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ISSN 2414-3146

1-[(*E*)-1-(2-Carboxy-6-methylphenyl)diazen-1-ium-2-yl]naphthalen-2-olate

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Received 11 December 2025
Accepted 20 December 2025

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

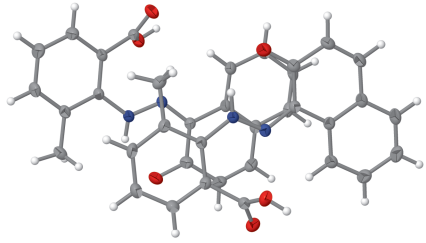
Keywords: azo compound; 2-naphthol; crystal structure.

CCDC reference: 2517770

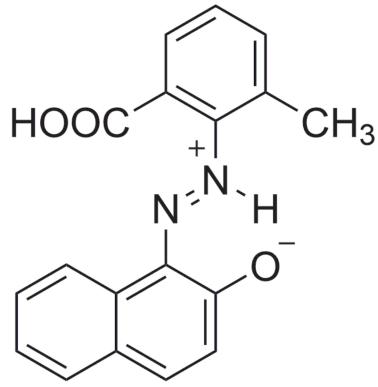
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{18}H_{14}N_2O_3$, crystallizes with two zwitterionic molecules (*A* and *B*) in the asymmetric unit, each featuring an intramolecular N—H···O hydrogen bond. In the extended structure, *A* + *A* and *B* + *B* carboxylic-acid inversion dimers linked by pairwise O—H···O hydrogen bonds arise, which generate $R_2^2(8)$ loops in each case. The dimers are linked by weak C—H···π and π–π stacking interactions.

3D view



Chemical scheme



Structure description

Azo compounds are crucial in dyes, pigments, and innovative materials (Wang *et al.*, 2003). As part of our studies in this area, we now describe the structure of the title compond, $C_{18}H_{14}N_2O_3$ (**I**), which crystallizes in the triclinic space group $P\overline{1}$ with two molecules (*A* and *B*) in the asymmetric unit (Fig. 1), in which nominal transfer of the hydrogen atom of the OH group to the N atom of the azo group has occurred to form a zwitterion in both molecules (Xu *et al.*, 2010). Intramolecular N2—H2*N*···O1 and N4—H4*N*···O4 hydrogen bonds arise within the zwitterions (Table 1). The dihedral angles between the benzene and naphthalene ring systems are 11.19 (9)° in *A* and 10.62 (9)° in *B*. The N=N bond lengths in the azo group [1.309 (2) Å in *A* and 1.311 (2) Å in *B*] are similar to those found in related structures, for example 1-(3-acetylphenyl)-2-(2-oxidonaphthalen-1-yl) diazen-1-ium (Bougueria *et al.*., 2013) and 1-[(*E*)-2-(2-fluorophenyl)diazan-1-ylidene] naphthalen-2(1*H*)-one (Akkache *et al.*, 2024). For molecule *A*, the C10—N1 [1.333 (2) Å] bond length is as expected for a C=N double bond and the C1—C10 and C9—C10 bond lengths are almost the same [1.457 (2) and 1.459 (2) Å, respectively]. However, the C1—C10—N1 bond angle [116.26 (13)°] is

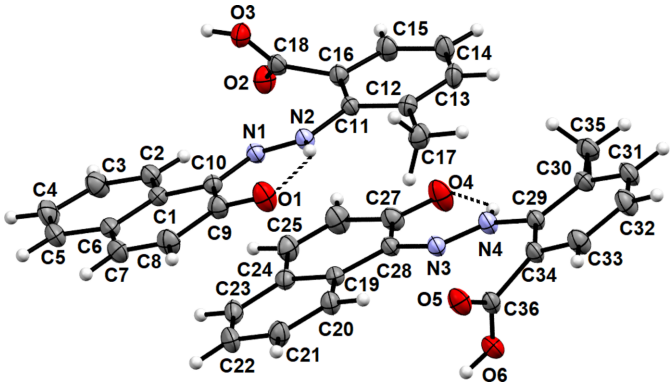


Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level.

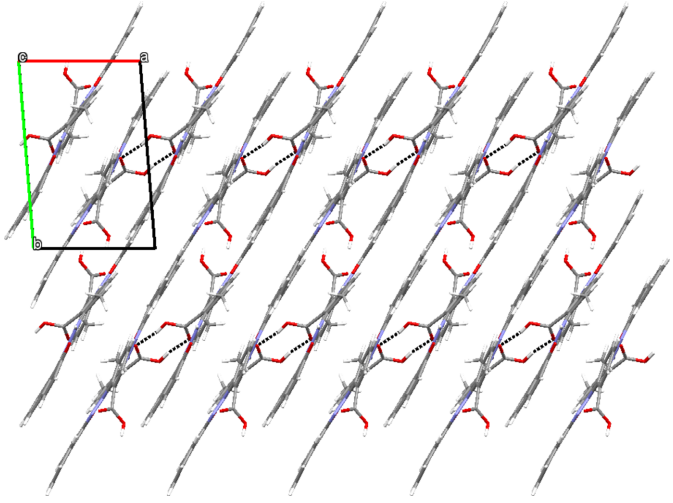


Figure 2
A packing diagram for (**I**) viewed along the *c*-axis direction (projection onto the *ab* plane), showing the O—H···O hydrogen bonds as black dashed lines.

