Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Nevirapininium picrate

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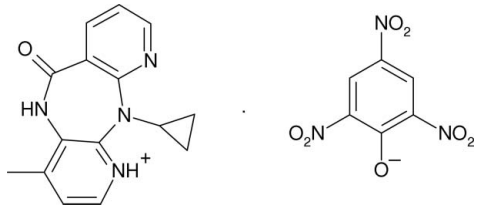
Received 15 August 2007; accepted 17 August 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.120; data-to-parameter ratio = 14.7.

The title compound, $C_{15}H_{15}N_4O^+\cdot C_6H_2N_3O_7^-$, is the picrate salt of nevirapine, in which the cation and anion are linked by an N—H···O hydrogen bond. A second N—H···O interaction leads to centrosymmetric dimers of cations. The dihedral angle between the aromatic ring planes in the cation is 48.27 (8)°.

Related literature

For the structure of nevirapine, see: Mui *et al.* (1992). For related structures, see: Harrison, Ashok *et al.* (2007); Harrison, Bindya *et al.* (2007). For background, see: Herbstein & Kaftory (1976); Bartlett (2005); Gazzard (2005); Manosuthi *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{15}N_4O^+\cdot C_6H_2N_3O_7^-$
M_r = 495.42
Triclinic, $P\overline{1}$
a = 9.9921 (5) Å
b = 10.2126 (5) Å
c = 11.5332 (6) Å
α = 70.716 (1)°
β = 77.980 (1)°
γ = 87.355 (1)°
V = 1086.19 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 291 (2) K
0.40 × 0.30 × 0.24 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.955, T_{max} = 0.972
8228 measured reflections
4911 independent reflections
3575 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.120
S = 1.05
4911 reflections
333 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···O1^{i}	0.881 (19)	2.063 (19)	2.9242 (17)	165.4 (16)
N4—H4N···O11	0.878 (19)	1.808 (19)	2.6656 (17)	164.9 (17)
C9—H9···O14^{ii}	0.93	2.46	3.386 (2)	174
C23—H23···O15^{iii}	0.93	2.49	3.335 (2)	151

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

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*.

TVS thanks Mangalore University for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2026).

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 doi:10.1107/S1600536807040731

supplementary materials

Acta Cryst. (2007). E**63**, o3871 [**doi:10.1107/S1600536807040731**]

Nevirapininium picrate

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Comment

Nevirapine, $C_{15}H_{14}N_4O$, has several important biological applications: it is a non-nucleoside reverse transcriptase inhibitor (NNRTI) used to treat HIV-1 infection and AIDS (Bartlett, 2005) and it is an inducer of cytochrome P450 isoenzymes CYP3A4 and CYP2B6 (Gazzard, 2005). Nevirapine in triple combination therapy has been shown to suppress viral load effectively when used as initial antiretroviral therapy (Manosuthi *et al.*, 2007).

The crystal structure of nevirapine was described earlier (Mui *et al.*, 1992). In continuation of our work on the structures of pharmaceutical compounds (Harrison, Ashok *et al.*, 2007; Harrison, Bindya *et al.*, 2007), we now report the crystal structure of the title compound, (I), a molecular salt of nevirapine and picric acid.

The structure of (I) (Fig. 1) shows that proton transfer from picric acid (pa) to nevirapine (np) has occurred, and that the N atom of the methyl-substituted pyridine ring has been protonated. The dihedral angle between the C1—C5/N1 and C7—C11/N4 ring planes is 48.27 (8)°, which is substantially different to the equivalent value of 59° (no s.u. stated) for unprotonated nevirapine (Mui *et al.*, 1992). This difference may arise due to the flexibility of the central seven-membered ring. In (I), the bond-angle sum about N2 (350.6°) is ambiguous with respect to the hybridization of the nitrogen atom. The equivalent value for N3 (358.3°) equates to sp^2 hybridization, perhaps due to delocalization with the adjacent pyridine ring (Mui *et al.*, 1992).

The significant variation of the C—C bond lengths around the picrate aromatic ring in (I) are normal and can be related to the contributions of various resonance forms involving the nitro groups (Herbstein & Kaftory, 976). The N11/O12/O13 nitro group in (I) is twisted from the benzene ring plane by 46.55 (12)°, whereas the other two nitro groups are close to co-planar with the ring [equivalent dihedral angles for N12/O14/O15 and N13/O16/O17 = 3.03 (11) and 12.2 (2)°, respectively].

