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(2*R*,3*R*)-3-*O*-Benzoyl-*N*-benzyl-tartramide¹

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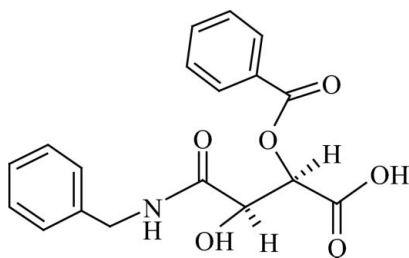
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.026; wR factor = 0.071; data-to-parameter ratio = 12.5.

The title compound, $\text{C}_{18}\text{H}_{17}\text{NO}_6$ [systematic name: (2*R*,3*R*)-4-benzylamino-2-benzoyloxy-3-hydroxy-4-oxobutanoic acid], is the first structurally characterized unsymmetrical monoamide–monoacyl tartaric acid derivative. The molecule shows a staggered conformation around the tartramide $\text{Csp}^3-\text{Csp}^3$ bond with *trans*-oriented carboxyl and amide groups. The molecular conformation is stabilized by an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond. In the crystal, molecules are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the carboxyl and amide carbonyl groups, forming translational chains along [001]. Further $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds as well as weaker $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ intermolecular interactions extend the supramolecular assembly into a double-layer structure parallel to (100). There are no directional interactions between the double layers.

Related literature

For crystal structures of *R,R*-tartaric mono amides, see: Rychlewska *et al.* (1999); Rychlewska & Warzajtis (2000, 2001). For examples of the crystal structures of monoacyl derivatives, see: Madura *et al.* (2010); Knyazev *et al.* (1988); Chekhlov *et al.* (1986); Ishihara *et al.* (1993). For the synthesis, see: Bell (1987); Bernaś *et al.* (2010).


¹ Tartaric acid and its *O*-acyl derivatives. Part 13.

Experimental

Crystal data

| | |
|---|-----------------------------------|
| $\text{C}_{18}\text{H}_{17}\text{NO}_6$ | $V = 1649.12$ (5) Å ³ |
| $M_r = 343.33$ | $Z = 4$ |
| Monoclinic, C_2 | Cu $K\alpha$ radiation |
| $a = 35.7118$ (6) Å | $\mu = 0.88$ mm ⁻¹ |
| $b = 6.17734$ (11) Å | $T = 100$ K |
| $c = 7.48599$ (15) Å | $0.54 \times 0.22 \times 0.18$ mm |
| $\beta = 93.0377$ (15)° | |

Data collection

| | |
|---|--|
| Agilent Gemini A Ultra diffractometer | 29440 measured reflections |
| Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2009) | 2945 independent reflections |
| $T_{\min} = 0.724$, $T_{\max} = 1.000$ | 2928 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.038$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.026$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.071$ | $\Delta\rho_{\text{max}} = 0.19$ e Å ⁻³ |
| $S = 1.06$ | $\Delta\rho_{\text{min}} = -0.17$ e Å ⁻³ |
| 2945 reflections | Absolute structure: Flack (1983), 1321 Friedel pairs |
| 235 parameters | Flack parameter: -0.01 (13) |
| 1 restraint | |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O1}-\text{H1}\cdots\text{O5}^{\text{i}}$ | 0.99 (2) | 1.55 (2) | 2.5317 (13) | 171.2 (17) |
| $\text{O4}-\text{H4}\cdots\text{O2}^{\text{ii}}$ | 0.81 (2) | 1.96 (2) | 2.7501 (14) | 165 (2) |
| $\text{N1}-\text{H1A}\cdots\text{O6}^{\text{iii}}$ | 0.88 (2) | 2.002 (19) | 2.7788 (17) | 146.4 (18) |
| $\text{N1}-\text{H1A}\cdots\text{O4}$ | 0.88 (2) | 2.12 (2) | 2.5894 (14) | 112.8 (15) |
| $\text{C12}-\text{H12A}\cdots\text{O1}^{\text{iv}}$ | 0.99 | 2.52 | 3.4827 (16) | 165 |
| $\text{C12}-\text{H12B}\cdots\text{O1}^{\text{v}}$ | 0.99 | 2.44 | 3.3117 (16) | 146 |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); 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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2491).