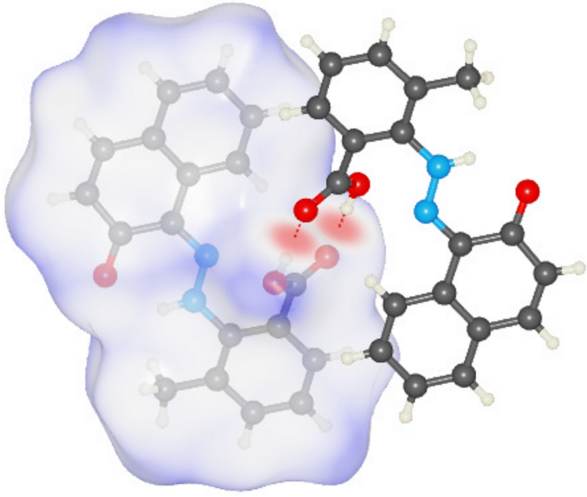


Figure 3
View of the three-dimensional Hirshfeld surface for molecule *A* over d_{norm} in the range of −0.73 to 1.45 a.u, showing the intense red spots associated with the carboxylic acid inversion dimer.

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

*Cg*1 and *Cg*5 are the centroids of the C1–C6 and C19–C24 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O1	0.88	1.86	2.5567 (17)	135
O3—H3*A*···O2^{i}	0.84	1.81	2.6480 (17)	173
N4—H4*N*···O4	0.88	1.86	2.5519 (18)	134
O6—H6*A*···O5^{ii}	0.84	1.81	2.6430 (18)	169
C17—H17*C*···*Cg*5^{iii}	0.98	2.81	3.6782 (18)	148
C35—H35*C*···*Cg*1^{iv}	0.98	2.70	3.5948 (18)	152

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

notably smaller than C9—C10—N1 [123.54 (14)°] possibly due to steric repulsion between N1 and O1.

In the crystal, the *A* and *B* molecules are linked by pairs of O—H···O hydrogen bonds associated with the carboxylic acid groups (Fig. 2, Table 1) to form *A* + *A* and *B* + *B* inversion dimers enclosing $R_2^2(8)$ loops in each case. The packing is consolidated by weak C—H···π interactions (Table 1) and aromatic π–π stacking, the shortest centroid–centroid separation being 3.6672 (10) Å between the C11–C16 and C19/C24–C28 rings.

The intermolecular interactions in (**I**) were further investigated and visualized using *CrystalExplorer* (Spackman *et al.*., 2021). Fig. 3 shows the Hirshfeld surface for molecule *A* and Fig. 4 illustrates the two-dimensional fingerprint plots for molecule *A* encompassing all intermolecular interactions and

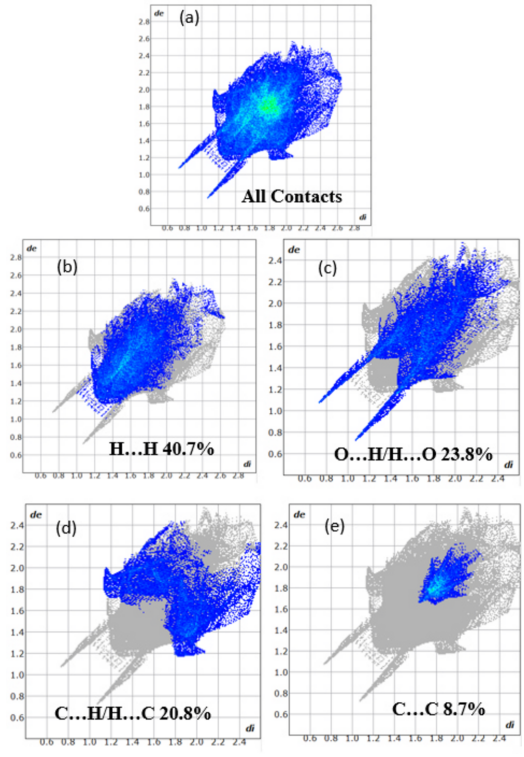


Figure 4
(*a*) The full two-dimensional fingerprint plot calculated for molecule *A* and those delineated into (*b*) H···H contacts, (*c*) O···H/H···O contacts, (*d*) C···H/H···C contacts and (*e*) C···C contacts.

the different contact types. The most significant contributions are H···H (40.7%), O···H/H···O (23.2%), C···H/H···C (20.8%) and C···C (8.7%) contacts. The Hirshfeld surface for molecule *B* is almost identical in appearance and the corresponding contact percentages are 39.4%, 25.5%, 20.7% and 8.3%, respectively.

