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Desipraminium picrate

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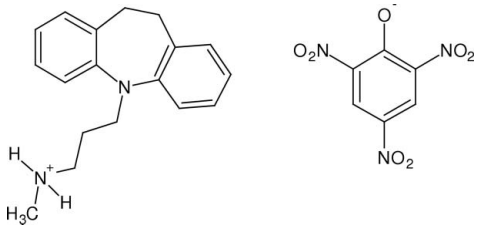
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.125; data-to-parameter ratio = 15.3.

In the title compound, $C_{18}H_{23}N_2^+\cdot C_6H_2N_3O_7^-$, the seven-membered ring of the cation adopts a boat conformation and the two aromatic rings form a dihedral angle of 52.79 (6)°. The anions and cations are connected *via* N—H···O hydrogen bonds.

Related literature

For related literature, see: Ahmed *et al.* (2002); Cohen *et al.* (1990); Cremer & Pople (1975); Eriksoo & Rohte (1970); Ware (1987); Yathirajan *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{23}N_2^+\cdot C_6H_2N_3O_7^-$
M_r = 495.49
Monoclinic, $C2/c$
a = 31.3356 (17) Å
b = 6.7952 (3) Å
c = 24.3100 (13) Å
β = 113.542 (4)°
V = 4745.5 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
0.38 × 0.27 × 0.01 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: none
20306 measured reflections
5107 independent reflections
4268 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.125
S = 1.04
5107 reflections
334 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2C···O31	0.90 (2)	1.89 (2)	2.7662 (16)	164 (2)
N2—H2C···O32	0.90 (2)	2.34 (2)	2.8580 (18)	117 (1)
N2—H2D···O31^{i}	0.92 (2)	2.06 (2)	2.8202 (15)	139 (2)
N2—H2D···O37^{i}	0.92 (2)	2.38 (2)	3.1873 (18)	148 (2)

Symmetry code: (i) $-x, y, -z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2047).

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supplementary materials

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Desipraminium picrate

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Comment

Desipramine, 10,11-dihydro-5-[3-(methylamino)propyl]-5*H*-dibenz[b,f]azepine, is a tricyclic antidepressant (TCA) that inhibits the reuptake of norepinephrine. It is an active metabolite of imipramine. Along with other tricyclics, it has found use in treating neuropathic pain (Eriksoo & Rohte, 1970). It is one of the most potent and selective medications in this respect (Ware, 1987). The effect of desipramine hydrochloride on peripheral sympathetic nerve activity is reported (Cohen *et al.*, 1990). Desipramine hydrochloride is proposed as a new reagent for detection of microamounts of blood in urine (Ahmed *et al.*, 2002).

In continuation of our work on picrates of some pharmaceutical compounds (Yathirajan *et al.*, 2007) and in view of the importance of desipramine, the paper reports the crystal structure of the title compound.

Geometric parameters of the title compound, are in the usual ranges. The seven-membered ring of the cation adopts a boat conformation with puckering parameters: q_2 = 0.7942 (14) Å, q_3 = 0.3246 (15) Å, φ_2 = 142.88 (11)°, φ_3 = 238.4 (3)° (Cremer & Pople, 1975) and the dihedral angle between the two aromatic rings is 52.79 (6)°. The anions and the cations are connected *via* N—H···O hydrogen bonds.

Experimental

Desipramine hydrochloride (3.03 g, 0.01 mol) was dissolved in 20 ml of water. Picric acid (2.29 g, 0.01 mol) was dissolved in 30 ml of water. Both the solutions were mixed and stirred for few minutes. The formed complex was filtered, dried and crystals were obtained by slow evaporation of the ethanolic solution (m. p.: 431–435 K).

Refinement

H atoms were found in a difference map, but those bonded to C were geometrically positioned and included in the refinements with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C) or U(H) = 1.5 U_{eq}(C_{methyl})] using a riding model with $C_{aromatic}$—H = 0.95 Å, $C_{methylene}$—H = 0.99Å and C_{methyl}—H = 0.98 Å. The methyl group was allowed to rotate but not to tip. The amino H atoms were freely refined.