The two constituents of (I) interact by a strong, near linear N4—H4N···O11 link (Table 1). Then, centrosymmetric associations of these ion pairs arise from the N3—HN3···O1^{i} (see Table 1 for symmetry code) bond (Fig. 2). Two short intermolecular C—H···O interactions also occur (Table 1) and a short π-π stacking interaction involving the C1—C5/N1 ring and its inversion-generated partner at (1 − *x*, 2 − *y*, −*z*) with a centroid···centroid separation of 3.5486 (9)Å completes the structure of (I).

Experimental

Nevirapine (2.66 g, 0.01 mol) was dissolved in 25 ml of ethanol. Picric acid (2.29 g, 0.01 mol) was dissolved in 10 ml of water. The solutions were mixed and 5 ml of 5*M* HCl was added to this mixture and stirred for few minutes. The resulting solid was filtered, dried and yellow crystals of (I) were obtained by slow evaporation of an ethanol solution (m.p.: 489–491 K; analysis for $C_{21}H_{17}N_7O_8$: Found (calculated): C 50.88 (50.91); H 3.39 (3.46); N 19.71% (19.79%).

Refinement

The N-bound H atoms were located in difference maps and their positions were freely refined 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.

Figures

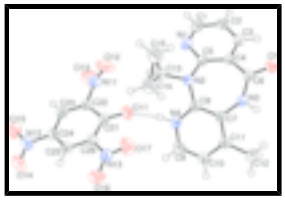

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The hydrogen bond is shown as a double-dashed line.

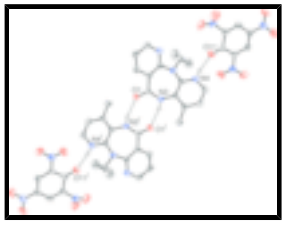

Fig. 2. View of a dimeric association of ion pairs in (I) with all C-bound H atoms omitted for clarity. Symmetry code as in Table 1.

Nevirapininium picrate

Crystal data

$C_{15}H_{15}N_4O^+·C_6H_2N_3O_7^-$	Z = 2
M_r = 495.42	F_{000} = 512
Triclinic, $P\overline{1}$	D_x = 1.515 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.9921 (5) Å	Cell parameters from 3856 reflections
b = 10.2126 (5) Å	θ = 2.3–27.5º
c = 11.5332 (6) Å	μ = 0.12 mm^{-1}
α = 70.716 (1)º	T = 291 (2) K
β = 77.980 (1)º	Block, yellow
γ = 87.355 (1)º	0.40 × 0.30 × 0.24 mm
V = 1086.19 (9) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	4911 independent reflections
Radiation source: fine-focus sealed tube	3575 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.015
T = 291(2) K	θ_{max} = 27.5º

ω scans	θ_{min} = 2.1°
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −12→12
T_{min} = 0.955, T_{max} = 0.972	k = −13→13
8228 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap (N-H) and geom (C-H)
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.120	$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.0965P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
4911 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
333 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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}
C1	0.30569 (18)	1.05420 (17)	0.08754 (17)	0.0448 (4)
H1	0.2861	1.1430	0.0909	0.054*
C2	0.30848 (17)	1.03144 (17)	−0.02357 (16)	0.0426 (4)
H2	0.2879	1.1015	−0.0925	0.051*
C3	0.34278 (16)	0.90120 (16)	−0.02970 (15)	0.0373 (4)
H3	0.3497	0.8833	−0.1047	0.045*
C4	0.36700 (14)	0.79642 (14)	0.07669 (13)	0.0300 (3)
C5	0.35655 (14)	0.83030 (14)	0.18620 (14)	0.0306 (3)
C6	0.41730 (15)	0.66361 (15)	0.05906 (14)	0.0329 (3)
C7	0.31537 (15)	0.51453 (14)	0.27735 (13)	0.0310 (3)
C8	0.30914 (15)	0.60597 (15)	0.34613 (13)	0.0313 (3)
C9	0.16245 (17)	0.44673 (17)	0.51808 (15)	0.0413 (4)

