Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# Crystal structure of (*Z*)-*N*-benzylidene-1-phenylmethanamine oxide hydrogen peroxide monosolvate

**Andrei V. Churakov,* Petr V. Prikhodchenko, Alexander G. Medvedev and Alexey A. Mikhaylov**

Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii prospekt 31, Moscow 119991, Russian Federation. *Correspondence e-mail: churakov@igic.ras.ru

Received 29 September 2017
Accepted 7 October 2017

Edited by G. Smith, Queensland University of Technology, Australia

**Keywords:** crystal structure; peroxosolvate; *N*-oxide; nitrone; hydrogen-bond motif.

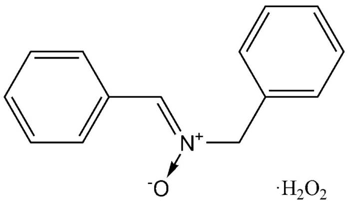
**CCDC reference**: 1578615

**Supporting information**: this article has supporting information at journals.iucr.org/e

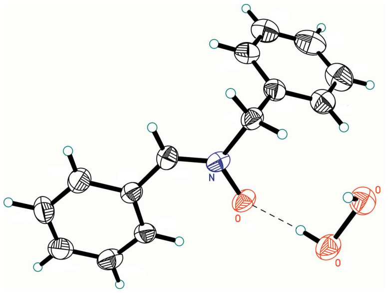
The title adduct, $C_{14}H_{13}NO{\cdot}H_2O_2$, consists of (*Z*)-*N*-benzylidene-1-phenylmethanamine oxide and hydrogen peroxide molecules in a 1:1 ratio. The organic coformer adopts a skew geometry with an inter-aryl-ring dihedral angle of 81.9 (2)°. In the crystal, the organic and peroxide molecules are linked through both peroxide O—H donor groups to oxide O-atom acceptors, giving one-dimensional chains extending along the *b* axis. Present also are weak intermolecular C—H⋯O hydrogen-bonding interactions.

## 1. Chemical context

Peroxosolvates are solid adducts that contain hydrogen peroxide molecules of crystallization in the same manner as the water in crystalline hydrates. Today, some of these are widely used as environmentally friendly bleaching compounds (Jakob *et al.*, 2012) and oxidizing agents in organic synthesis (Ahn *et al.*, 2015). Hydrogen bonding in peroxosolvates is of particular interest since it may be used for modelling of hydrogen peroxide behaviour in various significant biochemical processes (Kapustin *et al.*, 2014).



It is known that nitrones $R^1$–CH=N(O)–$R^2$ [$R^1$, $R^2$ = aryl (Ar) or alkyl (Alk)] are readily available by oxidation of secondary amines using hydrogen peroxide (Goti *et al.*, 2005). We supposed that the combination of oxidizing and cocrystallizing properties of hydrogen peroxide might afford an opportunity to obtain nitrone peroxosolvates in one step. We prepared the title 1:1 adduct of (*Z*)-*N*-benzylidene-1-phenylmethanamine oxide with hydrogen peroxide and the structure is reported herein.



## 2. Structural commentary

In the structure of the title adduct (Fig. 1), all bond lengths and angles in the organic coformer exhibit normal values for nitrone derivatives (Cambridge Structural Database, Version

https://doi.org/10.1107/S2056989017014499

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—H1$\cdots$O3 | 1.05 (5) | 1.66 (5) | 2.707 (5) | 174 (4) |
| O2—H2$\cdots$O3$^{i}$ | 1.06 (5) | 1.64 (5) | 2.681 (5) | 166 (4) |
| C21—H21$\cdots$O1$^{ii}$ | 0.95 | 2.46 | 3.304 (6) | 148 |
| C27—H27$\cdots$O3 | 0.95 | 2.29 | 2.902 (6) | 121 |
| C11—H111$\cdots$O1$^{ii}$ | 0.99 | 2.44 | 3.364 (7) | 155 |
| C11—H111$\cdots$O2$^{ii}$ | 0.99 | 2.47 | 3.394 (7) | 155 |
| C11—H112$\cdots$O2 | 0.99 | 2.52 | 3.407 (7) | 149 |

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

5.38, May 2017; Groom *et al.*, 2016). The nitrone fragment Ph—CH═N(O)—C is planar to within 0.128 (3) Å. It is almost perpendicular to the benzyl substituent C11–C17, with an O3—N1—C11—C12 torsion angle of 72.7 (4)°, and forms a dihedral angle between the two aryl rings of 81.9 (2)°. This is the same conformation as was previously observed in the structure of the pure coformer (Herrera *et al.*, 2001). The organic molecule forms two hydrogen bonds, involving the negatively charged oxide atom O3, with adjacent peroxide molecules and the conformation is stabilized by an aromatic C27—H$\cdots$O3 hydrogen bond (Table 1). As expected, the N1—O3$\cdots$O(peroxo) angles are close to trigonal [117.9 (2) and 126.2 (2)°].

