071 (5)	-0.1122 (4)	0.21074 (9)	0.0246 (8)
H12	-0.2376	-0.1722	0.2062	0.032*
C13	0.4498 (5)	0.0105 (4)	0.29752 (9)	0.0306 (9)
H13A	0.4353	0.1378	0.2987	0.040*
H13B	0.5647	-0.0137	0.2776	0.040*
C14	0.5151 (5)	-0.0751 (4)	0.33510 (9)	0.0339 (9)
H14A	0.5202	-0.2026	0.3338	0.044*
H14B	0.6662	-0.0372	0.3398	0.044*
C15	0.3848 (6)	0.1153 (5)	0.38376 (10)	0.0326 (9)
C16	0.2336 (6)	0.2855 (5)	0.43345 (11)	0.0469 (11)
H16A	0.2367	0.3934	0.4168	0.061*
H16B	0.3700	0.2795	0.4474	0.061*
C17	0.0265 (7)	0.2828 (5)	0.46030 (10)	0.0620 (13)
H17A	-0.1068	0.2913	0.4461	0.087*
H17B	0.0295	0.3811	0.4758	0.087*
H17C	0.0239	0.1741	0.4762	0.087*
O5	0.4018 (3)	0.4830 (3)	0.14990 (6)	0.0274 (6)
O6	0.7396 (3)	0.4935 (3)	0.29179 (6)	0.0293 (6)
O8	0.6720 (4)	0.6213 (3)	0.41569 (7)	0.0368 (6)
N2	0.8499 (4)	0.4950 (4)	0.36737 (8)	0.0337 (7)
H2N2	0.7451	0.4168	0.3709	0.044*
O7	0.9987 (4)	0.7375 (3)	0.38924 (7)	0.0562 (8)
C18	0.5324 (5)	0.5007 (4)	0.11550 (9)	0.0236 (8)
C19	0.4372 (5)	0.4303 (4)	0.08561 (9)	0.0256 (8)
H19	0.3003	0.3704	0.0894	0.033*
C20	0.5461 (5)	0.4492 (4)	0.05006 (9)	0.0285 (8)

H20	0.4841	0.3999	0.0298	0.037*
C21	0.7470 (5)	0.5407 (4)	0.04410 (9)	0.0310 (9)
H21	0.8186	0.5553	0.0199	0.040*
C22	0.8410 (5)	0.6104 (4)	0.07428 (9)	0.0296 (9)
H22	0.9779	0.6702	0.0704	0.039*
C23	0.7322 (5)	0.5919 (4)	0.11049 (9)	0.0243 (8)
H23	0.7938	0.6404	0.1308	0.032*
C24	0.5056 (5)	0.4846 (4)	0.18424 (9)	0.0218 (8)
C25	0.6998 (5)	0.3895 (4)	0.19176 (9)	0.0251 (8)
H25	0.7755	0.3277	0.1729	0.033*
C26	0.7829 (5)	0.3860 (4)	0.22775 (9)	0.0241 (8)
H26	0.9151	0.3227	0.2329	0.031*
C27	0.6686 (5)	0.4770 (4)	0.25594 (9)	0.0226 (8)
C28	0.4693 (5)	0.5692 (4)	0.24816 (9)	0.0252 (8)
H28	0.3905	0.6286	0.2671	0.033*
C29	0.3880 (5)	0.5730 (4)	0.21237 (9)	0.0240 (8)
H29	0.2548	0.6348	0.2072	0.031*
C30	0.9338 (5)	0.3936 (4)	0.30377 (9)	0.0307 (9)
H30A	1.0497	0.3917	0.2830	0.040*
H30B	0.8916	0.2726	0.3118	0.040*
C31	1.0216 (5)	0.4823 (5)	0.33638 (9)	0.0366 (9)
H31A	1.1511	0.4156	0.3456	0.048*
H31B	1.0726	0.6003	0.3275	0.048*
C32	0.8522 (6)	0.6256 (5)	0.39035 (10)	0.0343 (9)
C33	0.6632 (6)	0.7599 (5)	0.44188 (10)	0.0467 (11)
H33A	0.6760	0.8752	0.4280	0.061*
H33B	0.7870	0.7452	0.4586	0.061*
C34	0.4383 (6)	0.7456 (5)	0.46465 (10)	0.0549 (12)
H34A	0.3173	0.7534	0.4477	0.077*
H34B	0.4230	0.8402	0.4813	0.077*
H34C	0.4314	0.6339	0.4795	0.077*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0267 (13)	0.0378 (15)	0.0230 (14)	0.0023 (11)	-0.0034 (10)	-0.0012 (11)
O2	0.0311 (14)	0.0302 (14)	0.0238 (14)	-0.0025 (11)	-0.0039 (10)	0.0008 (11)
O4	0.0506 (17)	0.0392 (16)	0.0299 (16)	-0.0070 (13)	0.0029 (13)	-0.0109 (12)
N1	0.0404 (19)	0.0306 (18)	0.0310 (19)	-0.0116 (15)	0.0027 (14)	-0.0018 (14)
O3	0.0543 (18)	0.0398 (17)	0.069 (2)	-0.0240 (15)	0.0118 (15)	-0.0133 (15)
C1	0.0230 (19)	0.0206 (18)	0.025 (2)	0.0017 (15)	-0.0006 (15)	-0.0030 (15)
C2	0.026 (2)	0.0239 (19)	0.028 (2)	0.0029 (16)	-0.0054 (15)	-0.0003 (16)
C3	0.040 (2)	0.028 (2)	0.031 (2)	-0.0004 (18)	-0.0102 (17)	0.0003 (17)
C4	0.035 (2)	0.034 (2)	0.030 (2)	-0.0045 (18)	0.0020 (17)	-0.0077 (17)
C5	0.032 (2)	0.030 (2)	0.040 (2)	0.0057 (17)	-0.0042 (18)	-0.0065 (18)
C6	0.027 (2)	0.0239 (19)	0.029 (2)	0.0033 (16)	-0.0048 (16)	-0.0033 (16)
C7	0.027 (2)	0.0185 (18)	0.0217 (19)	0.0059 (15)	-0.0040 (15)	-0.0054 (15)
C8	0.031 (2)	0.0200 (19)	0.028 (2)	0.0001 (16)	0.0019 (15)	0.0026 (16)