H9	0.1121	0.4249	0.5998	0.050*
C10	0.16491 (17)	0.35632 (16)	0.45418 (15)	0.0409 (4)
H10	0.1151	0.2730	0.4916	0.049*
C11	0.24218 (16)	0.38812 (15)	0.33232 (14)	0.0354 (3)
C12	0.2488 (2)	0.28451 (18)	0.26464 (17)	0.0516 (5)
H12A	0.2239	0.3280	0.1848	0.077*
H12B	0.1865	0.2082	0.3138	0.077*
H12C	0.3402	0.2510	0.2520	0.077*
C13	0.40744 (19)	0.79138 (17)	0.39300 (15)	0.0436 (4)
H13	0.3329	0.8414	0.4286	0.052*
C14	0.4982 (2)	0.7114 (2)	0.47783 (19)	0.0604 (5)
H14A	0.5339	0.6250	0.4675	0.072*
H14B	0.4787	0.7122	0.5636	0.072*
C15	0.5491 (2)	0.8434 (3)	0.3756 (2)	0.0744 (7)
H15A	0.5606	0.9242	0.3995	0.089*
H15B	0.6157	0.8370	0.3034	0.089*
N1	0.32925 (14)	0.95717 (13)	0.19133 (13)	0.0408 (3)
N2	0.38048 (13)	0.73279 (12)	0.30040 (11)	0.0342 (3)
N3	0.40003 (14)	0.54343 (13)	0.15722 (12)	0.0355 (3)
H3N	0.4310 (18)	0.4702 (19)	0.1367 (16)	0.043*
N4	0.23304 (13)	0.56827 (14)	0.46307 (12)	0.0362 (3)
H4N	0.2240 (18)	0.6221 (19)	0.5097 (17)	0.043*
O1	0.47388 (13)	0.66345 (12)	−0.04642 (10)	0.0478 (3)
C21	0.14876 (16)	0.71482 (16)	0.73045 (14)	0.0356 (3)
C22	0.09458 (16)	0.83633 (15)	0.76164 (14)	0.0354 (3)
C23	0.05246 (16)	0.84102 (16)	0.88013 (15)	0.0373 (4)
H23	0.0128	0.9201	0.8940	0.045*
C24	0.07029 (16)	0.72442 (16)	0.98010 (14)	0.0361 (3)
C25	0.13314 (16)	0.60842 (16)	0.95952 (15)	0.0356 (3)
H25	0.1474	0.5326	1.0271	0.043*
C26	0.17471 (15)	0.60485 (15)	0.83888 (15)	0.0346 (3)
N11	0.08437 (17)	0.96236 (14)	0.65664 (14)	0.0460 (4)
N12	0.02625 (15)	0.72525 (16)	1.10704 (14)	0.0450 (3)
N13	0.24590 (15)	0.48166 (14)	0.82482 (14)	0.0432 (3)
O11	0.16134 (14)	0.71111 (13)	0.62164 (11)	0.0528 (3)
O12	0.18419 (18)	0.99914 (15)	0.57137 (15)	0.0781 (5)
O13	−0.02037 (18)	1.02612 (18)	0.65992 (16)	0.0875 (6)
O14	0.04083 (15)	0.61899 (16)	1.19367 (11)	0.0602 (4)
O15	−0.02505 (17)	0.83064 (15)	1.12454 (13)	0.0670 (4)
O16	0.24491 (16)	0.38042 (13)	0.91924 (13)	0.0620 (4)
O17	0.30523 (17)	0.48267 (15)	0.72064 (13)	0.0704 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0555 (10)	0.0274 (8)	0.0513 (10)	0.0056 (7)	−0.0086 (8)	−0.0145 (7)
C2	0.0482 (9)	0.0314 (8)	0.0428 (9)	0.0041 (7)	−0.0092 (7)	−0.0057 (7)
C3	0.0422 (8)	0.0363 (8)	0.0331 (8)	−0.0014 (6)	−0.0067 (7)	−0.0114 (7)