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(2*R*,3*R*)-3-*O*-Benzoyl-*N*-benzyltartramide

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S1. Comment

The title molecule crystallizes in non-centrosymmetric *C*2 space group as *R,R* enantiomer. Molecular structure with the atom numbering scheme is presented in Fig. 1. Similarly to the previously characterized tartaric acid mono amides (Rychlewska *et al.*, 1999; Rychlewska & Warżajtis, 2000, 2001), the title molecule adopts the staggered conformation around the C2–C3 bond (Fig. 2.). Thus, the carboxylic group is in *trans* (*T*) orientation with respect to the amide group, whereas the hydroxy and benzoyl substituents adopt the *gauche* counterclockwise orientation. The conformation on C–C* bond in amide fragment enables the formation of an intramolecular N–H···O hydrogen bond between N–H donor and the hydroxyl group. Hence the carbonyl group is on the opposite side of the proximal C*–O bond. Such orientation, called antiplanar (*a*) is also observed in the carboxylic fragment, and the overall conformation of molecules can be given as *T(a,a)*. It is worth noting that in case of dibenzoyl tartaric mono amides (Rychlewska & Warżajtis, 2001) or those with unsubstituted OH groups (Rychlewska *et al.*, 1999; Rychlewska & Warżajtis, 2000) the conformation of the acid fragment is such that the carbonyl group eclipses the nearest C–O bond (*synplanar* conformation, *s*), while the presence of at least one N–H bond forces the conformation of the amide fragment to be *antiplanar* with the intramolecular N–H···O bond.

The analysis of intermolecular interactions shows that the title molecules related by translation along [001] form infinite head-to-tail chains *via* hydrogen bonds between carboxylic OH donors and amide carbonyl groups. A topologically analogous chain motif was observed in the crystal structures of dibenzoyl mono amides by Rychlewska & Warżajtis (2001). Further, the O–H hydroxyl group acts as a donor to carboxyl C=O group joining the chains related by 2_1 screw axis into a double layer (Fig. 3). The layer is enhanced by N–H···O_{benzoyl carbonyl} as well as weaker C–H···O and C–H··· π intermolecular interactions. The neighbouring layers are held together by weak van der Waals forces only.

S2. Experimental

A (1:2 mol/mol) mixture of *O*-benzoyl-*L*-tartaric anhydride (Bernaś *et al.*, 2010) and benzylamine in acetonitrile was stirred at room temperature for 10 min. The mixture was then acidified with 10% HCl and filtered. The resulting white solid product was rinsed with water to give pure title compound with m.p. 465–467 K. Preparation and characterization of regioisomer of the title compound was described by Bell (1987), although its structure was defined incorrectly. $[\alpha]_D^{25} = +40.9^\circ$, (c 1, EtOH). IR (EtOH): $\nu = 693, 708$ (C=C, Ph); 1112, 1263 (C–O); 1663 (C=O, CONH); 1710 (C=O, COOH); 1723 (C=O, COBz) cm^{-1} . $^1\text{H NMR}$ (400 MHz, DMSO-*d*6): $\delta = 4.43\text{--}4.15$ (m, 2H), 4.62 (d, 1H), 5.53 (d, 1H), 6.42 (br), 6.81–6.91 (m, 5H), 7.42–7.99 (m, 5H), 8.69 (t, 1H) p.p.m.. $^{13}\text{C NMR}$ (400 MHz, DMSO-*d*6): $\delta = 41.87$ (CH₂), 71.33 (CH), 74.04 (CH), 126.44, 127.88, 128.76, 128.76, 129.06, 129.52, 133.71, 139.25 (Ph), 165.01, 168.93, 170.28 (C=O) p.p.m.. Anal. Calcd. (%) for C₁₈H₁₇NO₆: C 62.97; H 4.99; N 4.08. Found: C 62.92; H 4.99; N 4.11. Crystals suitable for single-crystal X-ray diffraction measurement were obtained from saturated ethyl acetate/methanol (3:1).

S3. Refinement

The position of the H atoms attached to O and N atoms were freely refined with $U_{\text{iso}}(\text{H}) = 1.2 \times U_{\text{eq}}(\text{N})$ and $U_{\text{iso}}(\text{H}) = 1.5 \times U_{\text{eq}}(\text{O})$. Other H atoms were positioned geometrically and refined using a riding model with $\text{C—H} = 0.95\text{--}0.99 \text{ \AA}$ and with $U_{\text{iso}}(\text{H}) = 1.2 \times U_{\text{eq}}(\text{C})$. The absolute structure was assigned on the basis of anomalous dispersion that confirmed the known chirality of the reagent. The estimated number of measured Friedel pairs amounts to 1321 with the fraction of 0.813.

