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# Pyrrolidinium-2-carboxylate–4-nitrophenol (1/2)

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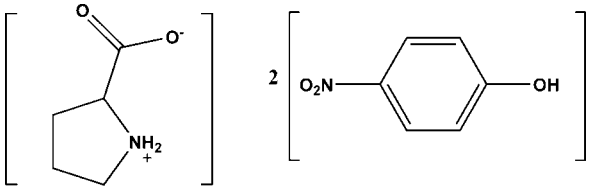
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.037; $wR$ factor = 0.094; data-to-parameter ratio = 13.7.

In the title compound, $C_5H_9NO_2\cdot 2C_6H_5NO_3$, the pyrrolidine ring of the pyrrolidinium-2-carboxylate zwitterion adopts a twisted conformation on the $-CH_2-CH_2-$ bond adjacent to the N atom. The mean plane of this pyrrolidine ring forms dihedral angles of 25.3 (3) and 32.1 (3)° with the two nitrophenol rings. An intramolecular N—H···O hydrogen bond occurs in the pyrrolidinium-2-carboxylate molecule. In the crystal, molecules are linked *via* O—H···O and N—H···O hydrogen bonds, enclosing $R_2^3(8)$ ring motifs, forming chains running parallel to the *a* axis. These chains are further cross-linked by O—H···O and C—H···O hydrogen bonds, forming undulating two-dimensional networks lying parallel to (001).

## Related literature

For the use of nitro-aromatics as intermediates in explosives, dyestuffs, pesticides and organic synthesis, see: Yan *et al.* (2006). For the occurrence of nitro-aromatics in industrial wastes and as direct pollutants in the environment, see: Yan *et al.* (2006); Soojhawon *et al.* (2005). For ring puckering analysis, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_5H_9NO_2\cdot 2C_6H_5NO_3$
$M_r$ = 393.35
Orthorhombic, $P2_12_12_1$
$a$ = 5.9045 (3) Å
$b$ = 15.6099 (7) Å
$c$ = 20.0424 (9) Å
$V$ = 1847.28 (15) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 K
0.35 × 0.25 × 0.25 mm

### Data collection

Bruker SMART APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.961, $T_{max}$ = 0.972
17765 measured reflections
3572 independent reflections
2987 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.094
$S$ = 1.06
3572 reflections
261 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3$A$···O8 | 0.94 (6) | 2.03 (6) | 2.606 (6) | 118 (5) |
| N3—H3$B$···O7$^{i}$ | 0.93 (5) | 1.87 (6) | 2.766 (6) | 160 (7) |
| O3—H3$C$···O7$^{i}$ | 0.82 | 1.92 | 2.656 (5) | 148 |
| O6—H6$A$···O8$^{ii}$ | 0.82 | 1.82 | 2.604 (5) | 159 |
| C11—H11···O1$^{i}$ | 0.93 | 2.59 | 3.503 (8) | 169 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. TS also thanks DST Inspire for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2657).

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# supporting information

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## Pyrrolidinium-2-carboxylate–4-nitrophenol (1/2)

**Narayanan Swarna Sowmya, Yechuri Vidyalakshmi, Sadasivam Sampathkrishnan, Thothadri Srinivasan and Devadasan Velmurugan**

### S1. Comment

Nitro-aromatics are widely used either as materials or as intermediates in explosives, dyestuffs, pesticides and organic synthesis (Yan *et al.*, 2006). They also occur as industrial wastes and direct pollutants in the environment. They are relatively soluble in water and detectable in rivers, ponds and soil (Yan *et al.*, 2006; Soojhawon *et al.*, 2005).

