IUCrData

ISSN 2414-3146

2,5-Dimethylanilinium 4-hydroxybenzoate–2,5-dimethylaniline–4-hydroxybenzoic acid (1/1/1)

A. Mani,[a,b] K. Rajesh,[c] P. Praveen Kumar[b]* and G. Chakkaravarthi[d]*

[a]Department of Physics, Sri Venkateswaraa College of Technology, Sriperumbudur 602 105, India, [b]Department of Physics, Presidency College, Chennai 600 005, India, [c]Department of Physics, AMET University, Chennai 603 112, India, and [d]Department of Physics, CPCL Polytechnic College, Chennai 600 068, India. *Correspondence e-mail: ppkpresidency@gmail.com, chakkaravarthi_2005@yahoo.com

Received 9 October 2016
Accepted 11 October 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

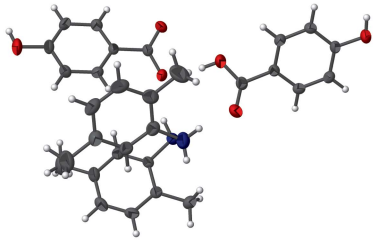
Keywords: crystal structure; hydrogen bonding,.

CCDC reference: 1509213

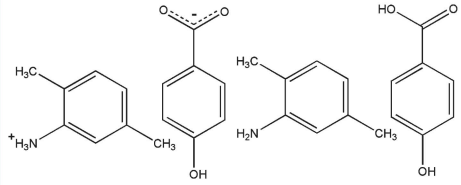
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_8H_{12}N^+\cdot C_7H_5O_3^-\cdot C_8H_{11}N\cdot C_7H_6O_3$, contains a 2,5-dimethylanilinium cation, 4-hydroxybenzoate anion and neutral 2,5-dimethylaniline and 4-hydroxybenzoic acid molecules. The components are connected by N—H···O, O—H···O and N—H···N hydrogen bonds, which generate $R_2^2(8)$, $R_4^4(20)$ and $R_4^4(24)$ loops, as part of a three-dimensional network. The crystal structure also features weak C—H···π interactions.

3D view



Chemical scheme



Structure description

We report the synthesis and structure of the title compound which comprises a 2,5-dimethylanilinium cation, a 4-hydroxybenzoate anion and neutral 2,5-dimethylaniline and 4-hydroxybenzoic acid molecules (Fig. 1). The benzene rings of the cation (C1–C6) and anion (C17–C22) are inclined at an angle of 15.00 (9) Å while the benzene rings of the neutral molecules (C9–C14) and (C24–C29) are inclined at an angle of 11.08 (9) Å. The bond lengths of the ions and molecules are comparable with those in previously reported structures (Fun *et al.*, 2011; Mani *et al.*, 2015; Mathlouthi *et al.*, 2014).

In the arbitrarily chosen asymmetric unit, the anion and cation are connected by an N1—H1*B*···O2 hydrogen bond and the ions are connected to the 4-hydroxybenzoic acid molecule through N1—H1*A*···O5 and O4—H4*A*···O2 hydrogen bonds, forming an $R_2^2(8)$ loop. Further, the 2,5-dimethylanilinium cation and 2,5-dimethylaniline molecule are connected through an N1—H1*A*···N2 hydrogen bond (Table 1 and Fig. 2).

In the extended structure, pairs of O—H···O [(O3—H3*A*···O6^{iv} and O6—H6*A*···O1^{iii}) and (O6—H6*A*···O1^{iii} and O4—H4*A*···O2); for symmetry codes, see Table 1] hydrogen bonds generate $R_4^4(20)$ and $R_4^4(24)$ ring-motifs, respectively (Fig. 3),

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

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C1–C6, C9–C14 and C24–C29 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5	0.87 (1)	2.55 (2)	3.015 (2)	114 (2)
N1—H1*A*···N2	0.87 (1)	2.24 (2)	3.016 (3)	149 (2)
N1—H1*B*···O2	0.87 (1)	2.00 (1)	2.792 (2)	151 (2)
O4—H4*A*···O2	0.83 (1)	1.78 (1)	2.6041 (17)	169 (3)
N1—H1*C*···O1^{i}	0.88 (1)	1.91 (1)	2.781 (2)	176 (2)
N2—H2*B*···O5^{ii}	0.86 (1)	2.49 (1)	3.306 (3)	159 (2)
O3—H3*A*···O6^{iii}	0.82 (1)	2.09 (2)	2.858 (2)	156 (3)
O6—H6*A*···O1^{iv}	0.83 (1)	1.88 (1)	2.7075 (19)	175 (3)
C13—H13···*Cg*1	0.93	2.80	3.437 (2)	127
C6—H6···*Cg*3^{ii}	0.93	2.87	3.554 (2)	132
C19—H19···*Cg*2^{v}	0.93	2.87	3.532 (2)	129