Figures

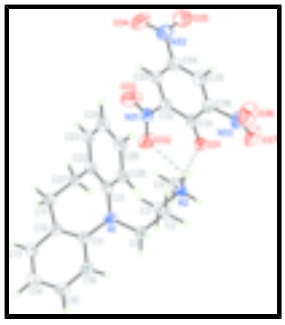


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. The hydrogen bonds are shown as dashed lines.

Desipraminium picrate

Crystal data

$C_{18}H_{23}N_2^+ \cdot C_6H_2N_3O_7^-$	F_{000} = 2080
M_r = 495.49	D_x = 1.387 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 431 - 435 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 31.3356 (17) Å	Cell parameters from 20595 reflections
b = 6.7952 (3) Å	θ = 2.6–27.1º
c = 24.3100 (13) Å	μ = 0.10 mm^{-1}
β = 113.542 (4)º	*T* = 173 (2) K
V = 4745.5 (4) Å^3	Plate, yellow
Z = 8	0.38 × 0.27 × 0.01 mm

Data collection

Stoe IPDSII two-circle diffractometer	4268 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.041
Monochromator: graphite	θ_{max} = 26.9º
T = 173(2) K	θ_{min} = 2.7º
ω scans	*h* = −39→39
Absorption correction: none	*k* = −7→8
20306 measured reflections	*l* = −30→30
5107 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.125	$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 2.222P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
5107 reflections	$\Delta\rho_{max} = 0.51$ e Å^{-3}
334 parameters	$\Delta\rho_{min} = -0.40$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Mol. Formula: $[C_{18}H_{23}N_2]^+[C_6H_2N_3O_7]^-$. Composition: Found (Calculated): C: 58.11 (58.18); H: 5.02 (5.09); N: 14.18 (14.13).

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}
N1	0.19958 (4)	0.69793 (17)	0.33576 (5)	0.0274 (2)
C1	0.16257 (4)	0.8250 (2)	0.29473 (6)	0.0307 (3)
H1A	0.1661	0.8353	0.2561	0.037*
H1B	0.1659	0.9587	0.3121	0.037*
C2	0.11411 (4)	0.7457 (2)	0.28317 (6)	0.0306 (3)
H2A	0.1100	0.7406	0.3215	0.037*
H2B	0.0903	0.8362	0.2558	0.037*
C3	0.10693 (4)	0.5419 (2)	0.25562 (6)	0.0282 (3)
H3A	0.1116	0.5462	0.2177	0.034*
H3B	0.1302	0.4504	0.2834	0.034*
N2	0.05907 (4)	0.46825 (18)	0.24327 (5)	0.0279 (2)
H2C	0.0533 (6)	0.476 (3)	0.2768 (8)	0.039 (4)*
H2D	0.0378 (6)	0.546 (3)	0.2147 (8)	0.038 (4)*
C4	0.05117 (5)	0.2612 (2)	0.22127 (7)	0.0358 (3)
H4A	0.0552	0.2517	0.1834	0.054*
H4B	0.0195	0.2210	0.2148	0.054*
H4C	0.0736	0.1746	0.2512	0.054*
C11	0.24440 (4)	0.7099 (2)	0.33351 (5)	0.0270 (3)
C12	0.27825 (4)	0.5601 (2)	0.35688 (6)	0.0284 (3)
C13	0.32069 (5)	0.5830 (2)	0.35128 (7)	0.0365 (3)
H13	0.3438	0.4844	0.3677	0.044*
C14	0.33045 (5)	0.7425 (3)	0.32298 (8)	0.0428 (4)
H14	0.3596	0.7526	0.3197	0.051*
C15	0.29706 (5)	0.8871 (3)	0.29952 (7)	0.0410 (4)

