organic compounds

Simple chains in methyl 3,5-dinitrobenzoate, isolated molecules in isopropyl 3,5-dinitrobenzoate, and a three-dimensional framework containing double and sextuple helices in benzyl 3,5-dinitrobenzoate, all at 120 K

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Molecules of methyl 3,5-dinitrobenzoate, $C_8H_6N_2O_6$, are linked into $C(7)$ chains by a single nearly linear C—H···O hydrogen bond, but there are no direction-specific interactions in the structure of isopropyl 3,5-dinitrobenzoate, $C_{10}H_{10}N_2O_6$. In benzyl 3,5-dinitrobenzoate, $C_{14}H_{10}N_2O_6$, the molecules are linked by four independent C—H···O hydrogen bonds into a complex three-dimensional framework structure, in which it is possible to identify simple substructures in the form of cyclic centrosymmetric dimers, double helices and sextuple helices.

Comment

We report here the structures of methyl, isopropyl and benzyl 3,5-dinitrobenzoate, (I)–(III), respectively, derived from low-temperature diffraction data. It has been recognized since the early days of experimental organic chemistry that the 3,5-nitrobenzoate esters of simple alcohols are crystalline solids

(I)   *R*=Me
(II) *R*=CHMe₂
(III) *R*=CH₂Ph
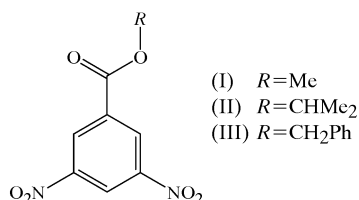
that are very readily purified and crystallized, and indeed these crystalline esters were for many decades utilized as an

aid to identification of such alcohols by means of their sharp and characteristic melting temperatures. The structures of a few of these esters have been the subject of isolated reports, including the ethyl ester (Hughes & Trotter, 1971) and the 2,2-dimethylbutyl ester, where $Z' = 2$ (Sax *et al.*, 1976); both of these structure determinations were based on the use of diffraction data collected at ambient temperature. More recently, the structures of the methyl ester, (I) (Jin & Xiao, 2000b), the *n*-propyl ester, where $Z' = 2$ (Jin & Xiao, 2005c), and the isopropyl ester, (II) (Jin & Xiao, 2005a), have been reported, also using diffraction data collected at ambient temperature. The structure of (I) was reported to contain π-stacked molecules related by translation along [100], with intermolecular C···C contacts as short as 3.440 (4) Å, despite the *a* repeat vector of 4.5833 (15) Å; no hydrogen bonds were reported in this compound. Although the unit-cell dimensions, the space group and the atomic coordinates for (I) indicate that no phase change has occurred between ambient
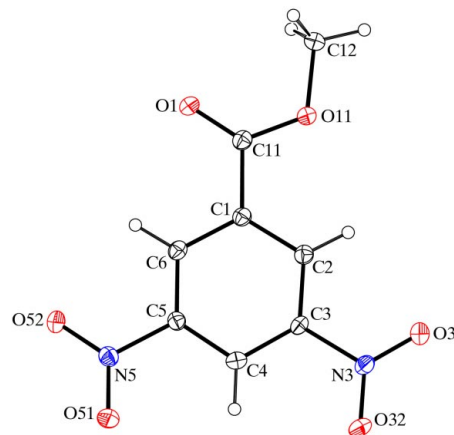

Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
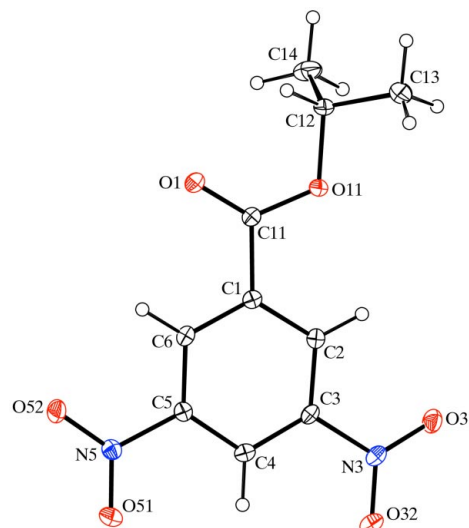

Figure 2
The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

temperature and 120 K, we find no π–π stacking in (I); however, a nearly linear C—H$\cdots$O hydrogen bond is present, in contrast to the findings reported previously (Jin & Xiao, 2005b).

The molecule of (I) (Fig. 1) is effectively planar, apart from the H atoms of the methyl group, as shown by the leading torsion angles (Table 3). In (II) and (III) (Figs. 2 and 3), the ester fragment up to and including atom C12 is effectively coplanar with the adjacent aryl ring, but in (III) in particular, the remaining torsion angles indicate a markedly non-planar conformation. The bond distances show no unusual values, but in each compound the internal angles at atoms C3 and C5, which are *ipso* to the nitro groups, are significantly larger than the corresponding angle at C2, which is *ipso* to the ester group.

There are no direction-specific intermolecular interactions in the structure of (II), but the molecules of (I) are linked into simple $C(7)$ chains by a single C—H$\cdots$O hydrogen bond (Table 1). Atom C4 in the molecule at (x, y, z) acts as a hydrogen-bond donor to carbonyl atom O1 in the molecule at $(\frac{3}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$, so forming a $C(7)$ (Bernstein *et al.*, 1995) chain running parallel to the [301] direction and generated by the *n*-glide plane at $y = \frac{3}{4}$ (Fig. 4). Two such chains, related to one another by inversion, pass through each unit cell, but there are no direction-specific interactions between adjacent chains; in particular, C—H$\cdots\pi$(arene) hydrogen bonds and aromatic π–π stacking interactions are both absent. The shortest ring-centroid separation, which involves the molecules at (x, y, z) and $(\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$, is 5.670 (2) Å and is clearly too large for effective π–π stacking.

The molecules of (III) are linked by four independent C—H$\cdots$O hydrogen bonds (Table 2) into a three-dimensional framework of considerable complexity. However, it is possible to identify several simple substructures, each generated by a limited number of hydrogen bonds. The formation of the framework is most s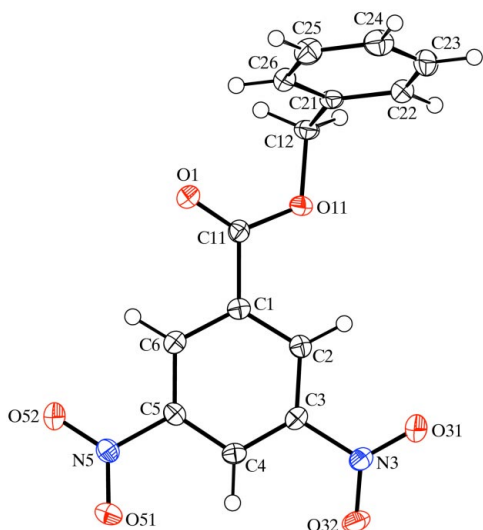imply analysed in terms of one finite zero-dimensional motif, which can be regarded as the basic building block; this motif is formed by the concerted action of two of the hydrogen bonds and two independent chain motifs, each containing a single hydrogen bond, one of which generates a double helix while the other generates a sextuple helix.

