

2-(3-Nitrophenylaminocarbonyl)-benzoic acid: hydrogen-bonded sheets of $R_4^4(22)$ rings

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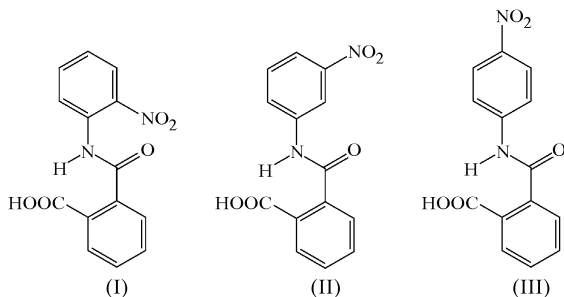
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In the title compound, $C_{14}H_{10}N_2O_5$, the molecules are linked by a combination of one $N-H\cdots O$ and one $O-H\cdots O$ hydrogen bond into sheets containing a single type of $R_4^4(22)$ ring.

Comment

We recently reported the molecular and supramolecular structures of 2-(2-nitrophenylaminocarbonyl)benzoic acid, (I), and two polymorphs, one orthorhombic with space group $P2_12_12_1$ and the other monoclinic with space group $P2_1/n$, of 2-(4-nitrophenylaminocarbonyl)benzoic acid, (III) (Glidewell *et al.*, 2004). In the 2-nitro isomer, compound (I), the molecules form $R_2^2(8)$ carboxylic acid dimers, which are linked into sheets by π - π stacking interactions, but intermolecular $N-H\cdots O$ hydrogen bonds are absent. In the orthorhombic polymorph of the 4-nitro isomer, (III), the molecules are linked into sheets of $R_4^4(22)$ rings built from a combination of two $C(7)$ chains, formed by $N-H\cdots O$ and $O-H\cdots O$ hydrogen bonds, respectively. In the monoclinic polymorph of (III), where $Z' = 2$, the molecules are linked into a continuous



three-dimensional framework by a combination of $O-H\cdots O$, $N-H\cdots O$ and $C-H\cdots O$ hydrogen bonds. We report here the structure of the 3-nitro isomer, (II), which, although it

crystallizes in a monoclinic space group, $P2_1$, with unit-cell dimensions very different from those of the orthorhombic polymorph of (III), nonetheless forms a supramolecular structure very similar to that of orthorhombic (III).

In compound (II), the C—O bond distances (Table 1) within the carboxyl group are consistent with the location of the carboxyl H atom as deduced from a difference map. The torsion angles (Table 1 and Fig. 1) indicate that the molecules of (II) have no internal symmetry and hence they are chiral. Thus, in the absence of any inversion twinning, each crystal will contain just a single enantiomorph of (II). However, this chirality in the solid state has no chemical significance.

The molecules of (II) (Fig. 1) are linked into sheets by a combination of one $N-H\cdots O$ hydrogen bond and one $O-H\cdots O$ hydrogen bond, and the formation of the sheet is most readily analysed in terms of the one-dimensional substructures generated by the two individual hydrogen bonds.

Amino atom N1 in the molecule at (x, y, z) acts as hydrogen-bond donor to carboxyl atom O22 in the molecule at $(-1 + x, y, z)$, so generating by translation a $C(7)$ (Bernstein *et al.*, 1995) chain running parallel to the $[100]$ direction (Fig. 2).

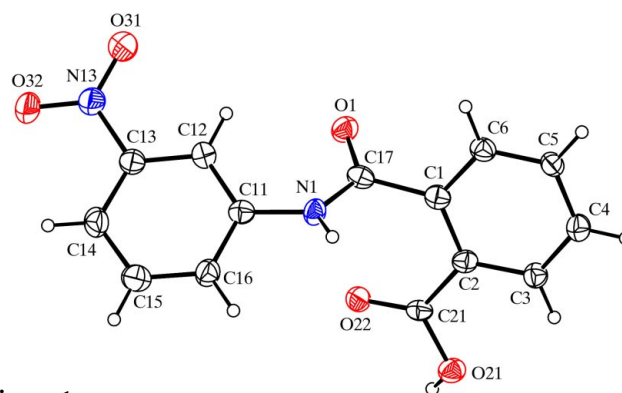


Figure 1
The molecule of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

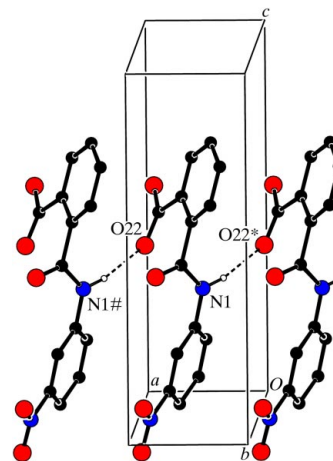


Figure 2
Part of the crystal structure of (II), showing the formation of a $C(7)$ chain along $[100]$. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-1 + x, y, z)$ and $(1 + x, y, z)$, respectively.

