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# Crystal structure of (*S*)-4-carbamoyl-4-(1,3-dioxoisoindolin-2-yl)butanoic acid

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Trivial name (*S*)-phthaloylisoglutamine, one of the hydrolysis products of thalidomide.

**Keywords**: crystal structure; thalidomide; hydrogen bonds

**CCDC reference**: 1038803
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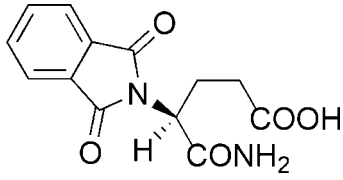
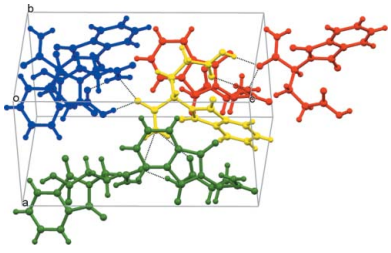
In the title compound, $C_{13}H_{12}N_2O_5$, the phthalimide ring system is essentially planar, with a maximum deviation of 0.0479 (14) Å. In the crystal, each molecule is linked *via* six neighbouring molecules into a three-dimensional network through N—H···O and O—H···O hydrogen bonds, which form an $R_3^2(8)$ ring motif.

## 1. Chemical context

The title compound, 5-(aminoxy)-4-(3-oxo-2*H*-isoindol-2-oyl)valeric acid (phthaloylisoglutamine), is one of the first-step hydrolysis products of thalidomide. Thalidomide was first synthesized in 1953 and was marketed as a hypnotic medicine in 1956. After that, the teratogenic side effect of thalidomide was proved and caused serious drug disaster (Lenz, 1961). Blashke *et al.* (1979) reported that only (*S*)-thalidomide exhibits teratogenicity while (*R*)-thalidomide exhibits sedative effects. In other words, the hypnotic and teratogenic mechanisms of thalidomide are different. For a long time, the target protein of thalidomide has not been clarified. However in 2010, the protein cereblon, which is one of the E3 ubiquitin ligase proteins, was identified as the primary target of thalidomide teratogenicity (Ito *et al.*, 2010). Furthermore, the conformation of a Cereblon and thalidomide complex has been reported (Fischer *et al.*, 2014).



Hydrolysis compounds of thalidomide are generated rapidly *in vivo* (Schumacher *et al.*, 1965; Nishimura *et al.*, 1994) and some of these showed TNF-α production-inhibitory activity (Nakamura *et al.*, 2007). Although the crystal structures of racemic and enantiomeric thalidomide were solved and reported earlier (Allen & Trotter, 1971; Suzuki *et al.*, 2010), the crystal structures of hydrolysis compounds of thalidomide have not been reported. Considering that knowing the structure of the molecule enables us to calculate the affinity between ligand and receptor using computer simulation, our report herein will be helpful in clarifying the differences between the biological effects of thalidomide and phthaloylisoglutamine.