H15	0.3030	0.9970	0.2795	0.049*
C16	0.25482 (5)	0.8717 (2)	0.30518 (6)	0.0333 (3)
H16	0.2324	0.9734	0.2895	0.040*
C17	0.27325 (5)	0.3698 (2)	0.38639 (6)	0.0323 (3)
H17A	0.2937	0.3779	0.4296	0.039*
H17B	0.2851	0.2612	0.3692	0.039*
C21	0.19701 (4)	0.6516 (2)	0.39189 (6)	0.0276 (3)
C22	0.20698 (4)	0.4590 (2)	0.41360 (6)	0.0297 (3)
C23	0.20271 (5)	0.4103 (2)	0.46694 (6)	0.0371 (3)
H23	0.2092	0.2797	0.4819	0.045*
C24	0.18910 (5)	0.5491 (3)	0.49853 (7)	0.0415 (4)
H24	0.1867	0.5141	0.5350	0.050*
C25	0.17903 (5)	0.7386 (3)	0.47653 (7)	0.0395 (3)
H25	0.1696	0.8342	0.4979	0.047*
C26	0.18265 (5)	0.7901 (2)	0.42310 (6)	0.0332 (3)
H26	0.1753	0.9202	0.4079	0.040*
C27	0.22439 (5)	0.3143 (2)	0.38077 (6)	0.0321 (3)
H27A	0.2029	0.3117	0.3379	0.039*
H27B	0.2250	0.1809	0.3975	0.039*
O31	0.03288 (3)	0.55588 (17)	0.33651 (4)	0.0368 (2)
O32	0.09643 (6)	0.2663 (2)	0.35570 (5)	0.0636 (4)
O33	0.10213 (8)	0.0736 (3)	0.42713 (9)	0.0966 (7)
O34	0.10268 (6)	0.3341 (2)	0.60773 (6)	0.0680 (4)
O35	0.05950 (5)	0.5845 (2)	0.60442 (5)	0.0610 (4)
O36	0.00360 (6)	0.9942 (2)	0.42802 (6)	0.0713 (5)
O37	−0.02365 (5)	0.8368 (2)	0.34441 (5)	0.0601 (4)
N31	0.09068 (5)	0.2297 (2)	0.40142 (6)	0.0425 (3)
N32	0.07745 (5)	0.4708 (2)	0.58073 (6)	0.0478 (4)
N33	0.00219 (5)	0.8483 (2)	0.39776 (6)	0.0432 (3)
C31	0.04474 (4)	0.5401 (2)	0.39216 (6)	0.0292 (3)
C32	0.07263 (5)	0.3815 (2)	0.42855 (6)	0.0321 (3)
C33	0.08277 (5)	0.3588 (2)	0.48851 (6)	0.0367 (3)
H33	0.1000	0.2479	0.5097	0.044*
C34	0.06763 (5)	0.4987 (3)	0.51768 (6)	0.0375 (3)
C35	0.04210 (5)	0.6613 (2)	0.48778 (7)	0.0381 (3)
H35	0.0325	0.7583	0.5087	0.046*
C36	0.03096 (5)	0.6790 (2)	0.42741 (6)	0.0338 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0255 (5)	0.0265 (6)	0.0293 (5)	−0.0007 (4)	0.0099 (4)	0.0052 (4)
C1	0.0287 (6)	0.0257 (7)	0.0332 (6)	−0.0006 (5)	0.0077 (5)	0.0038 (5)
C2	0.0266 (6)	0.0268 (7)	0.0343 (6)	0.0021 (5)	0.0078 (5)	−0.0008 (5)
C3	0.0243 (6)	0.0300 (7)	0.0304 (6)	−0.0008 (5)	0.0110 (5)	−0.0024 (5)
N2	0.0258 (5)	0.0297 (6)	0.0278 (5)	−0.0015 (4)	0.0103 (4)	−0.0019 (4)
C4	0.0409 (7)	0.0314 (7)	0.0380 (7)	−0.0079 (6)	0.0186 (6)	−0.0073 (6)
C11	0.0270 (6)	0.0265 (6)	0.0259 (6)	−0.0045 (5)	0.0089 (5)	−0.0014 (5)