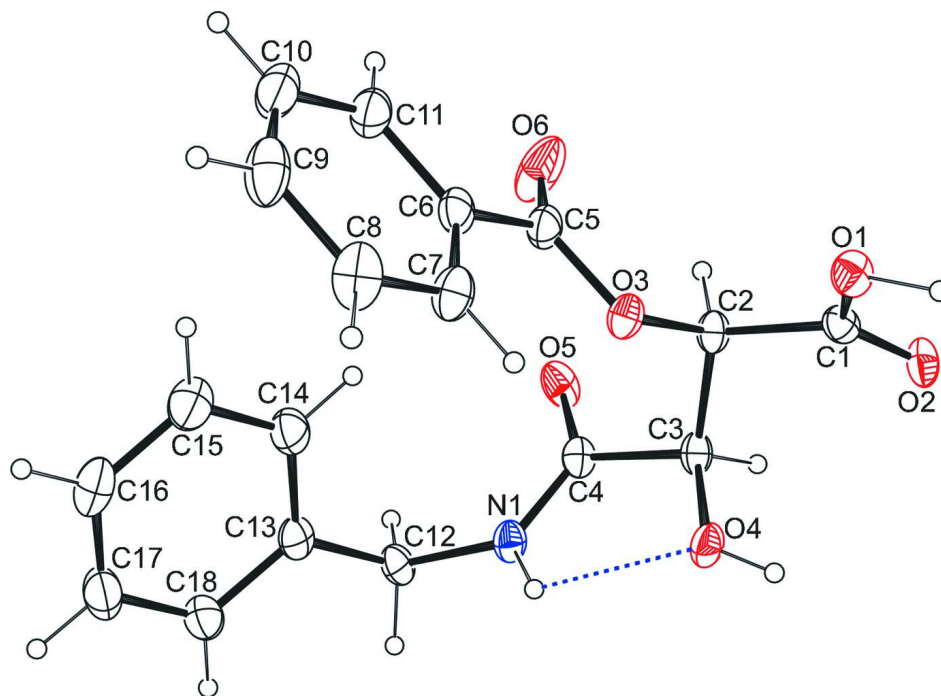


Figure 1

ORTEP plot of the molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are depicted as spheres with arbitrary radii. Dotted line indicates intramolecular hydrogen bond.

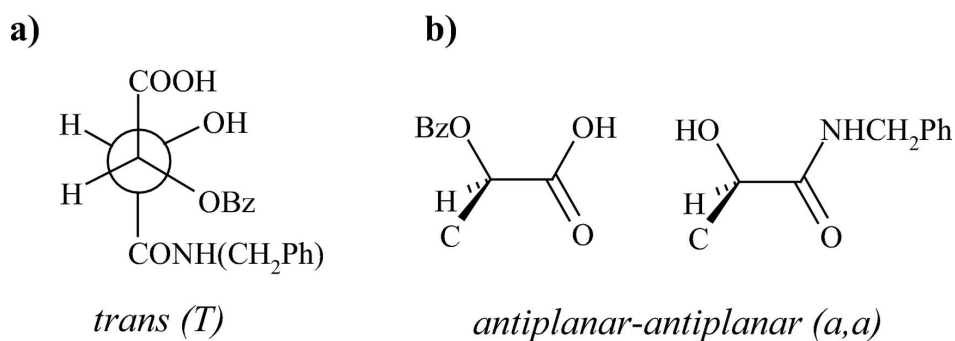


Figure 2

(a) The staggered conformation (*T*) around C2—C3 bond; (b) Antiplanar (*a*) conformations around C1—C2 and C4—C3 bonds.