At the same time, carboxyl atom O21 at (x, y, z) acts as donor to amide atom O1 in the molecule at $(2 - x, -\frac{1}{2} + y, 1 - z)$, so forming a second $C(7)$ chain, this time running parallel to the $[010]$ direction and generated by the 2_1 screw axis along $(1, y, \frac{1}{2})$ (Fig. 3). It is noteworthy that the $C(4)$ motif so characteristic of simple amides is absent. Likewise, the motifs characteristic of simple carboxylic acids, namely the $C(4)$ chain and the $R_2^2(8)$ cyclic dimer motif, are both absent.

The combination of these two simple chains along $[100]$ and $[010]$ generates an (001) sheet in the form of a $(4,4)$ -net built from a single type of $R_4^4(22)$ (Bernstein *et al.*, 1995) ring (Fig. 4). Despite the presence of two independent aryl rings, there are neither $C-H \cdots \pi$ (arene) hydrogen bonds nor aromatic $\pi-\pi$ stacking interactions present in the structure of isomer (II), and there are, in fact, no direction-specific interactions between adjacent (001) sheets.

It is of interest to note that, despite their different space groups and unit-cell dimensions, isomer (II) as reported here, and the orthorhombic polymer of isomer (III) (Glidewell *et al.*, 2004) form exactly the same two types of $C(7)$ chain built

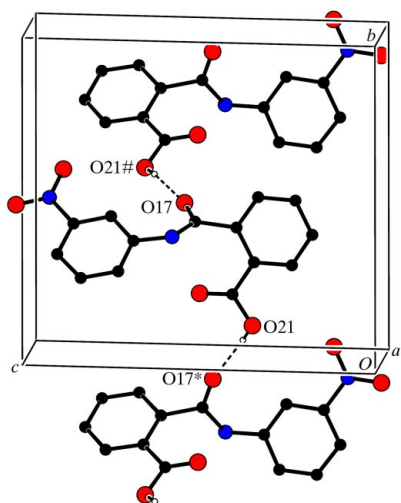


Figure 3
Part of the crystal structure of (II), showing the formation of a $C(7)$ chain along $[010]$. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(2 - x, -\frac{1}{2} + y, 1 - z)$ and $(2 - x, \frac{1}{2} + y, 1 - z)$, respectively.

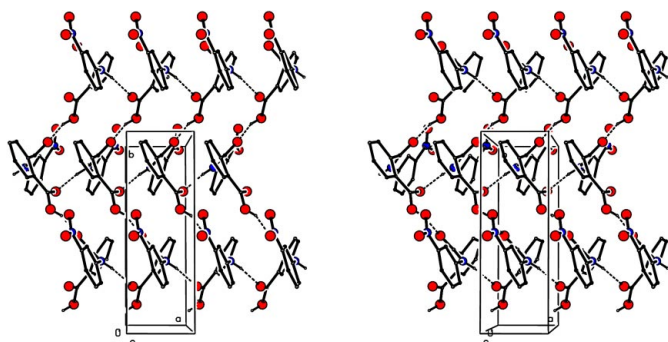


Figure 4
Stereoview of part of the crystal structure of (II), showing the formation of an (001) sheet of $R_4^4(22)$ rings.

from $N-H \cdots O$ and $O-H \cdots O$ hydrogen bonds, generated by translation along $[100]$ and by a 2_1 screw axis along $[010]$, respectively, with the combination of these two chains producing a sheet of $R_4^4(22)$ rings in both structures. Not only do the nitro groups play no direct role in the hydrogen-bonding scheme, but in these two examples they appear to exert no significant influence on the overall supramolecular aggregation.

Experimental

The title compound was prepared by the reaction of equimolar quantities of 3-nitroaniline and phthalic anhydride (2 mmol of each) in chloroform solution (20 ml), following the procedure used for the preparation of the 2-nitro and 4-nitro isomers (Glidewell *et al.*, 2004). Crystals of (II) suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in ethanol (m.p. 440–442 K).

