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2-(2-Methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol-3-ium 2,4,6-trinitrophenolate

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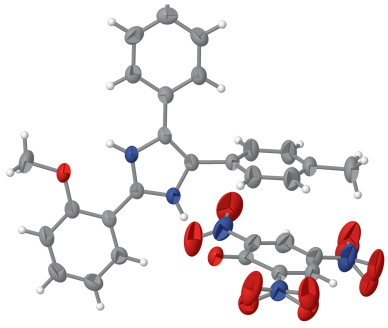
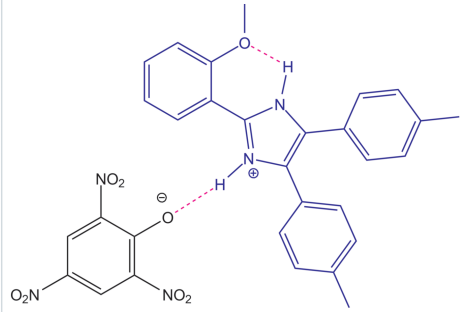
The title imidazolium picrate salt, $C_{24}H_{23}N_2O^+\cdot C_6H_2N_3O_7^-$, crystallizes in the triclinic space group $P\overline{1}$. The asymmetric unit consists of one imidazolium cation and one picrate anion. The molecular structure is consolidated by an intramolecular N—H···O hydrogen bond within the imidazolium cation and by an intermolecular N—H···O hydrogen bond between the imidazolium cation and the picrate anion. In the crystal, the ions are further associated through π–π stacking interactions, contributing to the supramolecular packing arrangement. Two nitro groups of the picrate anion were modelled as disordered over two positions.

3D view



Chemical scheme



Structure description

Imidazole and its derivatives are an important class of heterocycles in medicinal chemistry and materials science because of their diverse biological properties and their ability to participate in supramolecular assembly (Li *et al.*, 2023). Substituted imidazolium salts are of structural interest since their molecular conformations and crystal packing are often governed by non-covalent interactions such as hydrogen bonding and π–π stacking (Desiraju, 2002). Picric acid, 2,4,6-trinitrophenol, is a common acidic co-former for the formation of organic salts with nitrogen-containing bases (Bertolasi *et al.*, 2011). Related imidazolium picrate structures have also been reported previously (Du & Zhao, 2003; Dutkiewicz *et al.*, 2011; Solo *et al.*, 2025). In the present study, the crystal structure of 2-(2-methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol-3-ium 2,4,6-trinitrophenolate was determined in order to establish the molecular conformation of the imidazolium cation

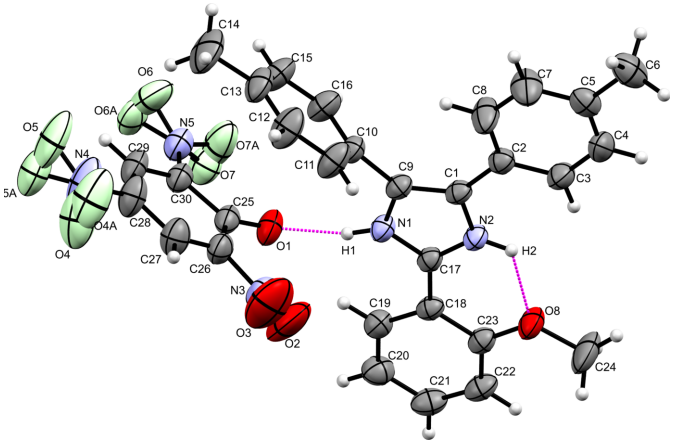


Figure 1
Asymmetric unit of the title imidazolium picrate salt, with displacement ellipsoids drawn at the 50% probability level. Dashed lines (magenta) indicate N—H···O hydrogen-bonding interactions. The split oxygen atoms of two nitro groups are coloured in green.

and to examine the hydrogen-bonding and π–π stacking interactions responsible for the crystal packing.

The asymmetric unit of the title salt, $C_{24}H_{23}N_2O^+\cdot C_6H_2N_3O_7^-$, contains one 2-(2-methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol-3-ium cation and one 2,4,6-trinitrophenolate anion (Fig. 1). Proton transfer from picric acid to the imidazole N atom gives the imidazolium cation and the picrate anion.

The molecular conformation is consolidated by an intramolecular N2—H2···O8 hydrogen bond within the imidazolium cation, involving the imidazolium N—H group and the methoxy O atom. In addition, an intermolecular N1—H1···O1 hydrogen bond links the imidazolium cation to the picrate anion. These N—H···O interactions help organize the cation–anion pairs and contribute to the crystal packing arrangement (Fig. 2). The hydrogen-bonding details are listed in Table 1.

