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Crystal structures of three co-crystals of 1,2-bis(pyridin-4-yl)ethane with 4-alkoxybenzoic acids: 4-ethoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), 4-*n*-propoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) and 4-*n*-butoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1)

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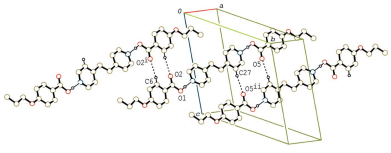
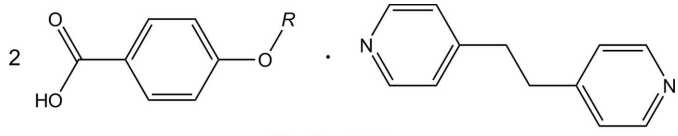
The crystal structures of three hydrogen-bonded co-crystals of 4-alkoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), namely, $2C_9H_{10}O_3\cdot C_{12}H_{12}N_2$, (I), $2C_{10}H_{12}O_3\cdot C_{12}H_{12}N_2$, (II), and $2C_{11}H_{14}O_3\cdot C_{12}H_{12}N_2$, (III), have been determined at 93, 290 and 93 K, respectively. In (I), the asymmetric unit consists of one 4-ethoxybenzoic acid molecule and one half-molecule of 1,2-bis(pyridin-4-yl)ethane, which lies on an inversion centre. In (II) and (III), the asymmetric units each comprise two crystallographically independent 4-alkoxybenzoic acid molecules and one 1,2-bis(pyridin-4-yl)ethane molecule. In each crystal, the two components are linked by O—H···N hydrogen bonds, forming a linear hydrogen-bonded 2:1unit of the acid and the base. Similar to the structure of 2:1 unit of (I), the units of (II) and (III) adopt nearly pseudo-inversion symmetry. The 2:1 units of (I), (II) and (III) are linked *via* C—H···O hydrogen bonds, forming tape structures.

1. Chemical context

Co-crystals of 4-alkoxybenzoic acid–4,4′-bipyridyl (2/1), in which the two acids and the base are held together by intermolecular O—H···N hydrogen bonds, show thermotropic liquid crystallinity (Kato *et al.*, 1990, 1993; Grunert *et al.*, 1997). Recently, we have reported the crystal structures of the three compounds of 4-ethoxy-, 4-*n*-propoxy- and 4-*n*-butoxybenzoic acid (Tabuchi *et al.*, 2015). As an expansion of our work on the structural characterization of hydrogen-bonded co-crystals which exhibit liquid phases, we have prepared compounds of 4-alkoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) and analyzed the crystal structures. DSC (differential scanning calorimetry) and polarizing microscope measurements show that the compounds of 4-methoxy-, 4-ethoxy- and 4-*n*-propoxybenzoic acid have nematic phases at 419 (1), 421 (1) and 419 (1) K, respectively, while the compound of 4-*n*-butoxybenzoic acid exhibits a smectic A phase at 413 (1) K and a nematic phase at 419 (1) K.





(I) *R* = ethyl
(II) *R* = *n*-propyl
(III) *R* = *n*-butyl

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We present here three structures of 4-ethoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), (I), 4-*n*-propoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), (II), and 4-*n*-butoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), (III). The structure of 4-methoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) has been reported recently (Mukherjee & Desiraju, 2014).

2. Structural commentary

The molecular structures of (I), (II) and (III) are shown in Fig. 1. The asymmetric unit of (I) consists of one 4-ethoxybenzoic acid molecule and one half-molecule of 1,2-bis(pyridin-4-yl)ethane which lies on an inversion centre. The two acid molecules and the base molecule are held together *via* O—H···N hydrogen bonds (Table 1) to afford a centrosymmetric linear 2:1 unit. The hydrogen-bonded asymmetric unit is approximately planar with dihedral angles of 9.40 (11), 4.38 (11) and 2.76 (9)°, respectively, between the N1/C10–C14 and O1/C7/O2 planes, the O1/C7/O2 and C1–C6 planes, and the C1–C6 and O3/C8/C9 planes.

