

6-Nitro-2,3-dihydro-1*H*-pyrrolo[2,1-*c*]-[1,4]benzodiazepine-5,11(10*H*,11*aH*)-dione

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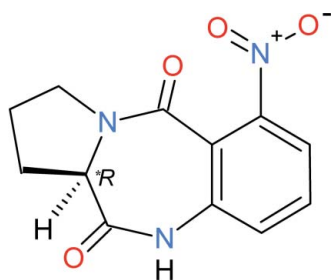
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Key indicators: single-crystal X-ray study; $T = 296$ K, $P = 0.0$ kPa; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 7.2.

In the two molecules of the asymmetric unit of the title compound, $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_4$, the seven-membered diazepine ring adopts a boat conformation (with the two phenylene C atoms representing the stern and the methine C atom the prow). The five-membered pyrrole ring, which has an envelope conformation, makes dihedral angles of 60.47 (10) and 54.69 (9)° with the benzene ring of the benzodiazepine unit in the two molecules. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ stacking interactions [centroid-centroid distance = 3.8023 (7)–3.8946 (7) Å] lead to the formation of a three-dimensional framework.

Related literature

For the biological activity of pyrrolo[2,1-*c*][1,4]benzodiazepine derivatives, see: Dervan (1986); Leimgruber *et al.* (1975); Da Settimo *et al.* (2007); Herpin *et al.* (2000); Arima *et al.* (1983).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_4$	$V = 1186.94$ (4) Å ³
$M_r = 261.24$	$Z = 4$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 10.7364$ (2) Å	$\mu = 0.11$ mm ⁻¹
$b = 6.8925$ (1) Å	$T = 296$ K
$c = 16.3901$ (3) Å	$0.23 \times 0.20 \times 0.15$ mm
$\beta = 101.870$ (1)°	

Data collection

Bruker APEXII CCD detector	2543 independent reflections
diffractometer	2452 reflections with $I > 2\sigma(I)$
14768 measured reflections	$R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.080$	$\Delta\rho_{\text{max}} = 0.17$ e Å ⁻³
$S = 1.04$	$\Delta\rho_{\text{min}} = -0.16$ e Å ⁻³
2543 reflections	
351 parameters	
1 restraint	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N10}-\text{H10N}\cdots\text{O11}^{\text{i}}$	0.85 (3)	1.98 (3)	2.821 (2)	169 (3)
$\text{N20}-\text{H20N}\cdots\text{O22}^{\text{ii}}$	0.84 (3)	2.15 (3)	2.980 (2)	169 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2102).

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supplementary materials

Acta Cryst. (2011). E67, o2003 [doi:10.1107/S1600536811024500]

6-Nitro-2,3-dihydro-1*H*-pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11(10*H*,11*aH*)-dione

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Comment

Benzodiazepines form a well known and widely applied class of biologically active compounds (Da Settimo *et al.*, 2007) and are representatives of the family of privileged structures (Herpin *et al.*, 2000). In the area of molecular recognition considerable efforts have been devoted to the synthesis of pyrrolo[2,1-*c*][1,4]benzodiazepines (PBDs) that can recognize and bind to specific sequences of DNA. They are potential regulators of gene expression with possible application as therapeutic agents in the treatment of genetic disorders including cancer. Furthermore, they can be used as affinity-cleavage reagents in molecular biology (Dervan, 1986). The PBD ring system is also found in natural antitumor antibiotics from *Streptomyces* species such as Anthramycin (Leimgruber *et al.*, 1975), Tomaymycine (Arima *et al.*, 1983).

The compound, C₂₄H₂₂N₆O₈, crystallizes with two reasonably similar molecules in the asymmetric unit (Fig. 1, r.m.s. deviation of 0.1051 Å for 19 non-H atoms fitted). The nitro and benzene systems are inclined at dihedral angles of 30.0 (3) and 41.0 (3)° in the first and second molecule of the asymmetric unit, respectively. The seven-membered diazepine ring adopts a boat conformation (with the two phenylene C atoms representing the stern and the methine C atom the prow). The five-membered pyrrolo ring, which has an envelope conformation, makes dihedral angles of 60.47 (10)° and 54.69 (9)° with the benzene ring of the benzodiazepine in the two independent molecules of the unit.