The title compound was synthesized by mixing eqimolar amounts of pyrrolidine carboxylic acid and para-nitrophenol in water. The crystals obtained were found to be composed of one molecule of pyrrolidinium-2-carboxylate, in the zwitterion form, and two molecules of para-nitrophenol, Fig. 1. The pyrrolidine ring (N3/C14-C17) adopts a twisted conformation on bond C17-C15, with puckering parameters (Cremer & Pople, 1975) $q_2$ = 0.373 (7) Å and $\varphi_2$ = 312.3 (11)°. The hydroxy group O atoms, O3 and O6, deviate slightly by -0.0380 (3) and 0.0160 (5) Å, respectively, from the mean planes of the benzene rings to which they are attached, (C1–C6) and (C7–C12).

The mean plane of the pyrrolidine ring (N3/C14-C17) forms dihedral angles of 25.3 (3)° and 32.1 (3)° with the nitrophenol rings (C1-C6) and (C7-C12), respectively.

In the crystal, molecules are linked via O—H···O and N—H···O intra- and inter-molecular hydrogen bonds (Table 1 and Fig. 2), with $R^3_2(8)$ ring motifs, forming chains running parallel to the a axis. These chains are further cross-linked by O—H···O and C—H···O hydrogen bond forming undulating two-dimensional networks lying parallel to the ab plane (Table 1 and Fig. 2).

### S2. Experimental

An equimolar (1:1:1) ratio of pyrrolidine carboxylic acid and para-nitrophenol were added to distilled water as solvent and the mixture stirred for 1 h, giving a clear solution. The solution was filtered into a clean beaker and sealed with parafilm and kept at room temperature for three days, after which block-like colourless crystals suitable for X-ray diffraction analysis were obtained.

### S3. Refinement

The NH H-atoms were located in difference electron-density maps and refined with distance restraints: N-H = 0.92 (2) Å. The OH and C-bound H-atoms were included in calculated positions and treated as riding atoms: O-H = 0.82 Å, C-H = 0.93, 0.97 and 0.98 Å for CH(aromatic), $CH_2$, and CH(methine) H-atoms, respectively, with $U_{iso}$(H) = 1.5$U_{eq}$(C-methyl and O), and = 1.2$U_{eq}$(C) for other H-atoms. In the final cycles of refinement, in the absence of significant anomalous scattering effects, 1490 Friedel pairs were merged and Δf ″ set to zero.