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

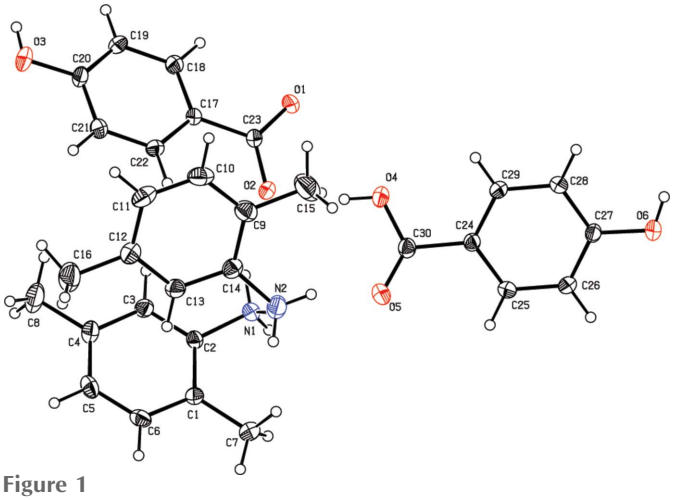


Figure 1
The molecular structure of the title compound, with 30% probability displacement ellipsoids.

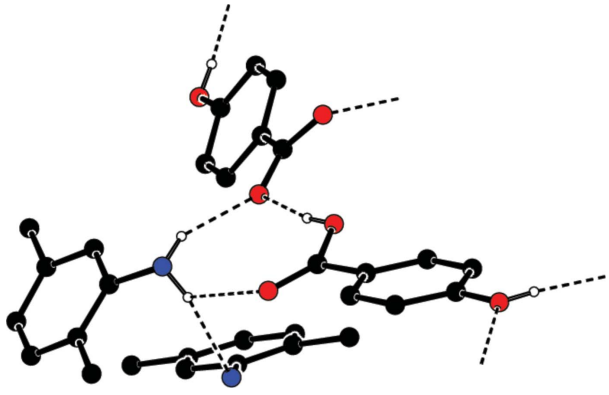


Figure 2
Hydrogen bonds within the asymmetric unit.

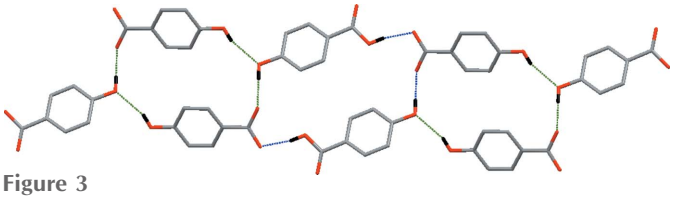


Figure 3
Partial crystal packing, showing the ring motif.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{12}N^+\cdot C_7H_5O_3^-\cdot C_8H_{11}N\cdot C_7H_6O_3$
M_r	518.59
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	295
a, b, c (Å)	9.6854 (5), 10.9169 (6), 13.1450 (7)
α, β, γ (°)	79.029 (4), 75.615 (3), 85.345 (4)
V (Å^3)	1320.86 (13)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.26 × 0.24 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.688, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	35698, 7024, 4026
R_{int}	0.070
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.693
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.059, 0.145, 1.01
No. of reflections	7024
No. of parameters	376
No. of restraints	8
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.30