C4	0.0326 (7)	0.0267 (7)	0.0297 (7)	−0.0021 (5)	−0.0026 (6)	−0.0100 (6)
C5	0.0312 (7)	0.0278 (7)	0.0327 (8)	−0.0010 (5)	−0.0037 (6)	−0.0112 (6)
C6	0.0379 (8)	0.0298 (7)	0.0309 (8)	0.0009 (6)	−0.0038 (6)	−0.0117 (6)
C7	0.0381 (8)	0.0268 (7)	0.0288 (7)	0.0045 (6)	−0.0078 (6)	−0.0098 (6)
C8	0.0363 (7)	0.0288 (7)	0.0294 (7)	0.0038 (6)	−0.0078 (6)	−0.0103 (6)
C9	0.0469 (9)	0.0410 (9)	0.0300 (8)	−0.0010 (7)	−0.0010 (7)	−0.0074 (7)
C10	0.0488 (9)	0.0312 (8)	0.0372 (9)	−0.0027 (7)	−0.0045 (7)	−0.0063 (7)
C11	0.0442 (8)	0.0278 (7)	0.0345 (8)	0.0023 (6)	−0.0101 (7)	−0.0094 (6)
C12	0.0739 (12)	0.0345 (9)	0.0462 (10)	−0.0101 (8)	−0.0026 (9)	−0.0174 (8)
C13	0.0577 (10)	0.0426 (9)	0.0363 (9)	−0.0056 (7)	−0.0103 (8)	−0.0192 (7)
C14	0.0669 (13)	0.0731 (14)	0.0477 (11)	−0.0071 (10)	−0.0223 (10)	−0.0210 (10)
C15	0.0817 (15)	0.0929 (17)	0.0526 (12)	−0.0393 (13)	−0.0131 (11)	−0.0242 (12)
N1	0.0524 (8)	0.0301 (7)	0.0415 (8)	0.0026 (6)	−0.0071 (6)	−0.0154 (6)
N2	0.0445 (7)	0.0297 (6)	0.0304 (7)	−0.0012 (5)	−0.0082 (5)	−0.0121 (5)
N3	0.0476 (7)	0.0256 (6)	0.0321 (7)	0.0034 (5)	−0.0009 (6)	−0.0127 (5)
N4	0.0456 (7)	0.0342 (7)	0.0297 (7)	0.0019 (6)	−0.0036 (6)	−0.0142 (6)
O1	0.0695 (8)	0.0366 (6)	0.0324 (6)	0.0056 (5)	0.0036 (6)	−0.0139 (5)
C21	0.0413 (8)	0.0334 (8)	0.0318 (8)	0.0001 (6)	−0.0045 (6)	−0.0122 (6)
C22	0.0437 (8)	0.0275 (7)	0.0341 (8)	−0.0004 (6)	−0.0097 (7)	−0.0078 (6)
C23	0.0430 (8)	0.0322 (8)	0.0405 (9)	0.0028 (6)	−0.0090 (7)	−0.0169 (7)
C24	0.0402 (8)	0.0380 (8)	0.0313 (8)	−0.0019 (6)	−0.0060 (6)	−0.0134 (7)
C25	0.0395 (8)	0.0335 (8)	0.0323 (8)	−0.0013 (6)	−0.0091 (6)	−0.0077 (6)
C26	0.0391 (8)	0.0296 (7)	0.0354 (8)	0.0020 (6)	−0.0074 (6)	−0.0114 (6)
N11	0.0612 (9)	0.0333 (7)	0.0423 (8)	0.0015 (7)	−0.0130 (7)	−0.0095 (6)
N12	0.0475 (8)	0.0534 (9)	0.0376 (8)	0.0016 (7)	−0.0078 (6)	−0.0203 (7)
N13	0.0508 (8)	0.0354 (7)	0.0445 (8)	0.0080 (6)	−0.0110 (7)	−0.0148 (6)
O11	0.0816 (9)	0.0462 (7)	0.0326 (6)	0.0143 (6)	−0.0118 (6)	−0.0172 (5)
O12	0.0899 (11)	0.0531 (9)	0.0639 (10)	−0.0085 (8)	0.0036 (8)	0.0066 (7)
O13	0.0858 (12)	0.0721 (11)	0.0801 (12)	0.0331 (9)	−0.0187 (9)	0.0045 (9)
O14	0.0723 (9)	0.0698 (9)	0.0320 (7)	0.0122 (7)	−0.0091 (6)	−0.0107 (6)
O15	0.0913 (11)	0.0638 (9)	0.0536 (8)	0.0143 (8)	−0.0067 (8)	−0.0359 (7)
O16	0.0835 (10)	0.0364 (7)	0.0571 (8)	0.0168 (6)	−0.0146 (7)	−0.0054 (6)
O17	0.0959 (11)	0.0658 (9)	0.0487 (8)	0.0363 (8)	−0.0084 (8)	−0.0262 (7)