C12	0.0287 (6)	0.0281 (7)	0.0279 (6)	−0.0029 (5)	0.0106 (5)	−0.0011 (5)
C13	0.0299 (7)	0.0395 (8)	0.0404 (7)	0.0013 (6)	0.0144 (6)	0.0037 (6)
C14	0.0327 (7)	0.0506 (10)	0.0499 (9)	−0.0043 (7)	0.0213 (7)	0.0064 (7)
C15	0.0392 (8)	0.0420 (9)	0.0448 (8)	−0.0076 (7)	0.0200 (6)	0.0090 (7)
C16	0.0330 (7)	0.0309 (7)	0.0347 (7)	−0.0029 (6)	0.0122 (5)	0.0045 (6)
C17	0.0321 (6)	0.0266 (7)	0.0381 (7)	0.0024 (5)	0.0139 (5)	0.0039 (5)
C21	0.0227 (6)	0.0303 (7)	0.0280 (6)	−0.0043 (5)	0.0084 (5)	0.0010 (5)
C22	0.0226 (6)	0.0319 (7)	0.0325 (6)	−0.0031 (5)	0.0087 (5)	0.0049 (5)
C23	0.0283 (6)	0.0448 (9)	0.0372 (7)	0.0023 (6)	0.0119 (6)	0.0138 (6)
C24	0.0318 (7)	0.0612 (11)	0.0322 (7)	0.0024 (7)	0.0136 (6)	0.0102 (7)
C25	0.0335 (7)	0.0504 (9)	0.0357 (7)	−0.0005 (6)	0.0151 (6)	−0.0050 (6)
C26	0.0299 (6)	0.0322 (7)	0.0372 (7)	−0.0026 (5)	0.0130 (5)	−0.0012 (6)
C27	0.0320 (7)	0.0256 (7)	0.0365 (7)	−0.0031 (5)	0.0112 (5)	0.0039 (5)
O31	0.0328 (5)	0.0484 (6)	0.0290 (5)	0.0081 (4)	0.0121 (4)	0.0044 (4)
O32	0.0868 (10)	0.0726 (10)	0.0390 (6)	0.0363 (8)	0.0332 (7)	0.0103 (6)
O33	0.1541 (18)	0.0715 (11)	0.1071 (13)	0.0735 (12)	0.0972 (14)	0.0479 (10)
O34	0.0879 (10)	0.0748 (10)	0.0363 (6)	0.0239 (8)	0.0193 (6)	0.0147 (6)
O35	0.0617 (8)	0.0845 (10)	0.0341 (6)	0.0097 (7)	0.0164 (6)	−0.0104 (6)
O36	0.0940 (11)	0.0473 (8)	0.0601 (8)	0.0245 (8)	0.0176 (8)	−0.0128 (7)
O37	0.0706 (8)	0.0502 (8)	0.0416 (6)	0.0220 (6)	0.0035 (6)	0.0007 (5)
N31	0.0410 (7)	0.0471 (8)	0.0428 (7)	0.0167 (6)	0.0203 (6)	0.0095 (6)
N32	0.0470 (8)	0.0627 (10)	0.0300 (6)	0.0009 (7)	0.0114 (6)	−0.0016 (6)
N33	0.0478 (7)	0.0355 (7)	0.0412 (7)	0.0072 (6)	0.0126 (6)	−0.0023 (6)
C31	0.0231 (6)	0.0332 (7)	0.0300 (6)	−0.0017 (5)	0.0094 (5)	−0.0002 (5)
C32	0.0279 (6)	0.0374 (8)	0.0320 (6)	0.0029 (5)	0.0128 (5)	0.0008 (6)
C33	0.0305 (7)	0.0436 (8)	0.0339 (7)	0.0040 (6)	0.0105 (5)	0.0076 (6)
C34	0.0342 (7)	0.0481 (9)	0.0272 (6)	−0.0012 (6)	0.0091 (5)	−0.0009 (6)
C35	0.0360 (7)	0.0415 (8)	0.0334 (7)	−0.0014 (6)	0.0104 (6)	−0.0084 (6)
C36	0.0314 (7)	0.0319 (7)	0.0337 (7)	0.0012 (5)	0.0084 (5)	−0.0016 (6)

Geometric parameters (Å, °)

N1—C11	1.4297 (16)	C21—C26	1.392 (2)
N1—C21	1.4335 (17)	C21—C22	1.399 (2)
N1—C1	1.4699 (16)	C22—C23	1.3955 (19)
C1—C2	1.5279 (18)	C22—C27	1.500 (2)
C1—H1A	0.9900	C23—C24	1.386 (2)
C1—H1B	0.9900	C23—H23	0.9500
C2—C3	1.5156 (19)	C24—C25	1.382 (2)
C2—H2A	0.9900	C24—H24	0.9500
C2—H2B	0.9900	C25—C26	1.393 (2)
C3—N2	1.4933 (16)	C25—H25	0.9500
C3—H3A	0.9900	C26—H26	0.9500
C3—H3B	0.9900	C27—H27A	0.9900
N2—C4	1.4906 (18)	C27—H27B	0.9900
N2—H2C	0.904 (19)	O31—C31	1.2564 (16)
N2—H2D	0.915 (19)	O32—N31	1.2203 (18)
C4—H4A	0.9800	O33—N31	1.211 (2)
C4—H4B	0.9800	O34—N32	1.227 (2)