Synthesis and crystallization

The title compound was synthesized according to a reported method (Wang *et al.*, 2003). The crude azo dye was recrystallized from hot ethanol solution in 80% yield and single crystals suitable for X-ray analysis were obtained by dissolving the compound in a minimum amount of tetrahydrofuran and water (1/1 *v*/*v*) at room temperature followed by slow evaporation.

Refinement

Crystallographic data and refinement parameters for the title compound are given in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{14}N_2O_3$
M_r	306.31
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, b, c (Å)	7.7505 (4), 11.8612 (7), 16.2866 (9)
α, β, γ (°)	83.111 (1), 79.292 (1), 84.218 (1)
V (Å^3)	1455.81 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.40 × 0.30 × 0.20
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.653, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23441, 8414, 5800
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.703
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.157, 1.04
No. of reflections	8414
No. of parameters	419
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.54, −0.34

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick 2015*a*), *SHELXL2014* (Sheldrick 2015*b*) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

The authors acknowledge the Algerian Ministry of Higher Education and Scientific Research, the Algerian Directorate for Scientific Research and Technological Development. Mme Corine Bailly from the University of Strasbourg, France, is thanked for collecting the data.

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full crystallographic data

IUCrData (2025). **10**, x251148 [https://doi.org/10.1107/S2414314625011484]

1-[(*E*)-1-(2-Carboxy-6-methylphenyl)diazen-1-ium-2-yl]naphthalen-2-olate

Nabila Benabila, Ouarda Brihi, Souheyla Chetioui and Jean-Pierre Djukic

(*E*)-1-(2-Carboxy-6-methylphenyl)-2-(2-oxidonaphthalen-1-yl)diazen-1-ium

Crystal data

$C_{18}H_{14}N_2O_3$
M_r = 306.31
Triclinic, $P\overline{1}$
a = 7.7505 (4) Å
b = 11.8612 (7) Å
c = 16.2866 (9) Å
α = 83.111 (1)°
β = 79.292 (1)°
γ = 84.218 (1)°
V = 1455.81 (14) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.7107 Å
Cell parameters from 8371 reflections
θ = 2.3–29.9°
μ = 0.10 mm^{-1}
T = 173 K
Block, red
0.40 × 0.30 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.0226 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.653, T_{max} = 0.746
23441 measured reflections
8414 independent reflections
5800 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 30.0°, θ_{min} = 1.3°
h = −10→10
k = −16→16
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.157
S = 1.04
8414 reflections
419 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 0.5416P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.38498 (18)	0.87201 (11)	0.39238 (7)	0.0344 (4)
O2	0.43533 (17)	0.86654 (10)	0.00964 (7)	0.0306 (4)
O3	0.62784 (15)	0.93350 (10)	0.07536 (7)	0.0278 (3)
N1	0.36970 (17)	0.88290 (11)	0.21732 (8)	0.0210 (3)
N2	0.46912 (17)	0.79838 (11)	0.24785 (8)	0.0218 (3)
C1	0.18485 (19)	1.05409 (13)	0.22892 (10)	0.0205 (4)
C2	0.1720 (2)	1.06406 (15)	0.14327 (10)	0.0261 (5)
C3	0.0828 (2)	1.15847 (16)	0.10777 (11)	0.0315 (5)
C4	0.0047 (2)	1.24658 (16)	0.15611 (12)	0.0345 (5)
O4	0.23104 (19)	0.50791 (12)	0.10738 (8)	0.0380 (4)
O5	0.22532 (16)	0.48158 (11)	0.48780 (8)	0.0320 (4)
C5	0.0109 (2)	1.23640 (15)	0.24043 (11)	0.0296 (5)
O6	0.03996 (15)	0.39734 (10)	0.42839 (8)	0.0298 (3)
C6	0.0985 (2)	1.14012 (14)	0.27841 (10)	0.0234 (4)
C7	0.1027 (2)	1.12783 (15)	0.36725 (10)	0.0280 (5)
C8	0.1932 (2)	1.03958 (15)	0.40481 (11)	0.0297 (5)
C9	0.2944 (2)	0.95199 (14)	0.35711 (10)	0.0251 (4)
C10	0.2868 (2)	0.95937 (13)	0.26776 (9)	0.0200 (4)
C11	0.55681 (19)	0.71804 (13)	0.19467 (9)	0.0201 (4)
C12	0.6230 (2)	0.61409 (13)	0.23267 (10)	0.0224 (4)
C13	0.7000 (2)	0.53091 (14)	0.18083 (11)	0.0282 (5)
C14	0.7088 (3)	0.54803 (15)	0.09399 (11)	0.0317 (5)
C15	0.6457 (2)	0.65149 (14)	0.05738 (10)	0.0281 (5)
C16	0.5745 (2)	0.73871 (13)	0.10678 (10)	0.0225 (4)
C17	0.6097 (2)	0.59299 (15)	0.32641 (10)	0.0290 (5)
C18	0.5367 (2)	0.85371 (13)	0.06156 (9)	0.0229 (4)
N3	0.21643 (17)	0.51973 (11)	0.28291 (8)	0.0214 (3)
N4	0.29368 (17)	0.42655 (11)	0.25216 (8)	0.0230 (4)
C19	0.05156 (19)	0.69943 (13)	0.27119 (9)	0.0200 (4)
C20	0.0364 (2)	0.70934 (14)	0.35725 (10)	0.0252 (5)
C21	−0.0629 (2)	0.80025 (15)	0.39276 (11)	0.0301 (5)
C22	−0.1516 (2)	0.88391 (15)	0.34388 (12)	0.0309 (5)
C23	−0.1325 (2)	0.87698 (14)	0.25871 (11)	0.0270 (5)
C24	−0.0298 (2)	0.78662 (13)	0.22098 (10)	0.0228 (4)
C25	−0.0049 (2)	0.78090 (15)	0.13164 (11)	0.0293 (5)
C26	0.0838 (3)	0.69271 (16)	0.09449 (11)	0.0332 (5)
C27	0.1578 (2)	0.59493 (15)	0.14226 (10)	0.0274 (5)
C28	0.14551 (19)	0.60074 (13)	0.23229 (9)	0.0203 (4)
C29	0.3755 (2)	0.34388 (13)	0.30435 (9)	0.0211 (4)
C30	0.4950 (2)	0.26046 (14)	0.26575 (10)	0.0238 (4)
C31	0.5887 (2)	0.18429 (15)	0.31662 (11)	0.0295 (5)
C32	0.5685 (2)	0.19012 (16)	0.40255 (11)	0.0329 (5)
C33	0.4463 (2)	0.27065 (15)	0.44036 (10)	0.0291 (5)
C34	0.3452 (2)	0.34531 (13)	0.39205 (10)	0.0228 (4)
C35	0.5201 (2)	0.25318 (15)	0.17265 (10)	0.0289 (5)