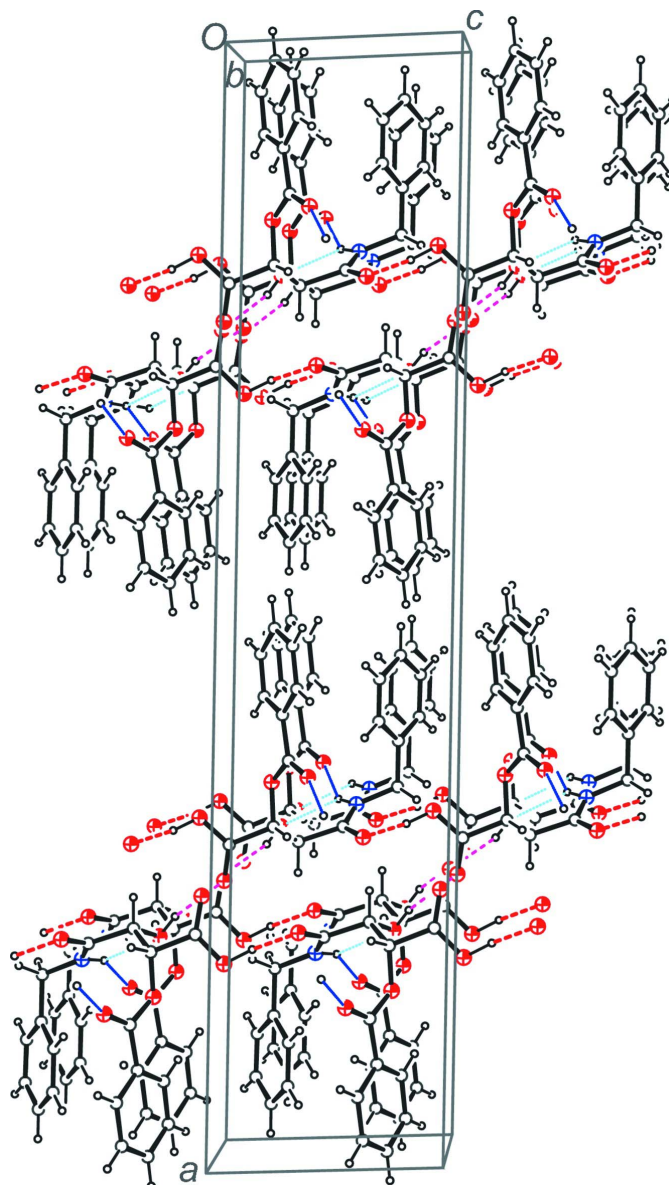


Figure 3

View along the [010] direction showing double-layers of molecules formed by two types of O—H...O (dashed lines) and N—H...O (dotted lines) bonds. Symmetry codes: (i) $x, y, z + 1$; (ii) $-x + 1/2, y + 1/2, -z + 2$; (iii) $x, y + 1, z$.