In the crystal structure, intermolecular N—H···O hydrogen bonds (Fig. 2, Table 1) and π – π stacking interactions (centroid-centroid distance = 3.8023 (7) Å to 3.8946 (7) Å) lead to the formation of a three-dimensional framework.

Experimental

5-Nitro-1*H*-benzo[*d*][1,3]oxazine-2,4-dione (0.5 g, 2.5 mmol) and *L*-proline (0.29 g, 2.5 mmol) were dissolved in DMF (10 ml) and were then heated under reflux for 3 h. After cooling, the solvent was removed under reduced pressure to yield an oily residue; the residue was then purified over silica gel column chromatography using a mixture of hexane and ethyl acetate (3:1) as eluent. Under these conditions the compound was obtained as colourless crystals.

Refinement

The H atoms bound to C were treated as riding with their parent atoms [C—H distances are 0.93 Å for CH groups with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$, and 0.97 Å for CH₃ groups with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$]. The nitrogen-bound H atoms were located in a difference Fourier map, and were refined with distance restraints of N—H 0.88±0.01. 2133 Friedel pairs were merged.

Figures

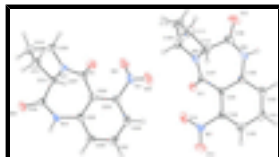


Fig. 1. Molecular view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

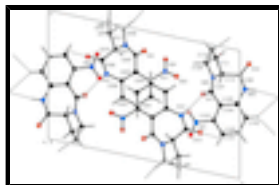


Fig. 2. Partial packing view showing the chain formed by N—H...O hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

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Crystal data

$C_{12}H_{11}N_3O_4$

$M_r = 261.24$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 10.7364$ (2) Å

$b = 6.8925$ (1) Å

$c = 16.3901$ (3) Å

$\beta = 101.870$ (1)°

$V = 1186.94$ (4) Å³

$Z = 4$

$F(000) = 544$

$D_x = 1.462$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 246 reflections

$\theta = 2.4$ – 26.3 °

$\mu = 0.11$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.23 \times 0.20 \times 0.15$ mm

Data collection

Bruker APEXII CCD detector
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω and ϕ scans

14768 measured reflections

2543 independent reflections

2452 reflections with $I > 2\sigma(I)$

$R_{int} = 0.021$

$\theta_{max} = 26.0$ °, $\theta_{min} = 2.5$ °

$h = -13$ → 13

$k = -8$ → 8

$l = -20$ → 20

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.080$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1616P]$
2543 reflections	where $P = (F_o^2 + 2F_c^2)/3$
351 parameters	$(\Delta/\sigma)_{\max} < 0.001$
1 restraint	$\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

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. 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C100	0.4871 (2)	0.4617 (3)	1.03240 (11)	0.0410 (4)
C101	0.3608 (2)	0.4649 (3)	0.99613 (13)	0.0445 (5)
C102	0.32175 (19)	0.4505 (3)	0.91009 (14)	0.0442 (5)
C103	0.41314 (17)	0.4215 (3)	0.86410 (11)	0.0359 (4)
C104	0.54297 (16)	0.4047 (3)	0.89854 (10)	0.0311 (4)
C105	0.58012 (18)	0.4374 (3)	0.98486 (10)	0.0336 (4)
C106	0.8095 (2)	0.5177 (3)	1.00041 (13)	0.0483 (5)
C107	0.63255 (16)	0.3127 (3)	0.85085 (10)	0.0316 (4)
C109	0.84963 (18)	0.3029 (4)	0.82441 (13)	0.0479 (5)
C110	0.9600 (2)	0.4396 (6)	0.8535 (2)	0.0749 (9)
C111	0.9040 (3)	0.6286 (5)	0.8754 (2)	0.0752 (9)
C112	0.7860 (2)	0.5679 (4)	0.90711 (14)	0.0485 (5)
C200	0.44163 (18)	0.4931 (3)	0.38760 (12)	0.0399 (4)
C201	0.40473 (19)	0.4968 (3)	0.46329 (13)	0.0440 (5)
C202	0.4948 (2)	0.5030 (3)	0.53689 (12)	0.0430 (4)
C203	0.62060 (19)	0.5134 (3)	0.53182 (11)	0.0374 (4)
C204	0.66333 (16)	0.5194 (3)	0.45691 (10)	0.0318 (4)
C205	0.57090 (16)	0.4986 (3)	0.38404 (10)	0.0318 (4)
C206	0.70022 (18)	0.4042 (3)	0.28050 (11)	0.0378 (4)
C207	0.79608 (17)	0.5876 (3)	0.45641 (11)	0.0360 (4)
C209	0.98134 (19)	0.5664 (4)	0.39103 (15)	0.0522 (6)
C210	1.0011 (2)	0.4484 (5)	0.31649 (15)	0.0655 (8)
C211	0.9243 (2)	0.2650 (4)	0.32000 (15)	0.0559 (6)
C212	0.80720 (17)	0.3350 (3)	0.35062 (11)	0.0369 (4)
H100	0.5118	0.4760	1.0899	0.049*
H101	0.3004	0.4767	1.0292	0.053*
H102	0.2362	0.4603	0.8845	0.053*

