organic compounds

Isomeric nitrophthalimides: sheets built from N—H⋯O and C—H⋯O hydrogen bonds

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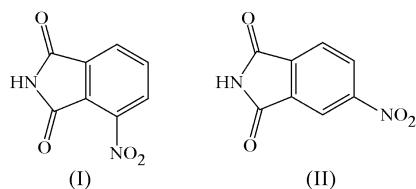
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Molecules of 3-nitrophthalimide, $C_8H_4N_2O_4$, are linked into sheets by a combination of one N—H⋯O hydrogen bond [H⋯O = 1.99 Å, N⋯O = 2.8043 (14) Å and N—H⋯O = 176°] and two independent C—H⋯O hydrogen bonds [H⋯O = 2.36 and 2.56 Å, C⋯O = 3.1639 (16) and 3.4386 (16) Å, and C—H⋯O = 142 and 153°], and these sheets are linked into pairs by a single π–π stacking interaction. Molecules of isomeric 4-nitrophthalimide are linked into sheets by a combination of one three-centre N—H⋯(O)₂ hydrogen bond [H⋯O = 2.14 and 2.55 Å, N⋯O = 2.974 (3) and 3.231 (3) Å, N—H⋯O = 151 and 131°, and O⋯H⋯O = 76°] and two independent two-centre C—H⋯O hydrogen bonds [H⋯O = 2.38 and 2.54 Å, C⋯O = 3.257 (4) and 3.452 (4) Å, and C—H⋯O = 156 and 168°].

Comment

The isomeric title compounds 3-nitrophthalimide, (I), and 4-nitrophthalimide, (II), contain, within very compact molecules, a wide variety of potential hydrogen-bond donors and acceptors (Fig. 1). Both N—H and C—H bonds provide potential donors, while carbonyl and nitro O atoms and the arene ring all provide potential acceptors, although there is a
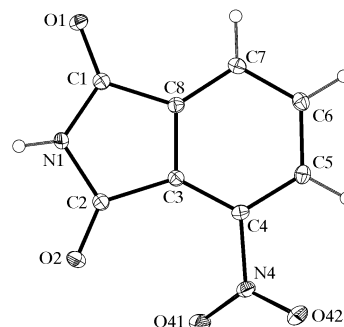
significant excess of hard acceptors over hard donors (Braga *et al.*, 1995; Desiraju & Steiner, 1999). In addition, aromatic π–π stacking interactions and non-bonded dipolar interactions

(Allen *et al.*, 1998) involving both carbonyl and nitro groups are possible, in principle.
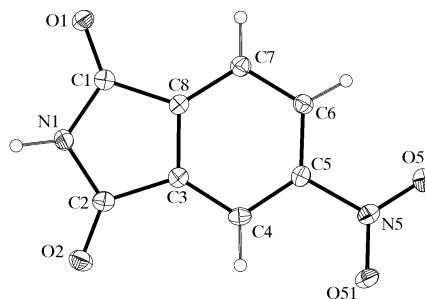
In the event, the supramolecular structure of (I) (Fig. 1) is dominated by a two-centre N—H⋯O hydrogen bond, in which the acceptor is a carbonyl O atom, and two independent C—H⋯O hydrogen bonds, one involving a carbonyl O atom and the other a nitro O atom as acceptor. By contrast, the supramolecular structure of (II) (Fig. 2) is dominated by one asymmetric three-centre N—H⋯(O)₂ hydrogen bond, involving both of the carbonyl O atoms as the acceptors, and two independent two-centre C—H⋯O hydrogen bonds, one involving a carbonyl O atom and the other a nitro O atom as acceptor.

In isomer (I), each of the independent hydrogen bonds (Table 1) can be regarded as producing a one-dimensional substructure (Gregson *et al.*, 2000), all parallel to the [100] direction but all generated by different symmetry operations; the combination of these three motifs generates a sheet. The formation of the sheets in (I) is most readily analysed by considering, in turn, the action of each hydrogen bond. Amine atom N1 in the molecule at (x, y, z) acts as a hydrogen-bond donor to carbonyl atom O1 in the molecule at $(\frac{1}{2} + x, y, \frac{1}{2} - z)$, so producing a C(4) chain running parallel to the [100] direction and generated by the *a*-glide plane at $z = \frac{1}{4}$ (Fig. 3).