Geometric parameters (Å, °)

C1—N1	1.335 (2)	C13—C15	1.486 (3)
C1—C2	1.370 (2)	C13—C14	1.490 (3)
C1—H1	0.9300	C13—H13	0.9800
C2—C3	1.379 (2)	C14—C15	1.493 (3)
C2—H2	0.9300	C14—H14A	0.9700
C3—C4	1.393 (2)	C14—H14B	0.9700
C3—H3	0.9300	C15—H15A	0.9700
C4—C5	1.399 (2)	C15—H15B	0.9700
C4—C6	1.4867 (19)	N3—H3N	0.881 (19)
C5—N1	1.3296 (18)	N4—H4N	0.878 (19)
C5—N2	1.4236 (19)	C21—O11	1.2480 (18)
C6—O1	1.2305 (17)	C21—C26	1.440 (2)
C6—N3	1.3559 (19)	C21—C22	1.454 (2)

C7—C11	1.399 (2)	C22—C23	1.360 (2)
C7—C8	1.4030 (19)	C22—N11	1.464 (2)
C7—N3	1.4097 (18)	C23—C24	1.392 (2)
C8—N4	1.3425 (19)	C23—H23	0.9300
C8—N2	1.3933 (18)	C24—C25	1.382 (2)
C9—N4	1.347 (2)	C24—N12	1.441 (2)
C9—C10	1.355 (2)	C25—C26	1.379 (2)
C9—H9	0.9300	C25—H25	0.9300
C10—C11	1.395 (2)	C26—N13	1.4557 (19)
C10—H10	0.9300	N11—O13	1.207 (2)
C11—C12	1.500 (2)	N11—O12	1.219 (2)
C12—H12A	0.9600	N12—O15	1.2294 (19)
C12—H12B	0.9600	N12—O14	1.2357 (19)
C12—H12C	0.9600	N13—O17	1.2211 (18)
C13—N2	1.4587 (19)	N13—O16	1.2278 (18)
N1—C1—C2	124.23 (14)	C15—C14—H14A	117.8
N1—C1—H1	117.9	C13—C14—H14B	117.8
C2—C1—H1	117.9	C15—C14—H14B	117.8
C1—C2—C3	117.77 (16)	H14A—C14—H14B	114.9
C1—C2—H2	121.1	C13—C15—C14	60.03 (13)
C3—C2—H2	121.1	C13—C15—H15A	117.8
C2—C3—C4	119.89 (15)	C14—C15—H15A	117.8
C2—C3—H3	120.1	C13—C15—H15B	117.8
C4—C3—H3	120.1	C14—C15—H15B	117.8
C3—C4—C5	117.31 (13)	H15A—C15—H15B	114.9
C3—C4—C6	116.49 (13)	C5—N1—C1	117.62 (14)
C5—C4—C6	125.80 (14)	C8—N2—C5	117.84 (12)
N1—C5—C4	123.08 (14)	C8—N2—C13	116.95 (12)
N1—C5—N2	114.28 (13)	C5—N2—C13	115.85 (12)
C4—C5—N2	122.58 (12)	C6—N3—C7	129.12 (13)
O1—C6—N3	120.04 (13)	C6—N3—H3N	113.9 (11)
O1—C6—C4	119.18 (13)	C7—N3—H3N	115.4 (11)
N3—C6—C4	120.77 (13)	C8—N4—C9	123.33 (13)
C11—C7—C8	119.18 (13)	C8—N4—H4N	122.4 (12)
C11—C7—N3	119.24 (12)	C9—N4—H4N	114.3 (12)
C8—C7—N3	121.48 (13)	O11—C21—C26	127.20 (14)
N4—C8—N2	118.15 (12)	O11—C21—C22	120.70 (15)
N4—C8—C7	118.47 (13)	C26—C21—C22	111.99 (13)
N2—C8—C7	123.36 (13)	C23—C22—C21	125.00 (14)
N4—C9—C10	119.90 (14)	C23—C22—N11	118.07 (14)
N4—C9—H9	120.1	C21—C22—N11	116.93 (13)
C10—C9—H9	120.1	C22—C23—C24	118.30 (14)
C9—C10—C11	120.04 (14)	C22—C23—H23	120.9
C9—C10—H10	120.0	C24—C23—H23	120.9
C11—C10—H10	120.0	C25—C24—C23	120.94 (14)
C10—C11—C7	119.08 (14)	C25—C24—N12	119.15 (15)
C10—C11—C12	119.49 (14)	C23—C24—N12	119.89 (14)
C7—C11—C12	121.41 (14)	C26—C25—C24	120.10 (15)
C11—C12—H12A	109.5	C26—C25—H25	120.0

