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Desipraminium picrate monohydrate

William T. A. Harrison,[a]* M. T. Swamy,[b] P. Nagaraja,[c] H. S. Yathirajan[c] and B. Narayana[d]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, [b]Department of Chemistry, Sambhram Institute of Technology, Bangalore 560 097, India, [c]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [d]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India
Correspondence e-mail: w.harrison@abdn.ac.uk

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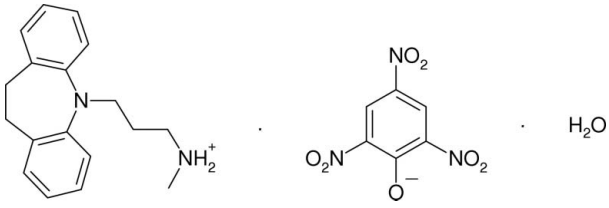
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; H-atom completeness 93%; disorder in main residue; R factor = 0.067; wR factor = 0.220; data-to-parameter ratio = 14.1.

In the title compound, $C_{18}H_{23}N_2^+\cdot C_6H_2N_3O_7^-\cdot H_2O$, the cation and anion are linked by a bifurcated N—H···(O,O) hydrogen bond. A second N—H···O interaction connects the cation to the solvent water molecule. The dihedral angle between the aromatic ring planes in the cation is 53.92 (8)°. One of the nitro groups of the anion is disordered over two orientations in a ratio of approximately 0.84:0.16.

Related literature

For related structures, see: Portalone *et al.* (2007); Harrison, Ashok *et al.* (2007); Harrison, Bindya *et al.* (2007). For background, see: Cohen *et al.* (1990); Ahmed *et al.* (2002).



Experimental

Crystal data

$C_{18}H_{23}N_2^+\cdot C_6H_2N_3O_7^-\cdot H_2O$
M_r = 513.51
Triclinic, $P\overline{1}$
a = 10.3126 (4) Å
b = 11.2594 (5) Å
c = 11.4304 (5) Å
α = 72.162 (1)°
β = 79.288 (1)°
γ = 87.233 (1)°
V = 1241.35 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 291 (2) K
0.60 × 0.50 × 0.45 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.943, T_{max} = 0.954
11334 measured reflections
4865 independent reflections
3688 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.220
S = 1.04
4865 reflections
344 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1N···O11	0.86	2.06	2.817 (3)	146
N2—H1N···O12	0.86	2.38	3.001 (3)	129
N2—H2N···O21	0.86	2.03	2.873 (4)	170

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2487).

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supplementary materials

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Desipraminium picrate monohydrate

W. T. A. Harrison, M. T. Swamy, P. Nagaraja, H. S. Yathirajan and B. Narayana

Comment

Desipramine, 10,11-dihydro-5-[3-(methylamino)propyl]-5*H*-dibenz[b,f]azepine, $C_{18}H_{22}N_2$, is a tricyclic antidepressant (TCA) and pain reliever that inhibits the reuptake of norepinephrine. Medical applications (Cohen *et al.*, 1990) usually utilize desipramine as its hydrochloride salt, *i.e.* $C_{18}H_{23}N_2^+\cdot Cl^-$. A recent proposed new application of desipramine hydrochloride as a reagent for the detection of microamounts of blood in urine (Ahmed *et al.*, 2002).

In continuation of our work on the picrate salts of pharmaceutical compounds (Portalone *et al.*, 2007; Harrison, Ashok *et al.*, 2007; Harrison, Bindya *et al.*, 2007), we now report the structure of the title compound, (I).

The structure of (I) (Fig. 1) shows that proton transfer from picric acid (pa) to desipramine (dp) has occurred, and that the secondary-amine N atom of the side chain has been protonated to yield an $–NH_2^+–$ grouping. The dihedral angle between the C1–C6 and C9–C14 ring planes in (I) is 53.92 (8)°. The bond-angle sum for N1 is 360.0°, indicating sp^2 hybridization. However, the unobserved p orbital appears not to be well aligned to overlap with the π clouds of the adjacent benzene rings, as the C1/C14/C15/N1 grouping makes dihedral angles of 65.28 (10)° and 44.78 (11)° with the C1–C6 and C9–C14 ring planes, respectively. The seven-membered ring in (I) approximates to a boat, with C1/C6/C8/C9 almost co-planar (r.m.s. deviation = 0.002 Å), C7 forming the prow [deviation = 0.721 (3) Å] and N1 and C14 the stern [deviations = 0.787 (4) and 0.351 (4) Å, respectively].