At the same time, aryl atom C6 in the molecule at (x, y, z) acts as a hydrogen-bond donor to the second carbonyl O atom, O2, in the molecule at (−1 + x, y, z), thus generating by translation a C(7) chain along [100]. The combination of the two motifs having carbonyl acceptors generates a column of $R_3^2(14)$ rings across $z = \frac{1}{4}$ (Fig. 3). Finally, aryl atom C5 at (x, y, z) acts as a hydrogen-bond donor to nitro atom O41 in the molecule at $(-\frac{1}{2} + x, y, \frac{3}{2} - z)$, so forming a C(5) chain along [100], this time generated by the *a*-glide plane at $z = \frac{3}{4}$. The
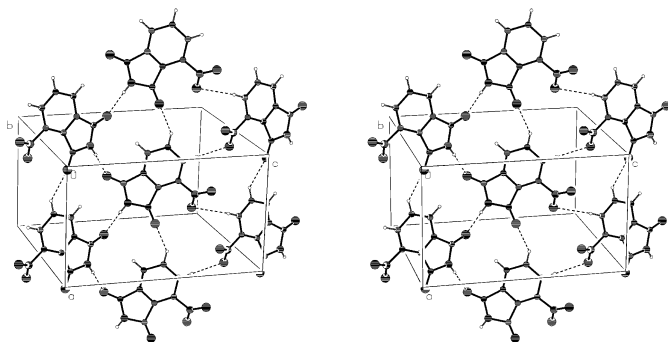
Figure 1
The molecule of isomer (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
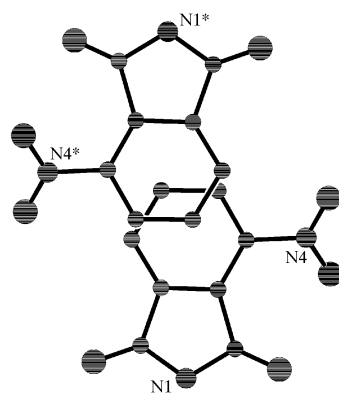
Figure 2
The molecule of isomer (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

combination of the two motifs involving aryl H atoms generates a column of $R_3^3(15)$ rings across $z = \frac{3}{4}$ (Fig. 3). Neither of the rings utilizes all three independent hydrogen bonds. However, the combination of all three hydrogen bonds generates a (010) sheet in which columns of $R_3^3(14)$ rings across $z = n + \frac{1}{4}$ (n = zero or integer) alternate with columns of $R_3^3(15)$ rings across $z = n + \frac{3}{4}$ (n = zero or integer) (Fig. 3). Four sheets of this type pass through each unit cell, and the sheets are linked weakly into pairs by a single aromatic π–π stacking interaction.

The C3–C8 aryl rings in the molecules at (x, y, z) and $(-x, -y, 1 - z)$ are strictly parallel, with an interplanar spacing of 3.309 (2) Å; the ring-centroid separation is 3.758 (2) Å, corresponding to a centroid offset of 1.781 (2) Å (Fig. 4). These two molecules lie in the (010) sheets within the domains $0.02 < y < 0.31$ and $-0.21 < y < -0.02$, respectively, and these two sheets are thus linked into a bilayer. The formation of the bilayer is reinforced by a dipolar interaction between the negatively polarized nitro atom O42 in the molecule at (x, y, z) and the positively polarized carbonyl atom C1 in the molecule at $(\frac{1}{2} - x, -y, \frac{1}{2} + z)$, which forms part of the sheet in the domain $-0.21 < y < -0.02$. The C$\cdots$O^i distance [symmetry code: (i) $\frac{1}{2} - x, -y, \frac{1}{2} + z$] is 2.980 (2) Å and the C$\cdots$O^i—N^i angle is 135.9 (2)°. However, there are no direction-specific interactions between adjacent bilayers

In isomer (II), the hard and soft hydrogen bonds independently generate two one-dimensional substructures, each in the form of a chain of rings, and these chains combine to form sheets. Amine atom N1 in the molecule of (II) at (x, y, z) acts as a hydrogen-bond donor to carbonyl atoms O1 and O2 in the molecules at $(2 - x, -\frac{1}{2} + y, \frac{3}{2} - z)$ and $(2 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, respectively. These two interactions (Table 2) are the strong and weak components of a markedly asymmetric, but essentially planar, three-centre interaction. The sum of the angles at atom H1 is 358°. Together these interactions produce a chain of edge-fused $R_2^2(8)$ rings running parallel to the [010] direction and generated by the 2_1 screw axis along $(1, y, \frac{3}{4})$ (Fig. 5).