C9	0.027 (2)	0.0198 (19)	0.033 (2)	0.0002 (16)	-0.0024 (16)	0.0009 (16)
C10	0.025 (2)	0.0218 (19)	0.024 (2)	0.0077 (16)	-0.0020 (15)	-0.0018 (15)
C11	0.027 (2)	0.0189 (18)	0.026 (2)	0.0015 (15)	0.0045 (15)	-0.0020 (15)
C12	0.0228 (19)	0.0198 (19)	0.031 (2)	0.0023 (15)	0.0022 (15)	-0.0040 (16)
C13	0.028 (2)	0.036 (2)	0.028 (2)	-0.0001 (17)	-0.0012 (15)	-0.0051 (17)
C14	0.037 (2)	0.036 (2)	0.029 (2)	0.0015 (18)	-0.0076 (17)	-0.0024 (18)
C15	0.040 (2)	0.026 (2)	0.032 (2)	-0.0031 (19)	-0.0043 (18)	0.0004 (17)
C16	0.065 (3)	0.037 (2)	0.041 (3)	0.005 (2)	-0.012 (2)	-0.014 (2)
C17	0.088 (4)	0.064 (3)	0.033 (3)	0.016 (3)	0.001 (2)	-0.008 (2)
O5	0.0264 (13)	0.0333 (14)	0.0226 (14)	-0.0003 (11)	-0.0027 (10)	-0.0021 (11)
O6	0.0344 (14)	0.0284 (14)	0.0259 (14)	0.0044 (11)	-0.0061 (10)	-0.0040 (11)
O8	0.0417 (16)	0.0343 (15)	0.0355 (16)	-0.0025 (13)	-0.0021 (12)	-0.0097 (12)
N2	0.0393 (19)	0.0333 (18)	0.0290 (19)	-0.0151 (15)	-0.0020 (14)	-0.0027 (15)
O7	0.0579 (18)	0.0566 (19)	0.056 (2)	-0.0336 (16)	0.0018 (14)	-0.0151 (15)
C18	0.029 (2)	0.0190 (19)	0.022 (2)	0.0051 (16)	-0.0019 (15)	0.0034 (15)
C19	0.027 (2)	0.0247 (19)	0.025 (2)	-0.0003 (16)	-0.0047 (15)	0.0028 (16)
C20	0.037 (2)	0.027 (2)	0.023 (2)	0.0020 (17)	-0.0100 (16)	-0.0037 (16)
C21	0.035 (2)	0.030 (2)	0.026 (2)	0.0056 (18)	0.0002 (16)	0.0062 (17)
C22	0.027 (2)	0.027 (2)	0.034 (2)	0.0011 (16)	0.0000 (16)	0.0015 (17)
C23	0.031 (2)	0.0177 (18)	0.025 (2)	0.0017 (16)	-0.0052 (15)	-0.0056 (15)
C24	0.0215 (19)	0.0222 (18)	0.0219 (19)	-0.0054 (15)	-0.0032 (15)	-0.0008 (15)
C25	0.033 (2)	0.0215 (19)	0.0206 (19)	0.0036 (16)	0.0019 (15)	-0.0045 (15)
C26	0.026 (2)	0.0194 (18)	0.027 (2)	0.0048 (15)	-0.0021 (15)	-0.0029 (15)
C27	0.031 (2)	0.0164 (18)	0.0206 (19)	-0.0041 (15)	-0.0032 (15)	0.0014 (14)
C28	0.028 (2)	0.0211 (19)	0.026 (2)	0.0004 (16)	0.0031 (15)	-0.0048 (15)
C29	0.0223 (19)	0.0155 (18)	0.034 (2)	0.0015 (15)	-0.0011 (15)	-0.0030 (15)
C30	0.033 (2)	0.035 (2)	0.025 (2)	0.0031 (17)	-0.0075 (16)	0.0015 (17)
C31	0.035 (2)	0.046 (2)	0.029 (2)	-0.0075 (19)	-0.0038 (17)	-0.0005 (18)
C32	0.035 (2)	0.039 (2)	0.028 (2)	-0.005 (2)	-0.0076 (18)	0.0056 (18)
C33	0.061 (3)	0.043 (3)	0.040 (3)	0.003 (2)	-0.013 (2)	-0.018 (2)
C34	0.066 (3)	0.056 (3)	0.044 (3)	0.014 (2)	-0.005 (2)	-0.011 (2)