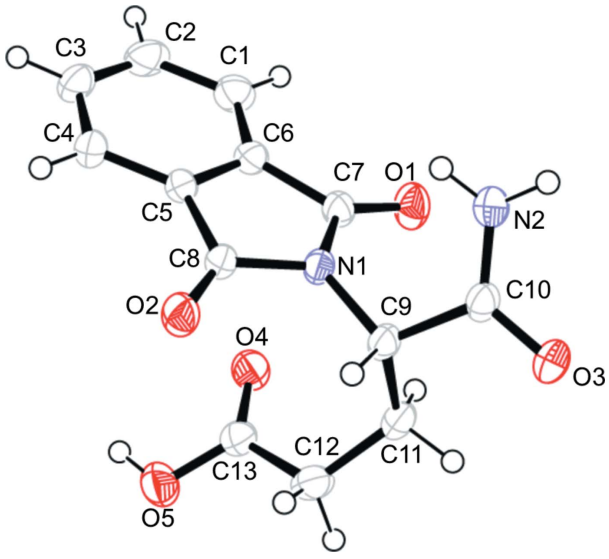



**Figure 1**
The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level.

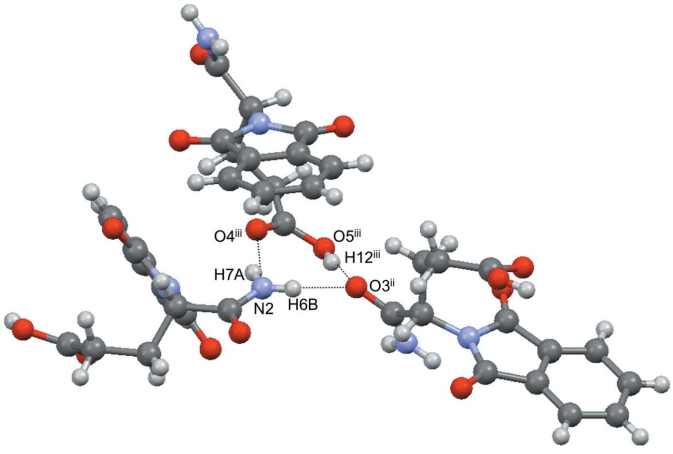



**Figure 2**
A trimer structure of the title compound and an $R_3^2(8)$ ring motif formed through O5$^{iii}$—H12$^{iii}$···O3$^{ii}$, N2—H6$B$···O3$^{ii}$ and N2—H7$A$···O4$^{iii}$ hydrogen bonds. [Symmetry codes: (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.]

## 2. Structural commentary

The molecular structure of the title molecule is shown in Fig. 1. The asymmetric center is *S* for atom C9. The phthalimide ring (N1/C1–C8) is essentially planar, with a maximum deviation of 0.0479 (14) Å for N1. The carbon–oxygen distances in the carboxy group (COOH) show different lengths [C13—O4 = 1.206 (2) and C13—O5 = 1.316 (2) Å]. This difference indicates that the C—O bonds in the carboxy group are non-delocalized. These bonds are slightly strengthened by intermolecular O5—H12···O3 and O4···H7$A$—N2 hydrogen bonding (Fig. 2). The conformation of C9—C11—C12—C13 chain is slightly twisted *gauche* [torsion angle = 77.4 (2)°].

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H12···O3$^{i}$ | 0.83 | 1.80 | 2.6230 (19) | 172 |
| N2—H6$B$···O3$^{ii}$ | 0.87 | 2.32 | 2.891 (2) | 123 |
| N2—H7$A$···O4$^{iii}$ | 0.87 | 2.05 | 2.886 (2) | 161 |

Symmetry codes: (i) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

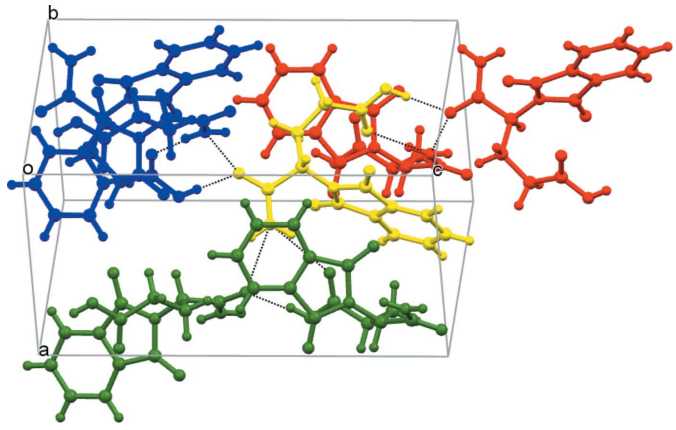



**Figure 3**
A crystal packing view of the title compound, showing the intermolecular hydrogen bonds. A yellow molecule is linked with two red, two green and two blue molecules.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{13}H_{12}N_2O_5$ |
| $M_r$ | 276.25 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 223 |
| $a, b, c$ (Å) | 8.4790 (3), 9.6751 (3), 15.4488 (5) |
| $V$ (Å$^3$) | 1267.35 (7) |
| $Z$ | 4 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.96 |
| Crystal size (mm) | 0.63 × 0.20 × 0.10 |
| | |
| Data collection | |
| Diffractometer | Rigaku R-AXIS RAPID |
| Absorption correction | Multi-scan (*ABSCOR*; Rigaku, 1995) |
| $T_{min}$, $T_{max}$ | 0.766, 0.908 |
| No. of measured, independent and observed [$F^2 > 2\sigma(F^2)$] reflections | 23228, 2320, 2245 |
| $R_{int}$ | 0.058 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.602 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.027, 0.067, 1.08 |
| No. of reflections | 2320 |
| No. of parameters | 183 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.16, −0.16 |
| Absolute structure | Flack $x$ determined using 914 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | 0.06 (4) |

