1,2-Bis(pyridin-4-yl)ethene–4-hydroxy-3-methoxy-benzoic acid (1/1)

Devin J. Angevine and Jason B. Benedict*

Department of Chemistry, The State University of New York at Buffalo, Buffalo, New York 14260-3000, USA.
*Correspondence e-mail: jbb6@buffalo.edu

In the title 1:1 co-crystal [alternatively called bipyridine ethylene–p-vanillic acid (1/1)], \( \text{C}_{12}\text{H}_{10}\text{N}_{2}\cdot\text{C}_{8}\text{H}_{8}\text{O}_{4} \), the dihedral angle between the pyridine rings is 59.51 (5)°. In the crystal, the molecules are linked by O—H⋯N hydrogen bonds, generating [401] chains of alternating \( \text{C}_{12}\text{H}_{10}\text{N}_{2} \) and \( \text{C}_{8}\text{H}_{8}\text{O}_{4} \) molecules.

Structure description

4-Hydroxy-3-methoxybenzoic acid, \( \text{C}_{8}\text{H}_{8}\text{O}_{4} \), known commonly as \( p \)-vanillic acid, is used as a flavoring agent and naturally found in a variety of fruits and edible plants (Ingole et al., 2021). In addition, \( p \)-vanillic acid is currently being investigated for its inflammatory pain-inhibiting properties (Calixto-Campos et al., 2015). Despite the prevalence of the molecule in our foods and its potential medicinal benefits, structural information on vanillic acid is sparse with few crystal structures being reported thus far. As such it is crucial to expand the number of structures containing vanillic acid in order to better understand the non-covalent interactions involving this molecule. Bipyridine ethylene (\( \text{C}_{12}\text{H}_{10}\text{N}_{2}; \text{BPyE} \)) was selected as a suitable coformer for the present study because of its ability to form both simple and complex hydrogen-bonded networks with organic acids (Delori et al., 2013; Bhattacharya et al., 2013).

When \( p \)-vanillic acid is combined with BPyE in a 1:1 molar ratio, the resulting 1:1 co-crystal possesses monoclinic (\( P2_1/c \)) symmetry at 90 K. The vanillic acid has two distinct O—H⋯N-type hydrogen-bonding interactions (Table 1); one of these involves the carboxylic acid group and a BPyE N atom acceptor and resulting in a 2.6295 (12) Å distance between heteroatoms (Fig. 1). The other hydrogen bond occurs between the \( \text{para} \)-position hydroxyl group and the other pyridine N atom of a BPyE molecule resulting in a 2.6868 (13) Å distance between heteroatoms (Fig. 2). The co-crystal structure may be described as dimolecular units made up of one acid plus one coformer, which form \( C_2(19) \) chain motifs. These chains propagate in the [401] direction, forming
Figure 1
A bimolecular unit consisting of p-vanillic acid and BPY-E with the hydrogen bond depicted as a blue dashed line. The BPY-E molecule illustrated is generated by the symmetry operation $x - 1, y, z$ from the asymmetric molecule.

twisting wires (Fig. 3). The wires stack along [010], forming sheets, which subsequently form layers parallel to (104), with every other sheet being rotated 180° about [010]. Two weak C—H⋯O contacts are also observed (Table 1).

Synthesis and crystallization
A 1:1 molar ratio of bipyridine ethylene (182.2 mg, 1 mmol) and p-vanillic acid (168.1 mg, 1 mmol) was added to a 25 ml scintillation vial to which methanol was added until both compounds dissolved (approximately 20 ml). The resulting solution was vortexed for 30 s at 3000 rpm on a VWR Mini Vortexer MV I. The solution was then stored in the dark uncapped to allow for crystal formation while the solvent slowly evaporated.