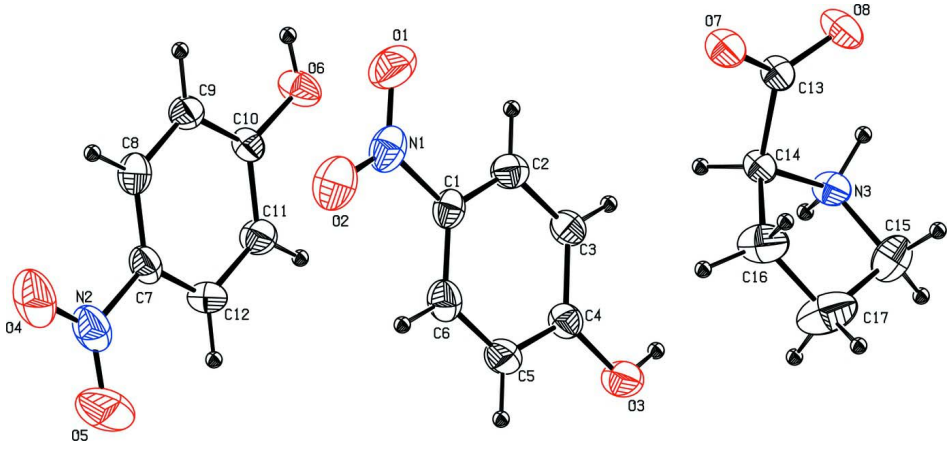


**Figure 1**

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

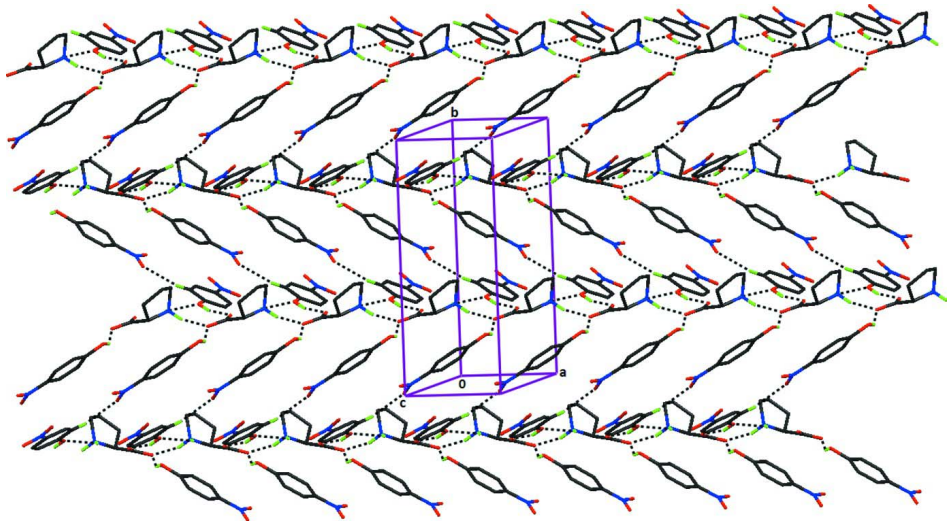


**Figure 2**

The crystal packing of the title compound viewed along the *c* axis. The hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been excluded for clarity).

### Pyrrolidinium-2-carboxylate–4-nitrophenol (1/2)

*Crystal data*

$C_5H_9NO_2·2C_6H_5NO_3$
$M_r$ = 393.35
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 5.9045 (3) Å
$b$ = 15.6099 (7) Å
$c$ = 20.0424 (9) Å
$V$ = 1847.28 (15) Å$^3$
$Z$ = 4
$F(000)$ = 824
$D_x$ = 1.414 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3572 reflections
$\theta$ = 2.4–25.9°
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 K
Block, colourless
0.35 × 0.25 × 0.25 mm