(2*R*,3*R*)-4-benzylamino-2-benzoyloxy-3-hydroxy-4-oxobutanoic acid

Crystal data

$C_{18}H_{17}NO_6$

$M_r = 343.33$

Monoclinic, $C2$

$a = 35.7118$ (6) Å

$b = 6.17734$ (11) Å

$c = 7.48599$ (15) Å

$\beta = 93.0377$ (15)°

$V = 1649.12$ (5) Å³

$Z = 4$

$F(000) = 720$

$D_x = 1.383$ Mg m⁻³

Melting point: 193 K

Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å

Cell parameters from 23556 reflections

$\theta = 5.0$ – 67.0 °

$\mu = 0.88$ mm⁻¹

$T = 100$ K $0.54 \times 0.22 \times 0.18$ mm
 Prism, colourless

Data collection

| | |
|--|--|
| Agilent Gemini A Ultra diffractometer | $T_{\min} = 0.724$, $T_{\max} = 1.000$ |
| Radiation source: Enhance Ultra (Cu) X-ray Source | 29440 measured reflections |
| Mirror monochromator | 2945 independent reflections |
| Detector resolution: 10.3347 pixels mm^{-1} | 2928 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.038$ |
| Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2009) | $\theta_{\max} = 67.1^\circ$, $\theta_{\min} = 5.0^\circ$ |
| | $h = -42 \rightarrow 42$ |
| | $k = -7 \rightarrow 7$ |
| | $l = -8 \rightarrow 8$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.026$ | $w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.6346P]$ |
| $wR(F^2) = 0.071$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{\max} = 0.001$ |
| 2945 reflections | $\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$ |
| 235 parameters | $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$ |
| 1 restraint | Absolute structure: Flack (1983), 1321 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Absolute structure parameter: -0.01 (13) |
| Secondary atom site location: difference Fourier map | |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| O1 | 0.31056 (3) | 0.28482 (16) | 1.07911 (12) | 0.0222 (2) |
| H1 | 0.2993 (5) | 0.315 (4) | 1.194 (3) | 0.046* |
| O2 | 0.25203 (3) | 0.35877 (17) | 0.97437 (12) | 0.0229 (2) |
| O3 | 0.33951 (2) | 0.36177 (15) | 0.77766 (11) | 0.0195 (2) |
| O4 | 0.29381 (3) | 0.72671 (16) | 0.76263 (12) | 0.0213 (2) |
| H4 | 0.2772 (6) | 0.764 (4) | 0.825 (3) | 0.046* |
| O5 | 0.28714 (3) | 0.38296 (17) | 0.38200 (11) | 0.0254 (2) |
| O6 | 0.34669 (3) | 0.0721 (2) | 0.60328 (18) | 0.0446 (3) |
| N1 | 0.31211 (3) | 0.7200 (2) | 0.43257 (14) | 0.0196 (2) |
| H1A | 0.3160 (5) | 0.818 (3) | 0.517 (3) | 0.037* |
| C1 | 0.28496 (3) | 0.3271 (2) | 0.95296 (16) | 0.0185 (3) |
| C2 | 0.29952 (3) | 0.3381 (2) | 0.76528 (16) | 0.0175 (3) |

| | | | | |
|------|-------------|------------|--------------|------------|
| H2 | 0.2924 | 0.2040 | 0.6969 | 0.021* |
| C3 | 0.28249 (3) | 0.5372 (2) | 0.67065 (16) | 0.0185 (3) |
| H3 | 0.2545 | 0.5262 | 0.6689 | 0.022* |