The significant variation of the C—C bond lengths around the picrate aromatic ring in (I) are normal (Harrison, Ashok *et al.*, 2007). The N13 nitro group is disordered over two orientations.

In the crystal, the cation and anion are linked by a bifurcated N—H···(O,*O*) hydrogen bond (Table 1), as also seen in related compounds (Harrison, Ashok *et al.*, 2007). The cation also bonds to the O21 water molecule with an N—H···O link. Unfortunately, the water molecule H atoms were not located in this study. One of the H atoms is probably involved in an H bond to O16 [O21···O16 = 2.667 (5) Å], but there is no obvious second H bond and geometrical placement of the H atoms was not attempted.

Experimental

Desipramine hydrochloride (0.8 g, 0.05 mol) and picric acid (0.6 g, 0.05 mol) were dissolved in distilled water (50 ml), mixed and stirred well in a beaker at room temperature. The separated bright yellow salt was washed well with distilled water, filtered and dried in a vacuum desiccator over P_2O_5. Yellow blocks of (I) were recrystallized from absolute ethanol (m.p.: 387 K). The water of crystallization in (I) was presumably incorporated from the atmosphere.

Refinement

One of the nitro groups of the anion is disordered over two orientations in a 0.840 (6):0.160 (6) ratio. The N-bound H atoms were located in a differenc map, then relocated to idealized locations (N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were geometrically placed (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl group was allowed to rotate, but not to tip, to best fit the electron density. The H atoms of the water molecule could not be located in the present study.

Figures

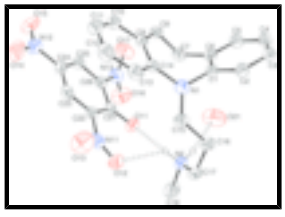


Fig. 1. View of the molecular structure of (I) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). The hydrogen bonds are shown as double-dashed lines. Only the N-bound H atoms and the major disorder component for the N13 nitro group are shown.

Desipraminium picrate monohydrate

Crystal data

$C_{18}H_{23}N_2^+ \cdot C_6H_2N_3O_7^- \cdot H_2O$	$Z = 2$
$M_r = 513.51$	$F_{000} = 540$
Triclinic, $P\overline{1}$	$D_x = 1.374$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.3126$ (4) Å	Cell parameters from 5773 reflections
$b = 11.2594$ (5) Å	$\theta = 2.3$–27.5º
$c = 11.4304$ (5) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 72.162$ (1)º	$T = 291$ (2) K
$\beta = 79.288$ (1)º	Chunk, yellow
$\gamma = 87.233$ (1)º	0.60 × 0.50 × 0.45 mm
$V = 1241.35$ (9) Å^3	

Data collection

Bruker SMART1000 CCD diffractometer	4865 independent reflections
Radiation source: fine-focus sealed tube	3688 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
$T = 291$(2) K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$h = -11 \rightarrow 12$
$T_{min} = 0.943$, $T_{max} = 0.954$	$k = -13 \rightarrow 13$

11334 measured reflections

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.220$

$S = 1.04$

4865 reflections

344 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1277P)^2 + 0.5257P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.47$ e Å^{-3}