*Data collection*

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.961, $T_{max}$ = 0.972
17765 measured reflections
3572 independent reflections
2987 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 25.9°, $\theta_{min}$ = 2.4°
$h = -7 \rightarrow 7$
$k = -17 \rightarrow 19$
$l = -23 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.094
$S$ = 1.06
3572 reflections
261 parameters
2 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.2454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 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 $F^2 > 2$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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.7666 (9) | 0.5876 (3) | 0.5668 (3) | 0.0468 (13) |
| C2 | 0.6897 (10) | 0.5897 (3) | 0.6313 (3) | 0.0502 (13) |
| H2 | 0.7673 | 0.5610 | 0.6649 | 0.060* |
| C3 | 0.4946 (9) | 0.6351 (3) | 0.6455 (2) | 0.0469 (12) |
| H3 | 0.4380 | 0.6362 | 0.6887 | 0.056* |
| C4 | 0.3835 (9) | 0.6789 (3) | 0.5951 (2) | 0.0455 (12) |
| C5 | 0.4649 (11) | 0.6750 (4) | 0.5306 (3) | 0.0556 (15) |
| H5 | 0.3892 | 0.7039 | 0.4967 | 0.067* |
| C6 | 0.6561 (11) | 0.6291 (4) | 0.5163 (3) | 0.0552 (14) |
| H6 | 0.7103 | 0.6262 | 0.4728 | 0.066* |
| C7 | 0.7088 (10) | 0.3784 (3) | 0.4091 (3) | 0.0491 (13) |
| C8 | 0.8784 (10) | 0.3384 (4) | 0.4433 (3) | 0.0518 (14) |
| H8 | 1.0042 | 0.3175 | 0.4207 | 0.062* |
| C9 | 0.8621 (9) | 0.3292 (3) | 0.5114 (3) | 0.0474 (12) |
| H9 | 0.9778 | 0.3028 | 0.5352 | 0.057* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.6715 (8) | 0.3596 (3) | 0.5440 (2) | 0.0404 (11) |
| C11 | 0.5015 (10) | 0.3997 (4) | 0.5088 (3) | 0.0508 (13) |
| H11 | 0.3744 | 0.4202 | 0.5311 | 0.061* |
| C12 | 0.5193 (11) | 0.4094 (4) | 0.4411 (3) | 0.0549 (14) |
| H12 | 0.4051 | 0.4364 | 0.4171 | 0.066* |
| C13 | 1.0014 (8) | 0.7956 (3) | 0.7647 (2) | 0.0421 (12) |
| C14 | 0.7877 (8) | 0.8049 (3) | 0.7237 (2) | 0.0401 (12) |
| H14 | 0.7464 | 0.7489 | 0.7054 | 0.048* |
| C15 | 0.5588 (13) | 0.9273 (4) | 0.7485 (4) | 0.0726 (19) |
| H15A | 0.6620 | 0.9654 | 0.7718 | 0.087* |
| H15B | 0.4042 | 0.9442 | 0.7583 | 0.087* |
| C16 | 0.8030 (11) | 0.8693 (5) | 0.6673 (3) | 0.0729 (19) |
| H16A | 0.9432 | 0.9014 | 0.6702 | 0.087* |
| H16B | 0.7977 | 0.8404 | 0.6245 | 0.087* |
| C17 | 0.6028 (15) | 0.9278 (5) | 0.6752 (4) | 0.094 (3) |
| H17A | 0.6379 | 0.9851 | 0.6596 | 0.113* |
| H17B | 0.4729 | 0.9063 | 0.6507 | 0.113* |
| N1 | 0.9739 (9) | 0.5403 (3) | 0.5517 (3) | 0.0627 (13) |
| N2 | 0.7296 (12) | 0.3891 (4) | 0.3376 (3) | 0.0710 (16) |
| N3 | 0.5998 (7) | 0.8362 (3) | 0.7673 (2) | 0.0465 (11) |
| O1 | 1.0691 (9) | 0.5029 (3) | 0.5975 (3) | 0.0833 (15) |
| O2 | 1.0428 (9) | 0.5400 (3) | 0.4942 (3) | 0.0866 (15) |
| O3 | 0.1964 (7) | 0.7261 (3) | 0.60652 (19) | 0.0642 (12) |
| H3C | 0.1632 | 0.7234 | 0.6462 | 0.096* |
| O4 | 0.8932 (11) | 0.3577 (4) | 0.3096 (2) | 0.0953 (18) |
| O5 | 0.5857 (13) | 0.4289 (4) | 0.3074 (3) | 0.119 (2) |
| O6 | 0.6447 (6) | 0.3513 (3) | 0.61037 (16) | 0.0571 (10) |
| H6A | 0.7548 | 0.3265 | 0.6262 | 0.086* |
| O7 | 1.1759 (6) | 0.7746 (3) | 0.73339 (18) | 0.0570 (10) |
| O8 | 0.9884 (7) | 0.8096 (3) | 0.82530 (17) | 0.0648 (12) |
| H3A | 0.653 (11) | 0.832 (4) | 0.811 (3) | 0.064 (17)* |
| H3B | 0.470 (11) | 0.804 (5) | 0.760 (4) | 0.10 (3)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.045 (3) | 0.038 (3) | 0.057 (3) | −0.005 (2) | 0.004 (2) | −0.007 (3) |
| C2 | 0.050 (3) | 0.046 (3) | 0.055 (3) | −0.004 (3) | −0.008 (3) | 0.004 (3) |
| C3 | 0.048 (3) | 0.053 (3) | 0.039 (3) | −0.003 (3) | 0.002 (2) | 0.001 (2) |
| C4 | 0.043 (3) | 0.050 (3) | 0.044 (3) | −0.002 (2) | −0.001 (2) | −0.003 (2) |
| C5 | 0.064 (4) | 0.064 (4) | 0.039 (3) | 0.009 (3) | −0.001 (3) | 0.001 (3) |
| C6 | 0.063 (4) | 0.057 (3) | 0.045 (3) | −0.001 (3) | 0.010 (3) | −0.006 (3) |
| C7 | 0.067 (4) | 0.046 (3) | 0.034 (3) | −0.007 (3) | 0.001 (2) | −0.002 (2) |
| C8 | 0.051 (3) | 0.055 (3) | 0.049 (3) | 0.002 (3) | 0.014 (3) | −0.008 (3) |
| C9 | 0.040 (3) | 0.054 (3) | 0.047 (3) | 0.006 (2) | −0.002 (2) | −0.003 (3) |
| C10 | 0.037 (3) | 0.049 (3) | 0.036 (2) | −0.004 (2) | 0.000 (2) | −0.004 (2) |
| C11 | 0.043 (3) | 0.061 (3) | 0.049 (3) | 0.009 (3) | 0.003 (2) | −0.001 (3) |
| C12 | 0.056 (4) | 0.057 (3) | 0.051 (3) | 0.008 (3) | −0.013 (3) | 0.006 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.031 (2) | 0.055 (3) | 0.040 (3) | −0.002 (2) | 0.003 (2) | −0.005 (2) |
| C14 | 0.028 (2) | 0.057 (3) | 0.035 (2) | 0.001 (2) | 0.0025 (19) | −0.005 (2) |
| C15 | 0.066 (4) | 0.064 (4) | 0.087 (5) | 0.013 (3) | 0.014 (4) | −0.005 (4) |
| C16 | 0.058 (4) | 0.104 (5) | 0.057 (4) | 0.017 (4) | 0.015 (3) | 0.022 (4) |
| C17 | 0.076 (5) | 0.103 (6) | 0.104 (6) | 0.025 (4) | 0.020 (4) | 0.046 (5) |
| N1 | 0.051 (3) | 0.049 (3) | 0.088 (4) | −0.005 (2) | 0.006 (3) | −0.012 (3) |
| N2 | 0.103 (5) | 0.067 (4) | 0.042 (3) | −0.013 (3) | 0.004 (3) | −0.001 (3) |
| N3 | 0.030 (2) | 0.068 (3) | 0.041 (2) | 0.004 (2) | 0.0055 (18) | 0.002 (2) |
| O1 | 0.065 (3) | 0.075 (3) | 0.110 (4) | 0.019 (2) | −0.008 (3) | −0.001 (3) |
| O2 | 0.074 (3) | 0.092 (3) | 0.094 (4) | 0.011 (3) | 0.028 (3) | −0.012 (3) |
| O3 | 0.055 (2) | 0.089 (3) | 0.048 (2) | 0.022 (2) | 0.0020 (19) | 0.000 (2) |
| O4 | 0.135 (5) | 0.101 (4) | 0.050 (3) | −0.013 (4) | 0.030 (3) | −0.009 (3) |
| O5 | 0.158 (6) | 0.145 (6) | 0.052 (3) | 0.023 (5) | −0.014 (4) | 0.028 (3) |
| O6 | 0.042 (2) | 0.092 (3) | 0.0365 (19) | 0.003 (2) | 0.0030 (16) | 0.0015 (19) |
| O7 | 0.0301 (18) | 0.089 (3) | 0.052 (2) | 0.0065 (19) | 0.0028 (16) | −0.010 (2) |
| O8 | 0.042 (2) | 0.113 (4) | 0.039 (2) | 0.005 (2) | −0.0036 (17) | −0.015 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.368 (8) | C13—O8 | 1.237 (6) |
| C1—C2 | 1.369 (8) | C13—O7 | 1.250 (6) |
| C1—N1 | 1.461 (8) | C13—C14 | 1.512 (7) |
| C2—C3 | 1.382 (8) | C14—N3 | 1.493 (6) |
| C2—H2 | 0.9300 | C14—C16 | 1.514 (8) |
| C3—C4 | 1.385 (7) | C14—H14 | 0.9800 |
| C3—H3 | 0.9300 | C15—N3 | 1.491 (8) |
| C4—O3 | 1.347 (6) | C15—C17 | 1.493 (11) |
| C4—C5 | 1.380 (7) | C15—H15A | 0.9700 |
| C5—C6 | 1.367 (8) | C15—H15B | 0.9700 |
| C5—H5 | 0.9300 | C16—C17 | 1.502 (10) |
| C6—H6 | 0.9300 | C16—H16A | 0.9700 |
| C7—C8 | 1.364 (8) | C16—H16B | 0.9700 |
| C7—C12 | 1.377 (8) | C17—H17A | 0.9700 |
| C7—N2 | 1.449 (7) | C17—H17B | 0.9700 |
| C8—C9 | 1.375 (7) | N1—O2 | 1.223 (7) |
| C8—H8 | 0.9300 | N1—O1 | 1.225 (7) |
| C9—C10 | 1.385 (7) | N2—O5 | 1.213 (8) |
| C9—H9 | 0.9300 | N2—O4 | 1.219 (8) |
| C10—O6 | 1.347 (6) | N3—H3A | 0.94 (6) |
| C10—C11 | 1.377 (7) | N3—H3B | 0.93 (5) |
| C11—C12 | 1.370 (7) | O3—H3C | 0.8200 |
| C11—H11 | 0.9300 | O6—H6A | 0.8200 |
| C12—H12 | 0.9300 | | |
| | | | |
| C6—C1—C2 | 121.9 (5) | N3—C14—C13 | 109.5 (4) |
| C6—C1—N1 | 119.0 (5) | N3—C14—C16 | 105.3 (4) |
| C2—C1—N1 | 119.0 (5) | C13—C14—C16 | 114.8 (5) |
| C1—C2—C3 | 118.8 (5) | N3—C14—H14 | 109.0 |