Geometric parameters (Å, °)

O1—C7	1.390 (3)	O5—C18	1.396 (3)
O1—C1	1.392 (3)	O5—C24	1.397 (3)
O2—C10	1.373 (3)	O6—C27	1.377 (3)
O2—C13	1.428 (3)	O6—C30	1.432 (3)
O4—C15	1.352 (4)	O8—C32	1.344 (4)
O4—C16	1.449 (4)	O8—C33	1.450 (4)
N1—C15	1.331 (4)	N2—C32	1.328 (4)
N1—C14	1.440 (4)	N2—C31	1.450 (4)
N1—H1N1	0.8600	N2—H2N2	0.8600
O3—C15	1.206 (4)	O7—C32	1.218 (4)
C1—C6	1.368 (4)	C18—C23	1.372 (4)
C1—C2	1.377 (4)	C18—C19	1.376 (4)
C2—C3	1.380 (4)	C19—C20	1.373 (4)
C2—H2	0.9300	C19—H19	0.9300

C3—C4	1.386 (4)	C20—C21	1.380 (4)
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.387 (4)	C21—C22	1.378 (4)
C4—H4	0.9300	C21—H21	0.9300
C5—C6	1.389 (4)	C22—C23	1.393 (4)
C5—H5	0.9300	C22—H22	0.9300
C6—H6	0.9300	C23—H23	0.9300
C7—C8	1.370 (4)	C24—C25	1.374 (4)
C7—C12	1.388 (4)	C24—C29	1.384 (4)
C8—C9	1.400 (4)	C25—C26	1.392 (4)
C8—H8	0.9300	C25—H25	0.9300
C9—C10	1.386 (4)	C26—C27	1.387 (4)
C9—H9	0.9300	C26—H26	0.9300
C10—C11	1.386 (4)	C27—C28	1.391 (4)
C11—C12	1.381 (4)	C28—C29	1.381 (4)
C11—H11	0.9300	C28—H28	0.9300
C12—H12	0.9300	C29—H29	0.9300
C13—C14	1.504 (4)	C30—C31	1.503 (4)
C13—H13A	0.9700	C30—H30A	0.9700
C13—H13B	0.9700	C30—H30B	0.9700
C14—H14A	0.9700	C31—H31A	0.9700
C14—H14B	0.9700	C31—H31B	0.9700
C16—C17	1.497 (5)	C33—C34	1.508 (5)
C16—H16A	0.9700	C33—H33A	0.9700
C16—H16B	0.9700	C33—H33B	0.9700
C17—H17A	0.9600	C34—H34A	0.9600
C17—H17B	0.9600	C34—H34B	0.9600
C17—H17C	0.9600	C34—H34C	0.9600
C7—O1—C1	120.0 (2)	C18—O5—C24	120.2 (2)
C10—O2—C13	117.9 (2)	C27—O6—C30	119.1 (2)
C15—O4—C16	116.0 (3)	C32—O8—C33	115.3 (3)
C15—N1—C14	120.6 (3)	C32—N2—C31	121.0 (3)
C15—N1—H1N1	119.7	C32—N2—H2N2	119.5
C14—N1—H1N1	119.7	C31—N2—H2N2	119.5
C6—C1—C2	121.6 (3)	C23—C18—C19	121.5 (3)
C6—C1—O1	124.1 (3)	C23—C18—O5	123.6 (3)
C2—C1—O1	114.1 (3)	C19—C18—O5	114.8 (3)
C1—C2—C3	119.5 (3)	C20—C19—C18	119.4 (3)
C1—C2—H2	120.2	C20—C19—H19	120.3
C3—C2—H2	120.2	C18—C19—H19	120.3
C2—C3—C4	119.8 (3)	C19—C20—C21	120.5 (3)
C2—C3—H3	120.1	C19—C20—H20	119.8
C4—C3—H3	120.1	C21—C20—H20	119.8
C3—C4—C5	120.0 (3)	C22—C21—C20	119.6 (3)
C3—C4—H4	120.0	C22—C21—H21	120.2
C5—C4—H4	120.0	C20—C21—H21	120.2
C4—C5—C6	120.0 (3)	C21—C22—C23	120.4 (3)