In the peroxide molecule, the O—O distance is 1.467 (4) Å. This value is close to those previously observed in the accurately determined structures of crystalline hydrogen peroxide [1.461 (3) Å; Savariault *et al.*, 1980] and urea perhydrate [1.4573 (8) Å; Fritchie & McMullan, 1981]. Partial substitutional disorder of hydrogen peroxide by water molecules (Pedersen, 1972) was not observed in the present structure since no residual peaks with an intensity of 0.14 e Å$^{-3}$ were seen in the hydrogen peroxide molecule region (Churakov *et al.*, 2005). The $H_2O_2$ molecule lies on a general position and presents a skew geometry, with the H—O—O—H torsion angle equal to 88 (4)°, and forms just two donor hydrogen bonds. It should be noted that the maximum possible number of hydrogen bonds formed by $H_2O_2$ is six (two donor and four acceptor; Chernyshov *et al.*, 2017).

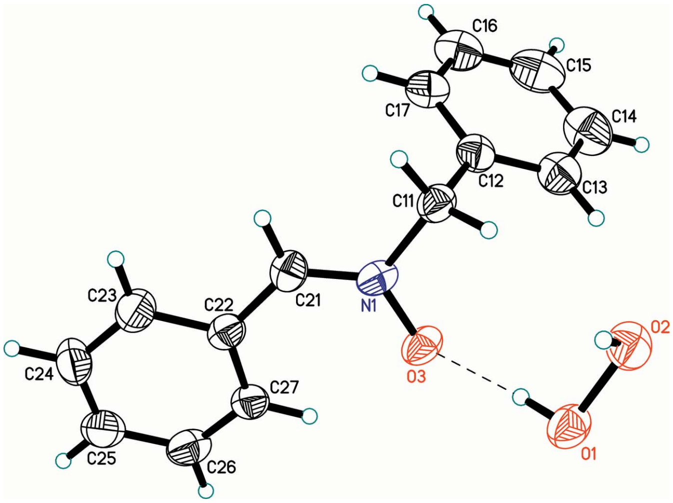


**Figure 1**
The asymmetric unit in the title structure. Displacement ellipsoids are shown at the 50% probability level and the hydrogen bond is drawn as a dashed line.

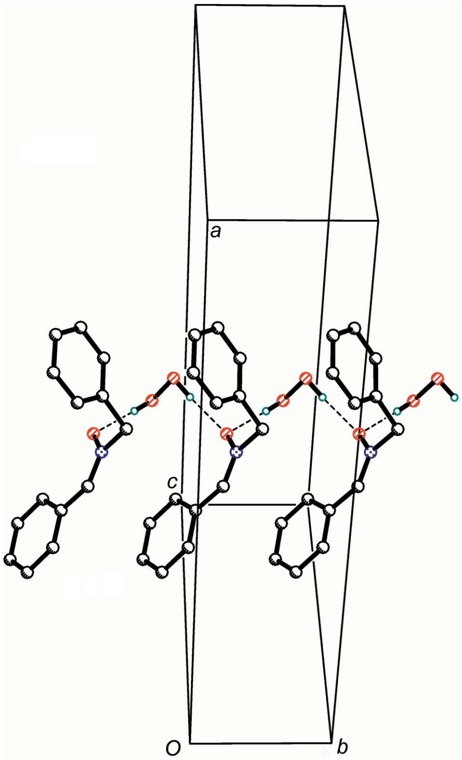


**Figure 2**
Hydrogen-bonded chains extending along the *b* axis. H atoms on C atoms have been omitted for clarity. Hydrogen bonds are drawn as dashed lines.

