Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

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

**Abdessamad Jebani,[a] Hafid Zouihri,[b] Ahmed El Hakmaoui,[a]* Saïd Lazar[c] and Mohamed Akssira[a]**

[a]Laboratoire de Chimie Biorganique & Analytique, URAC 22, Université Hassan II, Mohammedia-Casablanca, BP 146, 20800 Mohammedia, Morocco, [b]Laboratoires de Diffraction des Rayons X, Centre National pour la Recherche Scientifique et Technique, Rabat, Morocco, and [c]Laboratoire de Biochimie, Environnement & Agroalimentaire, URAC 36, Université Hassan II, Mohammedia-Casablanca, BP 146, 20800 Mohammedia, Morocco
Correspondence e-mail: a_elhakmaoui@yahoo.fr

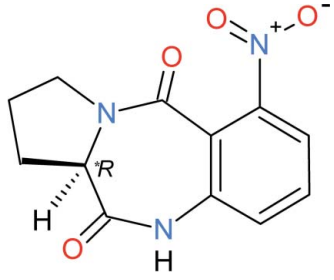
Received 10 June 2011; accepted 22 June 2011

Key indicators: single-crystal X-ray study; $T$ = 296 K, $P$ = 0.0 kPa; mean $\sigma$(C–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, $C_{12}H_{11}N_3O_4$, 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 N—H···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]benzodiaze-][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

$C_{12}H_{11}N_3O_4$
$M_r$ = 261.24
Monoclinic, $P2_1$
$a$ = 10.7364 (2) Å
$b$ = 6.8925 (1) Å
$c$ = 16.3901 (3) Å
$\beta$ = 101.870 (1)°
$V$ = 1186.94 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 296 K
0.23 × 0.20 × 0.15 mm

### Data collection

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

### Refinement

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N10—H10*N*···O11$^{i}$ | 0.85 (3) | 1.98 (3) | 2.821 (2) | 169 (3) |
| N20—H20*N*···O22$^{ii}$ | 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: *publCIF* (Westrip, 2010).

The authors thank the Unit of Support for Technical and Scientiﬁc Research (UATRS, CNRST) for the X-ray measurements.

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

## References

Arima, K., Kohsaka, M., Tamura, J., Imanaka, H. & Sakai, H. (1983). *J. Antibiot.* **25**, 437–444.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Da Settimo, F., Taliani, S., Trincavelli, M. L., Montali, M. & Martini, C. (2007). Curr. Med. Chem. 14, 2680–???.
Dervan, P. B. (1986). *Science* **232**, 464–471.
Herpin, T. F., Van Kirk, K. G., Salvino, J. M., Yu, S. T. & Labaudiniere, R. F. (2000). *J. Comb. Chem.* 2, 513–521.
Leimgruber, W., Stefanovic, V., Schenker, F., Karr, A. & Berger, J. (1975). *J. Am. Chem. Soc.* **87**, 5791–5793.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# supporting information

*Acta Cryst.* (2011). E**67**, o2003 [doi:10.1107/S1600536811024500]

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

**Abdessamad Jebani, Hafid Zouihri, Ahmed El Hakmaoui, Saïd Lazar and Mohamed Akssira**

## S1. 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, $C_{24}H_{22}N_6O_8$, 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 asymetric 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 $\pi$–$\pi$ stacking interactions (centroid-centroid distance = 3.8023 (7) Å to 3.8946 (7) Å) lead to the formation of a three-dimensional framework.

## S2. 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.

## S3. Refinement

The H atoms bound to C were treated as riding with their parent atoms [C—H distances are 0.93Å for CH groups with $U_{iso}$(H) = 1.2 $U_{eq}$(C), and 0.97 Å for CH3 groups with $U_{iso}$(H) = 1.5 $U_{eq}$(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.