In addition, aryl atoms C4 and C7 in the molecule at (x, y, z) act as donors, respectively, to atoms O1 and O51 in the molecules at $(-1 + x, -1 + y, z)$ and $(1 + x, 1 + y, z)$, so generating two individual $C(6)$ chains, whose combined action generates by translation a $C(6)C(6)[R_2^2(10)]$ chain of rings (Bernstein *et al.*, 1995) running parallel to the [110] direction (Fig. 6). The combination of the [010] and [110] chains generates a (001) sheet, which is modestly reinforced by a dipolar interaction between the negatively polarized atom O2 at (x, y, z) and the positively polarized atom C2 at $(1 - x, -\frac{1}{2} + y, \frac{3}{2} - z)$. The O2$\cdots$C2^{ii} distance is 2.906 (3) Å [symmetry code: (ii) $1 - x, -\frac{1}{2} + y, \frac{3}{2} - z$] and the C2—O2$\cdots$C2^{ii} angle is 156.1 (2)°, indicating an interaction whose geometry is intermediate between the perpendicular motif I and the sheared motif III described by Allen *et al.* (1998). The two molecules involved in this interaction lie in the [010] chains along $(1, y, \frac{3}{4})$ and $(\frac{1}{2}, y, \frac{3}{4})$, so that this dipolar interaction generates a motif along [100] within the (010) sheet. There are no direction-specific interactions between adjacent sheets.
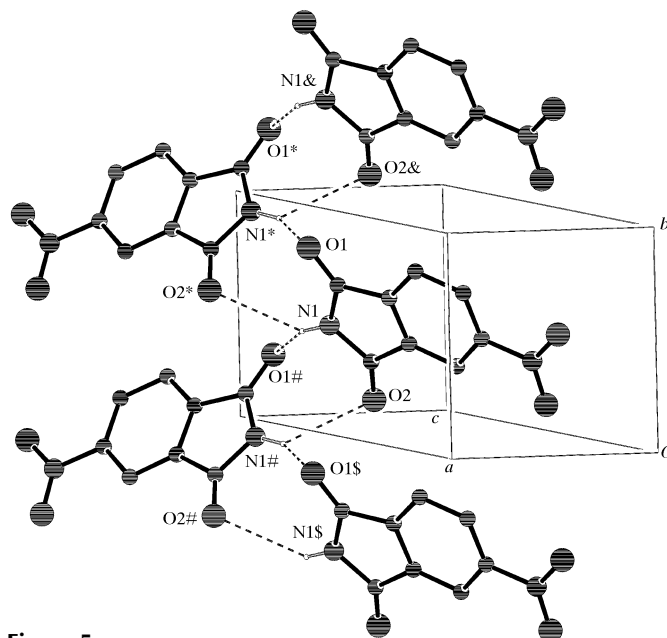

Figure 3
A stereoview of part of the crystal structure of isomer (I), showing the formation of a (010) sheet containing alternating columns of $R_3^3(14)$ and $R_3^3(15)$ rings.
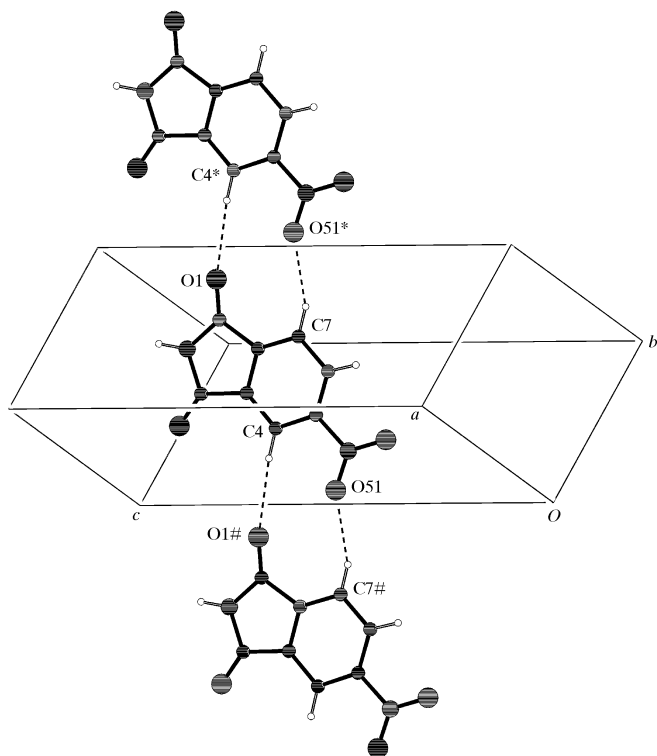