| C4 | 0.29469 (3) | 0.5431 (2) | 0.47812 (16) | 0.0176 (3) |
| C5 | 0.36003 (4) | 0.2140 (2) | 0.69603 (17) | 0.0217 (3) |
| C6 | 0.40098 (4) | 0.2478 (2) | 0.72820 (16) | 0.0220 (3) |
| C7 | 0.41579 (4) | 0.4261 (3) | 0.82131 (18) | 0.0256 (3) |
| H7 | 0.3996 | 0.5298 | 0.8704 | 0.031* |
| C8 | 0.45432 (4) | 0.4509 (3) | 0.8417 (2) | 0.0325 (4) |
| H8 | 0.4646 | 0.5732 | 0.9037 | 0.039* |
| C9 | 0.47800 (4) | 0.2982 (3) | 0.7721 (2) | 0.0341 (4) |
| H9 | 0.5044 | 0.3155 | 0.7874 | 0.041* |
| C10 | 0.46321 (4) | 0.1209 (3) | 0.6808 (2) | 0.0327 (3) |
| H10 | 0.4795 | 0.0163 | 0.6337 | 0.039* |
| C11 | 0.42477 (4) | 0.0950 (3) | 0.6574 (2) | 0.0275 (3) |
| H11 | 0.4147 | -0.0263 | 0.5935 | 0.033* |
| C12 | 0.32616 (4) | 0.7696 (2) | 0.25671 (16) | 0.0202 (3) |
| H12A | 0.3175 | 0.9160 | 0.2196 | 0.024* |
| H12B | 0.3154 | 0.6647 | 0.1680 | 0.024* |
| C13 | 0.36848 (4) | 0.7612 (2) | 0.25630 (16) | 0.0206 (3) |
| C14 | 0.38841 (4) | 0.5883 (2) | 0.33400 (19) | 0.0270 (3) |
| H14 | 0.3753 | 0.4735 | 0.3876 | 0.032* |
| C15 | 0.42720 (4) | 0.5825 (3) | 0.3336 (2) | 0.0310 (3) |
| H15 | 0.4406 | 0.4653 | 0.3885 | 0.037* |
| C16 | 0.44647 (4) | 0.7487 (3) | 0.25267 (19) | 0.0308 (3) |
| H16 | 0.4731 | 0.7458 | 0.2530 | 0.037* |
| C17 | 0.42679 (4) | 0.9175 (3) | 0.1720 (2) | 0.0317 (3) |
| H17 | 0.4399 | 1.0290 | 0.1141 | 0.038* |
| C18 | 0.38793 (4) | 0.9259 (2) | 0.17444 (19) | 0.0260 (3) |
| H18 | 0.3746 | 1.0441 | 0.1203 | 0.031* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0249 (4) | 0.0281 (5) | 0.0133 (4) | 0.0021 (4) | -0.0010 (3) | 0.0001 (4) |
| O2 | 0.0203 (4) | 0.0337 (5) | 0.0152 (4) | 0.0001 (4) | 0.0047 (3) | 0.0039 (4) |
| O3 | 0.0167 (4) | 0.0254 (5) | 0.0165 (4) | 0.0000 (4) | 0.0009 (3) | -0.0036 (4) |
| O4 | 0.0232 (4) | 0.0257 (5) | 0.0157 (4) | -0.0002 (4) | 0.0062 (3) | -0.0049 (4) |
| O5 | 0.0358 (5) | 0.0284 (5) | 0.0123 (4) | -0.0105 (4) | 0.0024 (4) | -0.0018 (4) |
| O6 | 0.0270 (5) | 0.0438 (7) | 0.0643 (8) | -0.0105 (5) | 0.0154 (5) | -0.0338 (6) |
| N1 | 0.0207 (5) | 0.0243 (6) | 0.0140 (5) | -0.0020 (5) | 0.0032 (4) | -0.0020 (5) |
| C1 | 0.0228 (6) | 0.0175 (6) | 0.0152 (6) | -0.0023 (5) | 0.0011 (5) | -0.0002 (5) |
| C2 | 0.0167 (6) | 0.0237 (6) | 0.0121 (6) | -0.0015 (5) | 0.0010 (4) | -0.0019 (5) |
| C3 | 0.0175 (6) | 0.0257 (7) | 0.0125 (6) | -0.0014 (6) | 0.0020 (4) | -0.0010 (5) |
| C4 | 0.0151 (5) | 0.0248 (6) | 0.0128 (6) | 0.0000 (5) | -0.0010 (4) | 0.0011 (5) |
| C5 | 0.0229 (6) | 0.0235 (7) | 0.0192 (6) | -0.0012 (6) | 0.0065 (5) | -0.0028 (6) |
| C6 | 0.0223 (6) | 0.0284 (8) | 0.0156 (6) | 0.0019 (6) | 0.0046 (5) | 0.0034 (5) |
| C7 | 0.0234 (7) | 0.0343 (8) | 0.0192 (6) | 0.0007 (6) | 0.0030 (5) | -0.0037 (6) |