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

| Table 1 | Hydrogen-bond geometry ($\AA$, °). |
|---------|-----------------------------------|
| D—H ·· A | D—H | H···A | D···A | D—H···A |
| O1—H1···N1 | 0.99 (2) | 1.65 (2) | 2.3295 (12) | 169 (2) |
| O4—H4···N2 | 0.92 (2) | 1.84 (2) | 2.6868 (13) | 154 (2) |
| C4—H4···O2 | 0.95 | 2.53 | 3.2341 (14) | 132 |
| C9—H9···O3 | 0.95 | 2.45 | 3.3520 (14) | 158 |

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

| Table 2 | Experimental details. |
|---------|-----------------------|
| Crystal data | C12H10N2C8H8O4 |
| Chemical formula | 350.36 |
| Crystal system, space group | Monoclinic, $P_{2}1/c$ |
| Temperature (K) | 90 |
| $a$, $b$, $c$ (Å) | 9.1486 (5), 9.2114 (5), 20.3429 (12) |
| $\beta$ (°) | 98.416 (1) |
| V (Å³) | 1695.86 (16) |
| Z | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.10 |
| Crystal size (mm) | 0.54 × 0.22 × 0.02 |

Data collection
Diffractometer | Bruker APEXI CCD |
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) |
| $T_{\text{min}}, T_{\text{max}}$ | 0.648, 0.746 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 33598, 5958, 4683 |
| $R_{\text{int}}$ | 0.084 |
| $\langle \sin \theta/\lambda \rangle_{\text{max}}$ (Å⁻¹) | 0.748 |

Refinement
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.047, 0.131, 1.03 |
| No. of reflections | 5958 |
| No. of parameters | 245 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta \rho_{\text{max}}, \Delta \rho_{\text{min}}$ (e Å⁻³) | 0.40, −0.26 |

Computer programs: APEXI2 and SAINT (Bruker, 2016), SHELXT2016/2 (Sheldrick, 2015a), SHELXL2016/3 (Sheldrick 2015b), and OLEX2 (Dolomanov et al., 2009).

Figure 2
Part of a [401] hydrogen-bonded chain of $p$-vanillic acid and BPY-E molecules. The O···N distances are shown for each O—H···N hydrogen-bonding interaction.

Figure 3
plane depicting twisting hydrogen-bonded wires running approximately parallel to (104). Hydrogen-bonding interactions are depicted as bright-blue dashed lines.
Funding information

Funding for this research was provided by: National Science Foundation, Directorate for Mathematical and Physical Sciences (award No. DMR-2003932).

References

Bhattacharya, S., Stojaković, J., Saha, B. K. & MacGillivray, L. R. (2013). Org. Lett. 15, 744–747.
Bruker (2016). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

Calixto-Campos, C., Carvalho, T. T., Hohmann, M. S. N., Pinho-Ribeiro, F. A., Fattori, V., Manchope, M. F., Zarpelon, A. C., Baracat, M. M., Georgetti, S. R., Casagrande, R. & Verri, W. A. (2015). J. Nat. Prod. 78, 1799–1808.
Delori, A., Eddleston, M. D. & Jones, W. (2013). CrystEngComm, 15, 73–77.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). J. Appl. Cryst. 42, 339–341.
Ingole, A., Kadam, M., Dalu, A., Kute, S., Mange, P., Theng, V., Lahane, R., Nikas, A., Kawal, Y., Nagrik, S. & Patil, P. (2021). J. Drug. Deliv. Ther. 11, 200–204.
Sheldrick, G. M. (2015a). Acta Cryst. C71, 3–8.
Sheldrick, G. M. (2015b). Acta Cryst. C71, 3–8.
full crystallographic data

IUCrData (2022). 7, x220304  [https://doi.org/10.1107/S2414314622003042]

1,2-Bis(pyridin-4-yl)ethene–4-hydroxy-3-methoxybenzoic acid (1/1)

Devin J. Angevine and Jason B. Benedict

1,2-Bis(pyridin-4-yl)ethene; 4-hydroxy-3-methoxybenzoic acid

Crystal data
C12H10N2·C8H8O4  F(000) = 736
Mr = 350.36
Monoclinic, P21/c  Dx = 1.372 Mg m−3
a = 9.1486 (5) Å  Mo Kα radiation, λ = 0.71073 Å
b = 9.2114 (5) Å  Cell parameters from 5974 reflections
MoKα radiation, λ = 0.71073 Å
θ = 2.4–32.1°
β = 98.416 (1)°  μ = 0.10 mm−1
V = 1695.86 (16) Å3  T = 90 K
Z = 4  Plate, clear colourless

Data collection
Bruker APEXII CCD  0.54 × 0.22 × 0.02 mm
φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2016)
θmin = 0.648, θmax = 0.746
4683 reflections with I > 2σ(I)
33598 measured reflections