$\Delta\rho_{min} = -0.58$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.3705 (2)	0.18454 (19)	0.4622 (2)	0.0422 (5)	
C2	0.4803 (3)	0.1592 (2)	0.5201 (3)	0.0557 (6)	
H2	0.5632	0.1876	0.4743	0.067*	
C3	0.4669 (3)	0.0915 (3)	0.6462 (3)	0.0676 (8)	
H3	0.5407	0.0748	0.6844	0.081*	
C4	0.3441 (4)	0.0493 (3)	0.7141 (3)	0.0735 (9)	
H4	0.3349	0.0041	0.7982	0.088*	
C5	0.2351 (3)	0.0740 (3)	0.6576 (2)	0.0614 (7)	
H5	0.1525	0.0456	0.7043	0.074*	
C6	0.2464 (2)	0.1412 (2)	0.5309 (2)	0.0447 (5)	
C7	0.1308 (2)	0.1630 (2)	0.4653 (2)	0.0499 (6)	
H7A	0.1287	0.2503	0.4171	0.060*	
H7B	0.0499	0.1436	0.5266	0.060*	
C8	0.1382 (2)	0.0830 (2)	0.3790 (2)	0.0515 (6)	
H8A	0.1503	−0.0027	0.4274	0.062*	
H8B	0.0533	0.0870	0.3531	0.062*	
C9	0.2437 (2)	0.11361 (19)	0.2627 (2)	0.0461 (5)	
C10	0.2280 (3)	0.0574 (2)	0.1725 (3)	0.0613 (7)	

