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# Conformational dimorphism of 2,2'-methylenebis(isoindoline-1,3-dione)

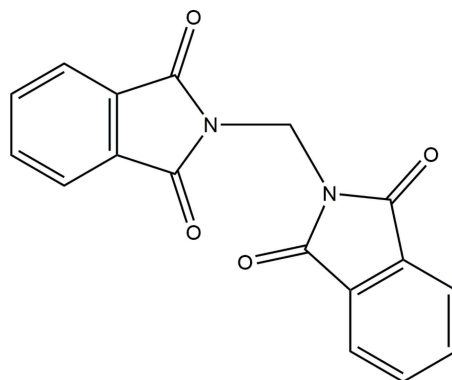
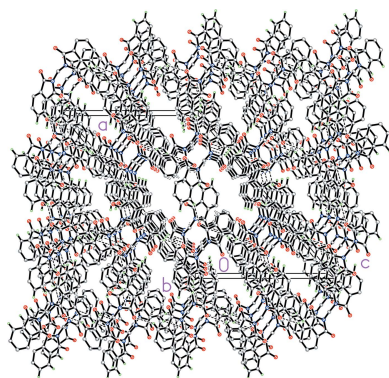
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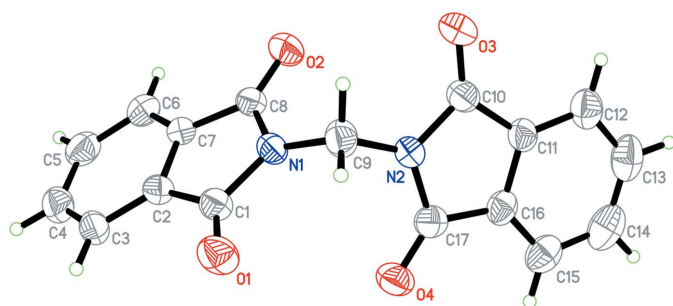
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In this study, a new monoclinic polymorph (space group  $C2/c$ ) of 2,2'-methylenebis(isoindoline-1,3-dione),  $C_{17}H_{10}N_2O_4$ , is reported and compared to the previously reported triclinic polymorph (space group  $P\bar{1}$ ). Similarly, both polymorphs consist of a unique molecule in the asymmetric unit ( $Z' = 1$ ). The molecular conformations of the two polymorphs are very similar, as shown by the r.m.s. deviation of 0.368 Å (excluding all H atoms). The intermolecular interactions of both polymorphs are described along with the Hirshfeld surface analysis, and the lattice energies are calculated.

## 1. Chemical context

Phthalimide (or isoindoline-1,3-dione) derivatives with five-membered *N*-heterocycles have been proven to exhibit significant biological and pharmaceutical activities, and have also been used as dyes and heat-resistant polymers in industry (Chidan Kumar *et al.*, 2015; Then *et al.*, 2018). The first reported crystal structure of 2,2'-methylenebis(isoindoline-1,3-dione) ( $1\alpha$ ; Jiang *et al.*, 2007) crystallizes in the centrosymmetric triclinic space group  $P\bar{1}$  [ $a = 7.6660(9)$  Å,  $b = 9.5810(8)$  Å,  $c = 10.2780(6)$  Å,  $\alpha = 104.325(3)^\circ$ ,  $\beta = 99.768(4)^\circ$ ,  $\gamma = 96.030(3)^\circ$ ,  $Z = 2$ ,  $Z' = 1$  and  $V = 712.23(11)$  Å<sup>3</sup>; Cambridge Structural Database (CSD; Groom *et al.*, 2016) refcode SINDID]. In this article, we report the second polymorphic form ( $1\beta$ ) of 2,2'-methylenebis(isoindoline-1,3-dione) with  $Z' = 1$  and compare its properties with those of  $1\alpha$ . According to the Online Dictionary of Crystallography, polymorphism is the phenomenon in which the same chemical compound exhibits different crystal structures (IUCr, 2018).





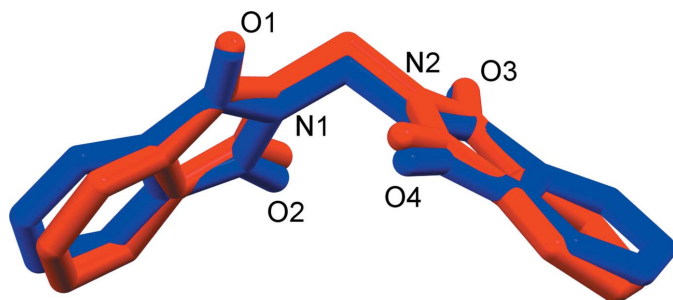
**Figure 1**  
Molecular structure of  $1\beta$  with atom labels and 30% probability displacement ellipsoids.