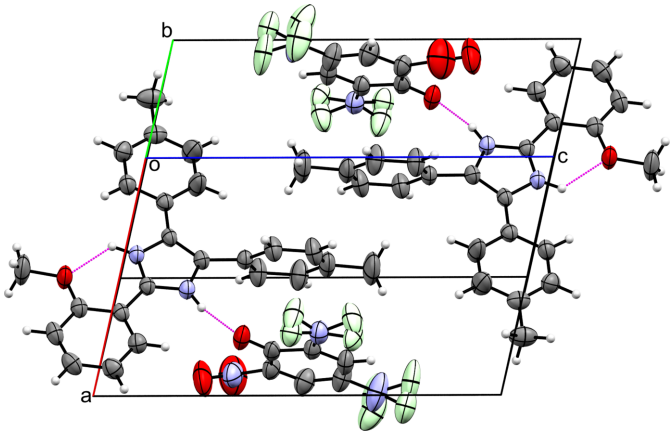


Figure 2
Unit-cell packing diagram of the title imidazolium picrate salt, showing the arrangement of the imidazolium cations and picrate anions in the triclinic $P\overline{1}$ unit cell. Hydrogen-bonding interactions are shown as magenta dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	1.82	2.661 (2)	167
N2—H2···O8	0.86	2.05	2.601 (2)	122

The packing is further consolidated by π–π stacking interactions between the picrate aromatic ring and an aromatic ring of the imidazolium cation (Fig. 3). The centroid–centroid separation is 3.712 (2) Å, the slippage is 0.69 Å and the dihedral angle between the interacting ring planes is 8.12 (14)°, indicating a nearly parallel arrangement of the aromatic rings (Janiak, 2000).

Two nitro groups of the picrate anion are disordered over two positions. The disorder was modelled using two sets of oxygen positions, with refined occupancies of 0.69 (4):0.31 (4) and 0.74 (3):0.26 (3) for the major and minor components, respectively. The disordered nitro groups were restrained to maintain chemically reasonable N—O and O···O distances and acceptable displacement parameters.

Synthesis and crystallization

2-(2-Methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol (**4**) was synthesized by a one-pot condensation reaction of 4,4-dimethylbenzil (**1**) (0.953 g, 0.004 mol), 2-methoxybenzaldehyde (**3**) (0.545 g, 0.004 mol), and ammonium acetate (**2**) (1.233 g, 0.016 mol) in the presence of ceric ammonium nitrate (CAN) as catalyst. Ethanol was used as the solvent and the reflux was carried out at 95°C. The progress of the reaction was monitored with TLC (hexane:ethyl acetate, 1:1) and at the completion of the reaction the mixture was poured into ice-cold water. The precipitate was collected and purified with multiple recrystallization in 90% ethanol. Equimolar amounts of 2-(2-methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol

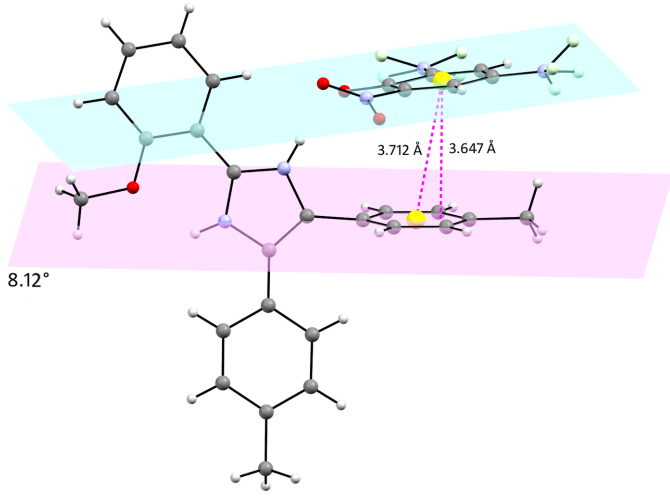


Figure 3
π–π stacking interaction between the picrate ring and the *p*-tolyl ring, showing a centroid–centroid distance of 3.712 (2) Å, and a dihedral angle of 8.12 (14)°.

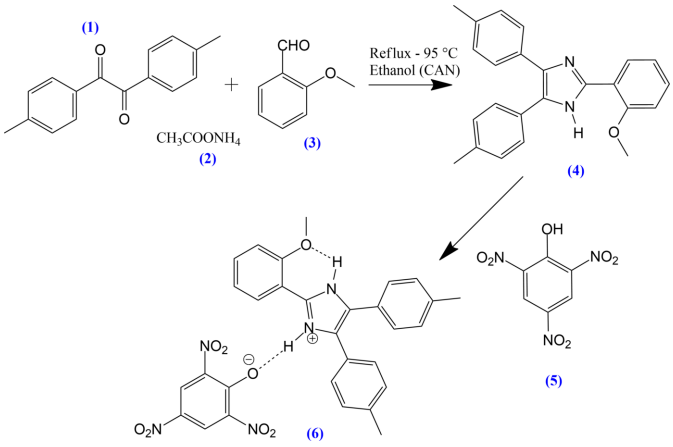


Figure 4
Synthesis of the title imidazolium picrate salt **(6)** from 4,4′-dimethylbenzil **(1)**, ammonium acetate **(2)**, 2-methoxybenzaldehyde **(3)** and picric acid **(5)**. The imidazole intermediate **(4)** was obtained in ethanol/CAN under reflux at 95°C, followed by salt formation with picric acid.