| | | | | | | |
|-----|------------|-------------|------------|-------------|-------------|-------------|
| C8 | 0.0243 (7) | 0.0486 (10) | 0.0243 (7) | -0.0042 (7) | -0.0010 (6) | -0.0054 (7) |
| C9 | 0.0181 (6) | 0.0575 (11) | 0.0266 (7) | 0.0026 (7) | 0.0009 (5) | 0.0055 (7) |
| C10 | 0.0269 (8) | 0.0434 (9) | 0.0286 (8) | 0.0110 (7) | 0.0083 (6) | 0.0052 (7) |
| C11 | 0.0269 (7) | 0.0314 (8) | 0.0250 (7) | 0.0035 (6) | 0.0073 (6) | 0.0009 (6) |
| C12 | 0.0241 (6) | 0.0237 (7) | 0.0131 (6) | -0.0025 (5) | 0.0027 (5) | 0.0028 (5) |
| C13 | 0.0241 (6) | 0.0255 (7) | 0.0124 (5) | -0.0019 (5) | 0.0029 (5) | -0.0020 (5) |
| C14 | 0.0270 (7) | 0.0307 (8) | 0.0237 (7) | 0.0014 (6) | 0.0059 (5) | 0.0052 (6) |
| C15 | 0.0272 (7) | 0.0399 (9) | 0.0261 (7) | 0.0062 (6) | 0.0029 (6) | 0.0027 (6) |
| C16 | 0.0203 (6) | 0.0437 (9) | 0.0289 (7) | -0.0010 (6) | 0.0049 (5) | -0.0088 (7) |
| C17 | 0.0289 (8) | 0.0345 (8) | 0.0329 (8) | -0.0083 (6) | 0.0111 (6) | -0.0021 (7) |
| C18 | 0.0278 (7) | 0.0267 (7) | 0.0241 (7) | -0.0017 (6) | 0.0055 (6) | 0.0019 (6) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|-------------|-------------|
| O1—C1 | 1.3053 (15) | C8—H8 | 0.9500 |
| O1—H1 | 0.99 (2) | C9—C8 | 1.387 (2) |
| O2—C1 | 1.2111 (16) | C9—H9 | 0.9500 |
| O3—C2 | 1.4336 (14) | C10—C9 | 1.381 (3) |
| O3—C5 | 1.3384 (16) | C10—C11 | 1.384 (2) |
| O4—C3 | 1.4067 (17) | C10—H10 | 0.9500 |
| O4—H4 | 0.81 (2) | C11—H11 | 0.9500 |
| O5—C4 | 1.2442 (17) | C12—H12A | 0.9900 |
| O6—C5 | 1.2010 (18) | C12—H12B | 0.9900 |
| N1—C4 | 1.3115 (18) | C13—C12 | 1.5126 (17) |
| N1—C12 | 1.4659 (16) | C13—C18 | 1.3922 (19) |
| N1—H1A | 0.88 (2) | C14—C13 | 1.393 (2) |
| C2—C1 | 1.5253 (17) | C14—C15 | 1.386 (2) |
| C2—C3 | 1.5293 (18) | C14—H14 | 0.9500 |
| C2—H2 | 1.0000 | C15—C16 | 1.392 (2) |
| C3—C4 | 1.5280 (16) | C15—H15 | 0.9500 |
| C3—H3 | 1.0000 | C16—H16 | 0.9500 |
| C6—C5 | 1.4841 (18) | C17—C16 | 1.379 (2) |
| C6—C7 | 1.393 (2) | C17—H17 | 0.9500 |
| C6—C11 | 1.393 (2) | C18—H18 | 0.9500 |
| C7—C8 | 1.385 (2) | C18—C17 | 1.390 (2) |
| C7—H7 | 0.9500 | | |
| C1—O1—H1 | 106.9 (12) | C9—C8—H8 | 119.8 |
| C5—O3—C2 | 117.93 (10) | C8—C9—C10 | 120.05 (13) |
| C3—O4—H4 | 108.7 (16) | C8—C9—H9 | 120.0 |
| C4—N1—C12 | 126.69 (12) | C10—C9—H9 | 120.0 |
| C4—N1—H1A | 116.4 (13) | C9—C10—C11 | 120.34 (14) |
| C12—N1—H1A | 116.9 (13) | C9—C10—H10 | 119.8 |
| O1—C1—O2 | 125.75 (11) | C11—C10—H10 | 119.8 |
| O1—C1—C2 | 114.55 (10) | C6—C11—C10 | 119.61 (15) |
| O2—C1—C2 | 119.70 (11) | C6—C11—H11 | 120.2 |
| O3—C2—C1 | 109.39 (9) | C10—C11—H11 | 120.2 |
| O3—C2—C3 | 108.56 (10) | N1—C12—C13 | 112.59 (10) |