C4—C5—H5	120.0	C21—C22—H22	119.8
C6—C5—H5	120.0	C23—C22—H22	119.8
C1—C6—C5	119.0 (3)	C18—C23—C22	118.6 (3)
C1—C6—H6	120.5	C18—C23—H23	120.7
C5—C6—H6	120.5	C22—C23—H23	120.7
C8—C7—C12	119.6 (3)	C25—C24—C29	120.7 (3)
C8—C7—O1	123.9 (3)	C25—C24—O5	123.0 (3)
C12—C7—O1	116.2 (3)	C29—C24—O5	116.0 (3)
C7—C8—C9	120.5 (3)	C24—C25—C26	119.7 (3)
C7—C8—H8	119.7	C24—C25—H25	120.1
C9—C8—H8	119.7	C26—C25—H25	120.1
C10—C9—C8	119.5 (3)	C27—C26—C25	120.0 (3)
C10—C9—H9	120.2	C27—C26—H26	120.0
C8—C9—H9	120.2	C25—C26—H26	120.0
O2—C10—C11	115.7 (3)	O6—C27—C26	125.9 (3)
O2—C10—C9	124.5 (3)	O6—C27—C28	114.5 (3)
C11—C10—C9	119.8 (3)	C26—C27—C28	119.6 (3)
C12—C11—C10	120.1 (3)	C29—C28—C27	120.2 (3)
C12—C11—H11	119.9	C29—C28—H28	119.9
C10—C11—H11	119.9	C27—C28—H28	119.9
C11—C12—C7	120.4 (3)	C28—C29—C24	119.8 (3)
C11—C12—H12	119.8	C28—C29—H29	120.1
C7—C12—H12	119.8	C24—C29—H29	120.1
O2—C13—C14	107.1 (3)	O6—C30—C31	107.3 (3)
O2—C13—H13A	110.3	O6—C30—H30A	110.3
C14—C13—H13A	110.3	C31—C30—H30A	110.3
O2—C13—H13B	110.3	O6—C30—H30B	110.3
C14—C13—H13B	110.3	C31—C30—H30B	110.3
H13A—C13—H13B	108.6	H30A—C30—H30B	108.5
N1—C14—C13	113.0 (3)	N2—C31—C30	112.0 (3)
N1—C14—H14A	109.0	N2—C31—H31A	109.2
C13—C14—H14A	109.0	C30—C31—H31A	109.2
N1—C14—H14B	109.0	N2—C31—H31B	109.2
C13—C14—H14B	109.0	C30—C31—H31B	109.2
H14A—C14—H14B	107.8	H31A—C31—H31B	107.9
O3—C15—N1	125.2 (4)	O7—C32—N2	125.0 (4)
O3—C15—O4	123.6 (3)	O7—C32—O8	122.8 (4)
N1—C15—O4	111.1 (3)	N2—C32—O8	112.2 (3)
O4—C16—C17	106.3 (3)	O8—C33—C34	107.3 (3)
O4—C16—H16A	110.5	O8—C33—H33A	110.2
C17—C16—H16A	110.5	C34—C33—H33A	110.2
O4—C16—H16B	110.5	O8—C33—H33B	110.2
C17—C16—H16B	110.5	C34—C33—H33B	110.2
H16A—C16—H16B	108.7	H33A—C33—H33B	108.5
C16—C17—H17A	109.5	C33—C34—H34A	109.5
C16—C17—H17B	109.5	C33—C34—H34B	109.5
H17A—C17—H17B	109.5	H34A—C34—H34B	109.5
C16—C17—H17C	109.5	C33—C34—H34C	109.5