C11—C12—H12B	109.5	C24—C25—H25	120.0
H12A—C12—H12B	109.5	C25—C26—C21	122.93 (14)
C11—C12—H12C	109.5	C25—C26—N13	116.69 (14)
H12A—C12—H12C	109.5	C21—C26—N13	120.37 (13)
H12B—C12—H12C	109.5	O13—N11—O12	123.37 (17)
N2—C13—C15	116.94 (15)	O13—N11—C22	118.84 (15)
N2—C13—C14	116.71 (15)	O12—N11—C22	117.77 (16)
C15—C13—C14	60.23 (14)	O15—N12—O14	122.93 (15)
N2—C13—H13	117.0	O15—N12—C24	118.83 (15)
C15—C13—H13	117.0	O14—N12—C24	118.24 (14)
C14—C13—H13	117.0	O17—N13—O16	122.36 (14)
C13—C14—C15	59.74 (14)	O17—N13—C26	119.31 (14)
C13—C14—H14A	117.8	O16—N13—C26	118.33 (14)
N1—C1—C2—C3	−2.5 (3)	C15—C13—N2—C5	−81.3 (2)
C1—C2—C3—C4	2.9 (2)	C14—C13—N2—C5	−149.74 (15)
C2—C3—C4—C5	−0.8 (2)	O1—C6—N3—C7	−165.46 (15)
C2—C3—C4—C6	−173.88 (14)	C4—C6—N3—C7	13.8 (2)
C3—C4—C5—N1	−2.2 (2)	C11—C7—N3—C6	138.44 (16)
C6—C4—C5—N1	170.24 (14)	C8—C7—N3—C6	−45.4 (2)
C3—C4—C5—N2	−179.24 (13)	N2—C8—N4—C9	−178.36 (15)
C6—C4—C5—N2	−6.8 (2)	C7—C8—N4—C9	0.2 (2)
C3—C4—C6—O1	22.0 (2)	C10—C9—N4—C8	−0.8 (3)
C5—C4—C6—O1	−150.49 (15)	O11—C21—C22—C23	166.67 (16)
C3—C4—C6—N3	−157.26 (14)	C26—C21—C22—C23	−9.7 (2)
C5—C4—C6—N3	30.3 (2)	O11—C21—C22—N11	−13.0 (2)
C11—C7—C8—N4	0.2 (2)	C26—C21—C22—N11	170.67 (13)
N3—C7—C8—N4	−175.92 (14)	C21—C22—C23—C24	4.5 (2)
C11—C7—C8—N2	178.69 (14)	N11—C22—C23—C24	−175.84 (14)
N3—C7—C8—N2	2.5 (2)	C22—C23—C24—C25	2.1 (2)
N4—C9—C10—C11	1.0 (3)	C22—C23—C24—N12	−179.67 (14)
C9—C10—C11—C7	−0.6 (3)	C23—C24—C25—C26	−2.4 (2)
C9—C10—C11—C12	177.43 (17)	N12—C24—C25—C26	179.29 (14)
C8—C7—C11—C10	0.0 (2)	C24—C25—C26—C21	−3.8 (2)
N3—C7—C11—C10	176.22 (15)	C24—C25—C26—N13	177.17 (13)
C8—C7—C11—C12	−178.01 (16)	O11—C21—C26—C25	−166.96 (16)
N3—C7—C11—C12	−1.8 (2)	C22—C21—C26—C25	9.1 (2)
N2—C13—C14—C15	107.25 (18)	O11—C21—C26—N13	12.1 (3)
N2—C13—C15—C14	−106.86 (18)	C22—C21—C26—N13	−171.83 (13)
C4—C5—N1—C1	2.7 (2)	C23—C22—N11—O13	−46.4 (2)
N2—C5—N1—C1	180.00 (14)	C21—C22—N11—O13	133.22 (18)
C2—C1—N1—C5	−0.3 (3)	C23—C22—N11—O12	131.86 (18)
N4—C8—N2—C5	−124.09 (15)	C21—C22—N11—O12	−48.5 (2)
C7—C8—N2—C5	57.4 (2)	C25—C24—N12—O15	177.36 (15)
N4—C8—N2—C13	21.2 (2)	C23—C24—N12—O15	−0.9 (2)
C7—C8—N2—C13	−157.30 (15)	C25—C24—N12—O14	−3.3 (2)
N1—C5—N2—C8	130.07 (13)	C23—C24—N12—O14	178.44 (15)
C4—C5—N2—C8	−52.61 (19)	C25—C26—N13—O17	−167.53 (16)
N1—C5—N2—C13	−15.56 (19)	C21—C26—N13—O17	13.4 (2)
C4—C5—N2—C13	161.75 (14)	C25—C26—N13—O16	11.8 (2)