| | | | |
|---|---|---|---|
| C1—C2—H2 | 120.6 | C13—C14—H14 | 109.0 |
| C3—C2—H2 | 120.6 | C16—C14—H14 | 109.0 |
| C2—C3—C4 | 119.9 (5) | N3—C15—C17 | 102.9 (5) |
| C2—C3—H3 | 120.1 | N3—C15—H15A | 111.2 |
| C4—C3—H3 | 120.1 | C17—C15—H15A | 111.2 |
| O3—C4—C5 | 118.0 (5) | N3—C15—H15B | 111.2 |
| O3—C4—C3 | 122.3 (5) | C17—C15—H15B | 111.2 |
| C5—C4—C3 | 119.8 (5) | H15A—C15—H15B | 109.1 |
| C6—C5—C4 | 120.4 (5) | C17—C16—C14 | 106.1 (5) |
| C6—C5—H5 | 119.8 | C17—C16—H16A | 110.5 |
| C4—C5—H5 | 119.8 | C14—C16—H16A | 110.5 |
| C5—C6—C1 | 119.1 (5) | C17—C16—H16B | 110.5 |
| C5—C6—H6 | 120.4 | C14—C16—H16B | 110.5 |
| C1—C6—H6 | 120.4 | H16A—C16—H16B | 108.7 |
| C8—C7—C12 | 121.6 (5) | C15—C17—C16 | 103.7 (6) |
| C8—C7—N2 | 119.2 (6) | C15—C17—H17A | 111.0 |
| C12—C7—N2 | 119.3 (6) | C16—C17—H17A | 111.0 |
| C7—C8—C9 | 119.7 (5) | C15—C17—H17B | 111.0 |
| C7—C8—H8 | 120.2 | C16—C17—H17B | 111.0 |
| C9—C8—H8 | 120.2 | H17A—C17—H17B | 109.0 |
| C8—C9—C10 | 119.2 (5) | O2—N1—O1 | 123.5 (6) |
| C8—C9—H9 | 120.4 | O2—N1—C1 | 118.5 (6) |
| C10—C9—H9 | 120.4 | O1—N1—C1 | 118.0 (6) |
| O6—C10—C11 | 117.6 (5) | O5—N2—O4 | 122.1 (6) |
| O6—C10—C9 | 121.9 (5) | O5—N2—C7 | 119.5 (6) |
| C11—C10—C9 | 120.5 (4) | O4—N2—C7 | 118.4 (6) |
| C12—C11—C10 | 120.1 (5) | C15—N3—C14 | 106.6 (4) |
| C12—C11—H11 | 120.0 | C15—N3—H3A | 111 (4) |
| C10—C11—H11 | 120.0 | C14—N3—H3A | 106 (4) |
| C11—C12—C7 | 119.0 (5) | C15—N3—H3B | 110 (5) |
| C11—C12—H12 | 120.5 | C14—N3—H3B | 110 (5) |
| C7—C12—H12 | 120.5 | H3A—N3—H3B | 112 (6) |
| O8—C13—O7 | 126.2 (5) | C4—O3—H3C | 109.5 |
| O8—C13—C14 | 117.7 (4) | C10—O6—H6A | 109.5 |
| O7—C13—C14 | 116.1 (4) | | |