H10	0.1550	0.0060	0.1876	0.074*	
C11	0.3157 (4)	0.0751 (3)	0.0634 (3)	0.0779 (9)	
H11	0.3026	0.0356	0.0059	0.093*	
C12	0.4236 (4)	0.1518 (3)	0.0391 (3)	0.0789 (10)	
H12	0.4837	0.1651	−0.0354	0.095*	
C13	0.4424 (3)	0.2094 (2)	0.1266 (2)	0.0606 (7)	
H13	0.5158	0.2608	0.1098	0.073*	
C14	0.3542 (2)	0.19202 (19)	0.2385 (2)	0.0436 (5)	
C15	0.4229 (2)	0.3765 (2)	0.2848 (2)	0.0475 (5)	
H15A	0.3560	0.4218	0.2395	0.057*	
H15B	0.5017	0.3791	0.2226	0.057*	
C16	0.4542 (3)	0.4549 (2)	0.3614 (3)	0.0588 (7)	
H16A	0.5492	0.4615	0.3522	0.071*	
H16B	0.4193	0.4139	0.4488	0.071*	
C17	0.3969 (3)	0.5852 (2)	0.3232 (3)	0.0618 (7)	
H17A	0.4266	0.6343	0.3698	0.074*	
H17B	0.4294	0.6253	0.2351	0.074*	
C18	0.1933 (4)	0.7103 (3)	0.3209 (4)	0.0896 (11)	
H18A	0.0988	0.7040	0.3386	0.134*	
H18B	0.2221	0.7567	0.2348	0.134*	
H18C	0.2221	0.7523	0.3732	0.134*	
N1	0.3828 (2)	0.2574 (2)	0.3275 (2)	0.0554 (5)	
N2	0.2508 (2)	0.5830 (2)	0.3460 (2)	0.0559 (5)	
H1N	0.2230	0.5440	0.3014	0.067*	
H2N	0.2161	0.5385	0.4198	0.067*	
C21	0.0489 (2)	0.4204 (2)	0.1836 (2)	0.0428 (5)	
C22	0.1382 (2)	0.4268 (2)	0.0688 (2)	0.0442 (5)	
C23	0.1227 (3)	0.3566 (2)	−0.0082 (2)	0.0529 (6)	
H23	0.1833	0.3644	−0.0815	0.063*	
C24	0.0174 (3)	0.2750 (2)	0.0241 (2)	0.0551 (6)	
C25	−0.0728 (3)	0.2612 (2)	0.1328 (2)	0.0541 (6)	
H25	−0.1427	0.2048	0.1550	0.065*	
C26	−0.0576 (2)	0.3315 (2)	0.2070 (2)	0.0462 (5)	
N11	0.2504 (2)	0.5128 (2)	0.0260 (2)	0.0598 (6)	
N12	−0.0004 (3)	0.2036 (3)	−0.0585 (3)	0.0804 (8)	
N13	−0.1562 (2)	0.3130 (2)	0.3202 (2)	0.0631 (6)	
O11	0.05959 (19)	0.47933 (17)	0.25813 (17)	0.0595 (5)	
O12	0.2615 (2)	0.5890 (2)	0.0801 (2)	0.0795 (6)	
O13	0.3289 (3)	0.5073 (3)	−0.0639 (3)	0.1196 (11)	
O14	0.0784 (4)	0.2197 (3)	−0.1557 (3)	0.1102 (10)	
O15	−0.0951 (4)	0.1312 (3)	−0.0257 (3)	0.1176 (11)	
O16	−0.2029 (3)	0.4031 (3)	0.3489 (3)	0.1007 (13)	0.840 (6)
O17	−0.1854 (3)	0.2071 (3)	0.3862 (3)	0.0971 (13)	0.840 (6)
O18	−0.1390 (18)	0.2968 (17)	0.4109 (18)	0.092 (6)*	0.160 (6)
O19	−0.278 (3)	0.301 (2)	0.301 (2)	0.144 (10)*	0.160 (6)
O21	0.1305 (4)	0.4099 (3)	0.5790 (3)	0.1427 (15)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0435 (12)	0.0359 (10)	0.0494 (12)	0.0018 (8)	−0.0119 (9)	−0.0144 (9)
C2	0.0515 (14)	0.0504 (13)	0.0714 (16)	0.0057 (10)	−0.0231 (12)	−0.0210 (12)
C3	0.087 (2)	0.0567 (15)	0.0725 (18)	0.0144 (14)	−0.0477 (17)	−0.0217 (13)
C4	0.111 (3)	0.0626 (17)	0.0477 (14)	0.0047 (17)	−0.0259 (16)	−0.0120 (12)
C5	0.0774 (18)	0.0564 (15)	0.0467 (13)	−0.0057 (13)	−0.0035 (12)	−0.0138 (11)