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H10A	0.8258	0.2995	0.7640	0.058*
H10B	0.8700	0.1724	0.8451	0.058*
H10N	0.539 (3)	0.528 (5)	0.2657 (18)	0.056 (8)*
H112	0.7192	0.6665	0.8932	0.058*
H11A	0.8815	0.7115	0.8267	0.090*
H11B	0.9636	0.6972	0.9182	0.090*
H11C	1.0174	0.3867	0.9019	0.090*
H11D	1.0070	0.4595	0.8096	0.090*
H200	0.3803	0.4870	0.3385	0.048*
H201	0.3187	0.4950	0.4649	0.053*
H202	0.4709	0.5002	0.5883	0.052*
H20A	1.0469	0.5388	0.4399	0.063*
H20B	0.9812	0.7043	0.3793	0.063*
H20N	0.716 (2)	0.416 (5)	1.0780 (17)	0.058 (7)*
H212	0.7765	0.2341	0.3837	0.044*
H21A	0.9722	0.1718	0.3584	0.067*
H21B	0.9005	0.2058	0.2653	0.067*
H21C	0.9703	0.5183	0.2649	0.079*
H21D	1.0905	0.4183	0.3208	0.079*
N10	0.60011 (15)	0.4905 (3)	0.30409 (9)	0.0369 (4)
N11	0.71691 (17)	0.5079 (3)	0.61116 (9)	0.0464 (4)
N12	0.85585 (14)	0.5013 (3)	0.40290 (9)	0.0371 (4)
N20	0.70780 (16)	0.4399 (3)	1.02707 (10)	0.0413 (4)
N21	0.36959 (16)	0.4213 (3)	0.77185 (11)	0.0479 (4)
N22	0.74763 (15)	0.3889 (3)	0.86106 (10)	0.0380 (4)
O11	0.60185 (13)	0.1634 (2)	0.81012 (8)	0.0435 (4)
O12	0.91105 (16)	0.5401 (4)	1.04775 (12)	0.0739 (6)
O13	0.26234 (16)	0.3669 (4)	0.74408 (12)	0.0761 (6)
O14	0.44340 (18)	0.4853 (4)	0.73230 (10)	0.0743 (6)
O21	0.84147 (15)	0.7225 (3)	0.50119 (10)	0.0571 (4)
O22	0.70331 (15)	0.3858 (3)	0.20681 (8)	0.0581 (5)
O23	0.6951 (2)	0.5986 (4)	0.66967 (9)	0.0723 (6)
O24	0.80876 (17)	0.4033 (4)	0.61259 (10)	0.0725 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C100	0.0594 (12)	0.0344 (10)	0.0313 (8)	0.0026 (9)	0.0144 (8)	-0.0011 (8)
C101	0.0534 (12)	0.0395 (11)	0.0477 (11)	0.0024 (9)	0.0268 (9)	-0.0014 (9)
C102	0.0350 (9)	0.0462 (12)	0.0522 (11)	0.0019 (9)	0.0107 (8)	-0.0011 (10)
C103	0.0352 (9)	0.0384 (10)	0.0328 (8)	0.0023 (8)	0.0039 (7)	0.0010 (8)
C104	0.0335 (8)	0.0321 (9)	0.0275 (7)	-0.0022 (7)	0.0057 (6)	0.0010 (7)
C105	0.0420 (9)	0.0288 (9)	0.0290 (8)	0.0006 (8)	0.0054 (7)	-0.0002 (7)
C106	0.0483 (11)	0.0426 (11)	0.0478 (11)	-0.0081 (10)	-0.0043 (9)	-0.0095 (10)
C107	0.0311 (8)	0.0397 (10)	0.0225 (7)	-0.0023 (8)	0.0021 (6)	0.0011 (7)
C109	0.0339 (10)	0.0676 (15)	0.0441 (10)	0.0059 (10)	0.0124 (8)	0.0114 (11)
C110	0.0398 (12)	0.097 (2)	0.0907 (19)	-0.0091 (14)	0.0187 (12)	0.021 (2)
C111	0.0662 (17)	0.079 (2)	0.0801 (18)	-0.0344 (16)	0.0148 (14)	0.0114 (17)