In the first substructure, atoms C6 and C12 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to atoms O1 and O52 in the molecule at $(1 - x, 1 - y, -z)$, so gener-
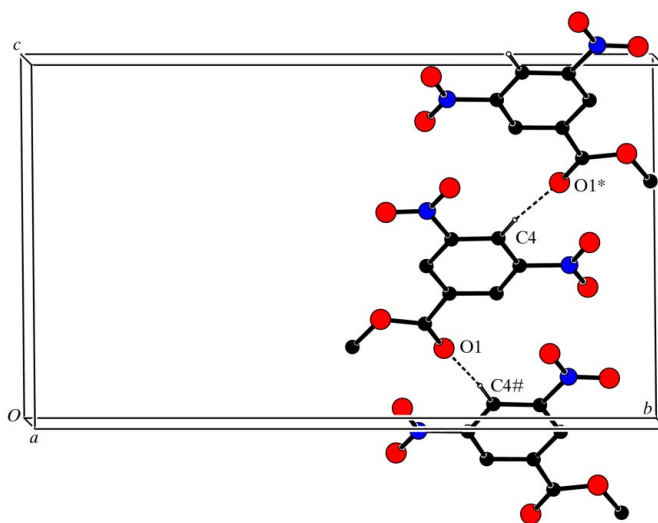
Figure 4
Part of the crystal structure of (I), showing the formation of a $C(7)$ chain along [301]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{3}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$ and $(-\frac{3}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$, respectively.

Figure 3
The molecule of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

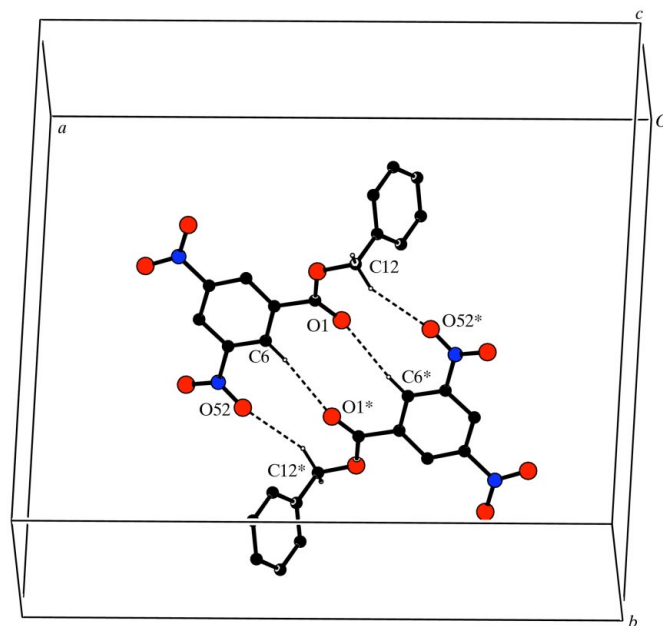**Figure 5**
Part of the crystal structure of (III), showing the formation of a centrosymmetric dimer. For the sake of clarity, H atoms bonded to C atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, -z)$.

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ating a centrosymmetric dimer centred at $(\frac{1}{2}, \frac{1}{2}, 0)$, in which inversion-related pairs of hydrogen bonds generate $R_2^2(10)$ and $R_2^2(18)$ rings (Fig. 5). In the second substructure, this time one-dimensional as opposed to zero-dimensional, atom C2 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O32 in the molecule at $(1 - y, -\frac{1}{2} + x, \frac{1}{2} + z)$, while atom C2 at $(1 - y, -\frac{1}{2} + x, \frac{1}{2} + z)$ in turn acts as a donor to atom O32 at $(\frac{3}{2} - x, \frac{1}{2} - y, 1 + z)$. Propagation of this hydrogen bond thus generates a $C(5)$ helical chain running parallel to the [001] direction and generated by the 4_2 screw axis along $(\frac{3}{4}, \frac{1}{4}, z)$ (Fig. 6). Because the screw axis is of the 4_2 type, this chain links the molecules at (x, y, z) and $(x, y, 2 + z)$, so that complete definition of this substructure requires two coaxial helices offset by a unit translation along [001].

The combination of this helical chain (Fig. 6) with the centrosymmetric dimer motif (Fig. 5) then links each helical chain to four adjacent helical chains; for example, the helix along $(\frac{3}{4}, \frac{1}{4}, z)$ is directly linked in this way to those along
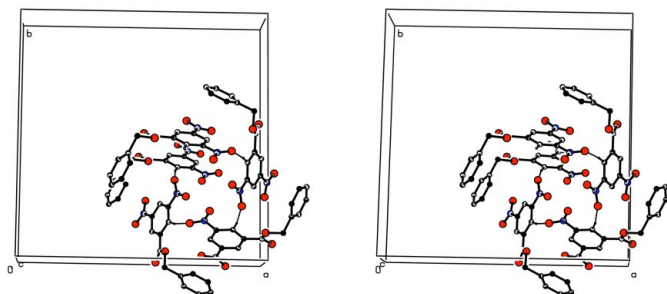
$(\frac{1}{4}, \frac{3}{4}, z)$, $(\frac{5}{4}, \frac{3}{4}, z)$, $(\frac{1}{4}, -\frac{1}{4}, z)$ and $(\frac{5}{4}, -\frac{1}{4}, z)$, so forming a continuous three-dimensional framework. Because of the double-helical nature of the [001] chain, there are in fact two such frameworks, intimately interwoven.