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| C6—C1—C2—C3 | 0.0 (8) | O8—C13—C14—N3 | 3.8 (7) |
| N1—C1—C2—C3 | 179.4 (5) | O7—C13—C14—N3 | −176.0 (5) |
| C1—C2—C3—C4 | −1.4 (8) | O8—C13—C14—C16 | 121.9 (6) |
| C2—C3—C4—O3 | −178.1 (5) | O7—C13—C14—C16 | −57.8 (7) |
| C2—C3—C4—C5 | 1.7 (8) | N3—C14—C16—C17 | −7.9 (7) |
| O3—C4—C5—C6 | 179.0 (5) | C13—C14—C16—C17 | −128.4 (6) |
| C3—C4—C5—C6 | −0.8 (8) | N3—C15—C17—C16 | −39.1 (8) |
| C4—C5—C6—C1 | −0.5 (9) | C14—C16—C17—C15 | 29.2 (8) |
| C2—C1—C6—C5 | 0.9 (9) | C6—C1—N1—O2 | 0.4 (8) |
| N1—C1—C6—C5 | −178.5 (5) | C2—C1—N1—O2 | −179.0 (5) |
| C12—C7—C8—C9 | −0.7 (9) | C6—C1—N1—O1 | −179.3 (5) |
| N2—C7—C8—C9 | 178.9 (5) | C2—C1—N1—O1 | 1.3 (7) |