## 3. Supramolecular features

In the title crystal, the organic and peroxide molecules are linked into hydrogen-bonded chains extending along the *b* axis through charge-supported moderate $\text{HOOH}\cdots{}^{-}\text{O—N}$ hydrogen bonds, with O$\cdots$O separations of 2.707 (5) and 2.681 (5) Å (Table 1 and Fig. 2). Similar chains formed by *N*-oxide and $H_2O_2$ molecules were previously observed in the structure of risperidone *N*-oxide hydrogen peroxide methanol solvate (Ravikumar *et al.*, 2005). In the present one-dimensional structure, minor weak non-aromatic C—H$\cdots$O(peroxide) hydrogen-bonding interactions are also present.

## 4. Database survey

The Cambridge Structural Database (Groom *et al.*, 2016) contains data for nine peroxosolvates of *N*- and *P*-oxides with one or two $R_3X^+ \rightarrow O^-$ functional groups ($X$ = N, P; $R$ = Alk, Ar). It is of interest that all of these were obtained by oxidation of the corresponding amines (phosphines) using hydrogen peroxide, followed by immediate crystallization from the reaction mixtures. Analysis of the crystal packing for these compounds reveals three main supramolecular motifs

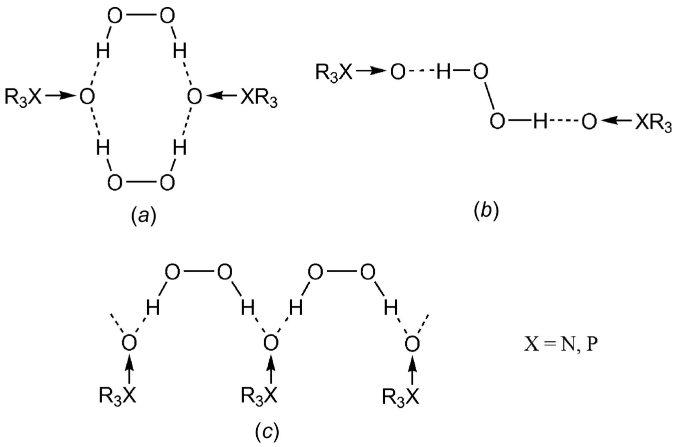


**Figure 3**
Hydrogen-bonded motifs in the structures of *N*- and *P*-oxides.

(Fig. 3*a*–3*c*). Compounds BAFGOH (Ahn *et al.*, 2015), BAFJUQ (Ahn *et al.*, 2015), VANVOX (Hilliard *et al.*, 2012) and XETSUK (Čermák *et al.*, 2001) belong to type *a* $[R_4^2(10)]$; compounds EKULUR (Chandrasekaran *et al.*, 2002), TPPOPH (Thierbach *et al.*, 1980) and UKEFEV (Sevcik *et al.*, 2003) represent type *b* $[D_2^2(6)]$. Finally, the title compound and DATHIQ (Ravikumar *et al.*, 2005) are of type *c* $[C_2^1(5)]$. The relative simplicity of these motifs is caused by the absence of active H atoms in coformers of the aforementioned compounds. The special case is the three-dimensional structure of triethylenediamine *N*,*N*′-dioxide triperoxosolvate (FURFIH; Kay Hon & Mak, 1987).

## 5. Synthesis and crystallization

Needle-shaped crystals of the title compound crystallized spontaneously from a saturated solution of dibenzylamine in 50% hydrogen peroxide after holding for 3 d at room temperature. **Caution!** Handling procedures for concentrated hydrogen peroxide (danger of explosion) are described in detail by Wolanov *et al.* (2010).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Peroxide H atoms were found from a difference electron-density map and refined with individual isotropic displacement parameters and restrained O—H distances. All other H atoms were placed in calculated positions, with C—H = 0.95 (aromatic) or 0.99 Å (methylene), and treated as riding atoms, with relative isotropic displacement parameters $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