In addition, there is a fourth C—H···O hydrogen bond, whose action in isolation is to generate a sextuple helix of $C(11)$ chains (Fig. 7), but which in combination with the first chain-forming hydrogen bond links the two interwoven frameworks into a single continuous structure. Atom C22 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O51 in the molecule at $(1 - y, -\frac{1}{2} + x, \frac{3}{2} + z)$, and atom C22 at $(1 - y, -\frac{1}{2} + x, \frac{3}{2} + z)$ likewise acts as a donor to atom O51 at $(\frac{3}{2} - x, \frac{1}{2} - y, 3 + z)$, and thence via $(\frac{1}{2} + y, 1 - x, \frac{9}{2} + z)$ to $(x, y, 6 + z)$, so generating the sextuple helix. At the same time, atom C2 in the molecule at $(1 - y, -\frac{1}{2} + x, \frac{3}{2} + z)$ acts as a hydrogen-bond donor to atom O32 in the molecule at $(\frac{3}{2} - x, \frac{1}{2} - y, 2 + z)$, thereby linking the two coaxial $C(5)$ helices along $(\frac{3}{4}, \frac{1}{4}, z)$ and hence linking the two frameworks.

Figure 6
A stereoview of part of the crystal structure of (III), showing a hydrogen-bonded helical $C(5)$ chain generated by the 4_2 screw axis along $(\frac{3}{4}, \frac{1}{4}, z)$ and forming one strand of a double helix. For the sake of clarity, H atoms not involved in the motif shown have been omitted.
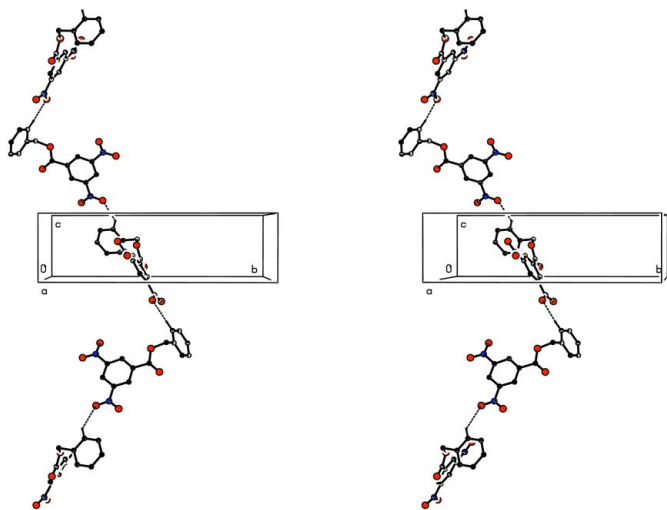

Figure 7
A stereoview of part of the crystal structure of (III), showing a hydrogen-bonded helical $C(11)$ chain generated by the 4_2 screw axis along $(\frac{3}{4}, \frac{1}{4}, z)$ and forming one strand of a sextuple helix. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

Experimental

Compound (I)

Crystal data

$C_8H_6N_2O_6$	$D_x = 1.655$ Mg m^{-3}
$M_r = 226.15$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2070
$a = 4.5664$ (4) Å	reflections
$b = 18.727$ (2) Å	$\theta = 2.9$–$27.6°$
$c = 10.8416$ (10) Å	$\mu = 0.14$ mm^{-1}
$\beta = 101.787$ (6)°	$T = 120$ (2) K
$V = 907.57$ (15) Å^3	Lath, colourless
$Z = 4$	$0.52 \times 0.12 \times 0.02$ mm

Data collection

Nonius KappaCCD diffractometer	1319 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.060$
Absorption correction: multi-scan	$\theta_{max} = 27.6°$
($SADABS$; Sheldrick, 2003)	$h = -5 \rightarrow 5$
$T_{min} = 0.935$, $T_{max} = 0.997$	$k = -24 \rightarrow 24$
9551 measured reflections	$l = -14 \rightarrow 14$
2070 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.058$	$+ 0.2307P]$
$wR(F^2) = 0.174$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
2070 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
146 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^i	0.95	2.46	3.399 (3)	171

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

Compound (II)

Crystal data

$C_{10}H_{10}N_2O_6$
$M_r = 254.20$
Monoclinic, $P2_1/n$
$a = 9.7037$ (3) Å
$b = 5.7152$ (2) Å
$c = 20.4739$ (9) Å
$\beta = 95.504$ (2)°
$V = 1130.22$ (7) Å^3
$Z = 4$

$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2596
  reflections
$\theta = 3.8–27.5°$
$\mu = 0.13$ mm^{-1}
$T = 120$ (2) K
Block, colourless
$0.42 \times 0.40 \times 0.38$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan
  (SADABS; Sheldrick, 2003)
  $T_{min} = 0.944$, $T_{max} = 0.954$
10022 measured reflections

2596 independent reflections
2045 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$; $\theta_{max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -24 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.105$
$S = 1.05$
2596 reflections
165 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.2191P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Table 2
Hydrogen-bond geometry (Å, °) for (III).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2$\cdots$O32^{ii}	0.95	2.52	3.460 (2)	170
C6—H6$\cdots$O1^{iii}	0.95	2.57	3.493 (2)	164
C12—H12A$\cdots$O52^{iii}	0.99	2.56	3.474 (2)	154
C22—H22$\cdots$O51^{iv}	0.95	2.57	3.360 (2)	140

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

Compound (III)

Crystal data

$C_{14}H_{10}N_2O_6$
$M_r = 302.24$
Tetragonal, $P4_2/n$
$a = 20.8531$ (5) Å
$c = 6.0377$ (2) Å
$V = 2625.50$ (12) Å^3
$Z = 8$
$D_x = 1.529$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3000
  reflections
$\theta = 3.1–27.5°$
$\mu = 0.12$ mm^{-1}
$T = 120$ (2) K
Needle, colourless
$0.22 \times 0.06 \times 0.04$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan
  (SADABS; Sheldrick, 2003)
  $T_{min} = 0.978$, $T_{max} = 0.995$
18873 measured reflections

3000 independent reflections
2413 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$; $\theta_{max} = 27.5°$
$h = -27 \rightarrow 26$
$k = -16 \rightarrow 27$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.103$
$S = 1.05$
3000 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 1.5651P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 3
Selected angles and torsion angles (°) for compounds (I)–(III).

	(I)	(II)	(III)
C6—C1—C2	120.2 (2)	120.34 (11)	120.82 (14)
C2—C3—C4	123.3 (2)	123.34 (11)	122.94 (15)
C4—C5—C6	123.3 (2)	122.58 (12)	123.37 (15)
C2—C1—C11—O11	2.0 (3)	−6.24 (16)	15.2 (2)
C1—C11—O11—C12	179.74 (19)	−176.88 (9)	177.03 (12)
C11—O11—C12—C13	–	152.12 (12)	–
C11—O11—C12—C14	–	−85.33 (13)	–
C11—O11—C12—C21	–	–	93.65 (16)
O11—C12—C21—C22	–	–	89.93 (7)
C2—C3—N3—O31	0.2 (3)	−4.88 (16)	9.1 (2)
C4—C5—N5—O51	4.2 (3)	6.13 (16)	7.5 (2)

For each of compounds (I) and (II), the space group $P2_1/n$ was uniquely assigned from the systematic absences. For compound (III), the space group $P4_2/n$ was uniquely assigned from the systematic absences, and the setting adopted had the origin coincident with a centre of inversion. All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic), 0.98 (methyl), 0.99 (CH$_2$) or 1.00 Å (aliphatic CH), and with U_{iso}(H) values of $1.2U_{eq}$(C), or $1.5U_{eq}$(C) for the methyl groups.