The asymmetric units of (II) and (III) are each composed of two crystallographically independent 4-alkoxybenzoic acid molecules and one 1,2-bis(pyridin-4-yl)ethane molecule, and the two acids and the base are held together by O—H···N hydrogen bonds (Tables 2 and 3), forming a linear hydrogen-bonded 2:1 aggregate. Similar to the 2:1 unit of (I), the units of (II) and (III) adopt nearly pseudo-inversion symmetry. The dihedral angles between the pyridine rings of 1,2-bis(pyridin-4-yl)ethane are 14.36 (6) and 29.92 (7)°, respectively, for (II) and (III). The pyridine ring and the carboxyl group hydrogen-bonded to it are twisted with respect to each other. In (II), the dihedral angles between the N1/C21–C25 and O1/C7/O2 planes, and the N2/C26–C30 and O4/C17/O5 planes are

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.967 (18)	1.659 (18)	2.6247 (17)	178.0 (14)
C6—H6···O2^{i}	0.95	2.60	3.406 (2)	144

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.966 (18)	1.657 (19)	2.6207 (17)	175.4 (16)
O4—H4···N2	1.010 (19)	1.610 (19)	2.6198 (17)	179 (2)
C6—H6···O2^{i}	0.93	2.55	3.376 (2)	149
C27—H27···O5^{ii}	0.93	2.52	3.389 (2)	156

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

Table 3
Hydrogen-bond geometry (Å, °) for (III).

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C12–C17 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	1.02 (2)	1.60 (2)	2.6209 (18)	177 (2)
O4—H4···N2	1.03 (3)	1.58 (3)	2.6092 (18)	178.1 (19)
C32—H32···O3^{i}	0.95	2.57	3.524 (2)	177
C11—H11*A*···*Cg*1^{ii}	0.98	2.80	3.662 (2)	148
C33—H33*A*···*Cg*2^{iii}	0.99	2.74	3.598 (2)	145

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

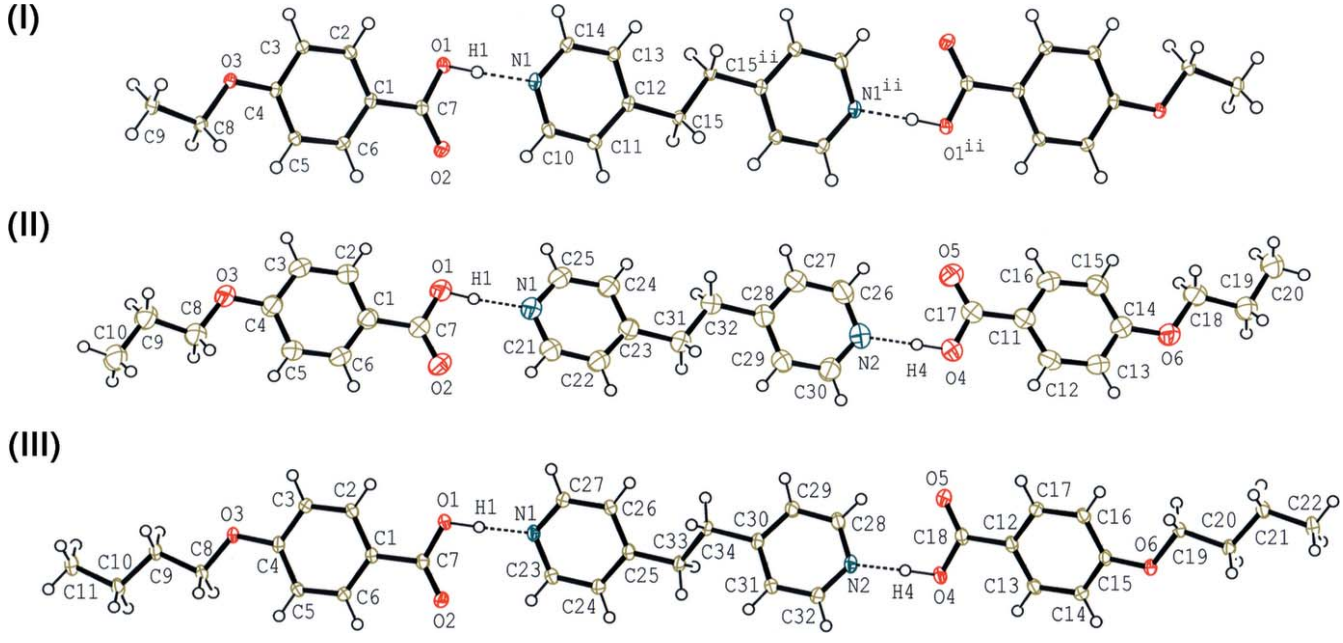

Figure 1
The molecular structures of compounds (I), (II) and (III) determined at 93, 290 and 93 K, respectively, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. O—H···N hydrogen bonds are indicated by dashed lines [symmetry code for (I): (ii) $-x, -y+2, -z$].