## 2. Structural commentary

The asymmetric units of polymorphs  $1\alpha$  and  $1\beta$  (Fig. 1) each contain a unique molecule of 2,2'-methylenebis(isoindoline-1,3-dione), which consists of two phthalimide groups and a methylene bridge. The phthalimide groups are nearly planar with maximum deviations of 0.029 (1) and 0.059 (1) Å for  $1\alpha$ , and 0.040 (4) and 0.064 (3) Å for  $1\beta$ . There are two degrees of freedom to characterize the molecular conformations of  $1\alpha$  and  $1\beta$ : these are the torsion angles C1–N1–C9–N2 [106.7 (1) and 117.4 (3)°, respectively] and N1–C9–N2–C10 [109.2 (1) and 117.6 (3)°, respectively]. Generally, the molecule of  $1\beta$  deviates only slightly from that of  $1\alpha$ , as indicated by a r.m.s. deviation of 0.368 Å (excluding all H atoms) (Fig. 2). The mean planes of the phthalimide rings for  $1\beta$  make a dihedral angle of 76.12 (8)°, which is smaller than that of 88.96 (4)° observed in polymorph  $1\alpha$ . The calculated density and Kitaigorodskii packing index (Spek, 2009) for  $1\beta$  (1.469 Mg m<sup>-3</sup> and 70.0%) are slightly higher than those observed for  $1\alpha$  (1.428 Mg m<sup>-3</sup> and 69.0%).

## 3. Supramolecular features

The crystal packing of  $1\alpha$  features weak intermolecular C–H···O hydrogen bonds and  $\pi$ – $\pi$  interactions between neighboring phthalimide units. In the crystal structure of  $1\beta$  (Fig. 3), the molecules are connected by weak intermolecular C–H···O hydrogen bonds (Table 1), forming a three-dimensional network. The crystal structure of  $1\beta$  also features weak  $\pi$ – $\pi$



**Figure 2**  
An overlay diagram for the molecules of  $1\alpha$  (red) and  $1\beta$  (blue).

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

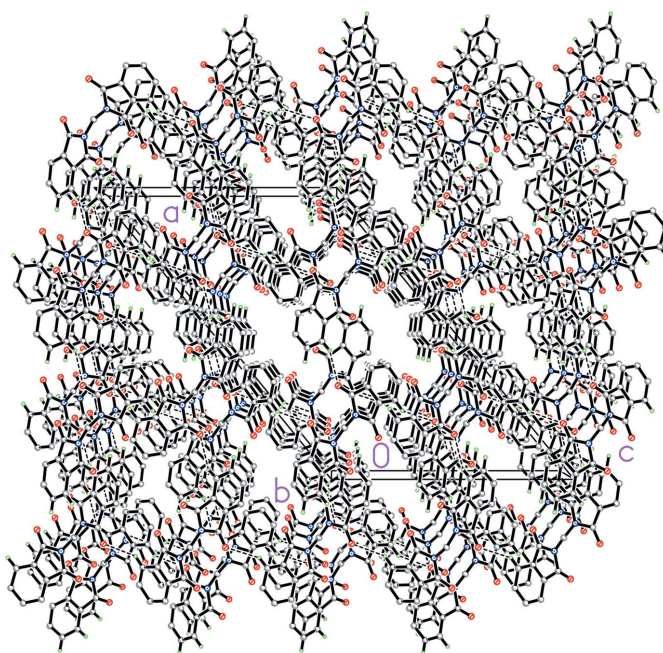
<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C3–H3A···O3 <sup>i</sup>	0.93	2.43	3.150 (6)	134
C4–H4A···O4 <sup>iii</sup>	0.93	2.60	3.300 (5)	133
C15–H15A···O2 <sup>iii</sup>	0.93	2.46	3.271 (7)	146

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

interactions between two C2–C7 phenyl rings (symmetry code:  $-x, -y + 1, -z$ ) and between N1/C1/C2/C7/C8 and C11–C16 rings (symmetry codes:  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$  and  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ ), with centroid-to-centroid distances of 3.664 (3) and 3.938 (3) Å, respectively.