H17A—C17—H17C	109.5	H34A—C34—H34C	109.5
H17B—C17—H17C	109.5	H34B—C34—H34C	109.5
C7—O1—C1—C6	30.9 (4)	C24—O5—C18—C23	-29.0 (4)
C7—O1—C1—C2	-154.4 (3)	C24—O5—C18—C19	155.6 (3)
C6—C1—C2—C3	-0.1 (5)	C23—C18—C19—C20	1.0 (5)
O1—C1—C2—C3	-174.9 (3)	O5—C18—C19—C20	176.5 (3)
C1—C2—C3—C4	-0.3 (5)	C18—C19—C20—C21	-1.2 (5)
C2—C3—C4—C5	0.1 (5)	C19—C20—C21—C22	1.3 (5)
C3—C4—C5—C6	0.4 (5)	C20—C21—C22—C23	-1.3 (5)
C2—C1—C6—C5	0.6 (5)	C19—C18—C23—C22	-0.9 (4)
O1—C1—C6—C5	174.9 (3)	O5—C18—C23—C22	-176.0 (3)
C4—C5—C6—C1	-0.7 (5)	C21—C22—C23—C18	1.0 (5)
C1—O1—C7—C8	42.6 (4)	C18—O5—C24—C25	-45.6 (4)
C1—O1—C7—C12	-143.3 (3)	C18—O5—C24—C29	141.2 (3)
C12—C7—C8—C9	0.6 (5)	C29—C24—C25—C26	-1.8 (5)
O1—C7—C8—C9	174.5 (3)	O5—C24—C25—C26	-174.7 (3)
C7—C8—C9—C10	-0.4 (5)	C24—C25—C26—C27	0.6 (5)
C13—O2—C10—C11	173.2 (3)	C30—O6—C27—C26	-7.7 (4)
C13—O2—C10—C9	-5.6 (4)	C30—O6—C27—C28	175.5 (3)
C8—C9—C10—O2	178.8 (3)	C25—C26—C27—O6	-175.7 (3)
C8—C9—C10—C11	0.0 (4)	C25—C26—C27—C28	0.9 (5)
O2—C10—C11—C12	-178.8 (3)	O6—C27—C28—C29	175.8 (3)
C9—C10—C11—C12	0.0 (5)	C26—C27—C28—C29	-1.2 (4)
C10—C11—C12—C7	0.2 (5)	C27—C28—C29—C24	0.0 (5)
C8—C7—C12—C11	-0.6 (5)	C25—C24—C29—C28	1.5 (5)
O1—C7—C12—C11	-174.9 (3)	O5—C24—C29—C28	174.9 (3)
C10—O2—C13—C14	-165.2 (3)	C27—O6—C30—C31	160.0 (3)
C15—N1—C14—C13	-85.2 (4)	C32—N2—C31—C30	-151.2 (3)
O2—C13—C14—N1	-64.6 (4)	O6—C30—C31—N2	57.3 (4)
C14—N1—C15—O3	-3.0 (5)	C31—N2—C32—O7	-4.1 (5)
C14—N1—C15—O4	178.8 (3)	C31—N2—C32—O8	176.0 (3)
C16—O4—C15—O3	-0.2 (5)	C33—O8—C32—O7	0.0 (5)
C16—O4—C15—N1	178.1 (3)	C33—O8—C32—N2	179.9 (3)
C15—O4—C16—C17	175.8 (3)	C32—O8—C33—C34	173.6 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1, Cg2, Cg3 and Cg4 are the centroids of the C1—C6, C7—C12, C18—C23 and C24—C29 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N1 \cdots O7 ⁱ	0.86	1.96	2.815 (4)	178
N2—H2N2 \cdots O3	0.86	1.94	2.795 (4)	173
C2—H2 \cdots Cg3 ⁱⁱ	0.93	2.85	3.562 (3)	134
C5—H5 \cdots Cg3 ⁱⁱⁱ	0.93	2.85	3.562 (3)	134
C9—H9 \cdots Cg4	0.93	2.86	3.622 (3)	139
C12—H12 \cdots Cg4 ⁱ	0.93	2.84	3.590 (3)	138
C19—H19 \cdots Cg1	0.93	2.92	3.657 (3)	137
C22—H22 \cdots Cg1 ^{iv}	0.93	2.84	3.583 (3)	137

C26—H26...Cg2 ^v	0.93	2.87	3.579 (3)	134
C29—H29...Cg2 ^{vi}	0.93	2.80	3.527 (3)	135

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