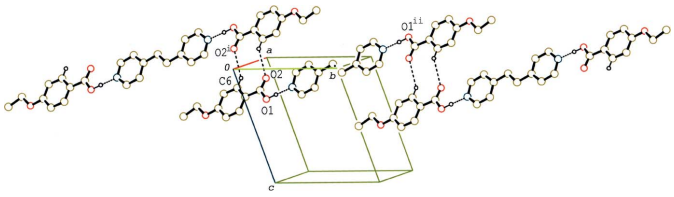

Figure 2
A partial packing diagram of compound (I), showing the tape structure formed by C—H···O hydrogen bonds. H atoms not involved in C—H···O and O—H···N hydrogen bonds (dashed lines) have been omitted [symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x, -y+2, -z$].

4.86 (14) and 7.71 (14)°, respectively, while those in (III) are 9.48 (16) and 25.25 (17)°, respectively, between the N1/C23–C27 and O1/C7/O2 planes, and the N2/C28–C32 and O4/C17/O5 planes.

The molecular structures of 4-*n*-propoxy- and 4-*n*-butoxybenzoic acids in (II) and (III) are approximately planar. The dihedral angles made by the benzene ring with the carboxyl group and the alkoxy group in each propoxybenzoic acid in (II) are 9.20 (14), 4.36 (14), 1.80 (11) and 5.98 (11)°, respectively, between the C1–C6 and O1/C7/O2 planes, the C11–C16 and O4/C17/O5 planes, the C1–C6 and O3/C8–C10 planes, and the C11–C16 and O6/C18–C20 planes. The corresponding dihedral angles in (III) are 0.67 (16), 15.05 (17), 2.83 (10) and 11.86 (10)°, respectively, between the C1–C6 and O1/C7/O2 planes, the C12–C17 and O4/C18/O5 planes, the C1–C6 and O3/C8–C11 planes, and the C12–C17 and O6/C19–C22 planes.

3. Supramolecular features

In the crystal of (I), the 2:1 units are linked by a pair of C—H···O hydrogen bonds (Table 1), forming a tape structure along [1$\bar{2}$0] (Fig. 2). In addition, the units are stacked in a column through π–π interactions between the acid and base rings along the *b* axis (Fig. 3). The centroid–centroid distance between the C1–C6 and N1/C10–C14($x, y-1, z$) rings is 3.592 (2) Å.

In the crystal of (II) and (III), the 2:1 units are linked by C—H···O interactions (Tables 2 and 3), forming tape structures along [310] (Fig. 4) and [001] (Fig. 5), respectively.

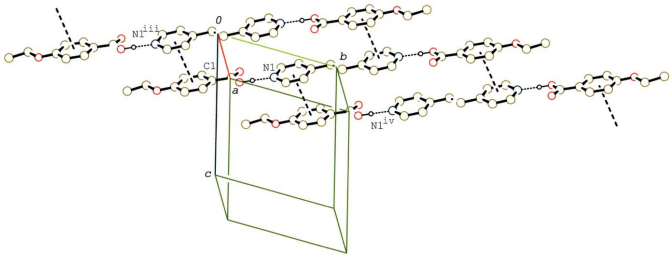

Figure 3
A partial packing diagram of compound (I), showing the column structure formed by π–π stacking interactions (dashed lines). H atoms not involved in O—H···N hydrogen bonds (dashed lines) have been omitted [symmetry codes: (iii) $x, y-1, z$; (iv) $x, y+1, z$].

