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2-(3-Hydroxyphenyl)-4,5-diphenyl-1*H*-imidazol-3-ium hemioxalate ethanol monosolvate

Peter Solo[a,c]* and M. Arockia doss[b]

[a]Department of Chemistry, St Joseph's College (A) Jakhama, Kohima 797001, Nagaland, India, [b]Department of Chemistry, St Joseph University, Chümoukedima, Nagaland 797115, India, and [c]Department of Environmental Studies, St Xavier College, Jalukie, Nagaland, India. *Correspondence e-mail: solopeter82@gmail.com

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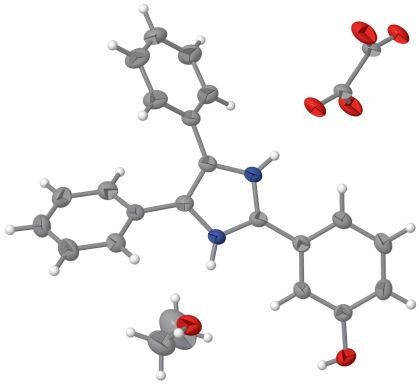
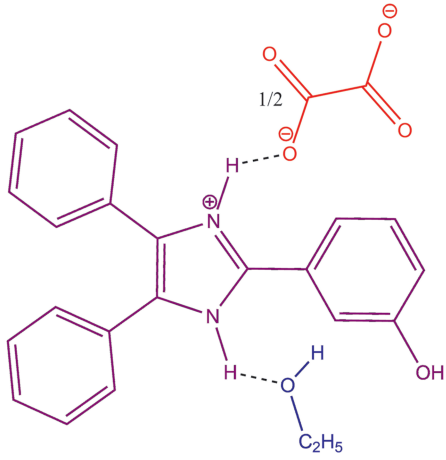
In the title imidazolium oxalate solvate, $C_{21}H_{17}N_2O^+\cdot0.5C_2O_4^{2-}\cdot C_2H_6O$, the cation and the ethanol solvent molecule are located on general positions, whereas the complete oxalate ion is generated by a crystallographic centre of symmetry. The study confirms two proton transfers from oxalic acid to the pyrimidine-type N atoms of two separate imidazole rings. The extended structure features strong N—H···O and O—H···O hydrogen-bond interactions.

3D view



Chemical scheme



Structure description

Compounds containing an imidazole moiety have therapeutic properties (Siwach & Verma, 2021), such as antiviral (Heinz & Vance, 1995), antihistaminic (Griffin *et al.*, 2017), antiulcer (Guerreiro *et al.*, 1990), antibacterial (Valls *et al.*, 2020), antifungal (Holt, 1976), anticancer (Kumar *et al.*, 2024), antioxidant (Pérez-González *et al.*, 2020) and antihypertension (Nikolic & Agbaba, 2012). A number of cocrystals of oxalic acids with different drugs are known (Hriňová *et al.*, 2025; Wenger & Bernstein, 2008; Othman *et al.*, 2018; Karthammaiah *et al.*, 2023), where the introduction of the cocrystallized substance improves the solubility, stability and tabletability of drugs (Chettri *et al.*, 2024).

In this article, we describe the synthesis and crystal structure of an imidazolium oxalate salt (Fig. 1). The crystal structure confirms two proton transfers from oxalic acid to the pyrimidine-type N atoms of two imidazole rings of 3-(4,5-diphenyl-1*H*-imidazol-2-yl) phenol. Each oxalate ion accepts hydrogen bonds from four protonated imidazolium cations *via* N—H···O and O—H···O interactions (Fig. 2 and Table 1). All three phenyl rings in the 2-(3-hydroxyphenyl)-4,5-diphenyl-1*H*-imidazol-3-ium cation are in different planes. The values of the torsion angles between the imidazole ring and the pendant phenyl rings are N3—C11—C6—C5 = 16.8 (2)°, N4—C17—C15—C22 = 47.4 (2)° and N3—C18—C14—C10 = 41.0 (2)°.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O8^{i}	0.82	1.83	2.613 (3)	161
N3—H3···O7	0.86	1.87	2.726 (3)	171
N4—H4···O2	0.86	1.88	2.711 (3)	161
O7—H7···O8^{ii}	0.82	2.03	2.797 (4)	154