Computer programs: *RAPID-AUTO* (Rigaku, 1998), *SHELXS2013* and *SHELXL2013* (Sheldrick, 2008) and *CrystalStructure* (Rigaku, 2014).

## 3. Supramolecular features

In the crystal structure, each molecule has six hydrogen bonds, which are divided into three types (Table 1). The three hydrogen bonds form a hydrogen-bonded ring with an $R_3^2(8)$ ring motif, which unites three molecules (Fig. 2). Taken together as shown in Fig. 3, one molecule (yellow) is linked to another six molecules (blue, red, and green) by three sets of circular hydrogen bonds.

## 4. Database survey

A search of the Cambridge Structural Database (Version 5.35 update in 2014; Groom & Allen, 2014) for the structure of thalidomide gave 11 hits, but there was no hydrolysis compound of thalidomide.

## 5. Synthesis and crystallization

The title compound was purchased from WuXi AppTec. The title compound (2 mg) was dissolved in ethanol (500 µl). After a few days of slow evaporation at 278 K, colourless single crystals suitable for X-ray diffraction were obtained.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were included in calculated positions [C—H (aromatic) = 0.93, C—H (methine) = 0.98, C—H (methylene) = 0.97, N—H = 0.87 and O—H = 0.82 Å] and treated as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(N, O)$.

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# supporting information

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## Crystal structure of (*S*)-4-carbamoyl-4-(1,3-dioxoisoindolin-2-yl)butanoic acid

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### Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *RAPID-AUTO* (Rigaku, 1998); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2014); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2014).

### (*S*)-4-Carbamoyl-4-(1,3-dioxoisoindolin-2-yl)butanoic acid

*Crystal data*

$C_{13}H_{12}N_2O_5$
$M_r$ = 276.25
Orthorhombic, $P2_12_12_1$
$a$ = 8.4790 (3) Å
$b$ = 9.6751 (3) Å
$c$ = 15.4488 (5) Å
$V$ = 1267.35 (7) Å$^3$
$Z$ = 4
$F(000)$ = 576.00
$D_x$ = 1.448 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 12101 reflections
$\theta$ = 4.6–68.3°
$\mu$ = 0.96 mm$^{-1}$
$T$ = 223 K
Needle, colorless
0.63 × 0.20 × 0.10 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
$T_{min}$ = 0.766, $T_{max}$ = 0.908
23228 measured reflections
2320 independent reflections
2245 reflections with $F^2 > 2\sigma(F^2)$
$R_{int}$ = 0.058
$\theta_{max}$ = 68.2°, $\theta_{min}$ = 5.4°
$h$ = −10→10
$k$ = −11→11
$l$ = −18→18

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.067
$S$ = 1.08
2320 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.1235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$
Extinction correction: SHELXL2013 (Sheldrick, 2008)
Extinction coefficient: 0.0357 (16)
Absolute structure: Flack $x$ determined using 914 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.06 (4)