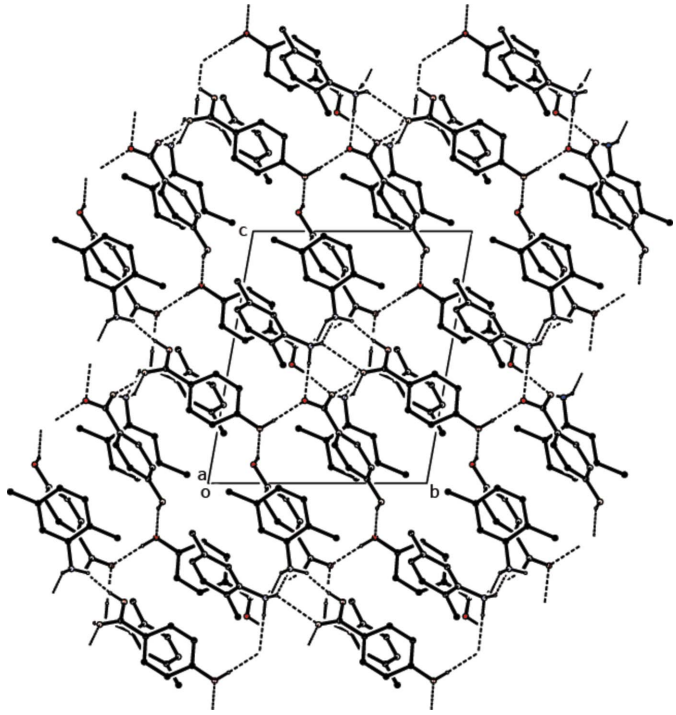


Figure 4
The crystal packing of the title compound viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

which generate a three-dimensional network (Fig. 4). The crystal structure also features weak C—H$\cdots\pi$ interactions.

Synthesis and crystallization

A mixture of 2,5-xylidine and 4-hydroxybenzoic acid dissolved in ethanol (molar ratio 1:1:1) was stirred for 4 h and then kept at room temperature. After three weeks, colourless blocks were obtained.

Refinement

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

Acknowledgements

The authors acknowledge the SAIF, IIT, Madras, for the data collection.

References

Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fun, H.-K., Yeap, C. S., Siddegowda, M. S., Yathirajan, H. S. & Narayana, B. (2011). *Acta Cryst.* E**67**, o1584.
Mani, A., Kumar, P. P. & Chakkaravarthi, G. (2015). *Acta Cryst.* E**71**, o643–o644.
Mathlouthi, M., Janzen, D. E., Rzaigui, M. & Smirani Sta, W. (2014). *Acta Cryst.* E**70**, o1183–o1184.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

full crystallographic data

IUCrData (2016). **1**, x161607 [https://doi.org/10.1107/S2414314616016072]

2,5-Dimethylanilinium 4-hydroxybenzoate–2,5-dimethylaniline–4-hydroxybenzoic acid (1/1/1)

A. Mani, K. Rajesh, P. Praveen Kumar and G. Chakkaravarthi

2,5-Dimethylanilinium 4-hydroxybenzoate–2,5-dimethylaniline–4-hydroxybenzoic acid (1/1/1)