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

Synthesis and crystallization

The synthesis of 3-(4,5-diphenyl-1*H*-imidazol-2-yl)phenol was performed by refluxing the reactants, *i.e.* benzil (0.21 g, 1 mmol), 3-hydroxybenzaldehyde (0.12 g, 1 mmol) and ammonium acetate (0.23 g, 3 mmol), in the presence of ceric ammonium nitrate (0.054 g, 0.1 mmol) as catalyst at 363 K in ethanol for about 4 h. The progress and completion of the reaction was monitored by thin-layer chromatography (TLC) using hexane–ethyl acetate solution (1:1 *v*/*v*). After the completion of the reaction, the reaction mixture was poured into ice-cold water and the precipitates were filtered off. The crude product was purified by recrystallization from a 90% ethanol solution to yield 3-(4,5-diphenyl-1*H*-imidazol-2-yl)phenol (yield: 0.23 g, 75%). Equimolar amounts of the imidazole compound and oxalic acid in an ethanol solution were heated to about 393 K and the mixture was cooled slowly to yield crystals of the title imidazolium oxalate salt.

Refinement

Details of the crystal data, data collection and refinement are given in Table 2. H atoms were located in a difference map and refined as riding on their parent atom, with U_{iso}(H) = 1.5U_{eq}(C,O) for the methyl group and the H atoms bonded to O atoms. All remaining H atoms were refined with U_{iso}(H) = 1.2U_{eq}(C,N). The methyl group was allowed to rotate but not to tip. Two reflections, *i.e.* (100) and (001), were omitted as bad reflections.

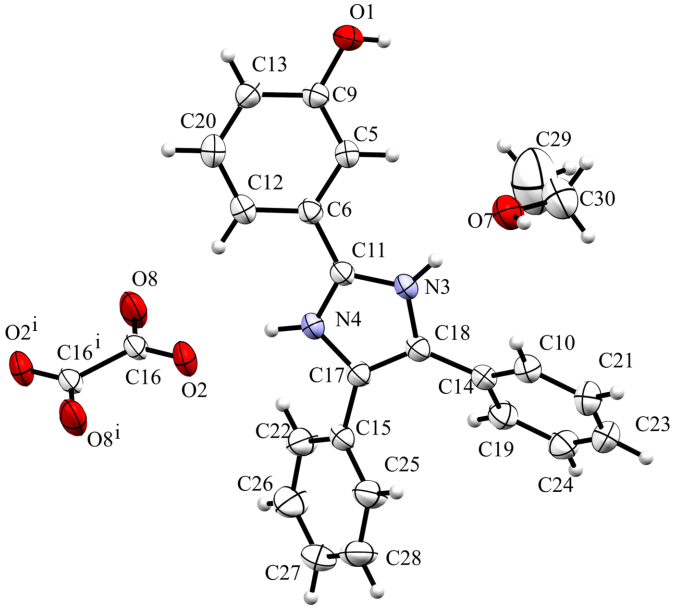


Figure 1
The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $-x+3, -y, -z$.]

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{17}N_2O^+\cdot0.5C_2O_4^{2-}\cdot C_2H_6O$
M_r	403.44
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	9.056 (9), 10.868 (11), 11.894 (12)
α, β, γ (°)	77.56 (3), 72.34 (2), 69.43 (2)
V (Å^3)	1036.4 (19)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.5 × 0.5 × 0.5
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.567, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31823, 5061, 3518
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.695
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.143, 1.05
No. of reflections	5061
No. of parameters	274
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.32

Computer programs: *APEX2* (Bruker, 2018), *SAINT* (Bruker, 2018), SHELXT (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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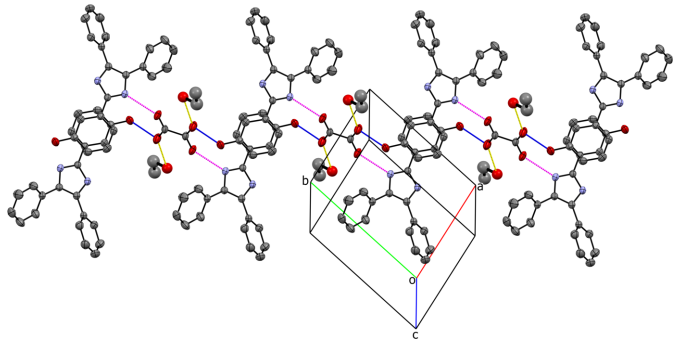