*Special details*

**Refinement**. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on $F^2$. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.60120 (16) | 0.50832 (14) | 0.65955 (9) | 0.0380 (4) |
| O2 | 0.22423 (15) | 0.29086 (15) | 0.81979 (8) | 0.0354 (3) |
| O3 | 0.28367 (17) | 0.34228 (17) | 0.49573 (8) | 0.0435 (4) |
| O4 | 0.32128 (15) | 0.66161 (14) | 0.78685 (8) | 0.0353 (3) |
| O5 | 0.08071 (16) | 0.6519 (2) | 0.84322 (9) | 0.0498 (4) |
| N1 | 0.39627 (16) | 0.38300 (15) | 0.71946 (9) | 0.0246 (3) |
| N2 | 0.48080 (19) | 0.24502 (17) | 0.57047 (10) | 0.0342 (4) |
| C1 | 0.7414 (2) | 0.4824 (2) | 0.84759 (13) | 0.0358 (4) |
| C2 | 0.7647 (2) | 0.4539 (2) | 0.93501 (13) | 0.0416 (5) |
| C3 | 0.6535 (2) | 0.3840 (2) | 0.98308 (13) | 0.0395 (5) |
| C4 | 0.5124 (2) | 0.3393 (2) | 0.94584 (11) | 0.0326 (4) |
| C5 | 0.4892 (2) | 0.36838 (17) | 0.85916 (11) | 0.0255 (4) |
| C6 | 0.6010 (2) | 0.43761 (18) | 0.81107 (11) | 0.0274 (4) |
| C7 | 0.5420 (2) | 0.45217 (18) | 0.72098 (11) | 0.0266 (4) |
| C8 | 0.3518 (2) | 0.33864 (17) | 0.80233 (11) | 0.0250 (4) |
| C9 | 0.2867 (2) | 0.38767 (19) | 0.64668 (11) | 0.0269 (4) |
| C10 | 0.3548 (2) | 0.32287 (19) | 0.56467 (11) | 0.0281 (4) |
| C11 | 0.2218 (2) | 0.5328 (2) | 0.62906 (12) | 0.0337 (4) |
| C12 | 0.1075 (2) | 0.5854 (2) | 0.69751 (13) | 0.0396 (5) |
| C13 | 0.1827 (2) | 0.63585 (19) | 0.77980 (12) | 0.0311 (4) |
| H1 | 0.81748 | 0.52994 | 0.8148 | 0.0429* |
| H2 | 0.85847 | 0.48296 | 0.96185 | 0.0499* |
| H3 | 0.67296 | 0.36605 | 1.04192 | 0.0475* |
| H4 | 0.43628 | 0.2915 | 0.97838 | 0.0392* |
| H5 | 0.19512 | 0.32999 | 0.66336 | 0.0323* |
| H6B | 0.51794 | 0.20427 | 0.52459 | 0.0513* |
| H7A | 0.52731 | 0.23407 | 0.62019 | 0.0513* |
| H8A | 0.31049 | 0.59737 | 0.62494 | 0.0404* |
| H9B | 0.16801 | 0.53233 | 0.5729 | 0.0404* |
| H10A | 0.04576 | 0.66122 | 0.67253 | 0.0475* |
| H11B | 0.03387 | 0.51082 | 0.71193 | 0.0475* |
| H12 | 0.1288 | 0.66053 | 0.8897 | 0.0747* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0368 (7) | 0.0480 (8) | 0.0294 (7) | −0.0115 (7) | 0.0044 (6) | 0.0064 (6) |
| O2 | 0.0285 (7) | 0.0478 (8) | 0.0299 (7) | −0.0078 (6) | 0.0024 (6) | 0.0079 (6) |
| O3 | 0.0411 (8) | 0.0673 (10) | 0.0221 (7) | 0.0098 (8) | −0.0079 (6) | −0.0063 (6) |
| O4 | 0.0307 (7) | 0.0439 (7) | 0.0313 (7) | −0.0037 (6) | −0.0003 (6) | −0.0053 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O5 | 0.0331 (7) | 0.0856 (12) | 0.0306 (8) | −0.0014 (8) | 0.0020 (6) | −0.0085 (8) |
| N1 | 0.0237 (7) | 0.0314 (7) | 0.0188 (7) | −0.0022 (6) | −0.0007 (6) | 0.0021 (6) |
| N2 | 0.0352 (9) | 0.0442 (9) | 0.0233 (8) | 0.0063 (7) | −0.0016 (7) | −0.0053 (7) |
| C1 | 0.0269 (9) | 0.0432 (10) | 0.0373 (11) | −0.0013 (9) | −0.0037 (8) | −0.0049 (9) |
| C2 | 0.0322 (10) | 0.0538 (12) | 0.0388 (11) | 0.0062 (10) | −0.0146 (9) | −0.0102 (10) |
| C3 | 0.0436 (12) | 0.0508 (11) | 0.0243 (10) | 0.0134 (10) | −0.0094 (9) | −0.0036 (9) |
| C4 | 0.0362 (10) | 0.0392 (10) | 0.0225 (9) | 0.0074 (9) | 0.0008 (8) | 0.0014 (8) |
| C5 | 0.0262 (9) | 0.0286 (9) | 0.0219 (9) | 0.0046 (7) | 0.0000 (7) | −0.0023 (7) |
| C6 | 0.0257 (8) | 0.0315 (8) | 0.0251 (9) | 0.0030 (8) | −0.0013 (7) | −0.0024 (8) |
| C7 | 0.0239 (8) | 0.0315 (8) | 0.0244 (9) | −0.0024 (7) | 0.0012 (7) | 0.0001 (8) |
| C8 | 0.0272 (9) | 0.0268 (8) | 0.0210 (8) | 0.0026 (7) | 0.0004 (7) | 0.0023 (7) |
| C9 | 0.0242 (8) | 0.0364 (9) | 0.0203 (9) | −0.0034 (7) | −0.0031 (7) | 0.0016 (8) |
| C10 | 0.0259 (9) | 0.0366 (9) | 0.0217 (9) | −0.0053 (8) | −0.0027 (7) | 0.0003 (7) |
| C11 | 0.0376 (10) | 0.0409 (10) | 0.0226 (9) | 0.0074 (9) | −0.0064 (8) | 0.0017 (8) |
| C12 | 0.0318 (9) | 0.0517 (12) | 0.0351 (11) | 0.0115 (9) | −0.0089 (9) | −0.0073 (9) |
| C13 | 0.0321 (10) | 0.0321 (9) | 0.0291 (10) | 0.0051 (8) | −0.0018 (8) | 0.0016 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.203 (2) | C9—C10 | 1.527 (2) |
| O2—C8 | 1.207 (2) | C9—C11 | 1.532 (3) |
| O3—C10 | 1.238 (2) | C11—C12 | 1.522 (3) |
| O4—C13 | 1.206 (2) | C12—C13 | 1.503 (3) |
| O5—C13 | 1.316 (2) | O5—H12 | 0.830 |
| N1—C7 | 1.405 (2) | N2—H6B | 0.870 |
| N1—C8 | 1.402 (2) | N2—H7A | 0.870 |
| N1—C9 | 1.460 (2) | C1—H1 | 0.940 |
| N2—C10 | 1.310 (2) | C2—H2 | 0.940 |
| C1—C2 | 1.393 (3) | C3—H3 | 0.940 |
| C1—C6 | 1.387 (3) | C4—H4 | 0.940 |
| C2—C3 | 1.378 (3) | C9—H5 | 0.990 |
| C3—C4 | 1.396 (3) | C11—H8A | 0.980 |
| C4—C5 | 1.382 (2) | C11—H9B | 0.980 |
| C5—C6 | 1.378 (2) | C12—H10A | 0.980 |
| C5—C8 | 1.487 (2) | C12—H11B | 0.980 |
| C6—C7 | 1.486 (2) | | |
| | | | |
| C7—N1—C8 | 111.52 (14) | O4—C13—C12 | 123.81 (17) |
| C7—N1—C9 | 123.92 (14) | O5—C13—C12 | 112.90 (16) |
| C8—N1—C9 | 122.81 (14) | C13—O5—H12 | 109.469 |
| C2—C1—C6 | 117.00 (17) | C10—N2—H6B | 120.005 |
| C1—C2—C3 | 121.55 (19) | C10—N2—H7A | 120.001 |
| C2—C3—C4 | 121.08 (18) | H6B—N2—H7A | 119.994 |
| C3—C4—C5 | 117.28 (17) | C2—C1—H1 | 121.519 |
| C4—C5—C6 | 121.54 (16) | C6—C1—H1 | 121.506 |
| C4—C5—C8 | 130.11 (16) | C1—C2—H2 | 119.202 |
| C6—C5—C8 | 108.34 (15) | C3—C2—H2 | 119.210 |
| C1—C6—C5 | 121.54 (16) | C2—C3—H3 | 119.482 |