C6	0.0486 (12)	0.0391 (11)	0.0457 (11)	−0.0002 (9)	−0.0054 (9)	−0.0136 (9)
C7	0.0376 (11)	0.0487 (12)	0.0564 (13)	−0.0044 (9)	−0.0003 (10)	−0.0099 (10)
C8	0.0492 (13)	0.0418 (12)	0.0627 (14)	−0.0085 (10)	−0.0182 (11)	−0.0086 (10)
C9	0.0554 (13)	0.0331 (10)	0.0525 (12)	0.0067 (9)	−0.0223 (10)	−0.0105 (9)
C10	0.0791 (19)	0.0462 (13)	0.0680 (16)	0.0080 (12)	−0.0317 (14)	−0.0214 (12)
C11	0.115 (3)	0.0649 (18)	0.0684 (18)	0.0157 (18)	−0.0309 (18)	−0.0348 (15)
C12	0.106 (3)	0.077 (2)	0.0487 (15)	0.0230 (19)	−0.0022 (15)	−0.0218 (14)
C13	0.0653 (17)	0.0540 (14)	0.0549 (14)	0.0046 (12)	0.0005 (12)	−0.0129 (11)
C14	0.0473 (12)	0.0356 (10)	0.0456 (11)	0.0053 (9)	−0.0098 (9)	−0.0089 (9)
C15	0.0358 (11)	0.0431 (12)	0.0589 (13)	−0.0047 (9)	−0.0044 (9)	−0.0105 (10)
C16	0.0502 (14)	0.0498 (13)	0.0802 (17)	−0.0091 (11)	−0.0226 (12)	−0.0174 (12)
C17	0.0684 (17)	0.0453 (13)	0.0765 (17)	−0.0121 (12)	−0.0238 (14)	−0.0172 (12)
C18	0.111 (3)	0.071 (2)	0.111 (3)	0.0295 (19)	−0.051 (2)	−0.050 (2)
N1	0.0438 (11)	0.0580 (12)	0.0614 (12)	0.0002 (9)	−0.0078 (9)	−0.0148 (10)
N2	0.0644 (13)	0.0542 (12)	0.0569 (12)	0.0019 (10)	−0.0198 (10)	−0.0230 (9)
C21	0.0434 (12)	0.0413 (11)	0.0434 (11)	0.0041 (9)	−0.0129 (9)	−0.0100 (9)
C22	0.0386 (11)	0.0465 (12)	0.0439 (11)	0.0008 (9)	−0.0108 (9)	−0.0065 (9)
C23	0.0573 (14)	0.0571 (14)	0.0418 (11)	0.0160 (11)	−0.0119 (10)	−0.0118 (10)
C24	0.0691 (16)	0.0481 (13)	0.0561 (14)	0.0089 (11)	−0.0261 (12)	−0.0201 (11)
C25	0.0542 (14)	0.0453 (13)	0.0645 (15)	−0.0034 (10)	−0.0236 (12)	−0.0108 (11)
C26	0.0408 (12)	0.0470 (12)	0.0470 (12)	0.0020 (9)	−0.0094 (9)	−0.0082 (9)
N11	0.0488 (12)	0.0693 (14)	0.0516 (12)	−0.0075 (10)	−0.0080 (10)	−0.0036 (10)
N12	0.109 (2)	0.0686 (16)	0.0815 (18)	0.0172 (16)	−0.0413 (17)	−0.0378 (14)
N13	0.0460 (12)	0.0755 (17)	0.0641 (14)	−0.0049 (11)	−0.0061 (10)	−0.0173 (12)
O11	0.0621 (11)	0.0657 (11)	0.0569 (10)	−0.0069 (9)	−0.0078 (8)	−0.0283 (9)
O12	0.0817 (15)	0.0814 (14)	0.0741 (13)	−0.0331 (11)	−0.0150 (11)	−0.0161 (11)
O13	0.0870 (18)	0.154 (3)	0.106 (2)	−0.0507 (18)	0.0476 (16)	−0.0526 (19)
O14	0.153 (3)	0.118 (2)	0.0835 (17)	0.0271 (19)	−0.0289 (18)	−0.0645 (16)
O15	0.154 (3)	0.096 (2)	0.134 (2)	−0.0217 (19)	−0.050 (2)	−0.0633 (18)
O16	0.087 (2)	0.103 (2)	0.111 (2)	−0.0076 (17)	0.0254 (18)	−0.055 (2)
O17	0.081 (2)	0.089 (2)	0.087 (2)	−0.0174 (15)	0.0176 (15)	0.0065 (16)
O21	0.215 (4)	0.099 (2)	0.0909 (19)	−0.009 (2)	0.043 (2)	−0.0353 (16)