Crystal data

$C_8H_{12}N^+·C_7H_5O_3^-·C_8H_{11}N·C_7H_6O_3$
M_r = 518.59
Triclinic, $P\overline{1}$
a = 9.6854 (5) Å
b = 10.9169 (6) Å
c = 13.1450 (7) Å
α = 79.029 (4)°
β = 75.615 (3)°
γ = 85.345 (4)°
V = 1320.86 (13) Å^3
Z = 2
$F(000)$ = 552
D_x = 1.304 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6719 reflections
θ = 2.3–29.4°
μ = 0.09 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.688, T_{max} = 0.746
35698 measured reflections
7024 independent reflections
4026 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 29.5°, θ_{min} = 2.2°
h = −13→13
k = −15→15
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.145
S = 1.01
7024 reflections
376 parameters
8 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.3498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 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}
C1	0.86255 (19)	0.17699 (16)	0.55783 (14)	0.0292 (4)
C2	0.72606 (19)	0.22707 (16)	0.59077 (14)	0.0277 (4)
C3	0.6211 (2)	0.16170 (17)	0.66719 (14)	0.0323 (4)
H3	0.529843	0.197520	0.685334	0.039*
C4	0.6513 (2)	0.04190 (18)	0.71738 (15)	0.0377 (5)
C5	0.7887 (2)	−0.00660 (18)	0.68915 (16)	0.0406 (5)
H5	0.812282	−0.085084	0.723802	0.049*
C6	0.8915 (2)	0.05903 (17)	0.61069 (15)	0.0358 (4)
H6	0.982781	0.023265	0.592701	0.043*
C7	0.9738 (2)	0.24619 (19)	0.46942 (16)	0.0400 (5)
H7A	1.060098	0.195534	0.457843	0.060*
H7B	0.992273	0.322696	0.488735	0.060*
H7C	0.940066	0.264569	0.405190	0.060*
C8	0.5368 (3)	−0.0305 (2)	0.8010 (2)	0.0635 (7)
H8A	0.503145	−0.092472	0.771013	0.095*
H8B	0.459104	0.025493	0.825236	0.095*
H8C	0.575279	−0.070556	0.860116	0.095*
C9	0.7449 (2)	0.49451 (19)	0.82967 (18)	0.0437 (5)
C10	0.6808 (2)	0.4508 (2)	0.93580 (18)	0.0498 (6)
H10	0.632124	0.507313	0.978034	0.060*
C11	0.6869 (2)	0.3264 (2)	0.98062 (17)	0.0492 (6)
H11	0.643147	0.300354	1.052185	0.059*
C12	0.7577 (2)	0.24010 (19)	0.91973 (16)	0.0405 (5)
C13	0.8226 (2)	0.28259 (18)	0.81374 (15)	0.0360 (4)
H13	0.869464	0.225492	0.771407	0.043*
C14	0.8192 (2)	0.40806 (18)	0.76934 (15)	0.0355 (5)
C15	0.7351 (3)	0.6308 (2)	0.7828 (2)	0.0734 (8)
H15A	0.672759	0.674199	0.834325	0.110*
H15B	0.698040	0.640291	0.720302	0.110*
H15C	0.828262	0.664817	0.763670	0.110*
C16	0.7672 (3)	0.1037 (2)	0.9659 (2)	0.0704 (8)
H16A	0.854196	0.085435	0.989195	0.106*
H16B	0.765939	0.055034	0.912536	0.106*
H16C	0.687379	0.083353	1.025583	0.106*
C17	0.29119 (19)	0.44302 (15)	0.80120 (13)	0.0267 (4)
C18	0.1466 (2)	0.45138 (17)	0.84845 (15)	0.0332 (4)
H18	0.089970	0.513913	0.818656	0.040*
C19	0.0851 (2)	0.36894 (17)	0.93867 (15)	0.0356 (5)
H19	−0.012149	0.375521	0.968950	0.043*
C20	0.1690 (2)	0.27650 (17)	0.98379 (15)	0.0365 (5)
C21	0.3135 (2)	0.26666 (17)	0.93802 (15)	0.0398 (5)
H21	0.369889	0.204221	0.968234	0.048*
C22	0.3739 (2)	0.34920 (16)	0.84780 (14)	0.0324 (4)
H22	0.471145	0.342192	0.817592	0.039*
C23	0.3543 (2)	0.53064 (16)	0.70188 (14)	0.0293 (4)