Figure 2
Hydrogen-bonding interactions in the title compound. N---H...O interactions are shown in magenta, O---H(imidazolium)...O interactions in blue and O---H(ethanol)...O interactions in yellow. H atoms have been omitted for clarity. Hydrogen-bonding interactions in the title compound. N—H···O interactions are in magenta and O—H···O interactions are in blue. H atoms and solvent molecules have been omitted for clarity.

References

Bruker (2016). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2018). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chettri, A., Subba, A., Singh, G. P. & Bag, P. P. (2024). *J. Pharm. Pharmacol.* **76**, 1–12.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Griffin, A., Hamling, K. R., Knupp, K., Hong, S., Lee, L. P. & Baraban, S. C. (2017). *Brain*, **140**, 669–683.

Guerreiro, A. S., Neves, B. C. & Quina, M. G. (1990). *Aliment. Pharmacol. Ther.* **4**, 309–313.

Heinz, B. A. & Vance, L. M. (1995). *J. Virol.* **69**, 4189–4197.

Holt, R. J. (1976). *J. Cutan. Pathol.* **3**, 45–59.

Hriňová, E., Čerňa, I., Zmeškalová, E., Ridvan, L. & Šoóš, M. (2025). *Org. Process Res. Dev.* **29**, 565–573.

Karthammaiah, G. N., Rao Amaraneni, S. & Solomon, A. K. (2023). *Acta Cryst.* E**79**, 319–322.

Kumar, A., Kaushal, A., Verma, P. K., Gupta, M. K., Chandra, G., Kumar, U., Yadav, A. K. & Kumar, D. (2024). *Eur. J. Med. Chem.* **280**, 116896.

Nikolic, K. & Agbaba, D. (2012). *Cardiovasc. Ther.* **30**, 209–216.

Othman, M. F., Anuar, N., Ad Rahman, S. & Ahmad Taifuddin, N. A. (2018). *IOP Conf. Ser. Mater. Sci. Eng.* **358**, 012065.

Pérez-González, A., García-Hernández, E. & Chigo-Anota, E. (2020). *J. Mol. Model.* **26**, 321.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Siwach, A. & Verma, P. K. (2021). *BMC Chem.* **15**, 12–80.

Valls, A., Andreu, J. J., Falomir, E., Luis, S. V., Atrián-Blasco, E., Mitchell, S. G. & Altava, B. (2020). *Pharmaceuticals*, **13**, 482–498.

Wenger, M. & Bernstein, J. (2008). *Cryst. Growth Des.* **8**, 1595–1598.

full crystallographic data

IUCrData (2025). **10**, x250315 [https://doi.org/10.1107/S2414314625003153]

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Peter Solo and M. Arockia doss