For all compounds, data collection: COLLECT (Hooft, 1999); 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).

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supporting information

Simple chains in methyl 3,5-dinitrobenzoate, isolated molecules in isopropyl 3,5-dinitrobenzoate, and a three-dimensional framework containing double and sextuple helices in benzyl 3,5-dinitrobenzoate, all at 120 K

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Computing details

For all compounds, data collection: *COLLECT* (Hooft, 1999); 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).

(I) Methyl 3,5-dinitrobenzoate

Crystal data

$C_8H_6N_2O_6$	$F(000) = 464$
$M_r = 226.15$	$D_x = 1.655$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2070 reflections
$a = 4.5664 (4)$ Å	$\theta = 2.9–27.6°$
$b = 18.727 (2)$ Å	$\mu = 0.15$ mm^{-1}
$c = 10.8416 (10)$ Å	$T = 120$ K
$\beta = 101.787 (6)°$	Lath, colourless
$V = 907.57 (15)$ Å^3	$0.52 \times 0.12 \times 0.02$ mm
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	$T_{min} = 0.935$, $T_{max} = 0.997$
Radiation source: Bruker-Nonius FR91 rotating anode	9551 measured reflections
	2070 independent reflections
Graphite monochromator	1319 reflections with $I > 2\sigma(I)$
Detector resolution: 9.091 pixels mm^{-1}	$R_{int} = 0.060$
φ and ω scans	$\theta_{max} = 27.6°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$h = -5 \rightarrow 5$
	$k = -24 \rightarrow 24$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.058$	Hydrogen site location: inferred from
$wR(F^2) = 0.174$	neighbouring sites
$S = 1.09$	H-atom parameters constrained
2070 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2 + 0.2307P]$
146 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.25$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -0.29$ e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	−0.2774 (3)	0.67000 (9)	0.18351 (15)	0.0278 (4)
O11	−0.0789 (4)	0.56667 (9)	0.26896 (16)	0.0297 (5)
O31	0.8123 (5)	0.56191 (10)	0.58930 (19)	0.0462 (6)
O32	1.0328 (4)	0.65946 (10)	0.66327 (15)	0.0315 (5)
O51	0.6277 (4)	0.88672 (9)	0.50125 (17)	0.0356 (5)
O52	0.2069 (4)	0.89026 (9)	0.36595 (17)	0.0343 (5)
N3	0.8306 (5)	0.62668 (11)	0.59376 (18)	0.0270 (5)
N5	0.4141 (4)	0.85809 (11)	0.43276 (18)	0.0251 (5)
C1	0.1600 (5)	0.67207 (13)	0.3470 (2)	0.0214 (5)
C2	0.3754 (5)	0.63231 (13)	0.4289 (2)	0.0230 (6)
C3	0.6005 (5)	0.66864 (13)	0.5090 (2)	0.0218 (5)
C4	0.6224 (5)	0.74254 (13)	0.5126 (2)	0.0224 (5)
C5	0.4021 (5)	0.77944 (13)	0.4308 (2)	0.0215 (5)
C6	0.1734 (5)	0.74635 (13)	0.3480 (2)	0.0228 (5)
C11	−0.0892 (5)	0.63688 (13)	0.2566 (2)	0.0233 (6)
C12	−0.3115 (6)	0.52557 (14)	0.1862 (2)	0.0333 (7)
H2	0.3673	0.5816	0.4295	0.028*
H4	0.7799	0.7664	0.5681	0.027*
H6	0.0277	0.7737	0.2926	0.027*
H12A	−0.5082	0.5398	0.2008	0.050*
H12B	−0.2803	0.4745	0.2042	0.050*
H12C	−0.3020	0.5348	0.0982	0.050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0261 (9)	0.0272 (10)	0.0266 (9)	0.0019 (7)	−0.0030 (8)	0.0002 (7)
O11	0.0302 (10)	0.0234 (10)	0.0302 (10)	−0.0011 (7)	−0.0062 (8)	0.0004 (7)
O31	0.0555 (13)	0.0244 (11)	0.0479 (12)	0.0059 (9)	−0.0151 (10)	0.0002 (9)
O32	0.0265 (10)	0.0376 (11)	0.0259 (9)	0.0003 (8)	−0.0052 (8)	0.0009 (8)
O51	0.0334 (11)	0.0275 (10)	0.0401 (11)	−0.0079 (8)	−0.0058 (9)	0.0009 (9)
O52	0.0358 (11)	0.0244 (10)	0.0378 (10)	0.0085 (8)	−0.0040 (9)	0.0004 (8)
N3	0.0277 (12)	0.0283 (13)	0.0224 (10)	0.0037 (9)	−0.0006 (9)	0.0010 (9)
N5	0.0276 (12)	0.0230 (11)	0.0253 (10)	−0.0014 (9)	0.0065 (9)	0.0000 (9)

C1	0.0229 (12)	0.0231 (13)	0.0184 (11)	0.0003 (9)	0.0048 (10)	0.0009 (9)
C2	0.0254 (13)	0.0236 (13)	0.0203 (11)	0.0013 (10)	0.0056 (10)	0.0003 (10)
C3	0.0196 (12)	0.0248 (13)	0.0198 (11)	0.0039 (10)	0.0012 (10)	0.0028 (10)
C4	0.0197 (12)	0.0278 (14)	0.0198 (12)	−0.0015 (10)	0.0039 (9)	−0.0022 (10)
C6	0.0229 (12)	0.0258 (13)	0.0196 (11)	0.0029 (10)	0.0041 (10)	0.0016 (10)
C5	0.0236 (13)	0.0200 (13)	0.0213 (12)	0.0005 (10)	0.0053 (10)	0.0000 (10)
C11	0.0247 (13)	0.0260 (13)	0.0197 (11)	0.0024 (10)	0.0057 (10)	0.0000 (10)
C12	0.0353 (16)	0.0242 (14)	0.0346 (15)	−0.0042 (11)	−0.0065 (12)	−0.0022 (11)

Geometric parameters (Å, º)