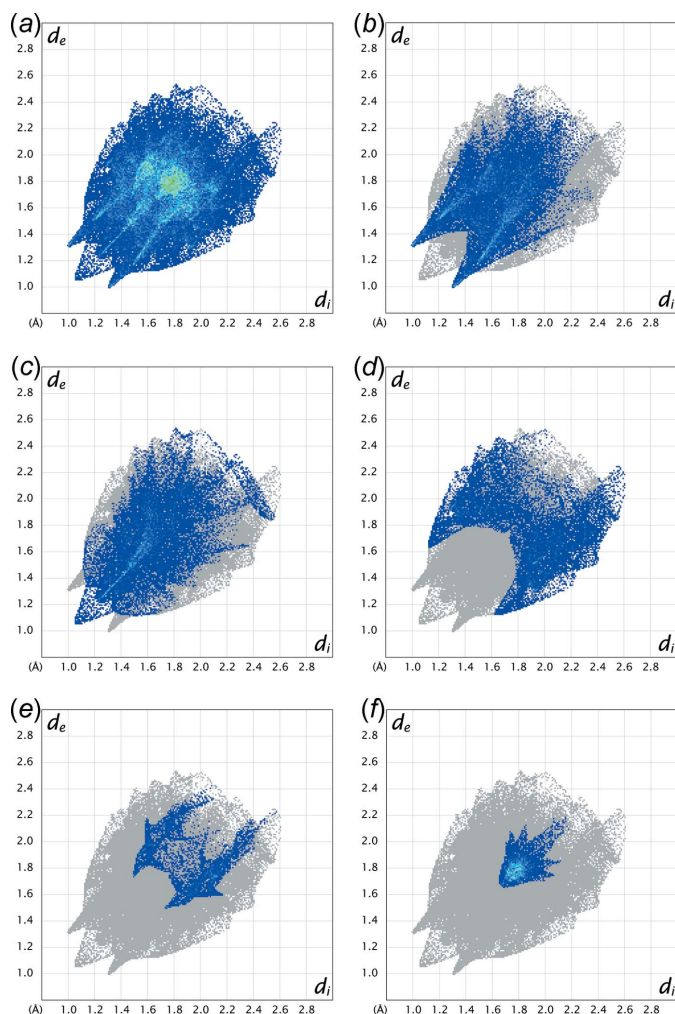
## 4. Hirshfeld surface analysis

The Hirshfeld surface analysis and two-dimensional fingerprint plots were performed using *CrystalExplorer* version 17.5 (Spackman & Jayatilaka, 2009; Spackman & McKinnon, 2002; Turner *et al.*, 2017). The H···O/O···H contact is the most populated contact and contributes 38.2 and 34.4% of the total intermolecular contacts of  $1\alpha$  and  $1\beta$  (Fig. 4), respectively. The large red spots on the Hirshfeld surface mapped over  $d_{\text{norm}}$  for  $1\beta$  (Fig. 5) correspond to the intermolecular C3–H3A···O3 and C15–H15A···O2 hydrogen-bonds. The tips of the pseudo-mirrored sharp spikes at  $d_e + d_i \approx 2.32$  Å represent the shortest H···O/O···H contacts, corresponding to the intermolecular C3–H3A···O3 hydrogen-bond. The H···H contact is the second most populated contact and contributes



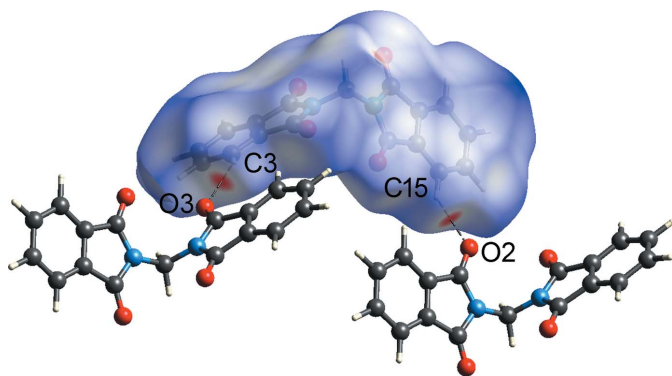
**Figure 3**  
A partial crystal packing diagram of  $1\beta$  viewed along the *b* axis. Dashed lines represent weak intermolecular C–H···O hydrogen bonds. Hydrogen atoms which are not involved in hydrogen bonding are omitted for clarity.