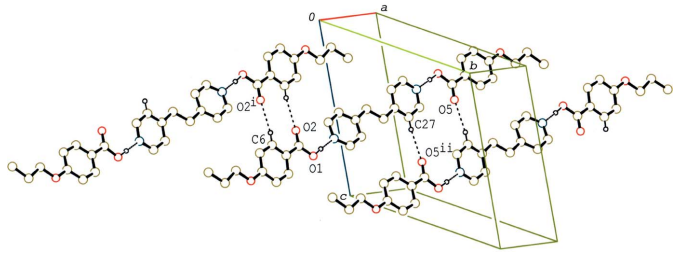

Figure 4
A partial packing diagram of compound (II), showing the tape structure formed by C—H···O interactions. H atoms not involved in C—H···O and O—H···N hydrogen bonds (dashed lines) have been omitted [symmetry codes: (i) $-x-2, -y, -z+1$; (ii) $-x+1, -y+1, -z+1$].

Between the tapes in (II), a weak π–π interaction is observed. The centroid–centroid distance between the C11–C16 benzene ring and the N2/C26–C30($x+1, y, z$) pyridine ring is 3.7115 (18) Å. On the other hand, between the tapes in (III) C—H···π interactions are observed (Table 3). Although the 2:1 units of the three compounds are arranged in the crystals with their long axes parallel to each other, the distinct layer structure leading to a smectic structure, as observed in 4-*n*-butoxybenzoic acid–4,4′-bipyridyl (2/1) (Tabuchi *et al.*, 2015), is not observed.

4. Database survey

A search of the Cambridge Structural Database (Version 5.36, last update February 2015; Groom & Allen, 2014) for co-crystals of 1,2-bis(pyridin-4-yl)ethane with 4-alkoxybenzoic acid gave three structures (Mukherjee & Desiraju, 2014; Aakeröy *et al.*, 2005). A similar compound, 4-pentylbenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), was reported to exhibit transitions to liquid-crystalline phases (smectic A at 421.3 K and nematic at 439.6 K) and the molecular motions were investigated by solid-state NMR (Duer *et al.*, 1996; Clauss *et al.*, 1996).

5. Synthesis and crystallization

Single crystals of compound (I) were obtained by slow evaporation from an acetone solution (150 ml) of 1,2-bis-(pyridin-4-yl)ethane (67 mg) with 4-ethoxybenzoic acid (120 mg) at room temperature. Crystals of compounds (II) and (III) were obtained from ethanol solutions of 1,2-bis-

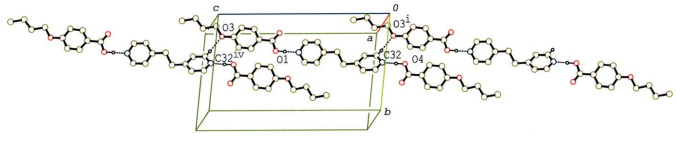

Figure 5
A partial packing diagram of compound (III), showing the tape structure formed by C—H···O interactions. H atoms not involved in C—H···O and O—H···N hydrogen bonds (dashed lines) have been omitted [symmetry codes: (i) $x, y, z-1$; (iv) $x, y, z+1$].

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$2C_9H_{10}O_3{\cdot}C_{12}H_{12}N_2$	$2C_{10}H_{12}O_3{\cdot}C_{12}H_{12}N_2$	$2C_{11}H_{14}O_3{\cdot}C_{12}H_{12}N_2$
M_r	516.57	544.63	572.68
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	93	290	93
a, b, c (Å)	6.967 (3), 9.163 (4), 10.813 (6)	9.121 (3), 12.552 (5), 13.306 (6)	7.702 (2), 10.726 (4), 19.010 (7)
α, β, γ (°)	75.41 (2), 74.97 (2), 77.801 (19)	71.328 (16), 75.076 (18), 89.817 (16)	83.861 (17), 78.794 (16), 73.612 (15)
V (Å^3)	637.3 (6)	1389.2 (9)	1475.5 (9)
Z	1	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.09	0.09
Crystal size (mm)	0.42 × 0.38 × 0.36	0.40 × 0.30 × 0.20	0.40 × 0.20 × 0.10
Data collection			
Diffractometer	Rigaku R-AXIS RAPIDII	Rigaku R-AXIS RAPIDII	Rigaku R-AXIS RAPIDII
Absorption correction	Multi-scan (*ABSCOR*; Higashi, 1995)	Multi-scan (*ABSCOR*; Higashi, 1995)	Multi-scan (*ABSCOR*; Higashi, 1995)
T_{min}, T_{max}	0.877, 0.967	0.601, 0.982	0.768, 0.991
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6408, 2908, 2628	14034, 6347, 4294	14444, 6670, 5219
R_{int}	0.014	0.022	0.021
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.649	0.649
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.113, 1.07	0.041, 0.122, 0.97	0.043, 0.129, 1.06
No. of reflections	2908	6347	6670
No. of parameters	177	371	389
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.40	0.18, −0.21	0.22, −0.36