C24	0.73147 (18)	0.84066 (16)	0.39196 (14)	0.0283 (4)
C25	0.82153 (19)	0.85457 (17)	0.29055 (15)	0.0321 (4)
H25	0.858731	0.784245	0.261371	0.039*
C26	0.8563 (2)	0.97198 (17)	0.23265 (15)	0.0348 (4)
H26	0.916804	0.980486	0.164794	0.042*
C27	0.80129 (19)	1.07684 (16)	0.27555 (14)	0.0303 (4)
C28	0.70977 (19)	1.06454 (16)	0.37624 (15)	0.0321 (4)
H28	0.671854	1.135002	0.404962	0.039*
C29	0.67551 (19)	0.94683 (17)	0.43341 (15)	0.0326 (4)
H29	0.613906	0.938508	0.500819	0.039*
C30	0.6966 (2)	0.71342 (17)	0.45436 (15)	0.0319 (4)
N1	0.69424 (19)	0.35629 (15)	0.54491 (14)	0.0348 (4)
N2	0.8895 (2)	0.44564 (19)	0.66002 (15)	0.0488 (5)
O1	0.28132 (14)	0.62590 (11)	0.67266 (10)	0.0380 (3)
O2	0.47869 (14)	0.50631 (11)	0.64920 (10)	0.0391 (3)
O3	0.11541 (18)	0.19274 (14)	1.07406 (13)	0.0578 (5)
O4	0.57699 (16)	0.71376 (13)	0.52863 (13)	0.0515 (4)
O5	0.77000 (15)	0.62033 (12)	0.43902 (12)	0.0455 (4)
O6	0.83921 (16)	1.19187 (12)	0.21635 (11)	0.0446 (4)
H1A	0.755 (2)	0.4040 (18)	0.5552 (18)	0.057 (7)*
H1B	0.6106 (14)	0.379 (2)	0.5801 (16)	0.052 (7)*
H1C	0.698 (3)	0.363 (2)	0.4768 (9)	0.063 (8)*
H2A	0.898 (3)	0.5244 (10)	0.643 (2)	0.080 (9)*
H2B	0.9714 (16)	0.409 (2)	0.644 (2)	0.077 (10)*
H3A	0.0300 (14)	0.206 (3)	1.099 (3)	0.116*
H4A	0.555 (3)	0.6430 (15)	0.564 (2)	0.116*
H6A	0.798 (3)	1.248 (2)	0.248 (2)	0.116*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0304 (10)	0.0291 (9)	0.0274 (9)	−0.0006 (8)	−0.0047 (7)	−0.0066 (7)
C2	0.0305 (10)	0.0246 (9)	0.0270 (9)	0.0016 (7)	−0.0079 (7)	−0.0017 (7)
C3	0.0280 (10)	0.0344 (10)	0.0321 (10)	0.0014 (8)	−0.0061 (8)	−0.0024 (8)
C4	0.0402 (12)	0.0352 (10)	0.0346 (11)	−0.0067 (9)	−0.0087 (9)	0.0033 (8)
C5	0.0507 (13)	0.0272 (10)	0.0431 (12)	0.0037 (9)	−0.0168 (10)	0.0016 (8)
C6	0.0351 (11)	0.0340 (10)	0.0386 (11)	0.0106 (8)	−0.0108 (9)	−0.0094 (8)
C7	0.0324 (11)	0.0423 (11)	0.0404 (11)	−0.0010 (9)	−0.0002 (9)	−0.0066 (9)
C8	0.0565 (16)	0.0587 (15)	0.0611 (16)	−0.0163 (12)	−0.0068 (12)	0.0209 (12)
C9	0.0435 (13)	0.0386 (11)	0.0511 (13)	0.0002 (9)	−0.0115 (10)	−0.0137 (10)
C10	0.0416 (13)	0.0555 (14)	0.0532 (14)	0.0027 (11)	−0.0014 (10)	−0.0263 (12)
C11	0.0422 (13)	0.0647 (15)	0.0369 (12)	−0.0092 (11)	0.0037 (9)	−0.0137 (11)
C12	0.0399 (12)	0.0432 (11)	0.0370 (11)	−0.0069 (9)	−0.0063 (9)	−0.0054 (9)
C13	0.0364 (11)	0.0356 (10)	0.0353 (11)	−0.0017 (9)	−0.0044 (8)	−0.0099 (8)
C14	0.0334 (11)	0.0396 (11)	0.0343 (10)	−0.0070 (9)	−0.0060 (8)	−0.0089 (9)
C15	0.099 (2)	0.0411 (14)	0.078 (2)	0.0072 (14)	−0.0171 (17)	−0.0165 (13)
C16	0.096 (2)	0.0541 (15)	0.0500 (15)	−0.0076 (14)	−0.0086 (14)	0.0071 (12)
C17	0.0291 (10)	0.0227 (8)	0.0254 (9)	−0.0010 (7)	−0.0025 (7)	−0.0021 (7)