C112	0.0483 (11)	0.0458 (12)	0.0499 (11)	-0.0153 (10)	0.0064 (9)	0.0005 (10)
C200	0.0364 (9)	0.0400 (10)	0.0417 (9)	-0.0023 (9)	0.0046 (7)	0.0022 (9)
C201	0.0381 (10)	0.0452 (11)	0.0519 (11)	-0.0033 (9)	0.0163 (8)	0.0039 (10)
C202	0.0537 (11)	0.0398 (10)	0.0407 (9)	0.0029 (10)	0.0217 (8)	0.0050 (9)
C203	0.0479 (10)	0.0353 (9)	0.0289 (8)	0.0061 (9)	0.0079 (7)	0.0014 (8)
C204	0.0358 (9)	0.0313 (9)	0.0282 (8)	0.0027 (7)	0.0059 (7)	0.0000 (7)
C205	0.0359 (9)	0.0294 (8)	0.0299 (8)	0.0005 (8)	0.0065 (7)	0.0018 (7)
C206	0.0412 (9)	0.0434 (10)	0.0273 (8)	-0.0008 (9)	0.0038 (7)	-0.0013 (8)
C207	0.0346 (9)	0.0420 (10)	0.0292 (8)	0.0010 (8)	0.0015 (7)	-0.0022 (8)
C209	0.0338 (10)	0.0653 (15)	0.0589 (12)	-0.0050 (10)	0.0124 (9)	-0.0021 (12)
C210	0.0449 (11)	0.100 (2)	0.0558 (13)	0.0031 (14)	0.0205 (10)	-0.0026 (15)
C211	0.0461 (12)	0.0721 (17)	0.0486 (11)	0.0161 (12)	0.0078 (9)	-0.0136 (12)
C212	0.0393 (10)	0.0408 (10)	0.0298 (8)	0.0036 (8)	0.0047 (7)	-0.0026 (8)
N10	0.0374 (8)	0.0469 (9)	0.0236 (7)	0.0050 (8)	-0.0001 (6)	0.0032 (7)
N11	0.0565 (10)	0.0552 (11)	0.0274 (7)	0.0124 (10)	0.0083 (7)	0.0032 (8)
N12	0.0316 (7)	0.0454 (9)	0.0339 (7)	-0.0014 (7)	0.0054 (6)	-0.0026 (8)
N20	0.0489 (9)	0.0438 (10)	0.0264 (7)	-0.0011 (8)	-0.0033 (6)	-0.0014 (7)
N21	0.0385 (9)	0.0599 (11)	0.0410 (8)	0.0143 (9)	-0.0017 (7)	-0.0011 (9)
N22	0.0335 (7)	0.0454 (9)	0.0350 (7)	-0.0040 (7)	0.0070 (6)	-0.0002 (7)
O11	0.0392 (7)	0.0551 (9)	0.0365 (7)	-0.0099 (7)	0.0085 (5)	-0.0170 (7)
O12	0.0528 (9)	0.0925 (16)	0.0643 (10)	-0.0183 (10)	-0.0158 (8)	-0.0133 (11)
O13	0.0474 (9)	0.1032 (18)	0.0660 (11)	0.0102 (11)	-0.0155 (8)	-0.0227 (12)
O14	0.0711 (11)	0.1173 (18)	0.0343 (7)	0.0125 (13)	0.0102 (7)	0.0148 (11)
O21	0.0456 (8)	0.0651 (11)	0.0592 (9)	-0.0130 (8)	0.0075 (7)	-0.0283 (9)
O22	0.0600 (9)	0.0874 (13)	0.0262 (6)	0.0125 (10)	0.0070 (6)	-0.0042 (8)
O23	0.0948 (13)	0.0859 (14)	0.0326 (7)	0.0271 (12)	0.0053 (8)	-0.0093 (9)
O24	0.0681 (10)	0.1011 (16)	0.0439 (8)	0.0377 (12)	0.0012 (7)	0.0013 (10)