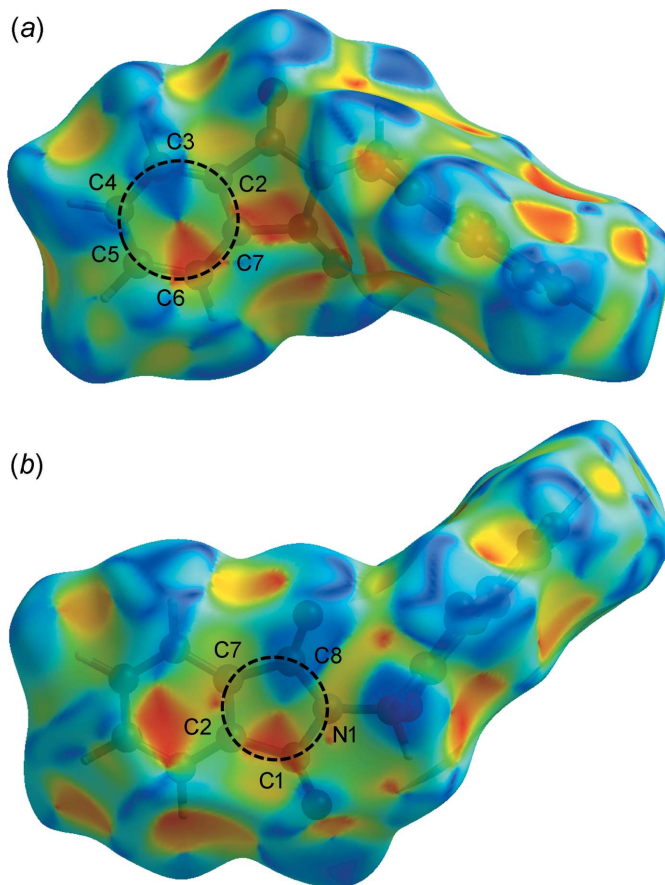


**Figure 4**  
The two-dimensional fingerprint plots of  $1\beta$  for (a) all, (b)  $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$ , (c)  $\text{H}\cdots\text{H}$ , (d)  $\text{H}\cdots\text{C}/\text{C}\cdots\text{H}$ , (e)  $\text{O}\cdots\text{C}/\text{C}\cdots\text{O}$  and (f)  $\text{C}\cdots\text{C}$  contacts.  $d_i$  and  $d_e$  are the distances from the Hirshfeld surface to the nearest atom interior and exterior, respectively, to the surface.

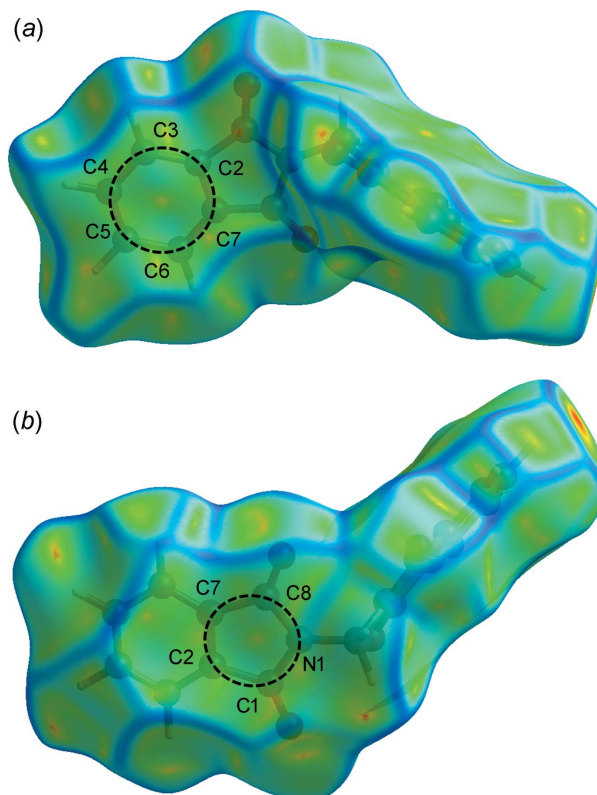
25.4 and 26.5% of the total intermolecular contacts of  $1\alpha$  and  $1\beta$ , respectively. The shortest  $\text{H}\cdots\text{H}$  contacts of  $1\alpha$  (symmetry code:  $-x, -y, -z + 1$ ) and  $1\beta$  (symmetry code:  $-x, y, -z - \frac{1}{2}$ )



**Figure 5**  
The Hirshfeld surface mapped over  $d_{\text{norm}}$  for the molecule in the asymmetric unit of  $1\beta$  hydrogen-bonded to two neighbouring molecules.