C4—H4C	0.9800	O35—N32	1.226 (2)
C11—C16	1.4038 (19)	O36—N33	1.2245 (19)
C11—C12	1.4146 (19)	O37—N33	1.2267 (18)
C12—C13	1.3986 (19)	N31—C32	1.455 (2)
C12—C17	1.5171 (19)	N32—C34	1.4504 (19)
C13—C14	1.382 (2)	N33—C36	1.462 (2)
C13—H13	0.9500	C31—C32	1.446 (2)
C14—C15	1.381 (2)	C31—C36	1.452 (2)
C14—H14	0.9500	C32—C33	1.371 (2)
C15—C16	1.388 (2)	C33—C34	1.379 (2)
C15—H15	0.9500	C33—H33	0.9500
C16—H16	0.9500	C34—C35	1.387 (2)
C17—C27	1.5292 (19)	C35—C36	1.371 (2)
C17—H17A	0.9900	C35—H35	0.9500
C17—H17B	0.9900		
C11—N1—C21	118.58 (10)	C12—C17—H17B	108.0
C11—N1—C1	117.48 (10)	C27—C17—H17B	108.0
C21—N1—C1	116.48 (11)	H17A—C17—H17B	107.2
N1—C1—C2	112.00 (11)	C26—C21—C22	119.94 (13)
N1—C1—H1A	109.2	C26—C21—N1	121.60 (12)
C2—C1—H1A	109.2	C22—C21—N1	118.38 (12)
N1—C1—H1B	109.2	C23—C22—C21	118.83 (14)
C2—C1—H1B	109.2	C23—C22—C27	121.97 (13)
H1A—C1—H1B	107.9	C21—C22—C27	119.12 (12)
C3—C2—C1	111.37 (11)	C24—C23—C22	121.23 (15)
C3—C2—H2A	109.4	C24—C23—H23	119.4
C1—C2—H2A	109.4	C22—C23—H23	119.4
C3—C2—H2B	109.4	C25—C24—C23	119.52 (14)
C1—C2—H2B	109.4	C25—C24—H24	120.2
H2A—C2—H2B	108.0	C23—C24—H24	120.2
N2—C3—C2	110.79 (11)	C24—C25—C26	120.26 (15)
N2—C3—H3A	109.5	C24—C25—H25	119.9
C2—C3—H3A	109.5	C26—C25—H25	119.9
N2—C3—H3B	109.5	C21—C26—C25	120.21 (14)
C2—C3—H3B	109.5	C21—C26—H26	119.9
H3A—C3—H3B	108.1	C25—C26—H26	119.9
C4—N2—C3	113.46 (11)	C22—C27—C17	110.76 (11)
C4—N2—H2C	108.3 (12)	C22—C27—H27A	109.5
C3—N2—H2C	110.3 (11)	C17—C27—H27A	109.5
C4—N2—H2D	107.9 (11)	C22—C27—H27B	109.5
C3—N2—H2D	109.0 (11)	C17—C27—H27B	109.5
H2C—N2—H2D	107.8 (16)	H27A—C27—H27B	108.1
N2—C4—H4A	109.5	O33—N31—O32	121.83 (15)
N2—C4—H4B	109.5	O33—N31—C32	118.42 (14)
H4A—C4—H4B	109.5	O32—N31—C32	119.62 (14)
N2—C4—H4C	109.5	O35—N32—O34	122.98 (14)
H4A—C4—H4C	109.5	O35—N32—C34	118.08 (15)
H4B—C4—H4C	109.5	O34—N32—C34	118.94 (15)
C16—C11—C12	118.47 (12)	O36—N33—O37	122.47 (14)