Refinement
Refinement on F2
H atoms treated by a mixture of independent and constrained refinement
wR(F2) = 0.131
Largest |Δρ| = 0.40 e Å−3
S = 1.03
Extinction correction: SHELXL2018/3
Extinction coefficient: 0.0070 (15)

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. The O-bound H atoms were located in difference maps and their positions were freely refined. The C-bound H atoms were placed geometrically (C—H = 0.95–0.98 Å) and refined as riding atoms with Uiso(H) = 1.2Ueq(C) or 1.5Ueq(methyl C).
### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|      | x         | y         | z         | Uiso*/*Ueq |
|------|-----------|-----------|-----------|------------|
| O3   | 0.43471(9)| 0.32423(9)| 0.77939(4)| 0.01865(17)|
| O2   | −0.05986(9)| 0.39976(10)| 0.62580(4)| 0.01877(17)|
| O4   | 0.41561(9)| 0.50304(10)| 0.88365(4)| 0.02072(18)|
| O1   | −0.17538(9)| 0.55862(10)| 0.68391(4)| 0.02055(18)|
| N1   | 0.58124(10)| 0.49305(11)| 0.60383(5)| 0.01647(18)|
| N2   | −0.30499(10)| 0.09310(11)| 0.39212(5)| 0.0198(2)  |
| C11  | 0.30840(11)| 0.38151(12)| 0.54449(5)| 0.01433(19)|
| C5   | 0.30531(11)| 0.49848(12)| 0.83165(5)| 0.01447(19)|
| C1   | −0.06039(11)| 0.47842(12)| 0.67408(5)| 0.01454(19)|
| C2   | 0.06755(11)| 0.49121(12)| 0.72818(5)| 0.01337(19)|
| C3   | 0.06593(11)| 0.58633(12)| 0.78115(5)| 0.01466(19)|
| H3   | −0.016259| 0.648781| 0.782394| 0.018*|
| C7   | 0.19036(11)| 0.40137(12)| 0.72604(5)| 0.01356(19)|
| H7   | 0.192506| 0.337576| 0.689504| 0.016*|
| C16  | −0.01655(11)| 0.18988(12)| 0.43775(5)| 0.01485(19)|
| C12  | 0.44140(11)| 0.31630(12)| 0.53464(5)| 0.0158(2)  |
| H12  | 0.441106| 0.232395| 0.507463| 0.019*|
| C6   | 0.30870(11)| 0.40521(12)| 0.77703(5)| 0.01375(19)|
| C9   | 0.45404(12)| 0.55722(13)| 0.61287(5)| 0.0171(2)  |
| H9   | 0.458134| 0.641952| 0.639702| 0.021*|
| C10  | 0.31694(11)| 0.50520(12)| 0.58473(5)| 0.0154(2)  |
| H10  | 0.229348| 0.553233| 0.592721| 0.019*|
| C13  | 0.57369(11)| 0.37518(13)| 0.56488(5)| 0.0164(2)  |
| H13  | 0.663253| 0.329869| 0.557653| 0.020*|
| C15  | 0.13307(11)| 0.24385(13)| 0.46164(5)| 0.0163(2)  |
| H15  | 0.210971| 0.220751| 0.437269| 0.020*|
| C4   | 0.18456(11)| 0.58996(12)| 0.83222(5)| 0.0158(2)  |
| H4A  | 0.183227| 0.655853| 0.868048| 0.019*|
| C20  | −0.06769(12)| 0.17485(13)| 0.36998(5)| 0.0172(2)  |
| H20  | −0.004731| 0.196381| 0.338053| 0.021*|
| C14  | 0.16281(11)| 0.32450(13)| 0.51668(5)| 0.0163(2)  |
| H14  | 0.082548| 0.346774| 0.539750| 0.020*|
| C17  | −0.11344(12)| 0.15200(13)| 0.48206(6)| 0.0180(2)  |
| H17  | −0.083003| 0.159193| 0.528642| 0.022*|
| C18  | −0.25442(12)| 0.10380(13)| 0.45737(6)| 0.0192(2)  |
| H18  | −0.318447| 0.077004| 0.488118| 0.023*|
| C19  | −0.21164(12)| 0.12807(13)| 0.34974(6)| 0.0199(2)  |
| H19  | −0.245544| 0.120512| 0.303447| 0.024*|
| C8   | 0.44161(13)| 0.22430(14)| 0.72620(6)| 0.0221(2)  |
| H8A  | 0.430464| 0.277176| 0.683957| 0.033*|
| H8B  | 0.361902| 0.152884| 0.725170| 0.033*|
| H8C  | 0.537185| 0.174302| 0.732989| 0.033*|
| H4   | 0.499(2)| 0.458(3)| 0.8743(11)| 0.055(6)*|
| H1   | −0.259(3)| 0.528(3)| 0.6504(12)| 0.066(7)*|
### Atomic displacement parameters (Å$^2$)