(0.071 g, 0.0002 mol) and picric acid (0.046 g, 0.0002 mol) were dissolved in 100% ethanol and heated to 120°C. The solution was kept still in a dark environment for days until yellow crystals of imidazolium picrate (**6**) appeared (Fig. 4).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were placed in calculated positions and refined using a riding model, with methyl groups treated as rotating groups. The picrate anion showed disorder affecting two nitro groups. The O6/O7 and O4/O5 nitro oxygen atoms were modelled over two sets of positions, with refined occupancies of 0.69 (4)(major): 0.31 (4)(minor) and 0.74 (3)(major):0.26 (3)(minor), respectively. The corresponding disordered atoms were assigned to PART 1 and PART 2 using linked free variables. SADI restraints were applied to maintain chemically reasonable N—O and O···O distances within the disordered nitro groups, while SIMU and RIGU restraints were used to restrain the anisotropic displacement parameters of the nitro-group atoms. Similar ADP restraints were also applied to the remaining nitro group to account for enlarged displacement parameters. The final weighting scheme was applied and the refinement converged with low residual electron density.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{23}N_2O^+\cdot C_6H_2N_3O_7^-$
M_r	583.55
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	8.4517 (19), 12.359 (3), 13.626 (3)
α, β, γ (°)	91.961 (6), 103.983 (6), 91.860 (7)
V (Å^3)	1379.1 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.25 × 0.18 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	58614, 6492, 3785
R_{int}	0.079
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.657
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.155, 1.00
No. of reflections	6492
No. of parameters	430
No. of restraints	206
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.16

Computer programs: *APEX2* (and *SAINT* (Bruker, 2018), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2025/1* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *OLEX2* (Dolomanov *et al.*, 2009).

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References

Bertolasi, V., Gilli, P. & Gilli, G. (2011). *Cryst. Growth Des.* **11**, 2724–2735.
Bruker (2018). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Desiraju, G. R. (2002). *Acc. Chem. Res.* **35**, 565–573.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Du, M. & Zhao, X.-J. (2003). *Acta Cryst.* E**59**, o1898–o1900.
Dutkiewicz, G., Samshuddin, S., Narayana, B., Yathirajan, H. S. & Kubicki, M. (2011). *Acta Cryst.* E**67**, o235.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
Li, S.-R., Tan, Y.-M., Zhang, L. & Zhou, C.-H. (2023). *Pharmaceutics* **15**, 1348.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* C**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* A**71**, 3–8.
Solo, P., Pillay, M., Arockia doss, M. & William Raja, T. R. (2025). *IUCrData* **10**, x250877.

full crystallographic data

IUCrData (2026). **11**, x260560 [https://doi.org/10.1107/S2414314626005602]

2-(2-Methoxyphenyl)-4,5-bis(4-methylphenyl)-1*H*-imidazol-3-ium 2,4,6-trinitrophenolate

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Crystal data

$C_{24}H_{23}N_2O^+\cdot C_6H_2N_3O_7^-$
M_r = 583.55
Triclinic, $P\overline{1}$
a = 8.4517 (19) Å
b = 12.359 (3) Å
c = 13.626 (3) Å
α = 91.961 (6)°
β = 103.983 (6)°
γ = 91.860 (7)°
V = 1379.1 (5) Å^3
Z = 2
$F(000)$ = 608
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5303 reflections
θ = 2.3–21.5°
μ = 0.10 mm^{-1}
T = 296 K
Block, clear yellowish orange
0.25 × 0.18 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
58614 measured reflections
6492 independent reflections
3785 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 27.8°, θ_{min} = 1.5°
h = −11→11
k = −16→16
l = −17→17

Refinement

Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.155
S = 1.00
6492 reflections
430 parameters
206 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: SHELXL-2019/2 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0146 (18)