## Acknowledgements

X-ray diffraction studies were performed at the Centre of Shared Equipment of IGIC RAS. Publication was supported by the Federal Agency of Scientific Organizations within the State Assignment on Fundamental Research to the Kurnakov Institute of General and Inorganic Chemistry.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{14}H_{13}NO\cdot H_2O_2$ |
| $M_r$ | 245.27 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 150 |
| $a$, $b$, $c$ (Å) | 21.802 (15), 4.597 (3), 12.742 (9) |
| $\beta$ (°) | 97.598 (11) |
| $V$ (Å$^3$) | 1265.8 (16) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.09 |
| Crystal size (mm) | 0.40 × 0.04 × 0.04 |
| Data collection | |
| Diffractometer | Bruker *SMART* APEXII area-detector |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2008) |
| $T_{\rm min}$, $T_{\rm max}$ | 0.965, 0.996 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 7458, 2227, 1113 |
| $R_{\rm int}$ | 0.108 |
| $(\sin \theta/\lambda)_{\rm max}$ (Å$^{-1}$) | 0.596 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.077, 0.218, 1.05 |
| No. of reflections | 2227 |
| No. of parameters | 172 |
| No. of restraints | 1 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å$^{-3}$) | 0.27, −0.26 |

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008).

## References

Ahn, S. H., Cluff, K. J., Bhuvanesh, N. & Blümel, J. (2015). *Angew. Chem. Int. Ed.* **54**, 13341–13345.

Bruker (2008). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Čermák, J., Kvíčalová, M., Šabata, S., Blechta, V., Vojtíšek, P., Podlaha, J. & Shaw, B. L. (2001). *Inorg. Chim. Acta*, **313**, 77–86.

Chandrasekaran, A., Timosheva, N. V., Day, R. O. & Holmes, R. R. (2002). *Inorg. Chem.* **41**, 5235–5240.

Chernyshov, I. Yu., Vener, M. V., Prikhodchenko, P. V., Medvedev, A. G., Lev, O. & Churakov, A. V. (2017). *Cryst. Growth Des.* **17**, 214–220.

Churakov, A. V., Prikhodchenko, P. V. & Howard, J. A. K. (2005). *CrystEngComm*, **7**, 664–669.

Fritchie, C. J. & McMullan, R. K. (1981). *Acta Cryst.* B**37**, 1086–1091.

Goti, A., Cardona, F. & Soldaini, G. (2005). *Org. Synth.* **81**, 204–212.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Herrera, R., Nagarajan, A., Morales, M. A., Méndez, F., Jiménez-Vázquez, H. A., Zepeda, L. G. & Tamariz, J. (2001). *J. Org. Chem.* **66**, 1252–1263.

Hilliard, C. R., Bhuvanesh, N. J., Gladysz, A. & Blümel, J. (2012). *Dalton Trans.* **41**, 1742–1754.

Jakob, H., Leininger, S., Lehmann, T., Jacobi, S. & Gutewort, S. (2012). *Ullmann's Encyclopedia of Industrial Chemistry*, pp. 1–33. Weinheim: Wiley-VCH Verlag GmbH & Co. KGaA.

Kapustin, E. A., Minkov, V. S. & Boldyreva, E. V. (2014). *CrystEngComm*, **16**, 10165–10168.

Kay Hon, P. & Mak, T. C. W. (1987). *J. Crystallogr. Spectrosc. Res.* **17**, 419–429.

Pedersen, B. F. (1972). *Acta Cryst.* B**28**, 746–754.

Ravikumar, K., Sridhar, B., Manjunatha, S. G. & Thomas, S. (2005). *Acta Cryst.* E**61**, o2515–o2517.

Savariault, J. & Lehmann, M. S. (1980). *J. Am. Chem. Soc.* **102**, 1298–1303.

Sevcik, R., Necas, M. & Novosad, J. (2003). *Polyhedron*, **22**, 1585–1593.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Thierbach, D., Huber, F. & Preut, H. (1980). *Acta Cryst.* B**36**, 974–977.

Wolanov, Y., Lev, O., Churakov, A. V., Medvedev, A. G., Novotortsev, V. M. & Prikhodchenko, P. V. (2010). *Tetrahedron*, **66**, 5130–5133.