C16—C11—N1	119.19 (12)	O36—N33—C36	118.27 (13)
C12—C11—N1	122.30 (12)	O37—N33—C36	119.21 (13)
C13—C12—C11	118.08 (12)	O31—C31—C32	124.70 (13)
C13—C12—C17	114.98 (12)	O31—C31—C36	123.38 (13)
C11—C12—C17	126.91 (12)	C32—C31—C36	111.91 (12)
C14—C13—C12	122.93 (14)	C33—C32—C31	124.14 (13)
C14—C13—H13	118.5	C33—C32—N31	115.80 (13)
C12—C13—H13	118.5	C31—C32—N31	120.04 (12)
C15—C14—C13	118.78 (14)	C32—C33—C34	119.20 (14)
C15—C14—H14	120.6	C32—C33—H33	120.4
C13—C14—H14	120.6	C34—C33—H33	120.4
C14—C15—C16	120.06 (14)	C33—C34—C35	121.66 (13)
C14—C15—H15	120.0	C33—C34—N32	118.82 (14)
C16—C15—H15	120.0	C35—C34—N32	119.48 (14)
C15—C16—C11	121.67 (14)	C36—C35—C34	118.49 (14)
C15—C16—H16	119.2	C36—C35—H35	120.8
C11—C16—H16	119.2	C34—C35—H35	120.8
C12—C17—C27	117.19 (11)	C35—C36—C31	124.50 (13)
C12—C17—H17A	108.0	C35—C36—N33	116.07 (13)
C27—C17—H17A	108.0	C31—C36—N33	119.41 (12)
C11—N1—C1—C2	157.46 (12)	C22—C21—C26—C25	1.2 (2)
C21—N1—C1—C2	−53.05 (15)	N1—C21—C26—C25	177.85 (12)
N1—C1—C2—C3	−59.70 (15)	C24—C25—C26—C21	−0.8 (2)
C1—C2—C3—N2	−178.70 (11)	C23—C22—C27—C17	−108.52 (14)
C2—C3—N2—C4	−174.77 (11)	C21—C22—C27—C17	68.09 (15)
C21—N1—C11—C16	−132.52 (13)	C12—C17—C27—C22	−65.78 (16)
C1—N1—C11—C16	16.31 (17)	O31—C31—C32—C33	−175.47 (14)
C21—N1—C11—C12	49.96 (17)	C36—C31—C32—C33	3.4 (2)
C1—N1—C11—C12	−161.21 (12)	O31—C31—C32—N31	2.6 (2)
C16—C11—C12—C13	0.94 (19)	C36—C31—C32—N31	−178.59 (13)
N1—C11—C12—C13	178.48 (12)	O33—N31—C32—C33	18.8 (2)
C16—C11—C12—C17	−176.99 (13)	O32—N31—C32—C33	−156.98 (16)
N1—C11—C12—C17	0.6 (2)	O33—N31—C32—C31	−159.42 (18)
C11—C12—C13—C14	−1.4 (2)	O32—N31—C32—C31	24.8 (2)
C17—C12—C13—C14	176.81 (14)	C31—C32—C33—C34	−3.1 (2)
C12—C13—C14—C15	0.5 (3)	N31—C32—C33—C34	178.74 (14)
C13—C14—C15—C16	0.7 (3)	C32—C33—C34—C35	0.5 (2)
C14—C15—C16—C11	−1.1 (2)	C32—C33—C34—N32	178.38 (14)
C12—C11—C16—C15	0.3 (2)	O35—N32—C34—C33	−172.16 (15)
N1—C11—C16—C15	−177.35 (13)	O34—N32—C34—C33	6.9 (2)
C13—C12—C17—C27	−166.15 (13)	O35—N32—C34—C35	5.8 (2)
C11—C12—C17—C27	11.8 (2)	O34—N32—C34—C35	−175.10 (16)
C11—N1—C21—C26	111.04 (14)	C33—C34—C35—C36	1.5 (2)
C1—N1—C21—C26	−38.11 (17)	N32—C34—C35—C36	−176.34 (14)
C11—N1—C21—C22	−72.31 (15)	C34—C35—C36—C31	−1.1 (2)
C1—N1—C21—C22	138.55 (12)	C34—C35—C36—N33	177.23 (14)
C26—C21—C22—C23	−0.66 (19)	O31—C31—C36—C35	177.65 (14)
N1—C21—C22—C23	−177.37 (11)	C32—C31—C36—C35	−1.2 (2)
C26—C21—C22—C27	−177.38 (12)	O31—C31—C36—N33	−0.6 (2)