**Figure 6**  
The Hirshfeld surface mapped over shape-index for  $1\beta$ .



**Figure 7**  
The Hirshfeld surface mapped over curvedness for  $1\beta$ .

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>17</sub> H <sub>10</sub> N <sub>2</sub> O <sub>4</sub>
<i>M</i> <sub>r</sub>	306.27
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	26.296 (5), 7.9996 (15), 16.987 (4)
β (°)	129.165 (10)
<i>V</i> (Å <sup>3</sup> )	2770.5 (10)
<i>Z</i>	8
Radiation type	Mo Kα
μ (mm <sup>-1</sup> )	0.11
Crystal size (mm)	0.44 × 0.13 × 0.02
Data collection	
Diffractometer	Bruker SMART APEXII DUO CCD area-detector
Absorption correction	Multi-scan (SADABS; Bruker, 2009)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.649, 0.745
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	29675, 2444, 1213
<i>R</i> <sub>int</sub>	0.128
(sin θ/λ) <sub>max</sub> (Å <sup>-1</sup> )	0.595
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.063, 0.158, 1.02
No. of reflections	2444
No. of parameters	208
H-atom treatment	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.14, -0.17

Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXS2013 and SHELXTL (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), Mercury (Macrae *et al.*, 2008), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

are illustrated as two sharp tips along the diagonal of their two-dimensional fingerprint plots at  $d_e \simeq d_i \simeq 1.06$  and  $1.21$  Å [Fig. 4(c)], respectively. The percentages of contribution of H...C/C...H, O...C/C...O and C...C contacts to the Hirshfeld surface are 20.6, 3.3 and 8.9%, respectively, for 1α, and 20.8, 7.9 and 6.7%, respectively, for 1β (Fig. 4). The absence of significant C—H...π interactions in the crystal structure of 1β is indicated by the absence of characteristic ‘wings’ in the fingerprint plot of H...C/C...H contacts [Fig. 4(d)]. The C...C contacts appear as a unique ‘triangle’ focused at  $d_e \simeq d_i \simeq 1.75$  Å [Fig. 4(f)]. The intermolecular π–π interactions are illustrated as unique patterns of red and blue ‘triangles’ on the shape-index surface (Fig. 6), and flat regions on the curvedness surface (Fig. 7), of the C2–C7, N1/C1/C2/C7/C8 and C11–C16 rings.

## 5. Lattice energy calculation

The C—H bond lengths in 1α and 1β were normalized to 1.08 Å and the lattice energies were calculated by using the CLP-PIXEL software package (Gavezzotti, 2003, 2008). The calculated lattice energy of 1α (130.3 kJ mol<sup>-1</sup>) is slightly larger than for 1β (128.5 kJ mol<sup>-1</sup>), indicating that 1α is slightly more stable than 1β under ambient conditions.

## 6. Synthesis and crystallization

Single crystals of 1β were obtained from an unsuccessful synthesis of 2-[(3-iodopyridin-4-yl)amino]methylisoindoline-1,3-dione by reacting *N*-(bromomethyl)phthalimide (1 mmol) and 4-amino-3-iodopyridine (1 mmol) in *N,N*-dimethylformamide (8 ml) with the presence of a catalytic amount of anhydrous potassium carbonate. The reaction solution was stirred for about 2 h at room temperature. Once the reaction was complete, the resultant mixture was poured into a beaker of ice-cooled water to obtain a precipitate (Then *et al.*, 2018), which was then filtered, washed with distilled water and dried. Crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

## 7. Refinement

Crystal data, data collection and structure refinement details of 1β are summarized in Table 2. All H atoms were positioned geometrically (C—H = 0.93 and 0.97 Å) and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

## Funding information

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## supporting information

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## Conformational dimorphism of 2,2'-methylenebis(isoindoline-1,3-dione)

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## Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE* (Bruker, 2009); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

## 2,2'-Methylenebis(isoindoline-1,3-dione)

## Crystal data

$C_{17}H_{10}N_2O_4$

$M_r = 306.27$

Monoclinic, *C2/c*

$a = 26.296$  (5) Å

$b = 7.9996$  (15) Å

$c = 16.987$  (4) Å

$\beta = 129.165$  (10)°

$V = 2770.5$  (10) Å<sup>3</sup>

$Z = 8$

$F(000) = 1264$

$D_x = 1.469$  Mg m<sup>-3</sup>

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

Cell parameters from 1601 reflections

$\theta = 2.4$ – $26.4$ °

$\mu = 0.11$  mm<sup>-1</sup>

$T = 296$  K

Block, colourless

$0.44 \times 0.13 \times 0.02$  mm

## Data collection

Bruker SMART APEXII DUO CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.649$ ,  $T_{\max} = 0.745$