# supporting information

*Acta Cryst.* (2017). E**73**, 1666-1669 [https://doi.org/10.1107/S2056989017014499]

## Crystal structure of (*Z*)-*N*-benzylidene-1-phenylmethanamine oxide hydrogen peroxide monosolvate

**Andrei V. Churakov, Petr V. Prikhodchenko, Alexander G. Medvedev and Alexey A. Mikhaylov**

### Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

### (*Z*)-*N*-Benzylidene-1-phenylmethanamine oxide hydrogen peroxide monosolvate

*Crystal data*

$C_{14}H_{13}NO\cdot H_2O_2$
$M_r = 245.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 21.802$ (15) Å
$b = 4.597$ (3) Å
$c = 12.742$ (9) Å
$\beta = 97.598$ (11)°
$V = 1265.8$ (16) Å$^3$
$Z = 4$

$F(000) = 520$
$D_x = 1.287$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 737 reflections
$\theta = 3.2–21.9$°
$\mu = 0.09$ mm$^{-1}$
$T = 150$ K
Needle, colourless
0.40 × 0.04 × 0.04 mm

*Data collection*

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{\min} = 0.965$, $T_{\max} = 0.996$

7458 measured reflections
2227 independent reflections
1113 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.108$
$\theta_{\max} = 25.1$°, $\theta_{\min} = 1.9$°
$h = -25 \rightarrow 25$
$k = -5 \rightarrow 5$
$l = -15 \rightarrow 14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.218$
$S = 1.05$
2227 reflections
172 parameters
1 restraint

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.034 (6)

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.23321 (15) | 0.7522 (7) | 1.0077 (3) | 0.0427 (9) |
| O2 | 0.28942 (16) | 0.9116 (7) | 0.9911 (3) | 0.0442 (10) |
| O3 | 0.22608 (15) | 0.3093 (6) | 0.8666 (2) | 0.0377 (9) |
| N1 | 0.23366 (18) | 0.3713 (7) | 0.7676 (3) | 0.0319 (10) |
| C11 | 0.2883 (2) | 0.5529 (9) | 0.7569 (4) | 0.0342 (12) |
| H112 | 0.2894 | 0.7210 | 0.8057 | 0.041* |
| H111 | 0.2855 | 0.6284 | 0.6836 | 0.041* |
| C12 | 0.3463 (2) | 0.3775 (9) | 0.7821 (4) | 0.0344 (12) |
| C13 | 0.3802 (2) | 0.3866 (10) | 0.8826 (4) | 0.0438 (13) |
| H13 | 0.3672 | 0.5083 | 0.9356 | 0.053* |
| C14 | 0.4332 (2) | 0.2183 (12) | 0.9054 (5) | 0.0573 (16) |
| H14 | 0.4563 | 0.2229 | 0.9741 | 0.069* |
| C15 | 0.4521 (3) | 0.0431 (12) | 0.8270 (5) | 0.0592 (17) |
| H15 | 0.4888 | −0.0694 | 0.8422 | 0.071* |
| C16 | 0.4190 (2) | 0.0295 (12) | 0.7281 (5) | 0.0521 (15) |
| H16 | 0.4317 | −0.0952 | 0.6756 | 0.063* |
| C17 | 0.3671 (2) | 0.1997 (10) | 0.7062 (4) | 0.0423 (13) |
| H17 | 0.3447 | 0.1954 | 0.6370 | 0.051* |
| C21 | 0.1988 (2) | 0.2713 (9) | 0.6851 (4) | 0.0334 (11) |
| H21 | 0.2083 | 0.3366 | 0.6184 | 0.040* |
| C22 | 0.1470 (2) | 0.0714 (9) | 0.6825 (4) | 0.0330 (12) |
| C23 | 0.1155 (2) | 0.0141 (11) | 0.5825 (4) | 0.0432 (13) |
| H23 | 0.1280 | 0.1064 | 0.5221 | 0.052* |
| C24 | 0.0658 (2) | −0.1774 (11) | 0.5704 (4) | 0.0480 (14) |
| H24 | 0.0438 | −0.2129 | 0.5022 | 0.058* |
| C25 | 0.0486 (2) | −0.3157 (11) | 0.6579 (4) | 0.0479 (14) |
| H25 | 0.0149 | −0.4481 | 0.6499 | 0.057* |
| C26 | 0.0800 (2) | −0.2629 (11) | 0.7565 (4) | 0.0443 (14) |
| H26 | 0.0681 | −0.3606 | 0.8164 | 0.053* |
| C27 | 0.1284 (2) | −0.0708 (9) | 0.7696 (4) | 0.0356 (12) |
| H27 | 0.1495 | −0.0341 | 0.8384 | 0.043* |
| H1 | 0.232 (2) | 0.588 (9) | 0.950 (4) | 0.060 (15)* |