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Crystal data

$C_{21}H_{17}N_2O^+ \cdot 0.5C_2O_4^{2-} \cdot C_2H_6O$
M_r = 403.44
Triclinic, $P\overline{1}$
a = 9.056 (9) Å
b = 10.868 (11) Å
c = 11.894 (12) Å
α = 77.56 (3)°
β = 72.34 (2)°
γ = 69.43 (2)°
V = 1036.4 (19) Å^3
Z = 2
$F(000)$ = 426
D_x = 1.293 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5467 reflections
θ = 2.5–26.2°
μ = 0.09 mm^{-1}
T = 296 K
Block, orange
0.5 × 0.5 × 0.5 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.957, T_{max} = 0.957
31823 measured reflections
5061 independent reflections
3518 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 29.6°, θ_{min} = 2.0°
h = −11→11
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.143
S = 1.05
5061 reflections
274 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.2615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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}
O1	1.23020 (14)	0.27909 (12)	−0.20771 (11)	0.0444 (3)
H1	1.238649	0.221040	−0.151031	0.067*
O2	0.50008 (16)	0.83543 (11)	0.03285 (11)	0.0442 (3)
N3	0.66676 (16)	0.36104 (12)	0.11752 (12)	0.0314 (3)
H3	0.720090	0.282619	0.098274	0.038*
N4	0.58706 (16)	0.57339 (12)	0.11283 (12)	0.0312 (3)
H4	0.580136	0.655501	0.090389	0.037*
C5	0.96646 (19)	0.37050 (15)	−0.07708 (14)	0.0312 (3)
H5	0.970348	0.288935	−0.031343	0.037*
C6	0.83192 (19)	0.48199 (15)	−0.04782 (14)	0.0291 (3)
O7	0.82059 (18)	0.10267 (13)	0.08045 (14)	0.0559 (4)
H7	0.772174	0.068017	0.054917	0.084*
O8	0.67298 (17)	0.92601 (12)	0.05650 (14)	0.0534 (4)
C9	1.09467 (19)	0.38249 (16)	−0.17525 (14)	0.0318 (4)
C10	0.4602 (2)	0.18315 (16)	0.26039 (16)	0.0368 (4)
H10	0.496326	0.173701	0.179802	0.044*
C11	0.69811 (18)	0.47219 (15)	0.05714 (14)	0.0293 (3)
C12	0.8266 (2)	0.60359 (16)	−0.11698 (15)	0.0367 (4)
H12	0.738378	0.678219	−0.096977	0.044*
C13	1.0869 (2)	0.50419 (17)	−0.24494 (15)	0.0385 (4)
H13	1.171617	0.511972	−0.311018	0.046*
C14	0.47346 (19)	0.28902 (15)	0.30020 (14)	0.0318 (4)
C15	0.35217 (19)	0.61249 (15)	0.29465 (15)	0.0322 (4)
C16	0.5498 (2)	0.93044 (15)	0.02620 (15)	0.0336 (4)
C17	0.48478 (19)	0.52616 (15)	0.21215 (14)	0.0310 (3)
C18	0.53606 (19)	0.39090 (15)	0.21533 (14)	0.0312 (3)
C19	0.4229 (2)	0.29914 (17)	0.42203 (15)	0.0401 (4)
H19	0.433637	0.368164	0.450264	0.048*
C20	0.9534 (2)	0.61304 (17)	−0.21584 (16)	0.0416 (4)
H20	0.948217	0.693904	−0.263119	0.050*
C21	0.3938 (2)	0.09172 (18)	0.33962 (18)	0.0469 (5)
H21	0.384967	0.021362	0.312113	0.056*
C22	0.3787 (2)	0.70962 (18)	0.33806 (18)	0.0469 (5)
H22	0.478999	0.725234	0.310859	0.056*
C23	0.3404 (2)	0.10459 (19)	0.45989 (18)	0.0497 (5)
H23	0.293561	0.044057	0.512833	0.060*
C24	0.3567 (2)	0.20705 (19)	0.50099 (17)	0.0469 (5)
H24	0.323298	0.214432	0.581960	0.056*
C25	0.2006 (2)	0.59230 (19)	0.33520 (18)	0.0453 (5)
H25	0.181223	0.528125	0.305996	0.054*
C26	0.2560 (3)	0.7831 (2)	0.4217 (2)	0.0572 (6)
H26	0.274879	0.847358	0.451212	0.069*
C27	0.1063 (2)	0.7623 (2)	0.46189 (19)	0.0529 (5)
H27	0.024275	0.812452	0.517990	0.064*
C28	0.0785 (2)	0.6664 (2)	0.4183 (2)	0.0552 (5)