29675 measured reflections

2444 independent reflections

1213 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.128$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.0$ °

$h = -31 \rightarrow 31$

$k = -9 \rightarrow 9$

$l = -20 \rightarrow 20$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.063$

$wR(F^2) = 0.158$

$S = 1.02$

2444 reflections

208 parameters

0 restraints

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.5204P]$

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

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.14$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.17$  e Å<sup>-3</sup>

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.05520 (11)	0.9015 (3)	0.12678 (18)	0.0786 (8)
O2	0.20385 (12)	0.6374 (3)	0.11771 (17)	0.0769 (8)
O3	0.32974 (11)	0.7796 (3)	0.34998 (18)	0.0817 (8)
O4	0.15774 (12)	0.6563 (3)	0.34093 (18)	0.0765 (8)
N1	0.14191 (12)	0.7785 (3)	0.14765 (19)	0.0563 (8)
N2	0.23369 (12)	0.7434 (3)	0.32431 (19)	0.0577 (8)
C1	0.07580 (16)	0.8101 (4)	0.0966 (3)	0.0595 (9)
C2	0.04023 (16)	0.7104 (4)	0.0028 (2)	0.0547 (9)
C3	-0.02562 (16)	0.6944 (4)	-0.0726 (2)	0.0620 (10)
H3A	-0.0554	0.7553	-0.0718	0.074*
C4	-0.04608 (18)	0.5854 (5)	-0.1492 (3)	0.0777 (11)
H4A	-0.0908	0.5706	-0.2014	0.093*
C5	-0.0021 (2)	0.4972 (5)	-0.1507 (3)	0.0798 (12)
H5A	-0.0176	0.4222	-0.2033	0.096*
C6	0.06535 (19)	0.5173 (5)	-0.0753 (3)	0.0697 (10)
H6A	0.0955	0.4600	-0.0768	0.084*
C7	0.08469 (15)	0.6259 (4)	0.0011 (2)	0.0531 (9)
C8	0.15090 (17)	0.6749 (4)	0.0923 (2)	0.0568 (9)
C9	0.19381 (15)	0.8576 (5)	0.2405 (2)	0.0671 (10)
H9A	0.1752	0.9399	0.2578	0.081*
H9B	0.2216	0.9165	0.2305	0.081*
C10	0.30011 (17)	0.7197 (5)	0.3748 (3)	0.0625 (10)
C11	0.32396 (17)	0.6090 (4)	0.4617 (2)	0.0591 (9)
C12	0.38561 (19)	0.5506 (5)	0.5381 (3)	0.0765 (11)
H12A	0.4214	0.5788	0.5418	0.092*
C13	0.3923 (2)	0.4479 (6)	0.6095 (3)	0.0930 (14)
H13A	0.4336	0.4071	0.6628	0.112*
C14	0.3398 (3)	0.4050 (5)	0.6036 (3)	0.0956 (14)
H14A	0.3458	0.3341	0.6522	0.115*
C15	0.2779 (2)	0.4654 (5)	0.5263 (3)	0.0784 (11)
H15A	0.2420	0.4371	0.5222	0.094*
C16	0.27123 (18)	0.5674 (4)	0.4564 (3)	0.0607 (9)
C17	0.21289 (18)	0.6552 (4)	0.3696 (3)	0.0590 (9)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0699 (17)	0.0814 (19)	0.0879 (18)	0.0089 (14)	0.0514 (15)	-0.0122 (15)
O2	0.0556 (16)	0.101 (2)	0.0781 (17)	0.0107 (14)	0.0441 (14)	0.0021 (15)