Geometric parameters (Å, °)

C100—H100	0.9300	C203—N11	1.486 (2)
C100—C101	1.365 (3)	C203—C202	1.372 (3)
C101—H101	0.9300	C204—C207	1.502 (3)
C101—C102	1.390 (3)	C204—C203	1.397 (2)
C102—H102	0.9300	C204—C205	1.394 (2)
C103—N21	1.488 (2)	C205—N10	1.410 (2)
C103—C102	1.369 (3)	C205—C200	1.402 (3)
C104—C107	1.499 (2)	C206—N10	1.352 (3)
C104—C105	1.407 (2)	C206—O22	1.222 (2)
C104—C103	1.397 (2)	C207—O21	1.222 (3)
C105—N20	1.403 (2)	C209—H20B	0.9700
C105—C100	1.397 (3)	C209—H20A	0.9700
C106—N20	1.366 (3)	C209—C210	1.519 (4)
C106—O12	1.211 (3)	C210—H21D	0.9700
C107—N22	1.321 (2)	C210—H21C	0.9700
C107—O11	1.234 (2)	C211—H21B	0.9700
C109—H10B	0.9700	C211—H21A	0.9700
C109—H10A	0.9700	C211—C210	1.516 (5)
C109—C110	1.512 (4)	C212—H212	0.9800

supplementary materials

C110—H11D	0.9700	C212—C211	1.525 (3)
C110—H11C	0.9700	C212—C206	1.525 (3)
C111—H11B	0.9700	N10—H10N	0.85 (3)
C111—H11A	0.9700	N11—O24	1.218 (3)
C111—C110	1.508 (5)	N11—O23	1.207 (2)
C112—H112	0.9800	N12—C209	1.470 (2)
C112—C106	1.537 (3)	N12—C212	1.462 (3)
C112—C111	1.524 (3)	N12—C207	1.329 (3)
C200—H200	0.9300	N20—H20N	0.84 (3)
C200—C201	1.378 (3)	N21—O13	1.208 (3)
C201—H201	0.9300	N21—O14	1.206 (3)
C202—H202	0.9300	N22—C109	1.477 (3)
C202—C201	1.383 (3)	N22—C112	1.461 (3)
C105—C100—H100	119.4	C204—C203—N11	118.34 (17)
C101—C100—H100	119.4	C202—C203—N11	117.53 (17)
C101—C100—C105	121.28 (18)	C202—C203—C204	124.06 (18)
C102—C101—H101	119.8	C203—C204—C207	119.82 (16)
C100—C101—H101	119.8	C205—C204—C207	122.44 (15)
C100—C101—C102	120.41 (17)	C205—C204—C203	116.44 (16)
C101—C102—H102	121.0	C200—C205—N10	116.68 (16)
C103—C102—H102	121.0	C204—C205—N10	123.03 (15)
C103—C102—C101	117.92 (18)	C204—C205—C200	120.24 (16)
C104—C103—N21	119.29 (16)	N10—C206—C212	116.22 (15)
C102—C103—N21	116.65 (17)	O22—C206—C212	122.90 (18)
C102—C103—C104	123.93 (17)	O22—C206—N10	120.88 (18)
C105—C104—C107	120.83 (16)	N12—C207—C204	116.80 (17)
C103—C104—C107	121.21 (15)	O21—C207—C204	119.76 (17)
C103—C104—C105	116.48 (16)	O21—C207—N12	123.36 (18)
N20—C105—C104	122.95 (17)	H20A—C209—H20B	109.1
C100—C105—C104	119.53 (18)	C210—C209—H20B	111.1
C100—C105—N20	117.48 (16)	N12—C209—H20B	111.1
N20—C106—C112	115.31 (17)	C210—C209—H20A	111.1
O12—C106—C112	123.4 (2)	N12—C209—H20A	111.1
O12—C106—N20	121.2 (2)	N12—C209—C210	103.11 (19)
N22—C107—C104	116.70 (17)	H21C—C210—H21D	108.9
O11—C107—C104	120.41 (16)	C209—C210—H21D	110.9
O11—C107—N22	122.60 (18)	C211—C210—H21D	110.9
H10A—C109—H10B	109.1	C209—C210—H21C	110.9
C110—C109—H10B	111.2	C211—C210—H21C	110.9
N22—C109—H10B	111.2	C211—C210—C209	104.39 (19)
C110—C109—H10A	111.2	H21A—C211—H21B	109.0
N22—C109—H10A	111.2	C212—C211—H21B	111.0
N22—C109—C110	102.7 (2)	C210—C211—H21B	111.0
H11C—C110—H11D	108.6	C212—C211—H21A	111.0
C109—C110—H11D	110.4	C210—C211—H21A	111.0
C111—C110—H11D	110.4	C210—C211—C212	103.9 (2)
C109—C110—H11C	110.4	C211—C212—H212	110.6
C111—C110—H11C	110.4	C206—C212—H212	110.6
C111—C110—C109	106.8 (2)	N12—C212—H212	110.6