H28	−0.022498	0.651913	0.445019	0.066*
C29	0.9744 (6)	0.0693 (4)	0.2205 (4)	0.1308 (15)
H29A	1.027149	0.004373	0.276394	0.196*
H29B	0.898109	0.143431	0.259980	0.196*
H29C	1.054539	0.098600	0.157279	0.196*
C30	0.8899 (3)	0.0118 (2)	0.1725 (3)	0.0749 (7)
H30A	0.803803	−0.011942	0.235633	0.090*
H30B	0.964940	−0.068401	0.140023	0.090*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0326 (6)	0.0396 (7)	0.0446 (7)	−0.0014 (5)	0.0002 (5)	−0.0021 (5)
O2	0.0544 (8)	0.0217 (6)	0.0555 (8)	−0.0086 (5)	−0.0191 (6)	0.0001 (5)
N3	0.0321 (7)	0.0215 (6)	0.0356 (7)	−0.0052 (5)	−0.0039 (6)	−0.0050 (5)
N4	0.0310 (7)	0.0219 (6)	0.0374 (7)	−0.0060 (5)	−0.0057 (6)	−0.0049 (5)
C5	0.0322 (8)	0.0249 (7)	0.0347 (8)	−0.0086 (6)	−0.0080 (7)	−0.0008 (6)
C6	0.0299 (8)	0.0261 (7)	0.0317 (8)	−0.0084 (6)	−0.0080 (7)	−0.0044 (6)
O7	0.0552 (9)	0.0370 (7)	0.0726 (10)	−0.0130 (6)	−0.0076 (7)	−0.0147 (7)
O8	0.0493 (8)	0.0311 (7)	0.0812 (10)	−0.0078 (6)	−0.0318 (8)	0.0048 (6)
C9	0.0281 (8)	0.0309 (8)	0.0349 (8)	−0.0072 (6)	−0.0078 (7)	−0.0044 (7)
C10	0.0377 (9)	0.0317 (8)	0.0386 (9)	−0.0101 (7)	−0.0071 (7)	−0.0038 (7)
C11	0.0288 (8)	0.0243 (7)	0.0332 (8)	−0.0053 (6)	−0.0081 (7)	−0.0049 (6)
C12	0.0377 (9)	0.0260 (8)	0.0416 (9)	−0.0054 (7)	−0.0099 (8)	−0.0015 (7)
C13	0.0354 (9)	0.0407 (9)	0.0368 (9)	−0.0155 (8)	−0.0049 (7)	0.0013 (7)
C14	0.0270 (8)	0.0264 (8)	0.0368 (9)	−0.0045 (6)	−0.0055 (7)	−0.0026 (6)
C15	0.0293 (8)	0.0263 (8)	0.0375 (9)	−0.0044 (6)	−0.0065 (7)	−0.0065 (7)
C16	0.0356 (9)	0.0218 (8)	0.0359 (9)	−0.0021 (7)	−0.0056 (7)	−0.0050 (6)
C17	0.0290 (8)	0.0271 (8)	0.0356 (8)	−0.0068 (6)	−0.0075 (7)	−0.0052 (6)
C18	0.0290 (8)	0.0274 (8)	0.0347 (8)	−0.0059 (6)	−0.0059 (7)	−0.0064 (6)
C19	0.0415 (10)	0.0358 (9)	0.0390 (9)	−0.0085 (8)	−0.0068 (8)	−0.0071 (7)
C20	0.0477 (10)	0.0310 (9)	0.0427 (10)	−0.0140 (8)	−0.0108 (8)	0.0048 (7)
C21	0.0505 (11)	0.0352 (9)	0.0558 (12)	−0.0181 (8)	−0.0111 (9)	−0.0023 (8)
C22	0.0411 (10)	0.0417 (10)	0.0575 (11)	−0.0182 (8)	0.0044 (9)	−0.0198 (9)
C23	0.0457 (11)	0.0422 (10)	0.0516 (12)	−0.0170 (9)	−0.0028 (9)	0.0061 (9)
C24	0.0450 (11)	0.0468 (11)	0.0366 (9)	−0.0084 (9)	−0.0022 (8)	−0.0012 (8)
C25	0.0318 (9)	0.0438 (10)	0.0610 (12)	−0.0082 (8)	−0.0083 (9)	−0.0184 (9)
C26	0.0585 (13)	0.0478 (11)	0.0643 (13)	−0.0207 (10)	0.0064 (11)	−0.0287 (10)
C27	0.0445 (11)	0.0450 (11)	0.0540 (12)	−0.0036 (9)	0.0056 (9)	−0.0181 (9)
C28	0.0296 (9)	0.0576 (12)	0.0709 (14)	−0.0099 (9)	0.0020 (9)	−0.0192 (11)
C29	0.171 (4)	0.104 (3)	0.156 (4)	−0.061 (3)	−0.094 (3)	0.012 (2)
C30	0.0766 (17)	0.0528 (14)	0.0881 (19)	−0.0125 (12)	−0.0223 (15)	−0.0038 (13)