C1—C6	1.392 (3)	C3—C4	1.387 (3)
C1—C2	1.398 (3)	C3—N3	1.474 (3)
C1—C11	1.495 (3)	N3—O31	1.216 (3)
C11—O1	1.214 (3)	N3—O32	1.230 (2)
C11—O11	1.322 (3)	C4—C5	1.383 (3)
O11—C12	1.462 (3)	C4—H4	0.95
C12—H12A	0.98	C6—C5	1.378 (3)
C12—H12B	0.98	C6—H6	0.95
C12—H12C	0.98	C5—N5	1.474 (3)
C2—C3	1.381 (3)	N5—O51	1.223 (2)
C2—H2	0.95	N5—O52	1.226 (2)
C6—C1—C2	120.2 (2)	C2—C3—N3	118.3 (2)
C6—C1—C11	118.2 (2)	C4—C3—N3	118.4 (2)
C2—C1—C11	121.6 (2)	O31—N3—O32	124.0 (2)
O1—C11—O11	125.5 (2)	O31—N3—C3	118.2 (2)
O1—C11—C1	123.1 (2)	O32—N3—C3	117.8 (2)
O11—C11—C1	111.44 (19)	C5—C4—C3	116.2 (2)
C11—O11—C12	117.08 (18)	C5—C4—H4	121.9
O11—C12—H12A	109.5	C3—C4—H4	121.9
O11—C12—H12B	109.5	C5—C6—C1	118.7 (2)
H12A—C12—H12B	109.5	C5—C6—H6	120.6
O11—C12—H12C	109.5	C1—C6—H6	120.6
H12A—C12—H12C	109.5	C4—C5—C6	123.3 (2)
H12B—C12—H12C	109.5	C6—C5—N5	118.7 (2)
C3—C2—C1	118.3 (2)	C4—C5—N5	118.1 (2)
C3—C2—H2	120.9	O51—N5—O52	124.5 (2)
C1—C2—H2	120.9	O51—N5—C5	118.01 (19)
C2—C3—C4	123.3 (2)	O52—N5—C5	117.45 (19)
C6—C1—C11—O1	0.8 (3)	C4—C3—N3—O32	−0.5 (3)
C2—C1—C11—O1	−179.2 (2)	C2—C3—C4—C5	0.4 (3)
C6—C1—C11—O11	−178.01 (19)	N3—C3—C4—C5	179.70 (18)
C2—C1—C11—O11	2.0 (3)	C2—C1—C6—C5	0.0 (3)
O1—C11—O11—C12	1.0 (3)	C11—C1—C6—C5	−179.97 (19)
C1—C11—O11—C12	179.74 (19)	C1—C6—C5—C4	0.8 (3)
C6—C1—C2—C3	−0.6 (3)	C1—C6—C5—N5	−179.33 (19)

C11—C1—C2—C3	179.4 (2)	C3—C4—C5—C6	−1.0 (3)
C1—C2—C3—C4	0.4 (3)	C3—C4—C5—N5	179.16 (19)
C1—C2—C3—N3	−178.96 (19)	C6—C5—N5—O51	−175.71 (19)
C2—C3—N3—O31	0.2 (3)	C4—C5—N5—O51	4.2 (3)
C4—C3—N3—O31	−179.2 (2)	C6—C5—N5—O52	4.8 (3)
C2—C3—N3—O32	178.86 (19)	C4—C5—N5—O52	−175.29 (19)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4⋯O1^i	0.95	2.46	3.399 (3)	171

Symmetry code: (i) $x+3/2, -y+3/2, z+1/2$.

(II) Isopropyl 3,5-dinitrobenzoate

Crystal data

$C_{10}H_{10}N_2O_6$	$F(000) = 528$
$M_r = 254.20$	$D_x = 1.494$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2596 reflections
$a = 9.7037$ (3) Å	$\theta = 3.8–27.5°$
$b = 5.7152$ (2) Å	$\mu = 0.13$ mm^{-1}
$c = 20.4739$ (9) Å	$T = 120$ K
$\beta = 95.504$ (2)°	Block, colourless
$V = 1130.22$ (7) Å^3	$0.42 \times 0.40 \times 0.38$ mm
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	$T_{min} = 0.944$, $T_{max} = 0.954$
Radiation source: Bruker-Nonius FR91 rotating anode	10022 measured reflections
	2596 independent reflections
Graphite monochromator	2045 reflections with $I > 2\sigma(I)$
Detector resolution: 9.091 pixels mm^{-1}	$R_{int} = 0.031$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.8°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$h = -12 \rightarrow 12$
	$k = -7 \rightarrow 7$
	$l = -24 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.105$	
$S = 1.05$	H-atom parameters constrained
2596 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.2191P]$
165 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.30$ e Å^{-3}
	$\Delta\rho_{min} = -0.34$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27425 (9)	0.16199 (16)	0.63304 (5)	0.0258 (2)
O11	0.32924 (8)	0.47435 (15)	0.57331 (4)	0.0207 (2)
O31	0.63611 (10)	1.06799 (16)	0.67549 (5)	0.0288 (2)
O32	0.67594 (9)	1.08403 (16)	0.78148 (5)	0.0261 (2)
O51	0.52347 (10)	0.45491 (17)	0.91396 (5)	0.0314 (3)
O52	0.41232 (9)	0.16298 (16)	0.86658 (5)	0.0288 (2)
N3	0.62359 (10)	0.99248 (18)	0.73064 (6)	0.0208 (2)
N5	0.46624 (10)	0.35684 (19)	0.86574 (5)	0.0217 (3)
C1	0.39856 (12)	0.4773 (2)	0.68655 (6)	0.0178 (3)
C2	0.47145 (11)	0.6845 (2)	0.68063 (6)	0.0184 (3)
C3	0.54042 (11)	0.7796 (2)	0.73684 (6)	0.0180 (3)
C4	0.53871 (11)	0.6808 (2)	0.79833 (6)	0.0192 (3)
C5	0.46452 (12)	0.4756 (2)	0.80192 (6)	0.0182 (3)
C6	0.39433 (11)	0.3724 (2)	0.74748 (6)	0.0183 (3)
C11	0.32647 (11)	0.3529 (2)	0.62836 (6)	0.0184 (3)
C12	0.25439 (13)	0.3717 (2)	0.51385 (6)	0.0240 (3)
C13	0.21072 (19)	0.5760 (3)	0.47026 (8)	0.0435 (4)
C14	0.35003 (15)	0.2029 (3)	0.48437 (8)	0.0357 (4)
H2	0.4739	0.7588	0.6392	0.022*
H4	0.5861	0.7504	0.8362	0.023*
H6	0.3439	0.2316	0.7517	0.022*
H12	0.1705	0.2865	0.5260	0.029*
H13A	0.1584	0.6876	0.4946	0.065*
H13B	0.1523	0.5203	0.4317	0.065*
H13C	0.2930	0.6533	0.4561	0.065*
H14A	0.4342	0.2852	0.4745	0.053*
H14B	0.3036	0.1374	0.4438	0.053*
H14C	0.3746	0.0762	0.5156	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0150 (5)	0.0180 (6)	0.0205 (7)	0.0023 (5)	0.0023 (5)	−0.0018 (5)
C2	0.0164 (5)	0.0184 (6)	0.0211 (6)	0.0023 (5)	0.0045 (5)	0.0007 (5)
C3	0.0145 (5)	0.0142 (6)	0.0257 (7)	0.0004 (5)	0.0042 (5)	−0.0016 (5)
N3	0.0171 (5)	0.0175 (5)	0.0283 (6)	−0.0001 (4)	0.0043 (4)	−0.0031 (4)
O31	0.0308 (5)	0.0251 (5)	0.0311 (6)	−0.0060 (4)	0.0072 (4)	0.0035 (4)
O32	0.0231 (4)	0.0227 (5)	0.0325 (6)	−0.0042 (4)	0.0028 (4)	−0.0069 (4)
C4	0.0153 (5)	0.0197 (6)	0.0225 (7)	0.0017 (5)	0.0015 (5)	−0.0039 (5)
C5	0.0160 (5)	0.0191 (6)	0.0197 (7)	0.0035 (5)	0.0031 (4)	0.0008 (5)
N5	0.0179 (5)	0.0251 (6)	0.0219 (6)	0.0005 (4)	0.0007 (4)	0.0020 (5)
O51	0.0359 (5)	0.0350 (6)	0.0216 (5)	−0.0047 (4)	−0.0061 (4)	0.0003 (4)
O52	0.0301 (5)	0.0267 (5)	0.0296 (6)	−0.0074 (4)	0.0029 (4)	0.0062 (4)
C6	0.0142 (5)	0.0165 (6)	0.0247 (7)	0.0008 (5)	0.0038 (5)	0.0002 (5)
C11	0.0147 (5)	0.0192 (6)	0.0214 (7)	0.0002 (5)	0.0026 (5)	−0.0004 (5)