Crystal data

$C_{14}H_{10}N_2O_5$	$D_x = 1.522 \text{ Mg m}^{-3}$
$M_r = 286.24$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 1471 reflections
$a = 4.0511(9) \text{ \AA}$	$\theta = 3.2\text{--}27.5^\circ$
$b = 12.076(3) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 12.771(3) \text{ \AA}$	$T = 120(2) \text{ K}$
$\beta = 90.287(12)^\circ$	Plate, colourless
$V = 624.8(3) \text{ \AA}^3$	$0.40 \times 0.10 \times 0.04 \text{ mm}$
$Z = 2$	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	1471 independent reflections
φ and ω scans	923 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$R_{\text{int}} = 0.100$
$T_{\text{min}} = 0.943, T_{\text{max}} = 0.995$	$\theta_{\text{max}} = 27.5^\circ$
6940 measured reflections	$h = -5 \rightarrow 4$
	$k = -15 \rightarrow 15$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.105P)^2 + 0.132P]$
$R[F^2 > 2\sigma(F^2)] = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.201$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.11$	$\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
1471 reflections	$\Delta\rho_{\text{min}} = -0.37 \text{ e \AA}^{-3}$
192 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997)
H-atom parameters constrained	Extinction coefficient: 0.065 (18)

Table 1

Selected geometric parameters (\AA , $^\circ$).

C17–N1	1.356 (8)	C21–O21	1.338 (7)
C17–O1	1.232 (7)	C21–O22	1.229 (7)
C12–C11–N1–C17	–31.8 (9)	C1–C2–C21–O21	–175.9 (5)
C11–N1–C17–C1	–168.3 (5)	C12–C13–N13–O31	–4.0 (9)
N1–C17–C1–C2	72.8 (6)		

Table 2

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1–H1 \cdots O22 ⁱ	0.88	1.99	2.857 (6)	168
O21–H21 \cdots O1 ⁱⁱ	0.84	1.84	2.624 (6)	155

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

For compound (II), the systematic absences permitted $P2_1$ and $P2_1/m$ as possible space groups; $P2_1$ was selected and confirmed by the successful structure analysis. All H atoms were located in difference maps, but they were subsequently treated as riding atoms, with C—H = 0.95 Å, N—H = 0.88 Å and O—H = 0.84 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ or $1.5U_{\text{eq}}(\text{O})$. In the absence of significant anomalous scattering, the Flack (1983) parameter was indeterminate (Flack & Bernardinelli, 2000). Accordingly, it was not possible to determine the absolute configuration of the molecules in the crystal selected for data collection, although this has no chemical significance. Friedel-equivalent reflections were merged prior to the final refinement.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the

authors thank the staff for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1880). Services for accessing these data are described at the back of the journal.

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Computing details

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$C_{14}H_{10}N_2O_5$

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Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 4.0511$ (9) Å

$b = 12.076$ (3) Å

$c = 12.771$ (3) Å

$\beta = 90.287$ (12)°

$V = 624.8$ (3) Å³

$Z = 2$

$F(000) = 296$

$D_x = 1.522$ Mg m⁻³

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

Cell parameters from 1471 reflections

$\theta = 3.2$ – 27.5 °

$\mu = 0.12$ mm⁻¹

$T = 120$ K

Plate, colourless

$0.40 \times 0.10 \times 0.04$ mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.943$, $T_{\max} = 0.995$

6940 measured reflections

1471 independent reflections

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

$R_{\text{int}} = 0.100$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.2$ °

$h = -5 \rightarrow 4$

$k = -15 \rightarrow 15$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.201$