Geometric parameters (Å, º)

O1—C9	1.359 (2)	C15—C25	1.389 (3)
O1—H1	0.8200	C15—C17	1.475 (2)
O2—C16	1.243 (2)	C16—C16^{i}	1.570 (3)

N3—C11	1.341 (2)	C17—C18	1.373 (3)
N3—C18	1.385 (2)	C19—C24	1.385 (3)
N3—H3	0.8600	C19—H19	0.9300
N4—C11	1.339 (2)	C20—H20	0.9300
N4—C17	1.388 (2)	C21—C23	1.385 (3)
N4—H4	0.8600	C21—H21	0.9300
C5—C9	1.395 (2)	C22—C26	1.382 (3)
C5—C6	1.400 (2)	C22—H22	0.9300
C5—H5	0.9300	C23—C24	1.376 (3)
C6—C12	1.392 (2)	C23—H23	0.9300
C6—C11	1.468 (2)	C24—H24	0.9300
O7—C30	1.443 (3)	C25—C28	1.379 (3)
O7—H7	0.8200	C25—H25	0.9300
O8—C16	1.255 (2)	C26—C27	1.376 (3)
C9—C13	1.392 (3)	C26—H26	0.9300
C10—C21	1.382 (3)	C27—C28	1.384 (3)
C10—C14	1.389 (3)	C27—H27	0.9300
C10—H10	0.9300	C28—H28	0.9300
C12—C20	1.387 (3)	C29—C30	1.434 (4)
C12—H12	0.9300	C29—H29A	0.9600
C13—C20	1.378 (3)	C29—H29B	0.9600
C13—H13	0.9300	C29—H29C	0.9600
C14—C19	1.397 (3)	C30—H30A	0.9700
C14—C18	1.476 (2)	C30—H30B	0.9700
C15—C22	1.388 (3)		

C9—O1—H1	109.5	N3—C18—C14	122.98 (14)
C11—N3—C18	110.12 (13)	C24—C19—C14	120.41 (17)
C11—N3—H3	124.9	C24—C19—H19	119.8
C18—N3—H3	124.9	C14—C19—H19	119.8
C11—N4—C17	109.91 (14)	C13—C20—C12	120.86 (17)
C11—N4—H4	125.0	C13—C20—H20	119.6
C17—N4—H4	125.0	C12—C20—H20	119.6
C9—C5—C6	119.62 (15)	C10—C21—C23	120.18 (18)
C9—C5—H5	120.2	C10—C21—H21	119.9
C6—C5—H5	120.2	C23—C21—H21	119.9
C12—C6—C5	119.88 (15)	C26—C22—C15	120.00 (18)
C12—C6—C11	119.70 (14)	C26—C22—H22	120.0
C5—C6—C11	120.40 (15)	C15—C22—H22	120.0
C30—O7—H7	109.5	C24—C23—C21	119.94 (18)
O1—C9—C13	117.16 (15)	C24—C23—H23	120.0
O1—C9—C5	122.82 (15)	C21—C23—H23	120.0
C13—C9—C5	120.01 (15)	C23—C24—C19	120.14 (19)
C21—C10—C14	120.50 (18)	C23—C24—H24	119.9
C21—C10—H10	119.7	C19—C24—H24	119.9
C14—C10—H10	119.7	C28—C25—C15	120.71 (18)
N4—C11—N3	107.12 (15)	C28—C25—H25	119.6
N4—C11—C6	125.96 (15)	C15—C25—H25	119.6