C36	0.1952 (2)	0.41597 (13)	0.43802 (9)	0.0232 (4)
H2	0.22515	1.00542	0.10968	0.0313*
H2N	0.48081	0.79203	0.30097	0.0261*
H3	0.07416	1.16391	0.04998	0.0377*
H3A	0.60056	0.99462	0.04723	0.0416*
H4	−0.05205	1.31281	0.13065	0.0414*
H5	−0.04471	1.29505	0.27348	0.0355*
H7	0.03934	1.18407	0.40058	0.0336*
H8	0.19046	1.03473	0.46368	0.0356*
H13	0.74770	0.46065	0.20522	0.0338*
H14	0.75797	0.48889	0.06012	0.0381*
H15	0.65084	0.66315	−0.00174	0.0337*
H17A	0.65978	0.51569	0.34135	0.0435*
H17B	0.48567	0.60117	0.35334	0.0435*
H17C	0.67494	0.64834	0.34567	0.0435*
H4N	0.29410	0.41611	0.19953	0.0277*
H6A	−0.03481	0.44257	0.45454	0.0447*
H20	0.09483	0.65309	0.39134	0.0303*
H21	−0.07122	0.80622	0.45104	0.0361*
H22	−0.22375	0.94450	0.36909	0.0371*
H23	−0.18988	0.93441	0.22505	0.0324*
H25	−0.05344	0.84233	0.09776	0.0352*
H26	0.09873	0.69423	0.03515	0.0398*
H31	0.66866	0.12670	0.29178	0.0354*
H32	0.63793	0.13929	0.43535	0.0395*
H33	0.43175	0.27479	0.49917	0.0350*
H35A	0.60926	0.19113	0.15721	0.0433*
H35B	0.55890	0.32531	0.14245	0.0433*
H35C	0.40830	0.23836	0.15765	0.0433*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0439 (7)	0.0352 (7)	0.0232 (6)	0.0110 (6)	−0.0105 (5)	−0.0040 (5)
O2	0.0385 (7)	0.0277 (6)	0.0262 (6)	0.0018 (5)	−0.0117 (5)	0.0002 (5)
O3	0.0306 (6)	0.0207 (6)	0.0309 (6)	−0.0006 (5)	−0.0065 (5)	0.0021 (5)
N1	0.0206 (6)	0.0195 (6)	0.0218 (6)	0.0015 (5)	−0.0028 (5)	−0.0021 (5)
N2	0.0251 (6)	0.0205 (6)	0.0183 (6)	0.0046 (5)	−0.0035 (5)	−0.0025 (5)
C1	0.0187 (7)	0.0191 (7)	0.0228 (7)	−0.0004 (5)	−0.0019 (5)	−0.0018 (5)
C2	0.0267 (8)	0.0262 (8)	0.0241 (8)	0.0039 (7)	−0.0031 (6)	−0.0044 (6)
C3	0.0313 (9)	0.0343 (10)	0.0266 (8)	0.0061 (7)	−0.0064 (7)	0.0003 (7)
C4	0.0317 (9)	0.0295 (9)	0.0394 (10)	0.0096 (7)	−0.0086 (7)	0.0016 (8)
O4	0.0514 (8)	0.0378 (7)	0.0233 (6)	0.0138 (6)	−0.0080 (5)	−0.0096 (5)
O5	0.0361 (7)	0.0323 (7)	0.0280 (6)	0.0059 (5)	−0.0057 (5)	−0.0124 (5)
C5	0.0256 (8)	0.0233 (8)	0.0374 (9)	0.0052 (7)	−0.0017 (7)	−0.0056 (7)
O6	0.0260 (6)	0.0275 (6)	0.0332 (6)	0.0040 (5)	0.0007 (5)	−0.0069 (5)
C6	0.0202 (7)	0.0202 (8)	0.0287 (8)	−0.0003 (6)	−0.0015 (6)	−0.0039 (6)
C7	0.0307 (8)	0.0245 (8)	0.0275 (8)	0.0011 (7)	0.0003 (7)	−0.0092 (6)