| | | | | |
|---|---|---|---|---|
| H2 | 0.268 (2) | 1.058 (10) | 0.933 (4) | 0.057 (15)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.054 (2) | 0.0379 (19) | 0.038 (2) | −0.0100 (17) | 0.0135 (17) | −0.0039 (15) |
| O2 | 0.050 (2) | 0.0394 (19) | 0.044 (2) | −0.0075 (17) | 0.0069 (18) | −0.0001 (16) |
| O3 | 0.052 (2) | 0.0361 (17) | 0.026 (2) | −0.0051 (15) | 0.0106 (16) | 0.0026 (14) |
| N1 | 0.041 (2) | 0.027 (2) | 0.029 (2) | 0.0001 (18) | 0.012 (2) | −0.0011 (17) |
| C11 | 0.041 (3) | 0.033 (2) | 0.029 (3) | −0.005 (2) | 0.008 (2) | −0.001 (2) |
| C12 | 0.038 (3) | 0.030 (2) | 0.036 (3) | −0.007 (2) | 0.006 (2) | 0.004 (2) |
| C13 | 0.043 (3) | 0.045 (3) | 0.044 (4) | 0.000 (3) | 0.005 (3) | 0.005 (2) |
| C14 | 0.047 (3) | 0.064 (4) | 0.058 (4) | 0.003 (3) | −0.002 (3) | 0.019 (3) |
| C15 | 0.052 (4) | 0.048 (3) | 0.080 (5) | 0.004 (3) | 0.020 (4) | 0.016 (3) |
| C16 | 0.044 (3) | 0.054 (3) | 0.060 (4) | 0.004 (3) | 0.014 (3) | 0.006 (3) |
| C17 | 0.042 (3) | 0.043 (3) | 0.043 (3) | −0.002 (3) | 0.012 (3) | 0.004 (2) |
| C21 | 0.038 (3) | 0.031 (2) | 0.031 (3) | 0.004 (2) | 0.001 (2) | 0.001 (2) |
| C22 | 0.035 (3) | 0.034 (2) | 0.030 (3) | 0.004 (2) | 0.007 (2) | 0.000 (2) |
| C23 | 0.046 (3) | 0.047 (3) | 0.035 (3) | 0.000 (3) | 0.004 (3) | −0.003 (2) |
| C24 | 0.039 (3) | 0.056 (3) | 0.046 (4) | −0.005 (3) | −0.005 (3) | −0.007 (3) |
| C25 | 0.042 (3) | 0.051 (3) | 0.053 (4) | −0.008 (3) | 0.016 (3) | −0.013 (3) |
| C26 | 0.051 (3) | 0.042 (3) | 0.043 (3) | −0.002 (3) | 0.017 (3) | −0.005 (2) |
| C27 | 0.033 (3) | 0.038 (3) | 0.037 (3) | −0.001 (2) | 0.009 (2) | −0.004 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—O2 | 1.467 (4) | C16—C17 | 1.375 (7) |
| O1—H1 | 1.05 (4) | C16—H16 | 0.9500 |
| O2—H2 | 1.06 (4) | C17—H17 | 0.9500 |
| O3—N1 | 1.325 (4) | C21—C22 | 1.454 (6) |
| N1—C21 | 1.297 (6) | C21—H21 | 0.9500 |
| N1—C11 | 1.475 (6) | C22—C23 | 1.390 (6) |
| C11—C12 | 1.498 (6) | C22—C27 | 1.393 (6) |
| C11—H112 | 0.9900 | C23—C24 | 1.388 (7) |
| C11—H111 | 0.9900 | C23—H23 | 0.9500 |
| C12—C17 | 1.388 (6) | C24—C25 | 1.378 (7) |
| C12—C13 | 1.392 (7) | C24—H24 | 0.9500 |
| C13—C14 | 1.388 (7) | C25—C26 | 1.371 (7) |
| C13—H13 | 0.9500 | C25—H25 | 0.9500 |
| C14—C15 | 1.389 (8) | C26—C27 | 1.368 (7) |
| C14—H14 | 0.9500 | C26—H26 | 0.9500 |
| C15—C16 | 1.368 (8) | C27—H27 | 0.9500 |
| C15—H15 | 0.9500 | | |
| | | | |
| O2—O1—H1 | 102 (3) | C17—C16—H16 | 120.6 |
| O1—O2—H2 | 97 (3) | C16—C17—C12 | 122.0 (5) |
| C21—N1—O3 | 124.2 (4) | C16—C17—H17 | 119.0 |
| C21—N1—C11 | 121.3 (4) | C12—C17—H17 | 119.0 |