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 structure was solved with *SHELXT* 2018/2 (Sheldrick, 2015*b*) using intrinsic phasing in the triclinic space group $P\overline{1}$ and refined by full-matrix least-squares on F^2 using *SHELXL* 2025/1 (Sheldrick, 2015*a*) within *OLEX2*. All non-hydrogen atoms were refined anisotropically.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.4853 (2)	0.30529 (15)	0.09982 (13)	0.0363 (4)	
C2	0.3818 (2)	0.39655 (15)	0.06932 (14)	0.0388 (4)	
C3	0.3181 (3)	0.41538 (18)	−0.03149 (15)	0.0472 (5)	
H3	0.340672	0.368829	−0.080982	0.057*	
C4	0.2216 (3)	0.50208 (18)	−0.05992 (16)	0.0513 (6)	
H4	0.179581	0.512070	−0.128498	0.062*	
C5	0.1852 (3)	0.57407 (17)	0.00922 (17)	0.0470 (5)	
C6	0.0801 (3)	0.66833 (19)	−0.0219 (2)	0.0636 (7)	
H6A	0.080399	0.685042	−0.090154	0.095*	
H6B	0.122080	0.730101	0.022492	0.095*	
H6C	−0.029456	0.650259	−0.018000	0.095*	
C7	0.2475 (3)	0.5545 (2)	0.10897 (18)	0.0699 (8)	
H7	0.224295	0.601298	0.158085	0.084*	
C8	0.3434 (3)	0.4682 (2)	0.13924 (17)	0.0684 (8)	
H8	0.382946	0.457803	0.207963	0.082*	
C9	0.5620 (2)	0.26805 (15)	0.19136 (13)	0.0371 (4)	
C10	0.5798 (2)	0.30630 (15)	0.29722 (13)	0.0382 (4)	
C11	0.6787 (3)	0.39536 (18)	0.33662 (16)	0.0588 (6)	
H11	0.728526	0.435304	0.294933	0.071*	
C12	0.7047 (4)	0.4261 (2)	0.43808 (17)	0.0694 (7)	
H12	0.772396	0.486565	0.463553	0.083*	
C13	0.6336 (3)	0.3698 (2)	0.50166 (15)	0.0592 (6)	
C14	0.6675 (5)	0.4030 (3)	0.61299 (18)	0.0948 (11)	
H14A	0.566647	0.403341	0.633654	0.142*	
H14B	0.718963	0.474251	0.624110	0.142*	
H14C	0.738340	0.352523	0.651819	0.142*	
C15	0.5350 (3)	0.2809 (2)	0.46217 (17)	0.0644 (7)	
H15	0.485591	0.241248	0.504209	0.077*	
C16	0.5076 (3)	0.24905 (19)	0.36137 (16)	0.0546 (6)	
H16	0.439959	0.188458	0.336301	0.065*	
C17	0.6164 (2)	0.15653 (15)	0.07255 (13)	0.0347 (4)	
C18	0.6833 (2)	0.06963 (15)	0.02284 (13)	0.0364 (4)	
C19	0.7747 (3)	−0.00823 (16)	0.08002 (15)	0.0433 (5)	
H19	0.791751	−0.004604	0.150105	0.052*	
C20	0.8400 (3)	−0.09065 (18)	0.03382 (17)	0.0518 (6)	
H20	0.900649	−0.142369	0.072675	0.062*	
C21	0.8156 (3)	−0.09635 (18)	−0.06953 (17)	0.0534 (6)	
H21	0.860003	−0.152130	−0.100291	0.064*	
C22	0.7263 (3)	−0.02068 (18)	−0.12814 (16)	0.0498 (5)	
H22	0.710708	−0.025245	−0.198125	0.060*	
C23	0.6596 (2)	0.06255 (16)	−0.08264 (14)	0.0401 (5)	