C8	0.0370 (9)	0.0298 (9)	0.0219 (8)	0.0020 (7)	−0.0034 (7)	−0.0081 (7)
C9	0.0289 (8)	0.0238 (8)	0.0225 (7)	0.0004 (6)	−0.0040 (6)	−0.0044 (6)
C10	0.0213 (7)	0.0178 (7)	0.0204 (7)	−0.0006 (6)	−0.0029 (5)	−0.0022 (5)
C11	0.0199 (7)	0.0182 (7)	0.0210 (7)	0.0016 (5)	−0.0019 (5)	−0.0031 (5)
C12	0.0240 (7)	0.0189 (7)	0.0235 (7)	−0.0007 (6)	−0.0041 (6)	0.0001 (6)
C13	0.0345 (9)	0.0163 (7)	0.0312 (9)	0.0033 (7)	−0.0041 (7)	0.0010 (6)
C14	0.0413 (10)	0.0213 (8)	0.0302 (9)	0.0047 (7)	−0.0014 (7)	−0.0068 (7)
C15	0.0368 (9)	0.0247 (8)	0.0211 (7)	0.0023 (7)	−0.0025 (6)	−0.0041 (6)
C16	0.0252 (7)	0.0196 (7)	0.0216 (7)	0.0018 (6)	−0.0039 (6)	−0.0015 (6)
C17	0.0360 (9)	0.0255 (8)	0.0243 (8)	0.0009 (7)	−0.0071 (7)	0.0024 (6)
C18	0.0259 (7)	0.0210 (8)	0.0191 (7)	0.0031 (6)	0.0004 (5)	−0.0019 (6)
N3	0.0220 (6)	0.0188 (6)	0.0225 (6)	0.0024 (5)	−0.0029 (5)	−0.0033 (5)
N4	0.0257 (7)	0.0215 (7)	0.0207 (6)	0.0052 (5)	−0.0038 (5)	−0.0037 (5)
C19	0.0188 (7)	0.0170 (7)	0.0234 (7)	−0.0004 (5)	−0.0024 (5)	−0.0020 (5)
C20	0.0301 (8)	0.0204 (8)	0.0245 (8)	0.0025 (6)	−0.0049 (6)	−0.0031 (6)
C21	0.0388 (9)	0.0232 (8)	0.0276 (8)	−0.0001 (7)	−0.0038 (7)	−0.0055 (6)
C22	0.0315 (9)	0.0203 (8)	0.0394 (10)	0.0041 (7)	−0.0023 (7)	−0.0079 (7)
C23	0.0262 (8)	0.0181 (8)	0.0364 (9)	0.0020 (6)	−0.0086 (7)	−0.0004 (6)
C24	0.0216 (7)	0.0197 (7)	0.0272 (8)	−0.0014 (6)	−0.0055 (6)	−0.0014 (6)
C25	0.0324 (9)	0.0273 (9)	0.0279 (8)	0.0029 (7)	−0.0110 (7)	0.0029 (7)
C26	0.0423 (10)	0.0354 (10)	0.0205 (8)	0.0059 (8)	−0.0089 (7)	0.0001 (7)
C27	0.0289 (8)	0.0295 (9)	0.0226 (8)	0.0030 (7)	−0.0041 (6)	−0.0031 (6)
C28	0.0197 (7)	0.0198 (7)	0.0206 (7)	0.0017 (6)	−0.0029 (5)	−0.0024 (5)
C29	0.0219 (7)	0.0192 (7)	0.0209 (7)	0.0034 (6)	−0.0020 (5)	−0.0035 (6)
C30	0.0229 (7)	0.0220 (8)	0.0245 (8)	0.0028 (6)	0.0004 (6)	−0.0049 (6)
C31	0.0281 (8)	0.0261 (9)	0.0310 (9)	0.0100 (7)	−0.0007 (7)	−0.0066 (7)
C32	0.0331 (9)	0.0307 (9)	0.0322 (9)	0.0117 (7)	−0.0079 (7)	−0.0008 (7)
C33	0.0325 (9)	0.0301 (9)	0.0228 (8)	0.0074 (7)	−0.0049 (6)	−0.0029 (6)
C34	0.0240 (7)	0.0205 (7)	0.0221 (7)	0.0050 (6)	−0.0013 (6)	−0.0051 (6)
C35	0.0302 (8)	0.0293 (9)	0.0257 (8)	0.0028 (7)	0.0006 (6)	−0.0098 (7)
C36	0.0280 (8)	0.0202 (7)	0.0186 (7)	0.0053 (6)	−0.0013 (6)	−0.0014 (5)