| | | | |
|---|---|---|---|
| O3—N1—C11 | 114.4 (4) | N1—C21—C22 | 127.8 (4) |
| N1—C11—C12 | 110.1 (3) | N1—C21—H21 | 116.1 |
| N1—C11—H112 | 109.6 | C22—C21—H21 | 116.1 |
| C12—C11—H112 | 109.6 | C23—C22—C27 | 118.8 (4) |
| N1—C11—H111 | 109.6 | C23—C22—C21 | 115.4 (4) |
| C12—C11—H111 | 109.6 | C27—C22—C21 | 125.8 (5) |
| H112—C11—H111 | 108.1 | C24—C23—C22 | 120.3 (5) |
| C17—C12—C13 | 118.4 (5) | C24—C23—H23 | 119.8 |
| C17—C12—C11 | 120.8 (5) | C22—C23—H23 | 119.8 |
| C13—C12—C11 | 120.9 (4) | C25—C24—C23 | 119.6 (5) |
| C14—C13—C12 | 120.2 (5) | C25—C24—H24 | 120.2 |
| C14—C13—H13 | 119.9 | C23—C24—H24 | 120.2 |
| C12—C13—H13 | 119.9 | C26—C25—C24 | 120.2 (5) |
| C13—C14—C15 | 119.3 (6) | C26—C25—H25 | 119.9 |
| C13—C14—H14 | 120.3 | C24—C25—H25 | 119.9 |
| C15—C14—H14 | 120.3 | C27—C26—C25 | 120.6 (5) |
| C16—C15—C14 | 121.3 (6) | C27—C26—H26 | 119.7 |
| C16—C15—H15 | 119.4 | C25—C26—H26 | 119.7 |
| C14—C15—H15 | 119.4 | C26—C27—C22 | 120.4 (5) |
| C15—C16—C17 | 118.8 (5) | C26—C27—H27 | 119.8 |
| C15—C16—H16 | 120.6 | C22—C27—H27 | 119.8 |

| | | | |
|---|---|---|---|
| C21—N1—C11—C12 | −103.9 (5) | C11—N1—C21—C22 | 174.4 (4) |
| O3—N1—C11—C12 | 72.7 (4) | N1—C21—C22—C23 | 176.4 (4) |
| N1—C11—C12—C17 | 82.7 (5) | N1—C21—C22—C27 | −6.0 (7) |
| N1—C11—C12—C13 | −96.6 (5) | C27—C22—C23—C24 | 1.2 (7) |
| C17—C12—C13—C14 | −0.7 (7) | C21—C22—C23—C24 | 179.0 (4) |
| C11—C12—C13—C14 | 178.7 (4) | C22—C23—C24—C25 | −1.4 (7) |
| C12—C13—C14—C15 | 0.5 (7) | C23—C24—C25—C26 | 0.5 (7) |
| C13—C14—C15—C16 | −1.1 (8) | C24—C25—C26—C27 | 0.6 (7) |
| C14—C15—C16—C17 | 1.8 (8) | C25—C26—C27—C22 | −0.8 (7) |
| C15—C16—C17—C12 | −2.0 (7) | C23—C22—C27—C26 | −0.1 (7) |
| C13—C12—C17—C16 | 1.4 (7) | C21—C22—C27—C26 | −177.6 (4) |
| C11—C12—C17—C16 | −178.0 (4) | H1—O1—O2—H2 | 88 (4) |
| O3—N1—C21—C22 | −1.8 (7) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O3 | 1.05 (5) | 1.66 (5) | 2.707 (5) | 174 (4) |
| O2—H2···O3$^{i}$ | 1.06 (5) | 1.64 (5) | 2.681 (5) | 166 (4) |
| C21—H21···O1$^{ii}$ | 0.95 | 2.46 | 3.304 (6) | 148 |
| C27—H27···O3 | 0.95 | 2.29 | 2.902 (6) | 121 |
| C11—H111···O1$^{ii}$ | 0.99 | 2.44 | 3.364 (7) | 155 |
| C11—H111···O2$^{ii}$ | 0.99 | 2.47 | 3.394 (7) | 155 |
| C11—H112···O2 | 0.99 | 2.52 | 3.407 (7) | 149 |

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