$S = 1.11$

1471 reflections

192 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.105P)^2 + 0.132P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 1997), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.065 (18)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.7629 (10)	0.9904 (4)	0.4509 (3)	0.0445 (11)
O21	0.8329 (10)	0.6256 (4)	0.6456 (3)	0.0437 (11)
O22	0.8956 (9)	0.7250 (3)	0.4985 (3)	0.0405 (11)
O31	0.8557 (18)	1.0909 (5)	0.1112 (4)	0.0825 (19)
O32	0.8928 (14)	0.9815 (5)	-0.0220 (4)	0.0696 (16)
N1	0.3916 (12)	0.8572 (5)	0.4009 (4)	0.0382 (12)
N13	0.8164 (14)	1.0005 (5)	0.0701 (4)	0.0507 (15)
C1	0.4754 (13)	0.8968 (5)	0.5867 (5)	0.0375 (16)
C2	0.5731 (14)	0.7993 (5)	0.6402 (5)	0.0359 (14)
C3	0.4897 (14)	0.7868 (6)	0.7445 (4)	0.0381 (15)
C4	0.3134 (15)	0.8679 (6)	0.7953 (5)	0.0419 (15)
C5	0.2226 (15)	0.9644 (5)	0.7441 (4)	0.0379 (14)
C6	0.3040 (14)	0.9783 (6)	0.6415 (4)	0.0393 (14)
C11	0.4610 (14)	0.8472 (5)	0.2941 (5)	0.0372 (14)
C12	0.5986 (14)	0.9313 (6)	0.2359 (4)	0.0404 (15)
C13	0.6678 (15)	0.9111 (6)	0.1324 (5)	0.0411 (14)
C14	0.6006 (18)	0.8113 (6)	0.0833 (5)	0.0513 (18)
C15	0.4543 (18)	0.7291 (7)	0.1443 (5)	0.0551 (18)
C16	0.3852 (15)	0.7470 (6)	0.2479 (5)	0.0445 (16)
C17	0.5601 (14)	0.9181 (6)	0.4725 (5)	0.0396 (14)
C21	0.7805 (14)	0.7158 (5)	0.5869 (5)	0.0358 (14)
H1	0.2201	0.8199	0.4240	0.046*
H3	0.5545	0.7219	0.7812	0.046*
H4	0.2532	0.8575	0.8664	0.050*
H5	0.1044	1.0205	0.7802	0.046*
H6	0.2428	1.0447	0.6067	0.047*
H12	0.6445	1.0013	0.2666	0.049*
H14	0.6515	0.7994	0.0116	0.062*
H15	0.4018	0.6596	0.1136	0.066*
H16	0.2846	0.6902	0.2881	0.053*
H21	1.0037	0.5930	0.6248	0.066*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.047 (2)	0.043 (3)	0.043 (2)	-0.007 (2)	0.0020 (19)	0.004 (2)
O21	0.044 (2)	0.038 (3)	0.049 (3)	0.0054 (19)	0.0084 (19)	0.004 (2)
O22	0.042 (2)	0.040 (3)	0.040 (2)	-0.003 (2)	0.0052 (18)	-0.0015 (19)
O31	0.133 (6)	0.055 (4)	0.060 (3)	-0.015 (3)	0.031 (3)	-0.001 (3)
O32	0.105 (4)	0.061 (4)	0.043 (3)	0.017 (3)	0.022 (3)	0.006 (2)
N1	0.037 (3)	0.045 (3)	0.032 (3)	-0.007 (2)	0.007 (2)	0.002 (2)

N13	0.060 (4)	0.047 (4)	0.046 (3)	0.008 (3)	0.006 (3)	0.003 (3)
C1	0.034 (3)	0.037 (4)	0.042 (3)	-0.004 (3)	-0.003 (3)	-0.002 (3)
C2	0.033 (3)	0.033 (3)	0.042 (3)	-0.005 (2)	0.002 (2)	0.001 (3)
C3	0.038 (3)	0.040 (4)	0.036 (3)	0.002 (3)	0.001 (3)	0.000 (3)
C4	0.042 (3)	0.047 (4)	0.037 (3)	-0.001 (3)	0.004 (3)	-0.001 (3)
C5	0.043 (3)	0.036 (4)	0.035 (3)	0.004 (3)	-0.001 (3)	-0.005 (3)
C6	0.040 (3)	0.036 (4)	0.042 (3)	0.005 (3)	-0.004 (3)	-0.002 (3)
C11	0.034 (3)	0.035 (4)	0.042 (3)	0.001 (3)	-0.004 (3)	0.001 (3)
C12	0.041 (3)	0.043 (4)	0.037 (3)	0.000 (3)	0.001 (2)	-0.003 (3)
C13	0.042 (3)	0.040 (4)	0.042 (3)	0.006 (3)	0.001 (3)	0.001 (3)
C14	0.061 (4)	0.057 (5)	0.036 (3)	0.008 (4)	-0.006 (3)	-0.001 (3)
C15	0.067 (4)	0.044 (4)	0.055 (4)	0.002 (4)	-0.005 (3)	-0.002 (3)
C16	0.048 (3)	0.048 (4)	0.038 (3)	-0.008 (3)	0.003 (3)	0.002 (3)
C17	0.037 (3)	0.033 (3)	0.049 (4)	0.006 (3)	0.000 (3)	-0.004 (3)
C21	0.037 (3)	0.026 (3)	0.044 (4)	-0.004 (3)	-0.003 (3)	-0.001 (3)

Geometric parameters (Å, °)