C15—C13—N2—C8	132.73 (18)	C21—C26—N13—O16	−167.29 (15)
C14—C13—N2—C8	64.3 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···O1^{i}	0.881 (19)	2.063 (19)	2.9242 (17)	165.4 (16)
N4—H4N···O11	0.878 (19)	1.808 (19)	2.6656 (17)	164.9 (17)
C9—H9···O14^{ii}	0.93	2.46	3.386 (2)	174
C23—H23···O15^{iii}	0.93	2.49	3.335 (2)	151

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

Fig. 1

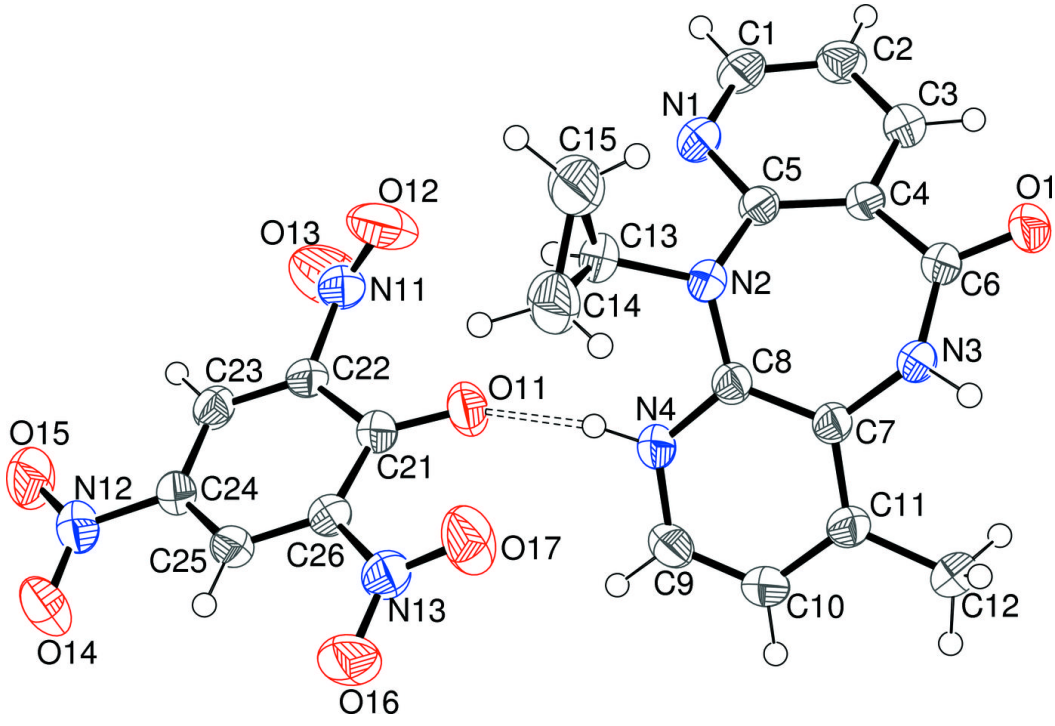
C1
C2
C3
N1
C15
C5
C4
O1
O12
O13
C13
N2
C6
N11
C14
C8
N3
C23
C22
O11
N4
C7
O15
C21
N12
C24
C11
C25
C26
O17
C9
C10
C12
N13
O14
O16

Fig. 2