Geometric parameters (Å, °)

C1—C2	1.390 (3)	C16—C17	1.521 (4)
C1—C6	1.394 (3)	C16—H16A	0.9700
C1—N1	1.491 (3)	C16—H16B	0.9700
C2—C3	1.393 (4)	C17—N2	1.481 (4)
C2—H2	0.9300	C17—H17A	0.9700

C3—C4	1.378 (5)	C17—H17B	0.9700
C3—H3	0.9300	C18—N2	1.489 (4)
C4—C5	1.373 (4)	C18—H18A	0.9600
C4—H4	0.9300	C18—H18B	0.9600
C5—C6	1.399 (3)	C18—H18C	0.9600
C5—H5	0.9300	N2—H1N	0.8596
C6—C7	1.495 (3)	N2—H2N	0.8577
C7—C8	1.516 (4)	C21—O11	1.252 (3)
C7—H7A	0.9700	C21—C22	1.440 (3)
C7—H7B	0.9700	C21—C26	1.453 (3)
C8—C9	1.513 (4)	C22—C23	1.385 (3)
C8—H8A	0.9700	C22—N11	1.457 (3)
C8—H8B	0.9700	C23—C24	1.379 (4)
C9—C10	1.401 (3)	C23—H23	0.9300
C9—C14	1.409 (3)	C24—C25	1.379 (4)
C10—C11	1.363 (5)	C24—N12	1.453 (3)
C10—H10	0.9300	C25—C26	1.357 (3)
C11—C12	1.376 (5)	C25—H25	0.9300
C11—H11	0.9300	C26—N13	1.456 (3)
C12—C13	1.394 (4)	N11—O13	1.200 (3)
C12—H12	0.9300	N11—O12	1.222 (3)
C13—C14	1.389 (3)	N12—O14	1.218 (4)
C13—H13	0.9300	N12—O15	1.229 (4)
C14—N1	1.502 (3)	N13—O18	1.044 (19)
C15—N1	1.336 (3)	N13—O16	1.211 (4)
C15—C16	1.502 (3)	N13—O17	1.217 (4)
C15—H15A	0.9700	N13—O19	1.33 (3)
C15—H15B	0.9700		
C2—C1—C6	119.7 (2)	C17—C16—H16B	109.1
C2—C1—N1	121.2 (2)	H16A—C16—H16B	107.8
C6—C1—N1	119.15 (19)	N2—C17—C16	112.1 (2)
C1—C2—C3	120.4 (3)	N2—C17—H17A	109.2
C1—C2—H2	119.8	C16—C17—H17A	109.2
C3—C2—H2	119.8	N2—C17—H17B	109.2
C4—C3—C2	119.8 (3)	C16—C17—H17B	109.2
C4—C3—H3	120.1	H17A—C17—H17B	107.9
C2—C3—H3	120.1	N2—C18—H18A	109.5
C5—C4—C3	120.1 (3)	N2—C18—H18B	109.5
C5—C4—H4	119.9	H18A—C18—H18B	109.5
C3—C4—H4	119.9	N2—C18—H18C	109.5
C4—C5—C6	121.0 (3)	H18A—C18—H18C	109.5
C4—C5—H5	119.5	H18B—C18—H18C	109.5
C6—C5—H5	119.5	C15—N1—C1	121.6 (2)
C1—C6—C5	119.0 (2)	C15—N1—C14	120.1 (2)
C1—C6—C7	118.9 (2)	C1—N1—C14	118.30 (18)
C5—C6—C7	122.1 (2)	C17—N2—C18	112.6 (2)
C6—C7—C8	111.2 (2)	C17—N2—H1N	110.2
C6—C7—H7A	109.4	C18—N2—H1N	109.9
C8—C7—H7A	109.4	C17—N2—H2N	113.6