|    | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$   | $U^{13}$   | $U^{23}$   |
|----|------------|------------|------------|------------|------------|------------|
| O3 | 0.0149 (3) | 0.0218 (4) | 0.0183 (4) | 0.0073 (3) | −0.0006 (3) | −0.0023 (3) |
| O2 | 0.0165 (4) | 0.0236 (4) | 0.0156 (4) | 0.0004 (3) | 0.0004 (3)  | −0.0028 (3) |
| O4 | 0.0136 (4) | 0.0285 (5) | 0.0181 (4) | 0.0034 (3) | −0.0043 (3) | −0.0043 (3) |
| O1 | 0.0116 (3) | 0.0257 (4) | 0.0226 (4) | 0.0033 (3) | −0.0031 (3) | −0.0063 (3) |
| N1 | 0.0135 (4) | 0.0191 (5) | 0.0156 (4) | −0.0005 (3)| −0.0016 (3) | 0.0012 (3)  |
| N2 | 0.0141 (4) | 0.0209 (5) | 0.0229 (5) | −0.0007 (3)| −0.0020 (3) | −0.0022 (4) |
| C11| 0.0129 (4) | 0.0171 (5) | 0.0125 (4) | 0.0002 (4) | 0.0003 (3)  | 0.0012 (4)  |
| C5 | 0.0123 (4) | 0.0158 (5) | 0.0148 (4) | −0.0010 (3)| 0.0006 (3)  | 0.0010 (4)  |
| C1 | 0.0119 (4) | 0.0158 (5) | 0.0157 (4) | −0.0006 (3)| 0.0012 (3)  | 0.0016 (4)  |
| C2 | 0.0107 (4) | 0.0147 (5) | 0.0144 (4) | −0.0004 (3)| 0.0005 (3)  | 0.0009 (3)  |
| C3 | 0.0118 (4) | 0.0152 (5) | 0.0167 (4) | 0.0017 (3) | 0.0010 (3)  | −0.0003 (4) |
| C7 | 0.0131 (4) | 0.0142 (5) | 0.0134 (4) | −0.0001 (3)| 0.0021 (3)  | 0.0002 (3)  |
| C16| 0.0125 (4) | 0.0154 (5) | 0.0159 (4) | 0.0013 (3) | −0.0004 (3) | −0.0015 (4) |
| C12| 0.0138 (4) | 0.0172 (5) | 0.0156 (4) | 0.0015 (4) | −0.0004 (3) | −0.0020 (4) |
| C6 | 0.0115 (4) | 0.0142 (5) | 0.0154 (4) | 0.0018 (3) | 0.0015 (3)  | 0.0016 (4)  |
| C9 | 0.0164 (5) | 0.0181 (5) | 0.0158 (5) | 0.0005 (4) | −0.0010 (4) | −0.0012 (4) |
| C10| 0.0126 (4) | 0.0189 (5) | 0.0145 (4) | 0.0015 (4) | 0.0008 (3)  | −0.0011 (4) |
| C13| 0.0120 (4) | 0.0199 (5) | 0.0166 (5) | 0.0021 (4) | 0.0000 (3)  | 0.0009 (4)  |
| C15| 0.0119 (4) | 0.0201 (5) | 0.0164 (5) | 0.0002 (4) | 0.0008 (3)  | 0.0002 (4)  |
| C4 | 0.0135 (4) | 0.0176 (5) | 0.0160 (4) | 0.0002 (4) | 0.0015 (3)  | −0.0030 (4) |
| C20| 0.0161 (5) | 0.0187 (5) | 0.0164 (5) | −0.0001 (4)| 0.0010 (4)  | −0.0027 (4) |
| C14| 0.0117 (4) | 0.0197 (5) | 0.0169 (5) | 0.0002 (4) | 0.0008 (3)  | −0.0007 (4) |
| C17| 0.0146 (4) | 0.0222 (5) | 0.0165 (5) | −0.0008 (4)| 0.0001 (4)  | 0.0001 (4)  |
| C18| 0.0135 (5) | 0.0221 (5) | 0.0218 (5) | −0.0008 (4)| 0.0018 (4)  | 0.0000 (4)  |
| C19| 0.0174 (5) | 0.0226 (6) | 0.0180 (5) | −0.0005 (4)| −0.0029 (4) | −0.0035 (4) |
| C8 | 0.0230 (5) | 0.0235 (6) | 0.0198 (5) | 0.0101 (4) | 0.0031 (4)  | −0.0025 (4) |