Figure 4
Part of the crystal structure of isomer (I), showing the π–π stacking interaction that links the (010) sheets into pairs. For clarity, H atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x, -y, 1 - z)$.

Figure 5
Part of the crystal structure of isomer (II), showing the formation of a chain of edge-fused rings along [010]. For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), a hash (#), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(2 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, $(2 - x, -\frac{1}{2} + y, \frac{3}{2} - z)$, $(x, -1 + y, z)$ and $(x, 1 + y, z)$, respectively.

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Figure 6
Part of the crystal structure of isomer (II), showing the formation of a chain of rings along [110]. Atoms marked with an asterisk (*) or a hash (#) are in the molecules at $(1 + x, 1 + y, z)$ and $(-1 + x, -1 + y, z)$, respectively.

The bond distances and angles within the molecules of (I) and (II) present no unusual values. The dihedral angle between the planes of the aryl ring and the nitro group is 31.6 (2)° in (I) and 8.0 (2)° in (II), so that each molecule has point group C_1; this is sufficient to render the molecules of both isomers chiral. Isomer (I) crystallizes in the centrosymmetric space group *Pbca*, as a racemate with equal numbers of both conformational enantiomers present in each single-crystal domain. Isomer (II) crystallizes in space group $P2_12_12_1$ as a fully ordered structure, having only one conformational enantiomer present in each single-crystal domain.

Experimental

Commercial samples of 3- and 4-nitrophthalimide were obtained from Aldrich, and crystals suitable for single-crystal X-ray diffraction were grown from solutions in ethyl acetate [for (I)] and acetone [for (II)].

Compound (I)

Crystal data

$C_8H_4N_2O_4$
$M_r = 192.13$
Orthorhombic, *Pbca*
$a = 8.2208$ (2) Å
$b = 13.0786$ (4) Å
$c = 13.9233$ (4) Å
$V = 1496.99$ (7) Å^3
$Z = 8$
$D_x = 1.705$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1703 reflections
$\theta = 3.1–27.5°$
$\mu = 0.14$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
$0.48 \times 0.32 \times 0.08$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.954$, $T_{max} = 0.989$
19 573 measured reflections
1703 independent reflections

1488 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 15$
$l = -18 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.099$
$S = 1.10$
1703 reflections
129 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.5962P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.033 (4)

Table 1
Hydrogen-bonding geometry (Å, °) for (I).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1···O1^{iii}	0.82	1.99	2.8043 (14)	176
C5—H5···O41^{iv}	0.95	2.56	3.4386 (16)	153
C6—H6···O2^{v}	0.95	2.36	3.1639 (16)	142

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

Compound (II)

Crystal data

$C_8H_4N_2O_4$
$M_r = 192.13$
Orthorhombic, $P2_12_12_1$
$a = 5.3114$ (3) Å
$b = 5.6812$ (5) Å
$c = 24.645$ (2) Å
$V = 743.67$ (10) Å^3
$Z = 4$
$D_x = 1.716$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1007 reflections
$\theta = 3.3–27.5°$
$\mu = 0.14$ mm^{-1}
$T = 291$ (2) K
Plate, colourless
$0.26 \times 0.22 \times 0.04$ mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.956$, $T_{max} = 0.994$
6348 measured reflections
1007 independent reflections

804 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 27.5°$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 7$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.110$
$S = 1.03$
1007 reflections
127 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.0553P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 2
Hydrogen-bonding geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1···O2^{vi}	0.92	2.55	3.231 (3)	131
N1—H1···O1^{vii}	0.92	2.14	2.974 (3)	151
C4—H4···O1^{viii}	0.93	2.54	3.452 (4)	168
C7—H7···O51^{ix}	0.93	2.38	3.257 (4)	156

Symmetry codes: (vi) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (vii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (viii) $x - 1, y - 1, z$; (ix) $1 + x, 1 + y, z$.