| | | | |
|---|---|---|---|
| C1—C6—C7 | 129.83 (16) | C4—C3—H3 | 119.476 |
| C5—C6—C7 | 108.63 (15) | C3—C4—H4 | 121.355 |
| O1—C7—N1 | 124.67 (16) | C5—C4—H4 | 121.352 |
| O1—C7—C6 | 129.86 (16) | N1—C9—H5 | 106.340 |
| N1—C7—C6 | 105.47 (14) | C10—C9—H5 | 106.336 |
| O2—C8—N1 | 124.22 (16) | C11—C9—H5 | 106.343 |
| O2—C8—C5 | 130.12 (16) | C9—C11—H8A | 108.681 |
| N1—C8—C5 | 105.63 (14) | C9—C11—H9B | 108.685 |
| N1—C9—C10 | 112.66 (14) | C12—C11—H8A | 108.682 |
| N1—C9—C11 | 113.19 (15) | C12—C11—H9B | 108.682 |
| C10—C9—C11 | 111.39 (14) | H8A—C11—H9B | 107.616 |
| O3—C10—N2 | 122.91 (17) | C11—C12—H10A | 108.474 |
| O3—C10—C9 | 117.83 (16) | C11—C12—H11B | 108.468 |
| N2—C10—C9 | 119.20 (15) | C13—C12—H10A | 108.470 |
| C9—C11—C12 | 114.32 (16) | C13—C12—H11B | 108.470 |
| C11—C12—C13 | 115.22 (16) | H10A—C12—H11B | 107.495 |
| O4—C13—O5 | 123.27 (17) | | |