C17—N1	1.356 (8)	C16—H16	0.9500
C17—O1	1.232 (7)	C1—C6	1.394 (8)
N1—C11	1.399 (7)	C1—C2	1.417 (8)
N1—H1	0.8800	C1—C17	1.522 (8)
C11—C12	1.377 (9)	C2—C3	1.384 (7)
C11—C16	1.380 (9)	C2—C21	1.480 (8)
C12—C13	1.375 (8)	C21—O21	1.338 (7)
C12—H12	0.9500	C21—O22	1.229 (7)
C13—C14	1.385 (10)	O21—H21	0.8400
C13—N13	1.470 (8)	C3—C4	1.376 (9)
N13—O31	1.222 (8)	C3—H3	0.9500
N13—O32	1.239 (7)	C4—C5	1.385 (9)
C14—C15	1.397 (10)	C4—H4	0.9500
C14—H14	0.9500	C5—C6	1.363 (8)
C15—C16	1.371 (8)	C5—H5	0.9500
C15—H15	0.9500	C6—H6	0.9500
C17—N1—C11	127.0 (5)	C6—C1—C17	118.5 (5)
C17—N1—H1	116.5	C2—C1—C17	122.6 (5)
C11—N1—H1	116.5	C3—C2—C1	119.1 (5)
C12—C11—C16	120.4 (6)	C3—C2—C21	120.7 (5)
C12—C11—N1	123.1 (6)	C1—C2—C21	120.2 (5)
C16—C11—N1	116.5 (6)	O22—C21—O21	121.9 (5)
C13—C12—C11	118.3 (6)	O22—C21—C2	125.4 (5)
C13—C12—H12	120.9	O21—C21—C2	112.7 (5)
C11—C12—H12	120.9	C21—O21—H21	109.5
C12—C13—C14	123.3 (6)	C4—C3—C2	120.4 (6)
C12—C13—N13	118.5 (6)	C4—C3—H3	119.8
C14—C13—N13	118.3 (5)	C2—C3—H3	119.8
O31—N13—O32	122.7 (6)	C3—C4—C5	120.9 (6)

O31—N13—C13	118.4 (5)	C3—C4—H4	119.6
O32—N13—C13	118.9 (6)	C5—C4—H4	119.6
C13—C14—C15	116.7 (6)	C6—C5—C4	119.5 (5)
C13—C14—H14	121.6	C6—C5—H5	120.3
C15—C14—H14	121.6	C4—C5—H5	120.3
C16—C15—C14	121.0 (7)	C5—C6—C1	121.3 (6)
C16—C15—H15	119.5	C5—C6—H6	119.4
C14—C15—H15	119.5	C1—C6—H6	119.4
C15—C16—C11	120.3 (6)	O1—C17—N1	124.6 (5)
C15—C16—H16	119.8	O1—C17—C1	119.2 (5)
C11—C16—H16	119.8	N1—C17—C1	116.1 (5)
C6—C1—C2	118.9 (5)		
C12—C11—N1—C17	-31.8 (9)	C6—C1—C2—C3	1.3 (8)
C11—N1—C17—C1	-168.3 (5)	C17—C1—C2—C3	179.2 (5)
N1—C17—C1—C2	72.8 (6)	C6—C1—C2—C21	-174.6 (5)
C1—C2—C21—O21	-175.9 (5)	C17—C1—C2—C21	3.3 (8)
C12—C13—N13—O31	-4.0 (9)	C3—C2—C21—O22	-172.0 (5)
C17—N1—C11—C16	147.9 (6)	C1—C2—C21—O22	3.8 (8)
C16—C11—C12—C13	-2.2 (8)	C3—C2—C21—O21	8.2 (7)
N1—C11—C12—C13	177.6 (5)	C1—C2—C3—C4	0.2 (8)
C11—C12—C13—C14	1.2 (9)	C21—C2—C3—C4	176.1 (5)
C11—C12—C13—N13	-179.7 (5)	C2—C3—C4—C5	-1.5 (9)
C14—C13—N13—O31	175.2 (7)	C3—C4—C5—C6	1.1 (9)
C12—C13—N13—O32	176.9 (6)	C4—C5—C6—C1	0.4 (8)
C14—C13—N13—O32	-3.9 (8)	C2—C1—C6—C5	-1.7 (8)
C12—C13—C14—C15	0.2 (9)	C17—C1—C6—C5	-179.7 (5)
N13—C13—C14—C15	-178.9 (6)	C11—N1—C17—O1	14.5 (10)
C13—C14—C15—C16	-0.6 (10)	C6—C1—C17—O1	68.1 (7)
C14—C15—C16—C11	-0.3 (10)	C2—C1—C17—O1	-109.9 (7)
C12—C11—C16—C15	1.7 (9)	C6—C1—C17—N1	-109.3 (7)
N1—C11—C16—C15	-178.0 (6)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O22 ⁱ	0.88	1.99	2.857 (6)	168
O21—H21 \cdots O1 ⁱⁱ	0.84	1.84	2.624 (6)	155

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