O3	0.0572 (16)	0.107 (2)	0.0841 (18)	-0.0104 (14)	0.0462 (15)	0.0008 (15)
O4	0.0587 (16)	0.094 (2)	0.0815 (17)	-0.0156 (14)	0.0465 (14)	-0.0159 (14)
N1	0.0423 (17)	0.072 (2)	0.0512 (17)	0.0008 (14)	0.0278 (15)	-0.0056 (15)
N2	0.0450 (18)	0.073 (2)	0.0508 (17)	-0.0036 (15)	0.0279 (15)	0.0004 (15)
C1	0.054 (2)	0.064 (3)	0.063 (2)	0.0040 (19)	0.038 (2)	0.003 (2)
C2	0.051 (2)	0.058 (2)	0.056 (2)	-0.0022 (18)	0.0348 (19)	0.0027 (18)
C3	0.049 (2)	0.068 (3)	0.058 (2)	0.0026 (18)	0.029 (2)	0.007 (2)
C4	0.060 (2)	0.093 (3)	0.062 (3)	-0.010 (2)	0.030 (2)	0.003 (2)
C5	0.089 (3)	0.093 (3)	0.063 (3)	-0.020 (3)	0.050 (3)	-0.010 (2)
C6	0.076 (3)	0.082 (3)	0.060 (2)	0.001 (2)	0.047 (2)	-0.002 (2)
C7	0.048 (2)	0.067 (2)	0.048 (2)	0.0001 (18)	0.0316 (18)	0.0034 (18)
C8	0.049 (2)	0.067 (2)	0.059 (2)	0.0068 (19)	0.036 (2)	0.0113 (19)
C9	0.059 (2)	0.072 (3)	0.057 (2)	-0.0047 (19)	0.030 (2)	-0.001 (2)
C10	0.052 (2)	0.075 (3)	0.061 (2)	-0.008 (2)	0.036 (2)	-0.012 (2)
C11	0.054 (2)	0.067 (2)	0.049 (2)	-0.0031 (19)	0.029 (2)	-0.0094 (19)
C12	0.070 (3)	0.080 (3)	0.063 (3)	0.009 (2)	0.034 (2)	-0.008 (2)
C13	0.091 (3)	0.090 (3)	0.063 (3)	0.022 (3)	0.032 (3)	0.003 (2)
C14	0.135 (4)	0.070 (3)	0.077 (3)	0.010 (3)	0.065 (3)	0.001 (2)
C15	0.103 (3)	0.065 (3)	0.075 (3)	-0.010 (2)	0.060 (3)	-0.012 (2)
C16	0.069 (2)	0.057 (2)	0.051 (2)	-0.009 (2)	0.036 (2)	-0.0096 (19)
C17	0.063 (2)	0.059 (2)	0.060 (2)	-0.014 (2)	0.041 (2)	-0.0183 (19)

*Geometric parameters (Å, °)*

O1—C1	1.201 (4)	C5—H5A	0.9300
O2—C8	1.207 (3)	C6—C7	1.365 (4)
O3—C10	1.195 (4)	C6—H6A	0.9300
O4—C17	1.203 (4)	C7—C8	1.476 (4)
N1—C8	1.381 (4)	C9—H9A	0.9700
N1—C1	1.391 (4)	C9—H9B	0.9700
N1—C9	1.425 (4)	C10—C11	1.480 (5)
N2—C17	1.386 (4)	C11—C12	1.369 (4)
N2—C10	1.389 (4)	C11—C16	1.372 (4)
N2—C9	1.441 (4)	C12—C13	1.380 (5)
C1—C2	1.473 (4)	C12—H12A	0.9300
C2—C3	1.362 (4)	C13—C14	1.363 (5)
C2—C7	1.366 (4)	C13—H13A	0.9300
C3—C4	1.364 (5)	C14—C15	1.382 (5)
C3—H3A	0.9300	C14—H14A	0.9300
C4—C5	1.369 (5)	C15—C16	1.358 (5)
C4—H4A	0.9300	C15—H15A	0.9300
C5—C6	1.394 (5)	C16—C17	1.470 (5)
C8—N1—C1	111.6 (3)	N1—C9—N2	113.7 (3)
C8—N1—C9	124.3 (3)	N1—C9—H9A	108.8
C1—N1—C9	123.8 (3)	N2—C9—H9A	108.8
C17—N2—C10	111.8 (3)	N1—C9—H9B	108.8
C17—N2—C9	125.1 (3)	N2—C9—H9B	108.8