C24	0.5352 (4)	0.1358 (3)	−0.24251 (17)	0.0837 (9)	
H24A	0.470770	0.195726	−0.268477	0.126*	
H24B	0.475928	0.069056	−0.268077	0.126*	
H24C	0.635683	0.139146	−0.263578	0.126*	
C25	0.8692 (3)	0.11473 (17)	0.41365 (14)	0.0450 (5)	
C26	0.9880 (3)	0.20295 (19)	0.42170 (15)	0.0517 (6)	
C27	1.0675 (3)	0.2554 (2)	0.51019 (17)	0.0627 (7)	
H27	1.141307	0.312900	0.510247	0.075*	
C28	1.0371 (3)	0.22234 (19)	0.59978 (16)	0.0628 (7)	
C29	0.9290 (3)	0.13748 (18)	0.60059 (15)	0.0551 (6)	
H29	0.910949	0.115200	0.661617	0.066*	
C30	0.8483 (3)	0.08592 (16)	0.51213 (14)	0.0442 (5)	
N1	0.6405 (2)	0.17613 (12)	0.17185 (11)	0.0380 (4)	
H1	0.696996	0.137103	0.217506	0.046*	
N2	0.52282 (19)	0.23449 (13)	0.02905 (11)	0.0380 (4)	
H2	0.489733	0.240122	−0.035337	0.046*	
N3	1.0314 (3)	0.2402 (2)	0.33090 (17)	0.0732 (6)	
N4	1.1240 (4)	0.2754 (2)	0.69380 (17)	0.1012 (10)	
N5	0.7348 (3)	−0.00302 (16)	0.51988 (14)	0.0564 (5)	
O1	0.7905 (2)	0.06989 (12)	0.33241 (10)	0.0566 (4)	
O2	1.0161 (3)	0.1779 (2)	0.25877 (16)	0.1128 (9)	
O3	1.0822 (4)	0.3330 (2)	0.33096 (19)	0.1259 (10)	
O4	1.2391 (12)	0.3402 (7)	0.6942 (9)	0.108 (2)	0.74 (3)
O5	1.070 (2)	0.2550 (9)	0.7706 (4)	0.120 (4)	0.74 (3)
O6	0.681 (2)	−0.0065 (12)	0.5949 (9)	0.095 (3)	0.69 (4)
O7	0.7000 (15)	−0.0726 (5)	0.4513 (5)	0.0689 (17)	0.69 (4)
O4A	1.189 (6)	0.366 (2)	0.687 (2)	0.133 (12)	0.26 (3)
O5A	1.157 (3)	0.2208 (14)	0.7717 (10)	0.087 (5)	0.26 (3)
O6A	0.743 (3)	−0.0316 (16)	0.6066 (8)	0.066 (4)	0.31 (4)
O7A	0.640 (5)	−0.045 (3)	0.4473 (8)	0.096 (8)	0.31 (4)
O8	0.5696 (2)	0.14085 (12)	−0.13492 (10)	0.0539 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0404 (11)	0.0385 (10)	0.0310 (9)	−0.0005 (8)	0.0114 (8)	−0.0056 (8)
C2	0.0404 (11)	0.0401 (11)	0.0361 (10)	0.0009 (9)	0.0100 (8)	−0.0016 (8)
C3	0.0547 (13)	0.0531 (13)	0.0370 (10)	0.0082 (11)	0.0162 (10)	0.0024 (9)
C4	0.0535 (14)	0.0588 (14)	0.0437 (11)	0.0080 (11)	0.0134 (10)	0.0161 (10)
C5	0.0401 (12)	0.0397 (11)	0.0611 (13)	−0.0011 (9)	0.0115 (10)	0.0080 (10)
C6	0.0535 (15)	0.0460 (13)	0.0926 (19)	0.0078 (11)	0.0175 (13)	0.0194 (13)
C7	0.0879 (19)	0.0646 (16)	0.0528 (14)	0.0339 (15)	0.0065 (13)	−0.0134 (12)
C8	0.0897 (19)	0.0739 (17)	0.0369 (11)	0.0383 (15)	0.0030 (12)	−0.0082 (11)
C9	0.0424 (11)	0.0370 (10)	0.0325 (9)	0.0002 (8)	0.0111 (8)	−0.0032 (8)
C10	0.0475 (12)	0.0381 (10)	0.0293 (9)	0.0041 (9)	0.0100 (8)	−0.0004 (8)
C11	0.0873 (18)	0.0495 (13)	0.0396 (11)	−0.0147 (12)	0.0188 (11)	−0.0049 (10)
C12	0.102 (2)	0.0575 (15)	0.0433 (13)	−0.0169 (14)	0.0124 (13)	−0.0134 (11)
C13	0.0885 (18)	0.0581 (14)	0.0312 (10)	0.0170 (13)	0.0135 (11)	−0.0031 (10)