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| C7—C8—C9—C10 | 1.0 (8) | C8—C7—N2—O5 | −175.9 (6) |
| C8—C9—C10—O6 | 179.1 (5) | C12—C7—N2—O5 | 3.7 (9) |
| C8—C9—C10—C11 | −0.9 (8) | C8—C7—N2—O4 | 3.9 (8) |
| O6—C10—C11—C12 | −179.6 (5) | C12—C7—N2—O4 | −176.5 (6) |
| C9—C10—C11—C12 | 0.3 (8) | C17—C15—N3—C14 | 34.8 (7) |
| C10—C11—C12—C7 | 0.1 (9) | C13—C14—N3—C15 | 107.3 (5) |
| C8—C7—C12—C11 | 0.1 (9) | C16—C14—N3—C15 | −16.6 (6) |
| N2—C7—C12—C11 | −179.5 (5) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3*A*···O8 | 0.94 (6) | 2.03 (6) | 2.606 (6) | 118 (5) |
| N3—H3*B*···O7$^{i}$ | 0.93 (5) | 1.87 (6) | 2.766 (6) | 160 (7) |
| O3—H3*C*···O7$^{i}$ | 0.82 | 1.92 | 2.656 (5) | 148 |
| O6—H6*A*···O8$^{ii}$ | 0.82 | 1.82 | 2.604 (5) | 159 |
| C11—H11···O1$^{i}$ | 0.93 | 2.59 | 3.503 (8) | 169 |

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