C18	0.0308 (10)	0.0305 (9)	0.0348 (10)	0.0013 (8)	−0.0058 (8)	−0.0007 (8)
C19	0.0267 (10)	0.0370 (10)	0.0372 (11)	−0.0046 (8)	0.0034 (8)	−0.0053 (8)
C20	0.0438 (12)	0.0292 (10)	0.0300 (10)	−0.0079 (9)	0.0010 (8)	0.0011 (8)
C21	0.0426 (12)	0.0315 (10)	0.0363 (11)	0.0069 (9)	−0.0038 (9)	0.0055 (8)
C22	0.0284 (10)	0.0324 (10)	0.0312 (10)	0.0037 (8)	−0.0015 (8)	−0.0025 (8)
C23	0.0328 (10)	0.0235 (9)	0.0287 (9)	−0.0025 (8)	−0.0032 (8)	−0.0021 (7)
C24	0.0236 (9)	0.0265 (9)	0.0325 (10)	−0.0014 (7)	−0.0054 (7)	−0.0009 (7)
C25	0.0303 (10)	0.0279 (9)	0.0356 (10)	0.0022 (8)	−0.0031 (8)	−0.0073 (8)
C26	0.0350 (11)	0.0338 (10)	0.0288 (10)	0.0009 (8)	0.0025 (8)	−0.0030 (8)
C27	0.0266 (10)	0.0280 (9)	0.0314 (10)	−0.0020 (7)	−0.0036 (7)	0.0027 (7)
C28	0.0320 (10)	0.0257 (9)	0.0356 (10)	0.0019 (8)	−0.0025 (8)	−0.0064 (8)
C29	0.0284 (10)	0.0326 (10)	0.0311 (10)	0.0001 (8)	0.0004 (8)	−0.0018 (8)
C30	0.0288 (10)	0.0296 (10)	0.0363 (10)	−0.0041 (8)	−0.0084 (8)	−0.0011 (8)
N1	0.0363 (10)	0.0284 (9)	0.0343 (10)	0.0036 (8)	−0.0029 (8)	−0.0013 (7)
N2	0.0612 (14)	0.0395 (11)	0.0398 (11)	−0.0093 (10)	−0.0009 (9)	−0.0037 (9)
O1	0.0447 (8)	0.0263 (7)	0.0345 (7)	0.0083 (6)	−0.0022 (6)	0.0023 (5)
O2	0.0354 (8)	0.0313 (7)	0.0373 (8)	0.0019 (6)	0.0073 (6)	0.0044 (6)
O3	0.0618 (11)	0.0443 (9)	0.0462 (9)	−0.0062 (8)	0.0100 (8)	0.0156 (7)
O4	0.0413 (9)	0.0330 (8)	0.0599 (10)	−0.0007 (7)	0.0115 (7)	0.0101 (7)
O5	0.0466 (9)	0.0274 (7)	0.0550 (9)	0.0009 (6)	−0.0031 (7)	−0.0017 (6)
O6	0.0520 (9)	0.0282 (7)	0.0404 (8)	−0.0031 (7)	0.0064 (7)	0.0036 (6)

Geometric parameters (Å, º)

C1—C6	1.385 (2)	C17—C18	1.387 (2)
C1—C2	1.386 (3)	C17—C22	1.392 (2)
C1—C7	1.503 (2)	C17—C23	1.489 (2)
C2—C3	1.375 (2)	C18—C19	1.379 (2)
C2—N1	1.465 (2)	C18—H18	0.9300
C3—C4	1.392 (3)	C19—C20	1.381 (3)
C3—H3	0.9300	C19—H19	0.9300
C4—C5	1.380 (3)	C20—O3	1.368 (2)
C4—C8	1.506 (3)	C20—C21	1.384 (3)
C5—C6	1.376 (3)	C21—C22	1.376 (2)
C5—H5	0.9300	C21—H21	0.9300
C6—H6	0.9300	C22—H22	0.9300
C7—H7A	0.9600	C23—O1	1.262 (2)
C7—H7B	0.9600	C23—O2	1.267 (2)
C7—H7C	0.9600	C24—C29	1.388 (2)
C8—H8A	0.9600	C24—C25	1.388 (2)
C8—H8B	0.9600	C24—C30	1.490 (2)
C8—H8C	0.9600	C25—C26	1.381 (2)
C9—C10	1.387 (3)	C25—H25	0.9300
C9—C14	1.389 (3)	C26—C27	1.382 (3)
C9—C15	1.503 (3)	C26—H26	0.9300
C10—C11	1.376 (3)	C27—O6	1.371 (2)
C10—H10	0.9300	C27—C28	1.387 (2)
C11—C12	1.381 (3)	C28—C29	1.380 (2)

C11—H11	0.9300	C28—H28	0.9300
C12—C13	1.385 (3)	C29—H29	0.9300
C12—C16	1.502 (3)	C30—O5	1.212 (2)
C13—C14	1.384 (3)	C30—O4	1.317 (2)
C13—H13	0.9300	N1—H1A	0.869 (9)
C14—N2	1.425 (3)	N1—H1B	0.871 (9)
C15—H15A	0.9600	N1—H1C	0.876 (10)
C15—H15B	0.9600	N2—H2A	0.852 (10)
C15—H15C	0.9600	N2—H2B	0.856 (10)
C16—H16A	0.9600	O3—H3A	0.824 (10)
C16—H16B	0.9600	O4—H4A	0.834 (10)
C16—H16C	0.9600	O6—H6A	0.830 (10)