Geometric parameters (Å, º)

O1—C9	1.268 (2)	C8—H8	0.9500
O2—C18	1.244 (2)	C13—H13	0.9500
O3—C18	1.300 (2)	C14—H14	0.9500
N1—N2	1.3089 (19)	C15—H15	0.9500
N1—C10	1.333 (2)	C17—H17C	0.9800
N2—C11	1.403 (2)	C17—H17B	0.9800
O3—H3A	0.8400	C17—H17A	0.9800
C1—C2	1.407 (2)	N4—H4N	0.8800
C1—C10	1.457 (2)	C19—C20	1.403 (2)
C1—C6	1.414 (2)	C19—C24	1.412 (2)
C2—C3	1.380 (2)	C19—C28	1.462 (2)
N2—H2N	0.8800	C20—C21	1.381 (2)
C3—C4	1.404 (3)	C21—C22	1.403 (2)
O4—C27	1.270 (2)	C22—C23	1.379 (3)

C4—C5	1.374 (3)	C23—C24	1.402 (2)
C5—C6	1.407 (2)	C24—C25	1.441 (2)
O5—C36	1.255 (2)	C25—C26	1.336 (3)
C6—C7	1.443 (2)	C26—C27	1.447 (3)
O6—C36	1.286 (2)	C27—C28	1.461 (2)
C7—C8	1.349 (2)	C29—C30	1.407 (2)
C8—C9	1.450 (2)	C29—C34	1.406 (2)
C9—C10	1.459 (2)	C30—C35	1.504 (2)
C11—C12	1.405 (2)	C30—C31	1.394 (2)
C11—C16	1.406 (2)	C31—C32	1.388 (2)
C12—C17	1.503 (2)	C32—C33	1.390 (2)
C12—C13	1.392 (2)	C33—C34	1.393 (2)
C13—C14	1.394 (2)	C34—C36	1.498 (2)
C14—C15	1.383 (2)	C20—H20	0.9500
C15—C16	1.394 (2)	C21—H21	0.9500
C16—C18	1.499 (2)	C22—H22	0.9500
C2—H2	0.9500	C23—H23	0.9500
C3—H3	0.9500	C25—H25	0.9500
N3—C28	1.333 (2)	C26—H26	0.9500
N3—N4	1.3110 (18)	C31—H31	0.9500
N4—C29	1.400 (2)	C32—H32	0.9500
C4—H4	0.9500	C33—H33	0.9500
C5—H5	0.9500	C35—H35A	0.9800
O6—H6A	0.8400	C35—H35B	0.9800
C7—H7	0.9500	C35—H35C	0.9800