### Geometric parameters (Å, °)

| Bond Pair | Distance (Å) | Angle (°) |
|-----------|--------------|-----------|
| O3—C6     | 1.3680 (12)  |           |
| O3—C8     | 1.4292 (14)  |           |
| O2—C1     | 1.2211 (13)  |           |
| O4—C5     | 1.3516 (12)  |           |
| O4—H4     | 0.92 (2)     |           |
| O1—C1     | 1.3243 (13)  |           |
| O1—H1     | 0.99 (2)     |           |
| N1—C9     | 1.3417 (14)  |           |
| N1—C13    | 1.3400 (15)  |           |
| N2—C18    | 1.3439 (15)  |           |
| N2—C19    | 1.3386 (16)  |           |
| C11—C12   | 1.3975 (15)  |           |
| C11—C10   | 1.3984 (15)  |           |
| C11—C14   | 1.4661 (14)  |           |
| C5—C6     | 1.4083 (15)  |           |
| C5—C4     | 1.3908 (15)  |           |
| Bond          | Distance     | Bond          | Distance     |
|---------------|--------------|---------------|--------------|
| C1—C2         | 1.4892 (14)  | C17—C18       | 1.3869 (15)  |
| C2—C3         | 1.3906 (15)  | C18—H18       | 0.9500       |
| C2—C7         | 1.4012 (14)  | C19—H19       | 0.9500       |
| C3—H3         | 0.9500       | C8—H8A        | 0.9800       |
| C3—C4         | 1.3886 (14)  | C8—H8B        | 0.9800       |
| C7—H7         | 0.9500       | C8—H8C        | 0.9800       |
| C7—C6         | 1.3858 (14)  | C6—O3—C8      | 117.07 (9)   |
| C5—O4—H4      | 112.0 (14)   | C10—C9—H9     | 118.6        |
| C1—O1—H1      | 107.0 (14)   | C11—C10—H10   | 120.2        |
| C13—N1—C9     | 117.86 (9)   | C9—C10—H10    | 120.2        |
| C19—N2—C18    | 117.30 (10)  | N1—C13—C12    | 123.09 (10)  |
| C12—C11—C10   | 117.32 (10)  | N1—C13—H13    | 118.5        |
| C12—C11—C14   | 123.47 (10)  | C12—C13—H13   | 118.5        |
| C10—C11—C14   | 119.18 (9)   | C16—C15—H15   | 119.0        |
| O4—C5—C6      | 122.43 (9)   | C14—C15—C16   | 121.99 (10)  |
| O4—C5—C4      | 118.53 (10)  | C14—C15—H15   | 119.0        |
| C4—C5—C6      | 119.04 (9)   | C5—C4—H4A     | 119.5        |
| O2—C1—O1      | 123.31 (10)  | C3—C4—C5      | 120.93 (10)  |
| O2—C1—C2      | 123.10 (10)  | C3—C4—H4A     | 119.5        |
| O1—C1—C2      | 113.58 (9)   | C16—C20—H20   | 120.4        |
| C3—C2—C1      | 121.74 (9)   | C19—C20—C16   | 119.30 (10)  |
| C3—C2—C7      | 119.71 (9)   | C19—C20—H20   | 120.4        |
| C7—C2—C1      | 118.52 (9)   | C11—C14—H14   | 117.1        |
| C2—C3—H3      | 120.0        | C15—C14—C11   | 125.72 (10)  |
| C4—C3—C2      | 119.94 (10)  | C15—C14—H14   | 117.1        |
| C4—C3—H3      | 120.0        | C16—C17—H17   | 120.3        |
| C2—C7—H7      | 119.9        | C18—C17—C16   | 119.35 (10)  |
| C6—C7—C2      | 120.26 (10)  | C18—C17—H17   | 120.3        |
| C6—C7—H7      | 119.9        | N2—C18—C17    | 123.28 (11)  |
| C20—C16—C15   | 121.40 (10)  | N2—C18—H18    | 118.4        |
| C20—C16—C17   | 117.35 (10)  | C17—C18—H18   | 118.4        |
| C17—C16—C15   | 121.24 (10)  | N2—C19—C20    | 123.38 (10)  |
| C11—C12—H12   | 120.3        | N2—C19—H19    | 118.3        |
| C13—C12—C11   | 119.36 (10)  | C20—C19—H19   | 118.3        |
| C13—C12—H12   | 120.3        | O3—C8—H8A     | 109.5        |
| O3—C6—C5      | 114.87 (9)   | O3—C8—H8B     | 109.5        |
| O3—C6—C7      | 125.05 (10)  | O3—C8—H8C     | 109.5        |
| C7—C6—C5      | 120.07 (9)   | H8A—C8—H8B    | 109.5        |
| N1—C9—H9      | 118.6        | H8A—C8—H8C    | 109.5        |
| N1—C9—C10     | 122.81 (10)  | H8B—C8—H8C    | 109.5        |