C6—C1—C2	116.45 (16)	C12—C16—H16C	109.5
C6—C1—C7	121.39 (17)	H16A—C16—H16C	109.5
C2—C1—C7	122.15 (16)	H16B—C16—H16C	109.5
C3—C2—C1	122.61 (16)	C18—C17—C22	118.19 (16)
C3—C2—N1	118.80 (17)	C18—C17—C23	120.40 (16)
C1—C2—N1	118.55 (16)	C22—C17—C23	121.39 (16)
C2—C3—C4	119.97 (18)	C19—C18—C17	121.40 (17)
C2—C3—H3	120.0	C19—C18—H18	119.3
C4—C3—H3	120.0	C17—C18—H18	119.3
C5—C4—C3	117.97 (17)	C18—C19—C20	119.55 (17)
C5—C4—C8	121.70 (19)	C18—C19—H19	120.2
C3—C4—C8	120.3 (2)	C20—C19—H19	120.2
C6—C5—C4	121.20 (17)	O3—C20—C19	122.63 (18)
C6—C5—H5	119.4	O3—C20—C21	117.39 (18)
C4—C5—H5	119.4	C19—C20—C21	119.98 (16)
C5—C6—C1	121.67 (18)	C22—C21—C20	120.05 (18)
C5—C6—H6	119.2	C22—C21—H21	120.0
C1—C6—H6	119.2	C20—C21—H21	120.0
C1—C7—H7A	109.5	C21—C22—C17	120.83 (17)
C1—C7—H7B	109.5	C21—C22—H22	119.6
H7A—C7—H7B	109.5	C17—C22—H22	119.6
C1—C7—H7C	109.5	O1—C23—O2	122.37 (16)
H7A—C7—H7C	109.5	O1—C23—C17	118.75 (16)
H7B—C7—H7C	109.5	O2—C23—C17	118.87 (16)
C4—C8—H8A	109.5	C29—C24—C25	118.81 (16)
C4—C8—H8B	109.5	C29—C24—C30	121.15 (16)
H8A—C8—H8B	109.5	C25—C24—C30	120.04 (16)
C4—C8—H8C	109.5	C26—C25—C24	120.55 (17)
H8A—C8—H8C	109.5	C26—C25—H25	119.7
H8B—C8—H8C	109.5	C24—C25—H25	119.7
C10—C9—C14	117.50 (19)	C25—C26—C27	119.99 (17)
C10—C9—C15	120.9 (2)	C25—C26—H26	120.0
C14—C9—C15	121.6 (2)	C27—C26—H26	120.0
C11—C10—C9	122.1 (2)	O6—C27—C26	118.45 (16)
C11—C10—H10	118.9	O6—C27—C28	121.39 (16)

C9—C10—H10	118.9	C26—C27—C28	120.16 (16)
C10—C11—C12	120.2 (2)	C29—C28—C27	119.41 (17)
C10—C11—H11	119.9	C29—C28—H28	120.3
C12—C11—H11	119.9	C27—C28—H28	120.3
C11—C12—C13	118.20 (19)	C28—C29—C24	121.08 (17)
C11—C12—C16	121.8 (2)	C28—C29—H29	119.5
C13—C12—C16	120.0 (2)	C24—C29—H29	119.5
C14—C13—C12	121.52 (19)	O5—C30—O4	123.77 (16)
C14—C13—H13	119.2	O5—C30—C24	124.02 (17)
C12—C13—H13	119.2	O4—C30—C24	112.21 (16)
C13—C14—C9	120.33 (18)	C2—N1—H1A	108.4 (16)
C13—C14—N2	118.65 (18)	C2—N1—H1B	108.7 (15)
C9—C14—N2	120.99 (19)	H1A—N1—H1B	106 (2)
C9—C15—H15A	109.5	C2—N1—H1C	110.5 (16)
C9—C15—H15B	109.5	H1A—N1—H1C	111 (2)
H15A—C15—H15B	109.5	H1B—N1—H1C	111 (2)
C9—C15—H15C	109.5	C14—N2—H2A	112.1 (19)
H15A—C15—H15C	109.5	C14—N2—H2B	112.1 (19)
H15B—C15—H15C	109.5	H2A—N2—H2B	109 (3)
C12—C16—H16A	109.5	C20—O3—H3A	113 (2)
C12—C16—H16B	109.5	C30—O4—H4A	113 (2)
H16A—C16—H16B	109.5	C27—O6—H6A	111 (2)