Space group *Pbca* for (I) and *P*2₁2₁2₁ for (II) were uniquely assigned from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.95 Å and N—H distances of 0.82 Å in (I) at 120 (2) K, and C—H distances of 0.93 Å and N—H distances of 0.92 Å in (II) at 291 (2) K, and with U_{iso}(H) values of 1.2U_{eq}(C,N). In the absence of any significant anomalous scattering, the Flack (1983) parameter for isomer (II) was indeterminate (Flack & Bernardinelli, 2000), and it was not possible to establish the absolute configuration (Jones, 1986). Accordingly, the Friedel equivalent reflections were merged prior to the final refinements.

For compound (I), data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*. For compound (II), data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*. For both compounds, program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1783). Services for accessing these data are described at the back of the journal.

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supporting information

Isomeric nitrophthalimides: sheets built from N—H⋯O and C—H⋯O hydrogen bonds

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Computing details

Data collection: *COLLECT* (Hooft, 1999) for (I); *KappaCCD Server Software* (Nonius, 1997) for (II). Cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (I); *DENZO*–SMN (Otwinowski & Minor, 1997) for (II). Data reduction: *DENZO* and *COLLECT* for (I); *DENZO*–SMN for (II). For both compounds, 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).

(I) 3-Nitrophthalimide

Crystal data

$C_8H_4N_2O_4$	$F(000) = 784$
$M_r = 192.13$	$D_x = 1.705$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1703 reflections
$a = 8.2208$ (2) Å	$\theta = 3.1$–27.5°
$b = 13.0786$ (4) Å	$\mu = 0.14$ mm^{-1}
$c = 13.9233$ (4) Å	$T = 120$ K
$V = 1496.99$ (7) Å^3	Plate, colourless
$Z = 8$	$0.48 \times 0.32 \times 0.08$ mm

Data collection

Bruker-Nonius 95mm CCD camera on κ-goniostat diffractometer	$T_{min} = 0.954$, $T_{max} = 0.989$
	19573 measured reflections
	1703 independent reflections
Radiation source: Bruker–Nonius FR591 rotating anode	1488 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.037$
Graphite monochromator	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
Detector resolution: 9.091 pixels mm^{-1}	$h = -10 \rightarrow 10$
φ & ω scans	$k = -16 \rightarrow 15$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$l = -18 \rightarrow 16$

Refinement

Refinement on F^2	1703 reflections
Least-squares matrix: full	129 parameters
$R[F^2 > 2\sigma(F^2)] = 0.039$	0 restraints
$wR(F^2) = 0.099$	Primary atom site location: structure-invariant
$S = 1.10$	direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.5962P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.33$ e Å^{-3}

$\Delta\rho_{min} = -0.42$ e Å^{-3}

Extinction correction: *SHELXL97*, Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$