N3—C11—C6	126.88 (14)	C27—C26—C22	120.74 (19)
C20—C12—C6	119.72 (16)	C27—C26—H26	119.6
C20—C12—H12	120.1	C22—C26—H26	119.6
C6—C12—H12	120.1	C26—C27—C28	119.60 (18)
C20—C13—C9	119.88 (16)	C26—C27—H27	120.2
C20—C13—H13	120.1	C28—C27—H27	120.2
C9—C13—H13	120.1	C25—C28—C27	119.95 (19)
C10—C14—C19	118.78 (15)	C25—C28—H28	120.0
C10—C14—C18	120.73 (16)	C27—C28—H28	120.0
C19—C14—C18	120.47 (15)	C30—C29—H29A	109.5
C22—C15—C25	118.99 (16)	C30—C29—H29B	109.5
C22—C15—C17	120.96 (16)	H29A—C29—H29B	109.5
C25—C15—C17	119.95 (15)	C30—C29—H29C	109.5
O2—C16—O8	126.35 (16)	H29A—C29—H29C	109.5
O2—C16—C16^{i}	117.1 (2)	H29B—C29—H29C	109.5
O8—C16—C16^{i}	116.57 (18)	C29—C30—O7	111.2 (2)
C18—C17—N4	106.51 (14)	C29—C30—H30A	109.4
C18—C17—C15	130.12 (15)	O7—C30—H30A	109.4
N4—C17—C15	123.35 (15)	C29—C30—H30B	109.4
C17—C18—N3	106.31 (14)	O7—C30—H30B	109.4
C17—C18—C14	130.72 (15)	H30A—C30—H30B	108.0

C9—C5—C6—C12	0.3 (2)	C15—C17—C18—N3	−179.02 (16)
C9—C5—C6—C11	−178.19 (14)	N4—C17—C18—C14	−179.89 (16)
C6—C5—C9—O1	178.87 (15)	C15—C17—C18—C14	1.5 (3)
C6—C5—C9—C13	−1.3 (2)	C11—N3—C18—C17	1.21 (18)
C17—N4—C11—N3	1.29 (18)	C11—N3—C18—C14	−179.25 (15)
C17—N4—C11—C6	−176.79 (15)	C10—C14—C18—C17	138.40 (19)
C18—N3—C11—N4	−1.55 (18)	C19—C14—C18—C17	−40.1 (3)
C18—N3—C11—C6	176.51 (15)	C10—C14—C18—N3	−41.0 (2)
C12—C6—C11—N4	−17.6 (2)	C19—C14—C18—N3	140.47 (17)
C5—C6—C11—N4	160.91 (16)	C10—C14—C19—C24	−1.7 (3)
C12—C6—C11—N3	164.72 (16)	C18—C14—C19—C24	176.82 (16)
C5—C6—C11—N3	−16.8 (2)	C9—C13—C20—C12	0.6 (3)
C5—C6—C12—C20	1.2 (3)	C6—C12—C20—C13	−1.7 (3)
C11—C6—C12—C20	179.69 (16)	C14—C10—C21—C23	−0.3 (3)
O1—C9—C13—C20	−179.32 (16)	C25—C15—C22—C26	1.0 (3)
C5—C9—C13—C20	0.9 (3)	C17—C15—C22—C26	−175.42 (19)
C21—C10—C14—C19	1.8 (3)	C10—C21—C23—C24	−1.4 (3)
C21—C10—C14—C18	−176.68 (16)	C21—C23—C24—C19	1.6 (3)
C11—N4—C17—C18	−0.55 (18)	C14—C19—C24—C23	0.0 (3)
C11—N4—C17—C15	178.19 (15)	C22—C15—C25—C28	−0.7 (3)
C22—C15—C17—C18	131.0 (2)	C17—C15—C25—C28	175.74 (18)
C25—C15—C17—C18	−45.4 (3)	C15—C22—C26—C27	−0.8 (3)
C22—C15—C17—N4	−47.4 (2)	C22—C26—C27—C28	0.2 (4)

C25—C15—C17—N4	136.21 (18)	C15—C25—C28—C27	0.2 (3)
N4—C17—C18—N3	−0.39 (17)	C26—C27—C28—C25	0.1 (3)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O8^{ii}	0.82	1.83	2.613 (3)	161
N3—H3···O7	0.86	1.87	2.726 (3)	171
N4—H4···O2	0.86	1.88	2.711 (3)	161
O7—H7···O8^{iii}	0.82	2.03	2.797 (4)	154

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