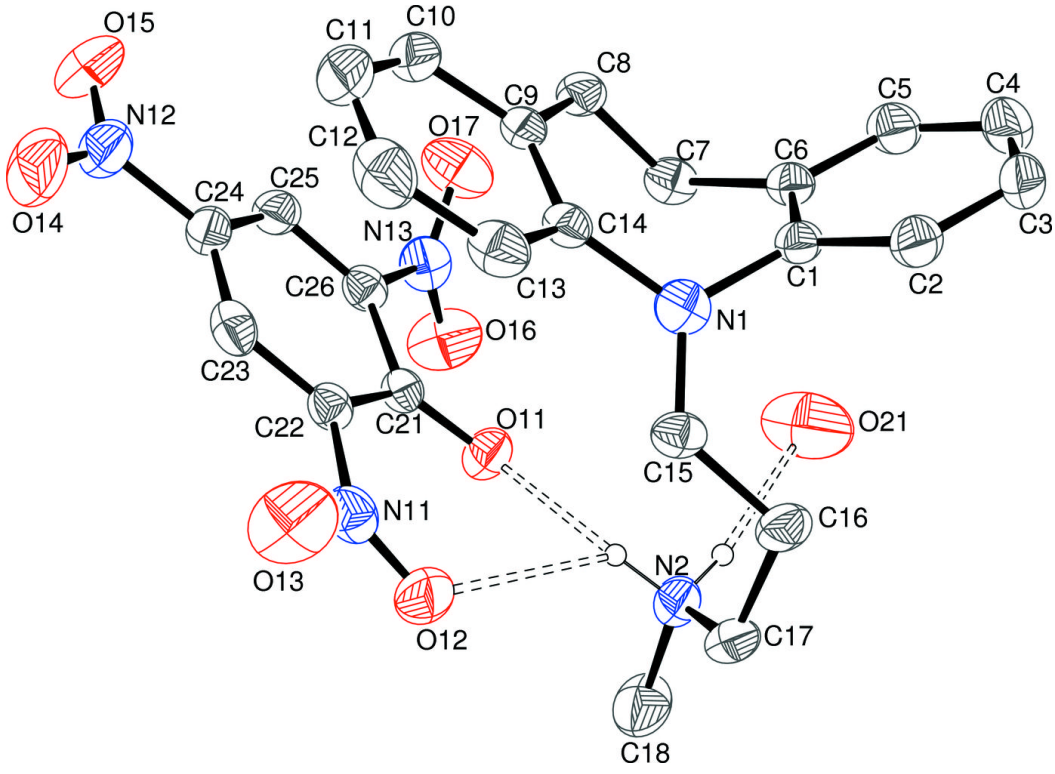
C6—C7—H7B	109.4	C18—N2—H2N	109.1
C8—C7—H7B	109.4	H1N—N2—H2N	100.9
H7A—C7—H7B	108.0	O11—C21—C22	126.3 (2)
C9—C8—C7	118.18 (18)	O11—C21—C26	122.4 (2)
C9—C8—H8A	107.8	C22—C21—C26	111.3 (2)
C7—C8—H8A	107.8	C23—C22—C21	123.6 (2)
C9—C8—H8B	107.8	C23—C22—N11	116.1 (2)
C7—C8—H8B	107.8	C21—C22—N11	120.3 (2)
H8A—C8—H8B	107.1	C24—C23—C22	119.8 (2)
C10—C9—C14	118.3 (2)	C24—C23—H23	120.1
C10—C9—C8	115.8 (2)	C22—C23—H23	120.1
C14—C9—C8	125.9 (2)	C23—C24—C25	121.0 (2)
C11—C10—C9	122.6 (3)	C23—C24—N12	119.7 (3)
C11—C10—H10	118.7	C25—C24—N12	119.3 (3)
C9—C10—H10	118.7	C26—C25—C24	118.7 (2)
C10—C11—C12	119.4 (3)	C26—C25—H25	120.7
C10—C11—H11	120.3	C24—C25—H25	120.7
C12—C11—H11	120.3	C25—C26—C21	125.6 (2)
C11—C12—C13	119.6 (3)	C25—C26—N13	116.1 (2)
C11—C12—H12	120.2	C21—C26—N13	118.3 (2)
C13—C12—H12	120.2	O13—N11—O12	121.8 (3)
C14—C13—C12	121.7 (3)	O13—N11—C22	118.2 (3)
C14—C13—H13	119.2	O12—N11—C22	120.0 (2)
C12—C13—H13	119.2	O14—N12—O15	124.2 (3)
C13—C14—C9	118.4 (2)	O14—N12—C24	118.2 (3)
C13—C14—N1	118.0 (2)	O15—N12—C24	117.6 (3)
C9—C14—N1	123.6 (2)	O18—N13—O16	76.0 (10)
N1—C15—C16	126.5 (2)	O18—N13—O17	67.5 (10)
N1—C15—H15A	105.7	O16—N13—O17	121.7 (3)
C16—C15—H15A	105.7	O18—N13—O19	119.9 (15)
N1—C15—H15B	105.7	O16—N13—O19	82.9 (11)
C16—C15—H15B	105.7	O17—N13—O19	78.2 (11)
H15A—C15—H15B	106.1	O18—N13—C26	127.0 (10)
C15—C16—C17	112.5 (2)	O16—N13—C26	119.3 (3)
C15—C16—H16A	109.1	O17—N13—C26	118.9 (3)
C17—C16—H16A	109.1	O19—N13—C26	112.5 (11)
C15—C16—H16B	109.1		
C6—C1—C2—C3	−0.4 (4)	C9—C14—N1—C15	−136.8 (2)
N1—C1—C2—C3	179.7 (2)	C13—C14—N1—C1	−133.9 (2)
C1—C2—C3—C4	0.0 (4)	C9—C14—N1—C1	45.6 (3)
C2—C3—C4—C5	0.0 (4)	C16—C17—N2—C18	174.3 (3)
C3—C4—C5—C6	0.4 (4)	O11—C21—C22—C23	178.2 (2)
C2—C1—C6—C5	0.8 (3)	C26—C21—C22—C23	0.1 (3)
N1—C1—C6—C5	−179.3 (2)	O11—C21—C22—N11	−3.4 (3)
C2—C1—C6—C7	−176.5 (2)	C26—C21—C22—N11	178.48 (19)
N1—C1—C6—C7	3.4 (3)	C21—C22—C23—C24	−0.1 (3)
C4—C5—C6—C1	−0.8 (4)	N11—C22—C23—C24	−178.6 (2)
C4—C5—C6—C7	176.4 (2)	C22—C23—C24—C25	−0.6 (4)
C1—C6—C7—C8	70.7 (3)	C22—C23—C24—N12	178.5 (2)