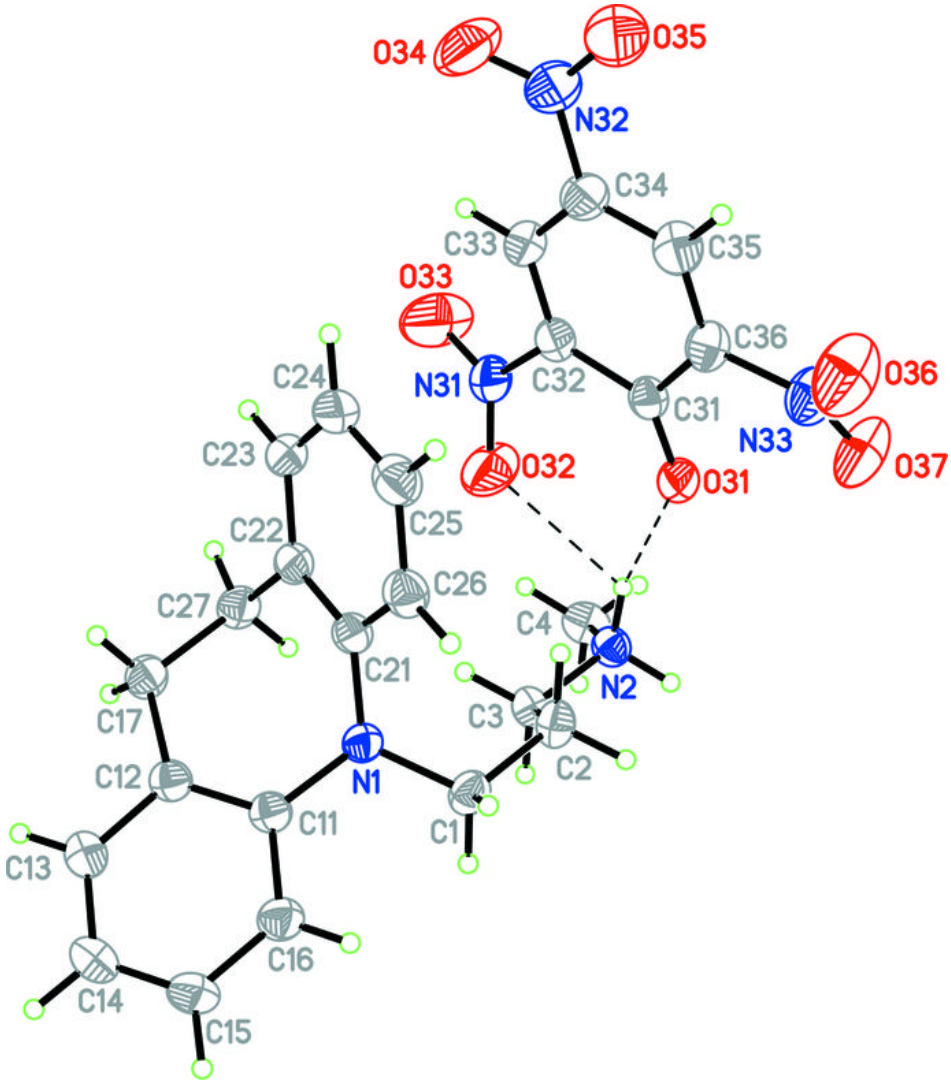
N1—C21—C22—C27	5.91 (17)	C32—C31—C36—N33	−179.48 (13)
C21—C22—C23—C24	−0.3 (2)	O36—N33—C36—C35	25.7 (2)
C27—C22—C23—C24	176.28 (13)	O37—N33—C36—C35	−151.61 (16)
C22—C23—C24—C25	0.8 (2)	O36—N33—C36—C31	−155.92 (16)
C23—C24—C25—C26	−0.2 (2)	O37—N33—C36—C31	26.8 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2C···O31	0.90 (2)	1.89 (2)	2.7662 (16)	164 (2)
N2—H2C···O32	0.90 (2)	2.34 (2)	2.8580 (18)	117 (1)
N2—H2D···O31^{i}	0.92 (2)	2.06 (2)	2.8202 (15)	139 (2)
N2—H2D···O37^{i}	0.92 (2)	2.38 (2)	3.1873 (18)	148 (2)

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

Fig. 1


O34
O35
N32
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O32
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C3
N1
C12
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C11
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C13
C16
C14
C15