C6—C1—C2—C3	−3.8 (3)	C23—C17—C18—C19	178.08 (17)
C7—C1—C2—C3	176.07 (18)	C17—C18—C19—C20	0.5 (3)
C6—C1—C2—N1	173.96 (17)	C18—C19—C20—O3	179.08 (19)
C7—C1—C2—N1	−6.1 (3)	C18—C19—C20—C21	−0.4 (3)
C1—C2—C3—C4	2.3 (3)	O3—C20—C21—C22	−179.26 (18)
N1—C2—C3—C4	−175.54 (18)	C19—C20—C21—C22	0.3 (3)
C2—C3—C4—C5	1.1 (3)	C20—C21—C22—C17	−0.2 (3)
C2—C3—C4—C8	−179.88 (19)	C18—C17—C22—C21	0.3 (3)
C3—C4—C5—C6	−2.7 (3)	C23—C17—C22—C21	−178.23 (17)
C8—C4—C5—C6	178.3 (2)	C18—C17—C23—O1	13.0 (3)
C4—C5—C6—C1	1.0 (3)	C22—C17—C23—O1	−168.48 (17)
C2—C1—C6—C5	2.2 (3)	C18—C17—C23—O2	−166.33 (17)
C7—C1—C6—C5	−177.70 (18)	C22—C17—C23—O2	12.2 (3)
C14—C9—C10—C11	−1.3 (3)	C29—C24—C25—C26	0.9 (3)
C15—C9—C10—C11	179.4 (2)	C30—C24—C25—C26	−179.00 (17)
C9—C10—C11—C12	−0.4 (4)	C24—C25—C26—C27	−0.1 (3)
C10—C11—C12—C13	0.6 (3)	C25—C26—C27—O6	179.34 (17)
C10—C11—C12—C16	179.7 (2)	C25—C26—C27—C28	−0.7 (3)
C11—C12—C13—C14	0.9 (3)	O6—C27—C28—C29	−179.42 (17)
C16—C12—C13—C14	−178.2 (2)	C26—C27—C28—C29	0.7 (3)
C12—C13—C14—C9	−2.6 (3)	C27—C28—C29—C24	0.2 (3)
C12—C13—C14—N2	179.36 (19)	C25—C24—C29—C28	−1.0 (3)
C10—C9—C14—C13	2.7 (3)	C30—C24—C29—C28	178.92 (17)
C15—C9—C14—C13	−177.9 (2)	C29—C24—C30—O5	−157.41 (19)
C10—C9—C14—N2	−179.3 (2)	C25—C24—C30—O5	22.5 (3)

C15—C9—C14—N2	0.1 (3)	C29—C24—C30—O4	22.3 (2)
C22—C17—C18—C19	−0.5 (3)	C25—C24—C30—O4	−157.83 (18)

Hydrogen-bond geometry (Å, º)

Cg1, Cg2 and Cg3 are the centroids of the C1–C6, C9–C14 and C24–C29 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5	0.87 (1)	2.55 (2)	3.015 (2)	114 (2)
N1—H1*A*···N2	0.87 (1)	2.24 (2)	3.016 (3)	149 (2)
N1—H1*B*···O2	0.87 (1)	2.00 (1)	2.792 (2)	151 (2)
O4—H4*A*···O2	0.83 (1)	1.78 (1)	2.6041 (17)	169 (3)
N1—H1*C*···O1^{i}	0.88 (1)	1.91 (1)	2.781 (2)	176 (2)
N2—H2*B*···O5^{ii}	0.86 (1)	2.49 (1)	3.306 (3)	159 (2)
O3—H3*A*···O6^{iii}	0.82 (1)	2.09 (2)	2.858 (2)	156 (3)
O6—H6*A*···O1^{iv}	0.83 (1)	1.88 (1)	2.7075 (19)	175 (3)
C13—H13···*Cg*1	0.93	2.80	3.437 (2)	127
C6—H6···*Cg*3^{ii}	0.93	2.87	3.554 (2)	132
C19—H19···*Cg*2^{v}	0.93	2.87	3.532 (2)	129

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