C5—C6—C7—C8	−106.4 (3)	C23—C24—C25—C26	1.3 (4)
C6—C7—C8—C9	−69.7 (3)	N12—C24—C25—C26	−177.8 (2)
C7—C8—C9—C10	−165.8 (2)	C24—C25—C26—C21	−1.4 (4)
C7—C8—C9—C14	14.5 (3)	C24—C25—C26—N13	179.8 (2)
C14—C9—C10—C11	0.4 (4)	O11—C21—C26—C25	−177.5 (2)
C8—C9—C10—C11	−179.3 (2)	C22—C21—C26—C25	0.7 (3)
C9—C10—C11—C12	−0.6 (5)	O11—C21—C26—N13	1.3 (3)
C10—C11—C12—C13	0.5 (5)	C22—C21—C26—N13	179.5 (2)
C11—C12—C13—C14	−0.3 (4)	C23—C22—N11—O13	−8.1 (4)
C12—C13—C14—C9	0.1 (4)	C21—C22—N11—O13	173.4 (3)
C12—C13—C14—N1	179.7 (2)	C23—C22—N11—O12	170.7 (2)
C10—C9—C14—C13	−0.2 (3)	C21—C22—N11—O12	−7.8 (3)
C8—C9—C14—C13	179.6 (2)	C23—C24—N12—O14	−1.0 (4)
C10—C9—C14—N1	−179.7 (2)	C25—C24—N12—O14	178.1 (3)
C8—C9—C14—N1	0.0 (3)	C23—C24—N12—O15	179.5 (3)
N1—C15—C16—C17	−136.0 (3)	C25—C24—N12—O15	−1.5 (4)
C15—C16—C17—N2	64.2 (3)	C25—C26—N13—O18	130.1 (13)
C16—C15—N1—C1	−3.1 (4)	C21—C26—N13—O18	−48.8 (14)
C16—C15—N1—C14	179.5 (2)	C25—C26—N13—O16	−135.5 (3)
C2—C1—N1—C15	−63.9 (3)	C21—C26—N13—O16	45.6 (4)
C6—C1—N1—C15	116.2 (2)	C25—C26—N13—O17	47.6 (4)
C2—C1—N1—C14	113.6 (2)	C21—C26—N13—O17	−131.3 (3)
C6—C1—N1—C14	−66.3 (3)	C25—C26—N13—O19	−40.9 (12)
C13—C14—N1—C15	43.6 (3)	C21—C26—N13—O19	140.1 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1N···O11	0.86	2.06	2.817 (3)	146
N2—H1N···O12	0.86	2.38	3.001 (3)	129
N2—H2N···O21	0.86	2.03	2.873 (4)	170

Fig. 1


O15
C11
C10
C8
C9
C5
C4
N12
C12
O17
C7
C6
C24
C25
C3
O14
N13
C14
C26
C13
C1
C2
O16
N1
C23
C22
C21
O11
O21
C15
N11
C16
O13
N2
O12
C17
C18