C10—N2—C9	122.9 (3)	H9A—C9—H9B	107.7
O1—C1—N1	124.6 (3)	O3—C10—N2	125.0 (4)
O1—C1—C2	130.0 (3)	O3—C10—C11	129.2 (3)
N1—C1—C2	105.4 (3)	N2—C10—C11	105.8 (3)
C3—C2—C7	122.1 (3)	C12—C11—C16	121.5 (4)
C3—C2—C1	129.0 (3)	C12—C11—C10	130.7 (4)
C7—C2—C1	108.9 (3)	C16—C11—C10	107.8 (3)
C2—C3—C4	117.3 (3)	C11—C12—C13	117.0 (4)
C2—C3—H3A	121.4	C11—C12—H12A	121.5
C4—C3—H3A	121.4	C13—C12—H12A	121.5
C3—C4—C5	121.3 (3)	C14—C13—C12	121.6 (4)
C3—C4—H4A	119.4	C14—C13—H13A	119.2
C5—C4—H4A	119.4	C12—C13—H13A	119.2
C4—C5—C6	121.5 (4)	C13—C14—C15	120.8 (4)
C4—C5—H5A	119.3	C13—C14—H14A	119.6
C6—C5—H5A	119.3	C15—C14—H14A	119.6
C7—C6—C5	116.2 (3)	C16—C15—C14	117.7 (4)
C7—C6—H6A	121.9	C16—C15—H15A	121.1
C5—C6—H6A	121.9	C14—C15—H15A	121.1
C6—C7—C2	121.6 (3)	C15—C16—C11	121.4 (4)
C6—C7—C8	130.6 (3)	C15—C16—C17	129.8 (4)
C2—C7—C8	107.7 (3)	C11—C16—C17	108.8 (3)
O2—C8—N1	124.1 (3)	O4—C17—N2	124.6 (3)
O2—C8—C7	129.7 (3)	O4—C17—C16	129.6 (4)
N1—C8—C7	106.2 (3)	N2—C17—C16	105.8 (3)
C8—N1—C1—O1	-177.0 (3)	C17—N2—C9—N1	-67.9 (4)
C9—N1—C1—O1	-3.1 (5)	C10—N2—C9—N1	117.6 (3)
C8—N1—C1—C2	2.8 (3)	C17—N2—C10—O3	178.7 (3)
C9—N1—C1—C2	176.7 (3)	C9—N2—C10—O3	-6.2 (5)
O1—C1—C2—C3	-1.7 (6)	C17—N2—C10—C11	-1.4 (3)
N1—C1—C2—C3	178.5 (3)	C9—N2—C10—C11	173.8 (3)
O1—C1—C2—C7	179.8 (4)	O3—C10—C11—C12	2.6 (6)
N1—C1—C2—C7	0.0 (3)	N2—C10—C11—C12	-177.4 (3)
C7—C2—C3—C4	2.1 (5)	O3—C10—C11—C16	-177.6 (4)
C1—C2—C3—C4	-176.2 (3)	N2—C10—C11—C16	2.5 (4)
C2—C3—C4—C5	-0.7 (5)	C16—C11—C12—C13	0.0 (5)
C3—C4—C5—C6	-1.2 (6)	C10—C11—C12—C13	179.8 (3)
C4—C5—C6—C7	1.7 (5)	C11—C12—C13—C14	0.7 (6)
C5—C6—C7—C2	-0.3 (5)	C12—C13—C14—C15	-1.0 (6)
C5—C6—C7—C8	179.1 (3)	C13—C14—C15—C16	0.5 (6)
C3—C2—C7—C6	-1.6 (5)	C14—C15—C16—C11	0.3 (5)
C1—C2—C7—C6	177.0 (3)	C14—C15—C16—C17	-177.0 (3)
C3—C2—C7—C8	178.8 (3)	C12—C11—C16—C15	-0.6 (5)
C1—C2—C7—C8	-2.5 (3)	C10—C11—C16—C15	179.6 (3)
C1—N1—C8—O2	175.4 (3)	C12—C11—C16—C17	177.2 (3)
C9—N1—C8—O2	1.5 (5)	C10—C11—C16—C17	-2.6 (4)
C1—N1—C8—C7	-4.3 (3)	C10—N2—C17—O4	179.2 (3)



C9—N1—C8—C7	-178.1 (3)	C9—N2—C17—O4	4.2 (5)
C6—C7—C8—O2	5.1 (6)	C10—N2—C17—C16	-0.2 (3)
C2—C7—C8—O2	-175.5 (3)	C9—N2—C17—C16	-175.2 (3)
C6—C7—C8—N1	-175.3 (3)	C15—C16—C17—O4	0.0 (6)
C2—C7—C8—N1	4.1 (3)	C11—C16—C17—O4	-177.6 (3)
C8—N1—C9—N2	-69.4 (4)	C15—C16—C17—N2	179.3 (3)
C1—N1—C9—N2	117.4 (3)	C11—C16—C17—N2	1.8 (4)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3 <i>A</i> ...O3 <sup>i</sup>	0.93	2.43	3.150 (6)	134
C4—H4 <i>A</i> ...O4 <sup>ii</sup>	0.93	2.60	3.300 (5)	133
C15—H15 <i>A</i> ...O2 <sup>iii</sup>	0.93	2.46	3.271 (7)	146

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