N2—N1—C10	119.06 (13)	H17B—C17—H17C	109.00
N1—N2—C11	119.05 (12)	C12—C17—H17A	109.00
C18—O3—H3A	109.00	H17A—C17—H17B	109.00
C2—C1—C6	118.73 (14)	N3—N4—H4N	121.00
C2—C1—C10	122.21 (14)	C29—N4—H4N	121.00
C6—C1—C10	119.04 (14)	C20—C19—C24	118.81 (14)
C11—N2—H2N	120.00	C20—C19—C28	122.14 (14)
N1—N2—H2N	120.00	C24—C19—C28	119.04 (13)
C1—C2—C3	120.41 (16)	C19—C20—C21	120.47 (15)
C2—C3—C4	120.73 (16)	C20—C21—C22	120.84 (16)
C3—C4—C5	119.57 (17)	C21—C22—C23	119.06 (16)
C4—C5—C6	120.76 (16)	C22—C23—C24	121.09 (15)
C1—C6—C5	119.68 (15)	C19—C24—C23	119.58 (15)
C1—C6—C7	119.26 (15)	C19—C24—C25	119.12 (14)
C5—C6—C7	121.07 (15)	C23—C24—C25	121.30 (15)
C6—C7—C8	122.53 (16)	C24—C25—C26	122.66 (16)
C7—C8—C9	121.29 (16)	C25—C26—C27	121.74 (16)
C8—C9—C10	117.48 (14)	C26—C27—C28	117.19 (15)
O1—C9—C8	120.79 (15)	O4—C27—C26	121.45 (15)
O1—C9—C10	121.73 (14)	O4—C27—C28	121.36 (15)
N1—C10—C1	116.26 (13)	N3—C28—C19	116.57 (13)
N1—C10—C9	123.54 (14)	N3—C28—C27	123.54 (14)

C1—C10—C9	120.21 (14)	C19—C28—C27	119.89 (14)
C12—C11—C16	120.85 (14)	C30—C29—C34	120.53 (14)
N2—C11—C12	117.39 (13)	N4—C29—C30	117.62 (13)
N2—C11—C16	121.75 (14)	N4—C29—C34	121.81 (14)
C11—C12—C17	121.07 (14)	C29—C30—C35	121.13 (14)
C11—C12—C13	117.90 (14)	C29—C30—C31	117.87 (15)
C13—C12—C17	121.03 (14)	C31—C30—C35	121.01 (15)
C12—C13—C14	121.69 (16)	C30—C31—C32	121.91 (16)
C13—C14—C15	119.71 (16)	C31—C32—C33	119.70 (16)
C14—C15—C16	120.32 (15)	C32—C33—C34	120.01 (15)
C15—C16—C18	116.92 (14)	C33—C34—C36	117.25 (14)
C11—C16—C15	119.33 (14)	C29—C34—C33	119.72 (14)
C11—C16—C18	123.49 (14)	C29—C34—C36	122.73 (14)
O2—C18—O3	123.85 (14)	O5—C36—O6	124.05 (15)
O2—C18—C16	120.62 (14)	O5—C36—C34	119.46 (14)
O3—C18—C16	115.27 (13)	O6—C36—C34	116.23 (13)
C1—C2—H2	120.00	C19—C20—H20	120.00
C3—C2—H2	120.00	C21—C20—H20	120.00
C2—C3—H3	120.00	C20—C21—H21	120.00
C4—C3—H3	120.00	C22—C21—H21	120.00
N4—N3—C28	119.03 (13)	C21—C22—H22	120.00
C3—C4—H4	120.00	C23—C22—H22	120.00
C5—C4—H4	120.00	C22—C23—H23	119.00
N3—N4—C29	118.99 (12)	C24—C23—H23	119.00
C4—C5—H5	120.00	C24—C25—H25	119.00
C6—C5—H5	120.00	C26—C25—H25	119.00
C36—O6—H6A	109.00	C25—C26—H26	119.00
C6—C7—H7	119.00	C27—C26—H26	119.00
C8—C7—H7	119.00	C30—C31—H31	119.00
C7—C8—H8	119.00	C32—C31—H31	119.00
C9—C8—H8	119.00	C31—C32—H32	120.00
C12—C13—H13	119.00	C33—C32—H32	120.00
C14—C13—H13	119.00	C32—C33—H33	120.00
C15—C14—H14	120.00	C34—C33—H33	120.00
C13—C14—H14	120.00	C30—C35—H35A	109.00
C14—C15—H15	120.00	C30—C35—H35B	109.00
C16—C15—H15	120.00	C30—C35—H35C	109.00
C12—C17—H17B	109.00	H35A—C35—H35B	110.00
C12—C17—H17C	109.00	H35A—C35—H35C	109.00
H17A—C17—H17C	109.00	H35B—C35—H35C	109.00

C10—N1—N2—C11	−179.82 (14)	C28—N3—N4—C29	176.97 (14)
N2—N1—C10—C9	−2.7 (2)	N4—N3—C28—C27	−3.8 (2)
N2—N1—C10—C1	177.27 (13)	N4—N3—C28—C19	176.25 (13)
N1—N2—C11—C12	−163.54 (14)	N3—N4—C29—C30	−163.68 (14)
N1—N2—C11—C16	14.9 (2)	N3—N4—C29—C34	14.2 (2)
C6—C1—C2—C3	2.6 (2)	C24—C19—C20—C21	2.9 (2)
C10—C1—C2—C3	−176.15 (15)	C28—C19—C20—C21	−175.81 (15)