C14	0.144 (3)	0.102 (2)	0.0364 (13)	0.022 (2)	0.0182 (16)	−0.0132 (14)
C15	0.093 (2)	0.0669 (16)	0.0419 (12)	0.0014 (14)	0.0328 (13)	0.0026 (11)
C16	0.0678 (15)	0.0568 (14)	0.0425 (11)	−0.0085 (12)	0.0222 (11)	−0.0029 (10)
C17	0.0370 (11)	0.0362 (10)	0.0306 (9)	−0.0013 (8)	0.0081 (8)	−0.0023 (7)
C18	0.0388 (11)	0.0368 (10)	0.0346 (9)	−0.0024 (8)	0.0124 (8)	−0.0044 (8)
C19	0.0491 (12)	0.0409 (11)	0.0411 (11)	0.0014 (9)	0.0133 (9)	0.0017 (9)
C20	0.0585 (14)	0.0431 (12)	0.0576 (13)	0.0089 (11)	0.0205 (11)	0.0024 (10)
C21	0.0632 (15)	0.0449 (12)	0.0590 (14)	0.0029 (11)	0.0293 (12)	−0.0069 (10)
C22	0.0617 (14)	0.0504 (13)	0.0410 (11)	−0.0039 (11)	0.0220 (10)	−0.0091 (9)
C23	0.0449 (12)	0.0409 (11)	0.0346 (10)	−0.0028 (9)	0.0113 (8)	−0.0029 (8)
C24	0.129 (3)	0.093 (2)	0.0289 (11)	0.0286 (19)	0.0165 (14)	0.0052 (12)
C25	0.0578 (13)	0.0454 (12)	0.0332 (10)	0.0175 (10)	0.0115 (9)	0.0047 (9)
C26	0.0612 (14)	0.0579 (14)	0.0376 (11)	0.0083 (11)	0.0131 (10)	0.0123 (10)
C27	0.0765 (18)	0.0567 (14)	0.0510 (13)	−0.0060 (13)	0.0083 (12)	0.0081 (11)
C28	0.0904 (19)	0.0514 (14)	0.0396 (12)	−0.0066 (13)	0.0034 (12)	0.0000 (10)
C29	0.0822 (17)	0.0513 (13)	0.0317 (10)	0.0077 (12)	0.0130 (11)	0.0030 (9)
C30	0.0558 (13)	0.0423 (11)	0.0353 (10)	0.0087 (10)	0.0113 (9)	0.0040 (8)
N1	0.0464 (10)	0.0380 (9)	0.0298 (8)	0.0029 (7)	0.0095 (7)	−0.0004 (6)
N2	0.0437 (9)	0.0429 (9)	0.0269 (7)	0.0040 (7)	0.0080 (7)	−0.0017 (7)
N3	0.0733 (15)	0.0994 (18)	0.0508 (12)	−0.0016 (13)	0.0207 (11)	0.0201 (12)
N4	0.161 (3)	0.0768 (17)	0.0483 (13)	−0.0366 (18)	−0.0017 (15)	−0.0009 (12)
N5	0.0672 (13)	0.0601 (12)	0.0426 (10)	0.0042 (10)	0.0139 (9)	0.0065 (9)
O1	0.0793 (11)	0.0561 (9)	0.0316 (7)	0.0080 (8)	0.0072 (7)	0.0024 (7)
O2	0.130 (2)	0.162 (2)	0.0567 (12)	−0.0289 (17)	0.0489 (13)	−0.0078 (13)
O3	0.173 (3)	0.117 (2)	0.1001 (18)	−0.0324 (18)	0.0585 (17)	0.0345 (15)
O4	0.143 (4)	0.078 (3)	0.078 (3)	−0.036 (3)	−0.020 (3)	0.005 (3)
O5	0.199 (8)	0.110 (5)	0.0367 (16)	−0.044 (5)	0.011 (3)	−0.0088 (19)
O6	0.114 (7)	0.118 (5)	0.066 (3)	−0.033 (5)	0.051 (4)	−0.009 (3)
O7	0.086 (4)	0.061 (2)	0.0554 (19)	−0.008 (2)	0.0120 (19)	−0.0028 (15)
O4A	0.21 (3)	0.086 (9)	0.071 (8)	−0.071 (14)	−0.008 (12)	−0.001 (7)
O5A	0.119 (13)	0.086 (7)	0.047 (4)	−0.017 (6)	0.007 (5)	0.005 (4)
O6A	0.071 (8)	0.080 (7)	0.046 (3)	−0.012 (5)	0.015 (3)	0.020 (3)
O7A	0.105 (12)	0.124 (14)	0.048 (4)	−0.051 (11)	0.005 (5)	0.002 (5)
O8	0.0731 (11)	0.0602 (9)	0.0280 (7)	0.0123 (8)	0.0106 (7)	−0.0008 (6)

Geometric parameters (Å, º)

C1—C9	1.362 (3)	C19—C20	1.378 (3)
C1—N2	1.380 (2)	C19—H19	0.9300
C1—C2	1.459 (3)	C20—C21	1.372 (3)
C2—C3	1.380 (3)	C20—H20	0.9300
C2—C8	1.381 (3)	C21—C22	1.374 (3)
C3—C4	1.377 (3)	C21—H21	0.9300
C3—H3	0.9300	C22—C23	1.387 (3)
C4—C5	1.369 (3)	C22—H22	0.9300
C4—H4	0.9300	C23—O8	1.362 (2)
C5—C7	1.367 (3)	C24—O8	1.423 (2)
C5—C6	1.498 (3)	C24—H24A	0.9600

C6—H6A	0.9600	C24—H24B	0.9600
C6—H6B	0.9600	C24—H24C	0.9600
C6—H6C	0.9600	C25—O1	1.246 (2)
C7—C8	1.375 (3)	C25—C26	1.441 (3)
C7—H7	0.9300	C25—C30	1.450 (3)
C8—H8	0.9300	C26—C27	1.361 (3)
C9—N1	1.384 (2)	C26—N3	1.458 (3)
C9—C10	1.473 (2)	C27—C28	1.379 (3)
C10—C11	1.372 (3)	C27—H27	0.9300
C10—C16	1.382 (3)	C28—C29	1.370 (3)
C11—C12	1.384 (3)	C28—N4	1.438 (3)
C11—H11	0.9300	C29—C30	1.359 (3)
C12—C13	1.365 (3)	C29—H29	0.9300
C12—H12	0.9300	C30—N5	1.459 (3)
C13—C15	1.368 (4)	N1—H1	0.8600
C13—C14	1.514 (3)	N2—H2	0.8600
C14—H14A	0.9600	N3—O2	1.207 (3)
C14—H14B	0.9600	N3—O3	1.211 (3)
C14—H14C	0.9600	N4—O4	1.238 (7)
C15—C16	1.378 (3)	N4—O4A	1.249 (12)
C15—H15	0.9300	N4—O5A	1.256 (9)
C16—H16	0.9300	N4—O5	1.268 (6)
C17—N2	1.329 (2)	N5—O7A	1.199 (9)
C17—N1	1.331 (2)	N5—O7	1.223 (5)
C17—C18	1.452 (3)	N5—O6	1.218 (5)
C18—C19	1.392 (3)	N5—O6A	1.231 (9)
C18—C23	1.402 (3)		