| | | | |
|---|---|---|---|
| C7—N1—C8—O2 | 171.27 (15) | C4—C5—C6—C7 | 179.74 (15) |
| C7—N1—C8—C5 | −6.74 (17) | C4—C5—C8—O2 | 5.8 (3) |
| C8—N1—C7—O1 | −174.78 (15) | C4—C5—C8—N1 | −176.37 (17) |
| C8—N1—C7—C6 | 5.92 (17) | C6—C5—C8—O2 | −172.97 (16) |
| C7—N1—C9—C10 | 63.76 (19) | C6—C5—C8—N1 | 4.88 (17) |
| C7—N1—C9—C11 | −63.74 (19) | C8—C5—C6—C1 | 178.32 (13) |
| C9—N1—C7—O1 | −9.5 (3) | C8—C5—C6—C7 | −1.38 (18) |
| C9—N1—C7—C6 | 171.19 (13) | C1—C6—C7—O1 | −1.5 (3) |
| C8—N1—C9—C10 | −132.59 (14) | C1—C6—C7—N1 | 177.71 (17) |
| C8—N1—C9—C11 | 99.91 (17) | C5—C6—C7—O1 | 178.13 (16) |
| C9—N1—C8—O2 | 5.8 (2) | C5—C6—C7—N1 | −2.62 (18) |
| C9—N1—C8—C5 | −172.20 (13) | N1—C9—C10—O3 | −167.77 (14) |
| C2—C1—C6—C5 | 0.2 (3) | N1—C9—C10—N2 | 15.1 (2) |
| C2—C1—C6—C7 | 179.85 (16) | N1—C9—C11—C12 | −70.37 (18) |
| C6—C1—C2—C3 | 0.2 (3) | C10—C9—C11—C12 | 161.47 (13) |
| C1—C2—C3—C4 | −0.2 (3) | C11—C9—C10—O3 | −39.3 (2) |
| C2—C3—C4—C5 | −0.1 (3) | C11—C9—C10—N2 | 143.54 (15) |
| C3—C4—C5—C6 | 0.5 (3) | C9—C11—C12—C13 | 77.4 (2) |
| C3—C4—C5—C8 | −178.13 (16) | C11—C12—C13—O4 | 15.2 (3) |
| C4—C5—C6—C1 | −0.6 (3) | C11—C12—C13—O5 | −166.20 (15) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H12···O3$^{i}$ | 0.83 | 1.80 | 2.6230 (19) | 172 |
| N2—H6*B*···O3$^{ii}$ | 0.87 | 2.32 | 2.891 (2) | 123 |
| N2—H7*A*···O4$^{iii}$ | 0.87 | 2.05 | 2.886 (2) | 161 |

Symmetry codes: (i) −*x*+1/2, −*y*+1, *z*+1/2; (ii) *x*+1/2, −*y*+1/2, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+3/2.