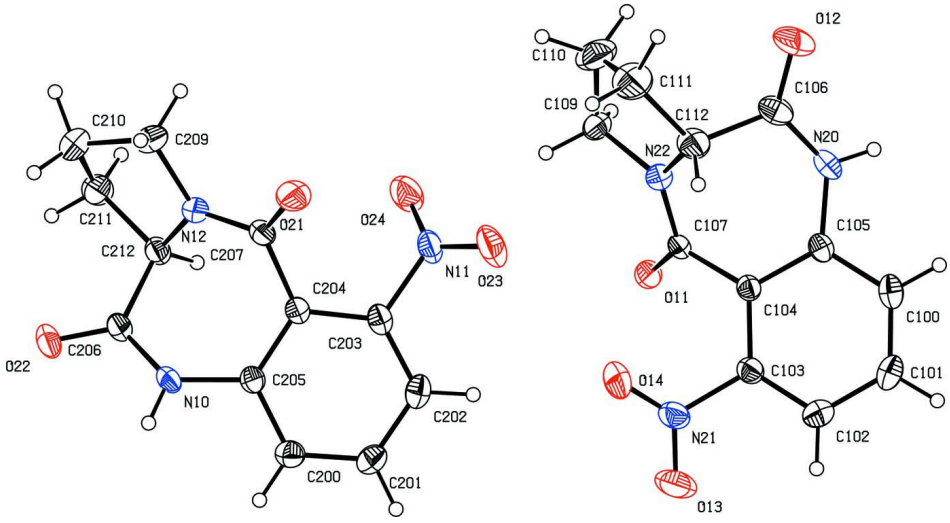



**Figure 1**

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.

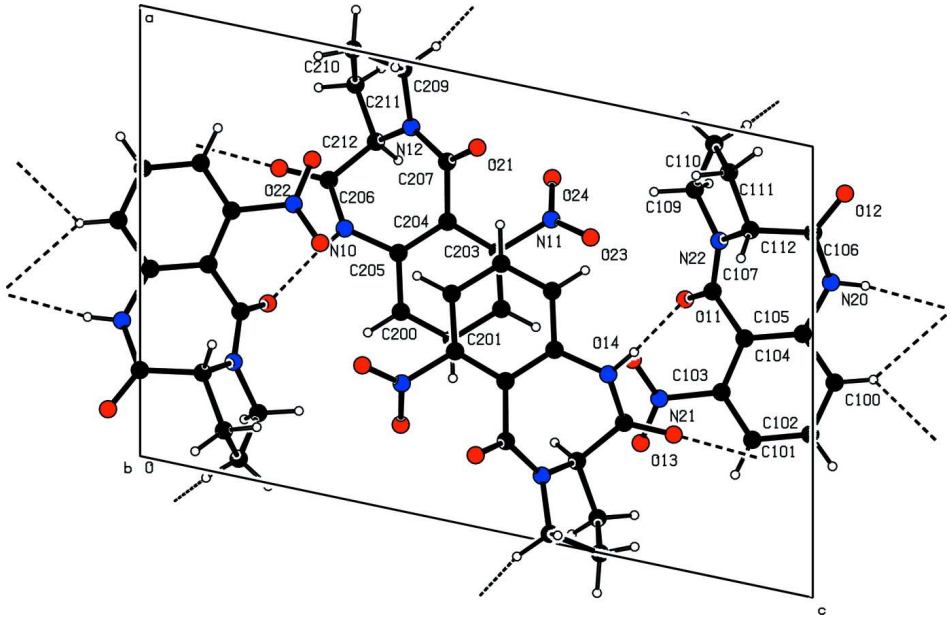



**Figure 2**

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.

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

*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) Å$^3$
$Z = 4$

$F(000) = 544$
$D_x = 1.462$ 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 × 0.20 × 0.15 mm

*Data collection*

Bruker APEXII CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
14768 measured reflections
2543 independent reflections

2452 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.04$
2543 reflections
351 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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* |
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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 |
| 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 (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N10—H10*N*···O11$^{i}$ | 0.85 (3) | 1.98 (3) | 2.821 (2) | 169 (3) |
| N20—H20*N*···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.