| | | | |
|---------------|--------------|-----------------|--------------|
| O3—C2—H2 | 110.1 | N1—C12—H12A | 109.1 |
| C1—C2—C3 | 108.41 (10) | N1—C12—H12B | 109.1 |
| C1—C2—H2 | 110.1 | C13—C12—H12A | 109.1 |
| C3—C2—H2 | 110.1 | C13—C12—H12B | 109.1 |
| O4—C3—C2 | 110.23 (9) | H12A—C12—H12B | 107.8 |
| O4—C3—C4 | 110.67 (10) | C14—C13—C12 | 120.92 (12) |
| O4—C3—H3 | 108.9 | C18—C13—C12 | 119.84 (12) |
| C2—C3—H3 | 108.9 | C18—C13—C14 | 119.23 (12) |
| C4—C3—C2 | 109.26 (10) | C13—C14—H14 | 119.8 |
| C4—C3—H3 | 108.9 | C15—C14—C13 | 120.48 (13) |
| O5—C4—N1 | 127.10 (11) | C15—C14—H14 | 119.8 |
| O5—C4—C3 | 117.56 (11) | C14—C15—C16 | 119.95 (14) |
| N1—C4—C3 | 115.34 (11) | C14—C15—H15 | 120.0 |
| O3—C5—O6 | 123.49 (12) | C16—C15—H15 | 120.0 |
| O3—C5—C6 | 112.86 (11) | C15—C16—H16 | 120.1 |
| O6—C5—C6 | 123.63 (12) | C17—C16—C15 | 119.71 (13) |
| C5—C6—C7 | 122.51 (12) | C17—C16—H16 | 120.1 |
| C5—C6—C11 | 117.27 (13) | C18—C17—H17 | 119.7 |
| C7—C6—C11 | 120.20 (13) | C16—C17—C18 | 120.58 (14) |
| C6—C7—C8 | 119.44 (14) | C16—C17—H17 | 119.7 |
| C6—C7—H7 | 120.3 | C13—C18—H18 | 120.0 |
| C8—C7—H7 | 120.3 | C17—C18—C13 | 120.01 (13) |
| C7—C8—C9 | 120.35 (15) | C17—C18—H18 | 120.0 |
| C7—C8—H8 | 119.8 | | |
| | | | |
| C5—O3—C2—C1 | 123.19 (12) | C11—C6—C5—O3 | 176.38 (12) |
| C5—O3—C2—C3 | -118.68 (11) | C11—C6—C5—O6 | -5.2 (2) |
| C2—O3—C5—O6 | 5.3 (2) | C11—C6—C7—C8 | 0.5 (2) |
| C2—O3—C5—C6 | -176.29 (10) | C5—C6—C7—C8 | -178.03 (13) |
| C12—N1—C4—C3 | -178.86 (11) | C5—C6—C11—C10 | 178.85 (13) |
| C12—N1—C4—O5 | 0.9 (2) | C7—C6—C11—C10 | 0.2 (2) |
| C4—N1—C12—C13 | -108.11 (14) | C6—C7—C8—C9 | -0.9 (2) |
| O3—C2—C1—O1 | -16.83 (15) | C10—C9—C8—C7 | 0.5 (2) |
| O3—C2—C1—O2 | 162.45 (12) | C11—C10—C9—C8 | 0.3 (2) |
| C3—C2—C1—O1 | -135.05 (11) | C9—C10—C11—C6 | -0.6 (2) |
| C3—C2—C1—O2 | 44.24 (16) | C14—C13—C12—N1 | 46.90 (17) |
| O3—C2—C3—O4 | -56.88 (12) | C18—C13—C12—N1 | -134.32 (13) |
| O3—C2—C3—C4 | 64.93 (12) | C12—C13—C18—C17 | -179.25 (12) |
| C1—C2—C3—O4 | 61.86 (12) | C14—C13—C18—C17 | -0.4 (2) |
| C1—C2—C3—C4 | -176.32 (10) | C15—C14—C13—C12 | -179.72 (13) |
| O4—C3—C4—O5 | 178.58 (11) | C15—C14—C13—C18 | 1.5 (2) |
| O4—C3—C4—N1 | -1.62 (15) | C13—C14—C15—C16 | -1.0 (2) |
| C2—C3—C4—O5 | 57.03 (14) | C14—C15—C16—C17 | -0.5 (2) |
| C2—C3—C4—N1 | -123.17 (12) | C18—C17—C16—C15 | 1.6 (2) |
| C7—C6—C5—O3 | -5.05 (18) | C13—C18—C17—C16 | -1.1 (2) |
| C7—C6—C5—O6 | 173.34 (14) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O1—H1 \cdots O5 ⁱ | 0.99 (2) | 1.55 (2) | 2.5317 (13) | 171.2 (17) |
| O4—H4 \cdots O2 ⁱⁱ | 0.81 (2) | 1.96 (2) | 2.7501 (14) | 165 (2) |
| N1—H1A \cdots O6 ⁱⁱⁱ | 0.88 (2) | 2.002 (19) | 2.7788 (17) | 146.4 (18) |
| N1—H1A \cdots O4 | 0.88 (2) | 2.12 (2) | 2.5894 (14) | 112.8 (15) |
| C2—H2 \cdots O6 | 1.00 | 2.25 | 2.6879 (17) | 105 |
| C12—H12A \cdots O1 ^{iv} | 0.99 | 2.52 | 3.4827 (16) | 165 |
| C12—H12B \cdots O1 ^v | 0.99 | 2.44 | 3.3117 (16) | 146 |

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