O1	0.0279 (5)	0.0233 (5)	0.0257 (5)	−0.0080 (4)	0.0008 (4)	0.0003 (4)
O11	0.0235 (4)	0.0212 (5)	0.0170 (5)	−0.0038 (4)	0.0001 (3)	−0.0005 (3)
C12	0.0267 (6)	0.0248 (7)	0.0193 (7)	−0.0063 (5)	−0.0032 (5)	−0.0014 (5)
C13	0.0634 (11)	0.0327 (8)	0.0302 (9)	−0.0079 (8)	−0.0173 (8)	0.0055 (7)
C14	0.0374 (8)	0.0390 (8)	0.0313 (8)	−0.0078 (7)	0.0065 (6)	−0.0141 (7)

Geometric parameters (Å, º)

C1—C6	1.3883 (17)	N5—O52	1.2261 (14)
C1—C2	1.3907 (17)	C6—H6	0.95
C1—C11	1.5014 (17)	C11—O1	1.2109 (15)
C2—C3	1.3856 (17)	C11—O11	1.3260 (15)
C2—H2	0.95	O11—C12	1.4778 (15)
C3—C4	1.3813 (17)	C12—C14	1.505 (2)
C3—N3	1.4721 (16)	C12—C13	1.5058 (19)
N3—O31	1.2257 (14)	C12—H12	1.00
N3—O32	1.2303 (14)	C13—H13A	0.98
C4—C5	1.3819 (17)	C13—H13B	0.98
C4—H4	0.95	C13—H13C	0.98
C5—C6	1.3815 (17)	C14—H14A	0.98
C5—N5	1.4710 (16)	C14—H14B	0.98
N5—O51	1.2216 (14)	C14—H14C	0.98
C6—C1—C2	120.34 (11)	C1—C6—H6	120.5
C6—C1—C11	117.19 (11)	O1—C11—O11	125.56 (11)
C2—C1—C11	122.43 (11)	O1—C11—C1	122.05 (11)
C3—C2—C1	118.09 (12)	O11—C11—C1	112.38 (10)
C3—C2—H2	121.0	C11—O11—C12	116.36 (10)
C1—C2—H2	121.0	O11—C12—C14	108.16 (11)
C4—C3—C2	123.34 (11)	O11—C12—C13	105.57 (11)
C4—C3—N3	118.10 (11)	C14—C12—C13	114.13 (13)
C2—C3—N3	118.53 (11)	O11—C12—H12	109.6
O31—N3—O32	124.07 (11)	C14—C12—H12	109.6
O31—N3—C3	118.30 (10)	C13—C12—H12	109.6
O32—N3—C3	117.63 (11)	C12—C13—H13A	109.5
C3—C4—C5	116.57 (11)	C12—C13—H13B	109.5
C3—C4—H4	121.7	H13A—C13—H13B	109.5
C5—C4—H4	121.7	C12—C13—H13C	109.5
C6—C5—C4	122.58 (12)	H13A—C13—H13C	109.5
C6—C5—N5	118.84 (11)	H13B—C13—H13C	109.5
C4—C5—N5	118.51 (11)	C12—C14—H14A	109.5
O51—N5—O52	124.39 (11)	C12—C14—H14B	109.5
O51—N5—C5	118.15 (11)	H14A—C14—H14B	109.5
O52—N5—C5	117.45 (10)	C12—C14—H14C	109.5
C5—C6—C1	119.07 (12)	H14A—C14—H14C	109.5
C5—C6—H6	120.5	H14B—C14—H14C	109.5
C6—C1—C2—C3	0.88 (17)	C6—C5—N5—O52	4.66 (16)

C11—C1—C2—C3	−176.74 (10)	C4—C5—N5—O52	−172.37 (11)
C1—C2—C3—C4	−0.94 (17)	C4—C5—C6—C1	0.32 (18)
C1—C2—C3—N3	176.99 (10)	N5—C5—C6—C1	−176.58 (10)
C4—C3—N3—O31	173.16 (10)	C2—C1—C6—C5	−0.59 (17)
C2—C3—N3—O31	−4.88 (16)	C11—C1—C6—C5	177.15 (10)
C4—C3—N3—O32	−7.08 (15)	C6—C1—C11—O1	−4.72 (17)
C2—C3—N3—O32	174.88 (10)	C2—C1—C11—O1	172.98 (11)
C2—C3—C4—C5	0.67 (17)	C6—C1—C11—O11	176.07 (10)
N3—C3—C4—C5	−177.27 (10)	C2—C1—C11—O11	−6.24 (16)
C3—C4—C5—C6	−0.34 (17)	O1—C11—O11—C12	3.94 (17)
C3—C4—C5—N5	176.57 (10)	C1—C11—O11—C12	−176.88 (9)
C6—C5—N5—O51	−176.84 (11)	C11—O11—C12—C14	−85.33 (13)
C4—C5—N5—O51	6.13 (16)	C11—O11—C12—C13	152.12 (12)

(III) Benzyl 3,5-dinitrobenzoate?