Extinction coefficient: 0.033 (4)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.41423 (13)	0.16003 (9)	0.34763 (7)	0.0189 (3)
C1	0.25286 (16)	0.17714 (9)	0.32887 (8)	0.0172 (3)
O1	0.19836 (10)	0.20978 (7)	0.25357 (6)	0.0209 (2)
C2	0.44360 (15)	0.12650 (10)	0.44109 (9)	0.0173 (3)
O2	0.57573 (11)	0.10362 (7)	0.47130 (7)	0.0229 (3)
C3	0.27874 (15)	0.12461 (9)	0.48828 (9)	0.0162 (3)
C4	0.22350 (15)	0.10039 (9)	0.57975 (9)	0.0169 (3)
C5	0.05837 (16)	0.09960 (9)	0.59990 (9)	0.0190 (3)
C6	−0.05338 (16)	0.12442 (10)	0.52875 (9)	0.0194 (3)
C7	−0.00199 (15)	0.15097 (9)	0.43675 (9)	0.0180 (3)
C8	0.16350 (15)	0.15055 (9)	0.41884 (8)	0.0160 (3)
N4	0.33533 (14)	0.07751 (8)	0.65906 (7)	0.0202 (3)
O41	0.46763 (13)	0.12029 (8)	0.65980 (7)	0.0284 (3)
O42	0.28788 (13)	0.01824 (8)	0.72135 (7)	0.0292 (3)
H1	0.4950	0.1735	0.3159	0.037 (5)*
H5	0.0216	0.0821	0.6624	0.023*
H6	−0.1664	0.1233	0.5431	0.023*
H7	−0.0779	0.1687	0.3881	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0147 (5)	0.0266 (6)	0.0154 (5)	−0.0013 (4)	0.0017 (4)	−0.0002 (4)
C1	0.0178 (6)	0.0167 (6)	0.0170 (6)	−0.0011 (5)	−0.0009 (5)	−0.0031 (5)
O1	0.0220 (5)	0.0258 (5)	0.0151 (4)	−0.0011 (4)	−0.0029 (3)	0.0018 (4)
C2	0.0163 (6)	0.0188 (6)	0.0167 (6)	−0.0005 (5)	0.0000 (5)	−0.0034 (5)
O2	0.0161 (5)	0.0294 (5)	0.0233 (5)	0.0022 (4)	−0.0028 (4)	−0.0004 (4)
C3	0.0159 (6)	0.0165 (6)	0.0163 (6)	0.0001 (5)	−0.0004 (5)	−0.0014 (5)
C4	0.0183 (6)	0.0173 (6)	0.0152 (6)	0.0002 (5)	−0.0025 (5)	−0.0006 (5)
C5	0.0211 (7)	0.0186 (6)	0.0173 (6)	−0.0008 (5)	0.0033 (5)	−0.0018 (5)
C6	0.0169 (6)	0.0197 (6)	0.0214 (6)	0.0000 (5)	0.0015 (5)	−0.0034 (5)
C7	0.0166 (6)	0.0179 (6)	0.0196 (6)	0.0009 (5)	−0.0023 (5)	−0.0019 (5)
C8	0.0171 (6)	0.0158 (6)	0.0151 (6)	0.0000 (4)	−0.0008 (5)	−0.0014 (4)
N4	0.0223 (6)	0.0224 (6)	0.0159 (5)	0.0020 (5)	−0.0023 (4)	−0.0002 (4)
O41	0.0241 (5)	0.0382 (6)	0.0230 (5)	−0.0062 (4)	−0.0080 (4)	0.0028 (4)
O42	0.0335 (6)	0.0331 (6)	0.0210 (5)	−0.0007 (4)	−0.0009 (4)	0.0104 (4)

Geometric parameters (Å, º)

N1—C1	1.3705 (16)	C4—N4	1.4678 (15)
N1—C2	1.3943 (16)	C5—C6	1.3895 (19)
N1—H1	0.8167	C5—H5	0.95
C1—O1	1.2174 (15)	C6—C7	1.3928 (18)
C1—C8	1.4933 (16)	C6—H6	0.95
C2—O2	1.2026 (15)	C7—C8	1.3832 (17)
C2—C3	1.5063 (17)	C7—H7	0.95
C3—C4	1.3887 (17)	N4—O41	1.2231 (15)
C3—C8	1.3954 (17)	N4—O42	1.2269 (14)
C4—C5	1.3862 (17)		
C1—N1—C2	113.38 (10)	C4—C5—C6	120.09 (12)
C1—N1—H1	130.4	C4—C5—H5	120.0
C2—N1—H1	115.6	C6—C5—H5	120.0
O1—C1—N1	125.29 (12)	C5—C6—C7	120.89 (12)
O1—C1—C8	128.54 (12)	C5—C6—H6	119.6
N1—C1—C8	106.16 (10)	C7—C6—H6	119.6
O2—C2—N1	124.13 (12)	C8—C7—C6	117.59 (11)
O2—C2—C3	131.00 (12)	C8—C7—H7	121.2
N1—C2—C3	104.86 (10)	C6—C7—H7	121.2
C4—C3—C8	117.96 (11)	C7—C8—C3	122.95 (11)
C4—C3—C2	134.26 (12)	C7—C8—C1	129.36 (11)
C8—C3—C2	107.74 (10)	C3—C8—C1	107.69 (11)
C5—C4—C3	120.51 (11)	O41—N4—O42	124.46 (11)
C5—C4—N4	117.36 (11)	O41—N4—C4	118.03 (10)
C3—C4—N4	122.12 (11)	O42—N4—C4	117.48 (11)
C2—N1—C1—O1	177.46 (12)	C5—C6—C7—C8	0.70 (18)
C2—N1—C1—C8	−1.07 (14)	C6—C7—C8—C3	0.22 (18)
C1—N1—C2—O2	177.25 (12)	C6—C7—C8—C1	−178.73 (12)
C1—N1—C2—C3	−1.49 (14)	C4—C3—C8—C7	−1.40 (18)
O2—C2—C3—C4	2.5 (2)	C2—C3—C8—C7	176.55 (11)
N1—C2—C3—C4	−178.89 (13)	C4—C3—C8—C1	177.75 (11)
O2—C2—C3—C8	−174.98 (14)	C2—C3—C8—C1	−4.30 (13)
N1—C2—C3—C8	3.63 (13)	O1—C1—C8—C7	4.0 (2)
C8—C3—C4—C5	1.68 (18)	N1—C1—C8—C7	−177.50 (12)
C2—C3—C4—C5	−175.60 (13)	O1—C1—C8—C3	−175.05 (12)
C8—C3—C4—N4	−176.64 (11)	N1—C1—C8—C3	3.42 (13)
C2—C3—C4—N4	6.1 (2)	C5—C4—N4—O41	−146.87 (12)
C3—C4—C5—C6	−0.82 (19)	C3—C4—N4—O41	31.51 (17)
N4—C4—C5—C6	177.58 (11)	C5—C4—N4—O42	31.58 (16)
C4—C5—C6—C7	−0.41 (19)	C3—C4—N4—O42	−150.05 (12)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1[i]	0.82	1.99	2.8043 (14)	176
C5—H5⋯O41[ii]	0.95	2.56	3.4386 (16)	153
C6—H6⋯O2[iii]	0.95	2.36	3.1639 (16)	142