IUCrData (2022). 7, x220304
O1—C1—C2—C3  \(-3.78 (15)\)  C13—N1—C9—C10  \(1.19 (16)\)
O1—C1—C2—C7  \(174.34 (10)\)  C15—C16—C20—C19  \(-177.84 (11)\)
N1—C9—C10—C11  \(-0.66 (17)\)  C15—C16—C17—C18  \(178.96 (11)\)
C11—C12—C13—N1  \(0.15 (17)\)  C4—C5—C6—O3  \(177.11 (9)\)
C1—C2—C3—C4  \(176.90 (10)\)  C4—C5—C6—C7  \(-2.45 (16)\)
C1—C2—C7—C6  \(-177.00 (9)\)  C20—C16—C15—C14  \(146.38 (12)\)
C2—C3—C4—C5  \(-0.61 (17)\)  C20—C16—C17—C18  \(-0.85 (17)\)
C2—C7—C6—O3  \(-178.84 (10)\)  C15—C16—C17—C18  \(178.96 (11)\)
C2—C7—C6—C5  \(0.68 (16)\)  C14—C11—C10—C9  \(178.05 (10)\)
C3—C2—C7—C6  \(1.15 (16)\)  C17—C16—C15—C14  \(-33.42 (17)\)
C7—C2—C3—C4  \(-1.20 (16)\)  C17—C16—C15—C18  \(1.97 (17)\)
C16—C15—C14—C11  \(179.32 (10)\)  C18—N2—C19—C20  \(-0.37 (18)\)
C16—C20—C19—N2  \(-1.42 (19)\)  C19—N2—C18—C17  \(1.59 (18)\)
C16—C17—C18—N2  \(-0.98 (19)\)  C8—O3—C6—C5  \(177.99 (10)\)
C12—C11—C10—C9  \(-0.15 (16)\)  C8—O3—C6—C7  \(-2.47 (16)\)

Hydrogen-bond geometry (Å, °)

| D—H···A     | D—H  | H···A  | D···A  | D—H···A |
|-------------|-------|--------|--------|--------|
| O1—H1···N1i | 0.99 (2) | 1.65 (2) | 2.6295 (12) | 169 (2) |
| O4—H4···N2ii | 0.92 (2) | 1.84 (2) | 2.6868 (13) | 154 (2) |
| C4—H4···O2iii | 0.95 | 2.53 | 3.2341 (14) | 132 |
| C9—H9···O3iv  | 0.95 | 2.45 | 3.3520 (14) | 158 |

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