Crystal data

$C_{14}H_{10}N_2O_6$	$D_x = 1.529$ Mg m^{-3}
$M_r = 302.24$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Tetragonal, $P4_2/n$	Cell parameters from 3000 reflections
Hall symbol: -P 4bc	$\theta = 3.1$–27.5°
$a = 20.8531$ (5) Å	$\mu = 0.12$ mm^{-1}
$c = 6.0377$ (2) Å	$T = 120$ K
$V = 2625.50$ (12) Å^3	Needle, colourless
$Z = 8$	$0.22 \times 0.06 \times 0.04$ mm
$F(000) = 1248$	

Data collection

Nonius KappaCCD diffractometer	$T_{min} = 0.978$, $T_{max} = 0.995$
Radiation source: Bruker-Nonius FR91 rotating anode	18873 measured reflections
	3000 independent reflections
Graphite monochromator	2413 reflections with $I > 2\sigma(I)$
Detector resolution: 9.091 pixels mm^{-1}	$R_{int} = 0.053$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$h = -27 \rightarrow 26$
	$k = -16 \rightarrow 27$
	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.103$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 1.5651P]$
3000 reflections	where $P = (F_o^2 + 2F_c^2)/3$
199 parameters	$(\Delta/\sigma)_{max} = 0.001$
0 restraints	$\Delta\rho_{max} = 0.24$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

	x	*y*	*z*	$U_{iso}*/U_{eq}$
O1	0.49125 (5)	0.44104 (6)	0.1932 (2)	0.0254 (3)
O11	0.52732 (5)	0.40805 (5)	0.52649 (18)	0.0211 (3)
O31	0.74509 (6)	0.32867 (7)	0.5879 (2)	0.0376 (3)
O32	0.81810 (5)	0.37069 (6)	0.3788 (2)	0.0284 (3)
O51	0.75704 (5)	0.47883 (6)	−0.2874 (2)	0.0273 (3)
O52	0.66120 (6)	0.51811 (6)	−0.3183 (2)	0.0287 (3)
N3	0.76221 (6)	0.36163 (7)	0.4312 (2)	0.0226 (3)
N5	0.70199 (6)	0.48641 (6)	−0.2208 (2)	0.0206 (3)
C1	0.60325 (7)	0.42401 (7)	0.2460 (3)	0.0172 (3)
C2	0.64954 (7)	0.39295 (7)	0.3724 (3)	0.0181 (3)
C3	0.71241 (7)	0.39371 (7)	0.2979 (3)	0.0188 (3)
C4	0.73094 (7)	0.42425 (7)	0.1063 (3)	0.0185 (3)
C5	0.68335 (7)	0.45441 (7)	−0.0135 (3)	0.0175 (3)
C6	0.61973 (7)	0.45520 (7)	0.0503 (3)	0.0180 (3)
C11	0.53428 (7)	0.42524 (7)	0.3149 (3)	0.0181 (3)
C12	0.46199 (7)	0.41048 (8)	0.6171 (3)	0.0209 (3)
C21	0.42942 (7)	0.34636 (7)	0.5979 (3)	0.0192 (3)
C22	0.43489 (8)	0.30202 (8)	0.7690 (3)	0.0242 (4)
C23	0.40476 (9)	0.24289 (9)	0.7537 (3)	0.0296 (4)
C24	0.36916 (8)	0.22777 (8)	0.5671 (3)	0.0293 (4)
C25	0.36404 (8)	0.27124 (8)	0.3950 (3)	0.0270 (4)
C26	0.39397 (8)	0.33054 (8)	0.4104 (3)	0.0219 (4)
H2	0.6384	0.3718	0.5063	0.022*
H4	0.7744	0.4246	0.0588	0.022*
H6	0.5882	0.4764	−0.0367	0.022*
H12A	0.4367	0.4431	0.5359	0.025*
H12B	0.4638	0.4234	0.7748	0.025*
H22	0.4594	0.3123	0.8968	0.029*
H23	0.4085	0.2128	0.8711	0.036*
H24	0.3482	0.1874	0.5573	0.035*
H25	0.3401	0.2605	0.2664	0.032*
H26	0.3902	0.3604	0.2924	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0169 (7)	0.0146 (7)	0.0201 (8)	−0.0015 (6)	−0.0002 (6)	−0.0025 (6)
C2	0.0196 (8)	0.0159 (7)	0.0187 (8)	−0.0025 (6)	−0.0007 (6)	−0.0005 (6)
C3	0.0183 (8)	0.0171 (8)	0.0210 (8)	0.0011 (6)	−0.0031 (6)	−0.0015 (6)
N3	0.0191 (7)	0.0253 (7)	0.0235 (7)	0.0027 (5)	−0.0016 (6)	0.0009 (6)
O32	0.0155 (6)	0.0409 (7)	0.0287 (7)	0.0048 (5)	−0.0011 (5)	0.0001 (6)
O31	0.0285 (7)	0.0473 (8)	0.0369 (8)	0.0044 (6)	−0.0004 (6)	0.0228 (7)
C4	0.0164 (7)	0.0173 (8)	0.0218 (8)	−0.0009 (6)	0.0004 (6)	−0.0026 (6)
C5	0.0207 (8)	0.0137 (7)	0.0182 (8)	−0.0025 (6)	0.0013 (6)	−0.0013 (6)
N5	0.0222 (7)	0.0188 (7)	0.0206 (7)	−0.0022 (5)	0.0009 (6)	0.0004 (6)