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

(II) 4-Nitrophthalimide

Crystal data

$C_8H_4N_2O_4$	$F(000) = 392$
$M_r = 192.13$	$D_x = 1.716$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 1007 reflections
$a = 5.3114$ (3) Å	$\theta = 3.3$–27.5°
$b = 5.6812$ (5) Å	$\mu = 0.14$ mm^{-1}
$c = 24.645$ (2) Å	$T = 291$ K
$V = 743.67$ (10) Å^3	Plate, colourless
$Z = 4$	0.26 × 0.22 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer	6348 measured reflections
Radiation source: rotating anode	1007 independent reflections
Graphite monochromator	804 reflections with $I > 2\sigma(I)$
φ scans, and ω scans with κ offsets	$R_{int} = 0.060$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)	$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
	$h = -6 \rightarrow 6$
	$k = -7 \rightarrow 7$
$T_{min} = 0.956$, $T_{max} = 0.994$	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.110$	
$S = 1.03$	H-atom parameters constrained
1007 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.0553P]$ where $P = (F_o^2 + 2F_c^2)/3$
127 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.26$ e Å^{-3}
	$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.8528 (5)	0.4545 (4)	0.71294 (9)	0.0268 (6)
C1	0.8471 (6)	0.6382 (5)	0.67636 (10)	0.0242 (6)
O1	0.9844 (4)	0.8077 (4)	0.67667 (8)	0.0306 (5)
C2	0.6641 (5)	0.2892 (5)	0.70364 (11)	0.0257 (7)
O2	0.6265 (4)	0.1134 (4)	0.73007 (8)	0.0336 (6)
C3	0.5294 (5)	0.3736 (5)	0.65413 (11)	0.0236 (6)