H11A—C111—H11B	109.0	C206—C212—C211	113.29 (16)
C112—C111—H11B	110.9	N12—C212—C211	102.79 (17)
C110—C111—H11B	110.9	N12—C212—C206	108.64 (17)
C112—C111—H11A	110.9	C205—N10—H10N	113.2 (18)
C110—C111—H11A	110.9	C206—N10—H10N	117.2 (18)
C110—C111—C112	104.1 (2)	C206—N10—C205	128.68 (16)
C106—C112—H112	110.5	O24—N11—C203	116.55 (17)
C111—C112—H112	110.5	O23—N11—C203	117.90 (18)
N22—C112—H112	110.5	O23—N11—O24	125.44 (18)
C111—C112—C106	115.0 (2)	C212—N12—C209	112.85 (16)
N22—C112—C106	107.16 (18)	C207—N12—C209	122.05 (19)
N22—C112—C111	102.8 (2)	C207—N12—C212	125.10 (16)
C205—C200—H200	119.8	C105—N20—H20N	112.6 (18)
C201—C200—H200	119.8	C106—N20—H20N	117.8 (19)
C201—C200—C205	120.49 (18)	C106—N20—C105	127.69 (17)
C202—C201—H201	119.8	O13—N21—C103	117.34 (19)
C200—C201—H201	119.8	O14—N21—C103	116.24 (18)
C200—C201—C202	120.46 (18)	O14—N21—O13	126.3 (2)
C201—C202—H202	121.0	C112—N22—C109	113.29 (17)
C203—C202—H202	121.0	C107—N22—C109	122.77 (19)
C203—C202—C201	118.00 (18)	C107—N22—C112	123.91 (18)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N10—H10N \cdots O11 ⁱ	0.85 (3)	1.98 (3)	2.821 (2)	169 (3)
N20—H20N \cdots O22 ⁱⁱ	0.84 (3)	2.15 (3)	2.980 (2)	169 (2)

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

Fig. 1

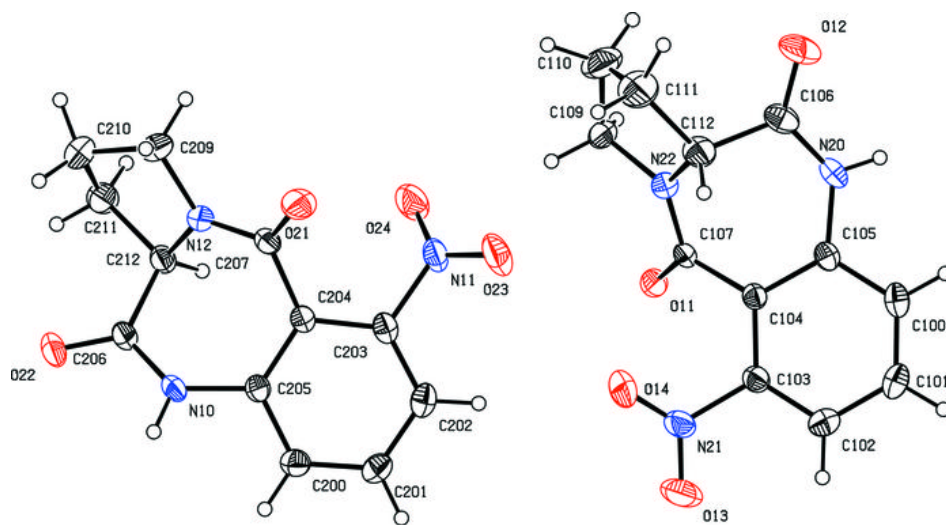


Fig. 2