C9—C1—N2	105.23 (16)	C20—C19—H19	119.7
C9—C1—C2	133.45 (17)	C18—C19—H19	119.7
N2—C1—C2	121.32 (16)	C21—C20—C19	120.0 (2)
C3—C2—C8	116.81 (19)	C21—C20—H20	120.0
C3—C2—C1	121.19 (17)	C19—C20—H20	120.0
C8—C2—C1	122.00 (18)	C20—C21—C22	120.8 (2)
C4—C3—C2	120.98 (19)	C20—C21—H21	119.6
C4—C3—H3	119.5	C22—C21—H21	119.6
C2—C3—H3	119.5	C21—C22—C23	119.80 (19)
C5—C4—C3	122.3 (2)	C21—C22—H22	120.1
C5—C4—H4	118.8	C23—C22—H22	120.1
C3—C4—H4	118.8	O8—C23—C22	123.66 (17)
C7—C5—C4	116.5 (2)	O8—C23—C18	116.19 (16)
C7—C5—C6	121.3 (2)	C22—C23—C18	120.16 (19)
C4—C5—C6	122.2 (2)	O8—C24—H24A	109.5
C5—C6—H6A	109.5	O8—C24—H24B	109.5
C5—C6—H6B	109.5	H24A—C24—H24B	109.5
H6A—C6—H6B	109.5	O8—C24—H24C	109.5
C5—C6—H6C	109.5	H24A—C24—H24C	109.5
H6A—C6—H6C	109.5	H24B—C24—H24C	109.5

H6B—C6—H6C	109.5	O1—C25—C26	124.71 (19)
C5—C7—C8	122.3 (2)	O1—C25—C30	123.6 (2)
C5—C7—H7	118.9	C26—C25—C30	111.63 (18)
C8—C7—H7	118.9	C27—C26—C25	124.5 (2)
C7—C8—C2	121.1 (2)	C27—C26—N3	115.8 (2)
C7—C8—H8	119.4	C25—C26—N3	119.7 (2)
C2—C8—H8	119.4	C26—C27—C28	119.2 (2)
C1—C9—N1	106.68 (15)	C26—C27—H27	120.4
C1—C9—C10	134.34 (18)	C28—C27—H27	120.4
N1—C9—C10	118.94 (16)	C29—C28—C27	120.9 (2)
C11—C10—C16	118.21 (18)	C29—C28—N4	119.6 (2)
C11—C10—C9	120.59 (18)	C27—C28—N4	119.4 (2)
C16—C10—C9	121.02 (18)	C30—C29—C28	119.9 (2)
C10—C11—C12	120.3 (2)	C30—C29—H29	120.0
C10—C11—H11	119.8	C28—C29—H29	120.0
C12—C11—H11	119.8	C29—C30—C25	123.8 (2)
C13—C12—C11	121.6 (2)	C29—C30—N5	116.31 (19)
C13—C12—H12	119.2	C25—C30—N5	119.84 (18)
C11—C12—H12	119.2	C17—N1—C9	110.54 (16)
C12—C13—C15	117.9 (2)	C17—N1—H1	124.7
C12—C13—C14	120.7 (3)	C9—N1—H1	124.7
C15—C13—C14	121.4 (2)	C17—N2—C1	111.74 (15)
C13—C14—H14A	109.5	C17—N2—H2	124.1
C13—C14—H14B	109.5	C1—N2—H2	124.1
H14A—C14—H14B	109.5	O2—N3—O3	122.4 (2)
C13—C14—H14C	109.5	O2—N3—C26	119.1 (3)
H14A—C14—H14C	109.5	O3—N3—C26	118.4 (2)
H14B—C14—H14C	109.5	O4A—N4—O5A	123.9 (13)
C13—C15—C16	121.3 (2)	O4—N4—O5	124.7 (6)
C13—C15—H15	119.3	O4—N4—C28	119.3 (6)
C16—C15—H15	119.3	O4A—N4—C28	115.9 (13)
C15—C16—C10	120.6 (2)	O5A—N4—C28	118.5 (7)
C15—C16—H16	119.7	O5—N4—C28	116.0 (4)
C10—C16—H16	119.7	O7—N5—O6	122.4 (5)
N2—C17—N1	105.81 (16)	O7A—N5—O6A	122.8 (11)
N2—C17—C18	127.47 (16)	O7A—N5—C30	122.4 (8)
N1—C17—C18	126.70 (17)	O7—N5—C30	118.5 (4)
C19—C18—C23	118.59 (17)	O6—N5—C30	119.1 (4)
C19—C18—C17	120.09 (16)	O6A—N5—C30	114.8 (8)
C23—C18—C17	121.32 (17)	C23—O8—C24	119.02 (17)
C20—C19—C18	120.67 (19)		