C4	0.3286 (5)	0.2744 (5)	0.62623 (11)	0.0243 (6)
C5	0.2516 (5)	0.4022 (5)	0.58086 (10)	0.0234 (6)
N5	0.0417 (5)	0.3041 (4)	0.54841 (9)	0.0277 (6)
O51	−0.0327 (4)	0.1058 (4)	0.55967 (9)	0.0377 (6)
O52	−0.0481 (4)	0.4261 (4)	0.51226 (8)	0.0320 (5)
C6	0.3542 (6)	0.6110 (5)	0.56411 (11)	0.0251 (6)
C7	0.5546 (6)	0.7071 (6)	0.59283 (11)	0.0269 (7)
C8	0.6376 (5)	0.5839 (5)	0.63799 (11)	0.0233 (6)
H1	0.9573	0.4249	0.7418	0.032*
H4	0.2521	0.1346	0.6369	0.029*
H6	0.2904	0.6879	0.5338	0.030*
H7	0.6296	0.8477	0.5822	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0289 (13)	0.0261 (14)	0.0253 (12)	0.0008 (12)	−0.0031 (10)	0.0017 (10)
C1	0.0249 (14)	0.0240 (15)	0.0238 (13)	0.0020 (14)	0.0031 (11)	−0.0024 (12)
O1	0.0331 (12)	0.0290 (12)	0.0298 (10)	−0.0076 (11)	−0.0022 (9)	−0.0014 (9)
C2	0.0290 (15)	0.0209 (15)	0.0271 (13)	0.0023 (14)	0.0032 (13)	−0.0034 (12)
O2	0.0424 (14)	0.0260 (11)	0.0325 (11)	−0.0001 (11)	−0.0002 (9)	0.0038 (10)
C3	0.0233 (14)	0.0221 (15)	0.0253 (12)	0.0022 (13)	0.0015 (11)	−0.0028 (13)
C4	0.0238 (14)	0.0183 (14)	0.0309 (13)	0.0003 (13)	0.0061 (12)	−0.0011 (12)
C5	0.0192 (13)	0.0264 (16)	0.0248 (13)	0.0022 (13)	0.0009 (11)	−0.0034 (12)
N5	0.0253 (12)	0.0277 (14)	0.0300 (12)	−0.0039 (13)	0.0026 (11)	−0.0034 (12)
O51	0.0387 (12)	0.0359 (13)	0.0385 (11)	−0.0171 (12)	−0.0039 (10)	0.0030 (10)
O52	0.0309 (11)	0.0337 (13)	0.0313 (10)	0.0008 (11)	−0.0061 (9)	0.0023 (9)
C6	0.0274 (14)	0.0232 (16)	0.0246 (13)	0.0003 (14)	−0.0001 (11)	0.0009 (12)
C7	0.0286 (15)	0.0261 (16)	0.0259 (13)	−0.0014 (14)	−0.0004 (12)	−0.0005 (13)
C8	0.0219 (14)	0.0238 (16)	0.0241 (13)	−0.0004 (13)	0.0015 (11)	−0.0029 (12)

Geometric parameters (Å, º)

N1—C1	1.379 (4)	C4—H4	0.93
N1—C2	1.392 (4)	C5—C6	1.369 (4)
N1—H1	0.92	C5—N5	1.481 (4)
C1—O1	1.208 (3)	N5—O52	1.225 (3)
C1—C8	1.493 (4)	N5—O51	1.226 (3)
C2—O2	1.209 (3)	C6—C7	1.390 (4)
C2—C3	1.494 (4)	C6—H6	0.93
C3—C8	1.384 (4)	C7—C8	1.387 (4)
C3—C4	1.388 (4)	C7—H7	0.93
C4—C5	1.395 (4)		
C1—N1—C2	112.8 (2)	C6—C5—C4	125.2 (3)
C1—N1—H1	131.2	C6—C5—N5	117.6 (3)
C2—N1—H1	116.0	C4—C5—N5	117.3 (3)
O1—C1—N1	125.9 (3)	O52—N5—O51	124.0 (3)

O1—C1—C8	128.2 (3)	O52—N5—C5	118.2 (3)
N1—C1—C8	105.9 (2)	O51—N5—C5	117.8 (3)
O2—C2—N1	126.0 (3)	C5—C6—C7	119.5 (3)
O2—C2—C3	128.7 (3)	C5—C6—H6	120.3
N1—C2—C3	105.3 (2)	C7—C6—H6	120.3
C8—C3—C4	121.8 (3)	C8—C7—C6	117.0 (3)
C8—C3—C2	108.2 (3)	C8—C7—H7	121.5
C4—C3—C2	130.0 (3)	C6—C7—H7	121.5
C3—C4—C5	114.3 (3)	C3—C8—C7	122.3 (3)
C3—C4—H4	122.9	C3—C8—C1	107.8 (2)
C5—C4—H4	122.9	C7—C8—C1	129.9 (3)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O2[i]	0.92	2.55	3.231 (3)	131
N1—H1⋯O1[ii]	0.92	2.14	2.974 (3)	151
C4—H4⋯O1[iii]	0.93	2.54	3.452 (4)	168
C7—H7⋯O51[iv]	0.93	2.38	3.257 (4)	156

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