C2—C1—C10—N1	0.9 (2)	C20—C19—C28—N3	0.2 (2)
C2—C1—C10—C9	−179.24 (15)	C20—C19—C28—C27	−179.80 (14)
C6—C1—C10—N1	−177.94 (14)	C24—C19—C28—N3	−178.51 (14)
C2—C1—C6—C5	−3.7 (2)	C20—C19—C24—C23	−4.0 (2)
C2—C1—C6—C7	176.68 (15)	C20—C19—C24—C25	175.84 (15)
C10—C1—C6—C5	175.15 (15)	C28—C19—C24—C23	174.73 (14)
C10—C1—C6—C7	−4.5 (2)	C28—C19—C24—C25	−5.5 (2)
C6—C1—C10—C9	2.0 (2)	C24—C19—C28—C27	1.5 (2)
C1—C2—C3—C4	0.6 (2)	C19—C20—C21—C22	0.5 (2)
C2—C3—C4—C5	−2.7 (3)	C20—C21—C22—C23	−2.7 (2)
C3—C4—C5—C6	1.7 (2)	C21—C22—C23—C24	1.5 (2)
C4—C5—C6—C7	−178.81 (16)	C22—C23—C24—C25	−178.01 (15)
C4—C5—C6—C1	1.6 (2)	C22—C23—C24—C19	1.8 (2)
C1—C6—C7—C8	3.2 (2)	C19—C24—C25—C26	4.1 (3)
C5—C6—C7—C8	−176.44 (16)	C23—C24—C25—C26	−176.08 (18)
C6—C7—C8—C9	0.9 (3)	C24—C25—C26—C27	1.5 (3)
C7—C8—C9—O1	177.00 (16)	C25—C26—C27—O4	174.93 (18)
C7—C8—C9—C10	−3.4 (2)	C25—C26—C27—C28	−5.4 (3)
O1—C9—C10—C1	−178.45 (15)	O4—C27—C28—C19	−176.53 (15)
C8—C9—C10—N1	−178.19 (15)	C26—C27—C28—N3	−176.17 (16)
C8—C9—C10—C1	1.9 (2)	C26—C27—C28—C19	3.8 (2)
O1—C9—C10—N1	1.5 (3)	O4—C27—C28—N3	3.5 (2)
N2—C11—C12—C13	175.89 (14)	N4—C29—C30—C31	174.30 (14)
N2—C11—C12—C17	−3.6 (2)	N4—C29—C30—C35	−6.1 (2)
C16—C11—C12—C13	−2.5 (2)	C34—C29—C30—C31	−3.6 (2)
C16—C11—C12—C17	177.99 (14)	C34—C29—C30—C35	176.01 (15)
N2—C11—C16—C15	−173.07 (14)	N4—C29—C34—C33	−171.64 (15)
N2—C11—C16—C18	13.0 (2)	N4—C29—C34—C36	14.8 (2)
C12—C11—C16—C15	5.3 (2)	C30—C29—C34—C33	6.2 (2)
C12—C11—C16—C18	−168.69 (15)	C30—C29—C34—C36	−167.36 (15)
C11—C12—C13—C14	−1.3 (2)	C29—C30—C31—C32	−0.9 (2)
C17—C12—C13—C14	178.15 (17)	C35—C30—C31—C32	179.48 (16)
C12—C13—C14—C15	2.4 (3)	C30—C31—C32—C33	2.8 (3)
C13—C14—C15—C16	0.4 (3)	C31—C32—C33—C34	−0.2 (3)
C14—C15—C16—C11	−4.2 (2)	C32—C33—C34—C29	−4.2 (2)
C14—C15—C16—C18	170.20 (17)	C32—C33—C34—C36	169.68 (15)
C11—C16—C18—O2	−127.69 (17)	C29—C34—C36—O5	−127.72 (17)
C11—C16—C18—O3	58.0 (2)	C29—C34—C36—O6	57.9 (2)
C15—C16—C18—O2	58.2 (2)	C33—C34—C36—O5	58.6 (2)
C15—C16—C18—O3	−116.11 (16)	C33—C34—C36—O6	−115.79 (17)

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*5 are the centroids of the C1–C6 and C19–C24 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O1	0.88	1.86	2.5567 (17)	135
O3—H3*A*···O2^{i}	0.84	1.81	2.6480 (17)	173
N4—H4*N*···O4	0.88	1.86	2.5519 (18)	134

O6—H6*A*···O5^{ii}	0.84	1.81	2.6430 (18)	169
C17—H17*C*···*Cg*5^{iii}	0.98	2.81	3.6782 (18)	148
C35—H35*C*···*Cg*1^{iv}	0.98	2.70	3.5948 (18)	152

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