C9—C1—C2—C3	−179.6 (2)	O1—C25—C26—C27	176.4 (2)
N2—C1—C2—C3	−0.1 (3)	C30—C25—C26—C27	−1.9 (3)
C9—C1—C2—C8	0.6 (4)	O1—C25—C26—N3	−4.5 (3)
N2—C1—C2—C8	−179.9 (2)	C30—C25—C26—N3	177.2 (2)
C8—C2—C3—C4	0.3 (3)	C25—C26—C27—C28	1.3 (4)
C1—C2—C3—C4	−179.5 (2)	N3—C26—C27—C28	−177.8 (2)

C2—C3—C4—C5	0.7 (3)	C26—C27—C28—C29	0.3 (4)
C3—C4—C5—C7	−1.2 (3)	C26—C27—C28—N4	178.3 (3)
C3—C4—C5—C6	−180.0 (2)	C27—C28—C29—C30	−1.1 (4)
C4—C5—C7—C8	0.7 (4)	N4—C28—C29—C30	−179.0 (2)
C6—C5—C7—C8	179.6 (3)	C28—C29—C30—C25	0.3 (4)
C5—C7—C8—C2	0.2 (5)	C28—C29—C30—N5	−179.6 (2)
C3—C2—C8—C7	−0.7 (4)	O1—C25—C30—C29	−177.2 (2)
C1—C2—C8—C7	179.1 (2)	C26—C25—C30—C29	1.1 (3)
N2—C1—C9—N1	−0.7 (2)	O1—C25—C30—N5	2.6 (3)
C2—C1—C9—N1	178.9 (2)	C26—C25—C30—N5	−179.02 (19)
N2—C1—C9—C10	176.9 (2)	N2—C17—N1—C9	−0.4 (2)
C2—C1—C9—C10	−3.5 (4)	C18—C17—N1—C9	177.88 (18)
C1—C9—C10—C11	−72.6 (3)	C1—C9—N1—C17	0.7 (2)
N1—C9—C10—C11	104.8 (2)	C10—C9—N1—C17	−177.36 (17)
C1—C9—C10—C16	112.4 (3)	N1—C17—N2—C1	−0.1 (2)
N1—C9—C10—C16	−70.3 (3)	C18—C17—N2—C1	−178.31 (18)
C16—C10—C11—C12	0.1 (4)	C9—C1—N2—C17	0.5 (2)
C9—C10—C11—C12	−175.1 (2)	C2—C1—N2—C17	−179.13 (17)
C10—C11—C12—C13	−0.2 (4)	C27—C26—N3—O2	153.3 (3)
C11—C12—C13—C15	0.2 (4)	C25—C26—N3—O2	−25.9 (4)
C11—C12—C13—C14	178.3 (3)	C27—C26—N3—O3	−26.5 (4)
C12—C13—C15—C16	−0.2 (4)	C25—C26—N3—O3	154.3 (3)
C14—C13—C15—C16	−178.3 (3)	C29—C28—N4—O4	168.8 (7)
C13—C15—C16—C10	0.2 (4)	C27—C28—N4—O4	−9.2 (8)
C11—C10—C16—C15	−0.1 (3)	C29—C28—N4—O4A	−163 (3)
C9—C10—C16—C15	175.1 (2)	C27—C28—N4—O4A	19 (3)
N2—C17—C18—C19	−177.60 (19)	C29—C28—N4—O5A	30.6 (17)
N1—C17—C18—C19	4.5 (3)	C27—C28—N4—O5A	−147.4 (17)
N2—C17—C18—C23	3.1 (3)	C29—C28—N4—O5	−14.3 (10)
N1—C17—C18—C23	−174.78 (18)	C27—C28—N4—O5	167.7 (10)
C23—C18—C19—C20	−0.1 (3)	C29—C30—N5—O7A	169 (3)
C17—C18—C19—C20	−179.47 (19)	C25—C30—N5—O7A	−11 (3)
C18—C19—C20—C21	0.1 (3)	C29—C30—N5—O7	−156.8 (6)
C19—C20—C21—C22	0.0 (3)	C25—C30—N5—O7	23.4 (7)
C20—C21—C22—C23	−0.1 (3)	C29—C30—N5—O6	21.8 (14)
C21—C22—C23—O8	−180.0 (2)	C25—C30—N5—O6	−158.1 (13)
C21—C22—C23—C18	0.1 (3)	C29—C30—N5—O6A	−10.5 (14)
C19—C18—C23—O8	−179.89 (17)	C25—C30—N5—O6A	169.7 (14)
C17—C18—C23—O8	−0.6 (3)	C22—C23—O8—C24	2.7 (3)
C19—C18—C23—C22	0.0 (3)	C18—C23—O8—C24	−177.4 (2)
C17—C18—C23—C22	179.34 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	1.82	2.661 (2)	167
N2—H2···O8	0.86	2.05	2.601 (2)	122