O52	0.0285 (7)	0.0305 (7)	0.0271 (7)	0.0026 (5)	−0.0010 (5)	0.0108 (5)
O51	0.0220 (6)	0.0325 (7)	0.0275 (7)	−0.0003 (5)	0.0074 (5)	0.0030 (5)
C6	0.0185 (8)	0.0151 (7)	0.0206 (8)	−0.0009 (6)	−0.0025 (6)	−0.0013 (6)
C11	0.0187 (8)	0.0152 (7)	0.0204 (8)	−0.0015 (6)	−0.0013 (6)	0.0012 (6)
O1	0.0184 (6)	0.0317 (7)	0.0261 (6)	0.0013 (5)	−0.0020 (5)	0.0065 (5)
O11	0.0150 (5)	0.0283 (6)	0.0199 (6)	−0.0014 (4)	0.0014 (5)	0.0028 (5)
C12	0.0160 (8)	0.0260 (8)	0.0207 (8)	0.0001 (6)	0.0035 (6)	−0.0016 (7)
C21	0.0147 (7)	0.0211 (8)	0.0218 (8)	0.0026 (6)	0.0037 (6)	0.0004 (6)
C22	0.0210 (8)	0.0288 (9)	0.0229 (9)	0.0031 (7)	0.0005 (7)	0.0040 (7)
C23	0.0294 (10)	0.0261 (9)	0.0334 (10)	0.0044 (7)	0.0049 (8)	0.0102 (8)
C24	0.0271 (9)	0.0192 (8)	0.0414 (11)	−0.0025 (7)	0.0035 (8)	0.0006 (8)
C25	0.0250 (9)	0.0243 (9)	0.0318 (10)	−0.0023 (7)	−0.0032 (7)	−0.0018 (7)
C26	0.0201 (8)	0.0227 (8)	0.0230 (9)	0.0017 (6)	−0.0011 (7)	0.0031 (7)

Geometric parameters (Å, º)

C1—C2	1.391 (2)	C11—O11	1.3350 (19)
C1—C6	1.392 (2)	O11—C12	1.4690 (18)
C1—C11	1.497 (2)	C12—C21	1.504 (2)
C2—C3	1.386 (2)	C12—H12A	0.99
C2—H2	0.95	C12—H12B	0.99
C3—C4	1.376 (2)	C21—C22	1.391 (2)
C3—N3	1.474 (2)	C21—C26	1.392 (2)
N3—O32	1.2223 (17)	C22—C23	1.387 (2)
N3—O31	1.2224 (18)	C22—H22	0.95
C4—C5	1.380 (2)	C23—C24	1.386 (3)
C4—H4	0.95	C23—H23	0.95
C5—C6	1.381 (2)	C24—C25	1.383 (3)
C5—N5	1.471 (2)	C24—H24	0.95
N5—O51	1.2263 (17)	C25—C26	1.388 (2)
N5—O52	1.2275 (17)	C25—H25	0.95
C6—H6	0.95	C26—H26	0.95
C11—O1	1.2053 (19)		
C2—C1—C6	120.82 (14)	O11—C11—C1	111.45 (13)
C2—C1—C11	121.48 (14)	C11—O11—C12	116.61 (12)
C6—C1—C11	117.70 (14)	O11—C12—C21	111.06 (12)
C3—C2—C1	118.23 (15)	O11—C12—H12A	109.4
C3—C2—H2	120.9	C21—C12—H12A	109.4
C1—C2—H2	120.9	O11—C12—H12B	109.4
C4—C3—C2	122.94 (15)	C21—C12—H12B	109.4
C4—C3—N3	118.11 (14)	H12A—C12—H12B	108.0
C2—C3—N3	118.93 (14)	C22—C21—C26	119.35 (15)
O32—N3—O31	124.45 (14)	C22—C21—C12	119.77 (15)
O32—N3—C3	117.39 (13)	C26—C21—C12	120.88 (14)
O31—N3—C3	118.15 (13)	C23—C22—C21	120.28 (16)
C3—C4—C5	116.73 (14)	C23—C22—H22	119.9
C3—C4—H4	121.6	C21—C22—H22	119.9

C5—C4—H4	121.6	C24—C23—C22	119.95 (16)
C4—C5—C6	123.37 (15)	C24—C23—H23	120.0
C4—C5—N5	117.57 (14)	C22—C23—H23	120.0
C6—C5—N5	119.06 (14)	C25—C24—C23	120.20 (16)
O51—N5—O52	124.14 (14)	C25—C24—H24	119.9
O51—N5—C5	117.88 (13)	C23—C24—H24	119.9
O52—N5—C5	117.96 (13)	C24—C25—C26	119.93 (17)
C5—C6—C1	117.92 (14)	C24—C25—H25	120.0
C5—C6—H6	121.0	C26—C25—H25	120.0
C1—C6—H6	121.0	C25—C26—C21	120.29 (16)
O1—C11—O11	125.14 (15)	C25—C26—H26	119.9
O1—C11—C1	123.40 (15)	C21—C26—H26	119.9
C6—C1—C2—C3	−0.2 (2)	C11—C1—C6—C5	179.75 (13)
C11—C1—C2—C3	179.86 (14)	C2—C1—C11—O1	−166.04 (15)
C1—C2—C3—C4	0.6 (2)	C6—C1—C11—O1	14.0 (2)
C1—C2—C3—N3	179.32 (13)	C2—C1—C11—O11	15.2 (2)
C4—C3—N3—O32	8.9 (2)	C6—C1—C11—O11	−164.72 (13)
C2—C3—N3—O32	−169.88 (14)	O1—C11—O11—C12	−1.7 (2)
C4—C3—N3—O31	−172.16 (15)	C1—C11—O11—C12	177.03 (12)
C2—C3—N3—O31	9.1 (2)	C11—O11—C12—C21	93.65 (16)
C2—C3—C4—C5	−0.6 (2)	O11—C12—C21—C22	89.93 (17)
N3—C3—C4—C5	−179.32 (13)	O11—C12—C21—C26	−89.78 (17)
C3—C4—C5—C6	0.2 (2)	C26—C21—C22—C23	−0.7 (2)
C3—C4—C5—N5	−178.99 (13)	C12—C21—C22—C23	179.61 (15)
C4—C5—N5—O51	7.5 (2)	C21—C22—C23—C24	0.2 (3)
C6—C5—N5—O51	−171.70 (14)	C22—C23—C24—C25	0.6 (3)
C4—C5—N5—O52	−173.68 (14)	C23—C24—C25—C26	−0.8 (3)
C6—C5—N5—O52	7.1 (2)	C24—C25—C26—C21	0.3 (3)
C4—C5—C6—C1	0.2 (2)	C22—C21—C26—C25	0.4 (2)
N5—C5—C6—C1	179.36 (13)	C12—C21—C26—C25	−179.86 (15)
C2—C1—C6—C5	−0.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O32[i]	0.95	2.52	3.460 (2)	170
C6—H6···O1[ii]	0.95	2.57	3.493 (2)	164
C12—H12A···O52[ii]	0.99	2.56	3.474 (2)	154
C22—H22···O51[iii]	0.95	2.57	3.360 (2)	140

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