Computer programs: *RAPID-AUTO* (Rigaku, 2006), *Il Milione* (Burla *et al.*, 2007), *SHELXL2014*/7 (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *CrystalStructure* (Rigaku, 2010) and *PLATON* (Spek, 2009).

(pyridin-4-yl)ethane with 4-*n*-propoxybenzoic acid and 4-*n*-butoxybenzoic acid, respectively, at room temperature [ethanol solution (150 ml) of 1,2-bis(pyridin-4-yl)ethane (62 mg) and 4-*n*-propoxybenzoic acid (120 mg) for (II), and ethanol solution (150 ml) of 1,2-bis(pyridin-4-yl)ethane (57 mg) and 4-*n*-butoxybenzoic acid (120 mg) for (III)].

6. DSC measurements

Phase transitions of 4-methoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) and the title three compounds were observed by DSC and the liquid phases were confirmed by polarizing microscopy. DSC measurements were performed by using a PerkinElmer Pyris 1 in the temperature range from 103 K to the melting temperature at a heating rate of 10 K min^{-1}. Phase transition temperatures (K) and enthalpies (kJ mol^{-1}) determined by DSC are as follows:

4-methoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) 419 (1) [56 (2)] $K_1 \rightarrow N$, 423 (1) [6.3 (13)] $N \rightarrow I$;

(I) 353 (3) [4.5 (5)] $K_1 \rightarrow K_2$, 373 (3) [6.55 (9)] $K_2 \rightarrow K_3$, 404 (1) [0.89 (15)] $K_3 \rightarrow K_4$, 421 (1) [49 (3)] $K_4 \rightarrow N$, 434 (1) [11.7 (10)] $N \rightarrow I$;

(II) 365 (1) [17.9 (9)] $K_1 \rightarrow K_2$, 419 (1) [39 (2)] $K_2 \rightarrow N$, 421 (1) [6.3 (2)] $N \rightarrow I$;

(III) 339 (2) [4.4 (2)] $K_1 \rightarrow K_2$, 399 (1) [0.33 (4)] $K_2 \rightarrow K_3$, 413 (1) [39 (3)] $K_3 \rightarrow S_A$, 419 (1) [0.74 (13)] $S_A \rightarrow N$, 424 (1) [9.8 (15)] $N \rightarrow I$.

K_i, S_A, N and I denote crystal, smectic A, nematic and isotropic phases, respectively.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. For all compounds, C-bound H atoms were positioned geometrically with C—H = 0.93–0.99 Å and were refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). The O-bound H atoms were located in difference Fourier maps and freely refined [refined O—H = 0.966 (18)–1.03 (3) Å].

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supporting information

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Crystal structures of three co-crystals of 1,2-bis(pyridin-4-yl)ethane with 4-alkoxybenzoic acids: 4-ethoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), 4-*n*-propoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) and 4-*n*-butoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1)

Yohei Tabuchi, Kazuma Gotoh and Hiroyuki Ishida

Computing details

For all compounds, data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO* (Rigaku, 2006); data reduction: *RAPID-AUTO* (Rigaku, 2006); program(s) used to solve structure: *Il Milione* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL2014*/7 (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2010) and *PLATON* (Spek, 2009).

(I) 4-Ethoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1)

Crystal data

$2C_9H_{10}O_3{\cdot}C_{12}H_{12}N_2$
M_r = 516.57
Triclinic, $P\overline{1}$
a = 6.967 (3) Å
b = 9.163 (4) Å
c = 10.813 (6) Å
α = 75.41 (2)°
β = 74.97 (2)°
γ = 77.801 (19)°
V = 637.3 (6) Å^3
Z = 1
$F(000)$ = 274.00
D_x = 1.346 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 7263 reflections
θ = 3.1–30.0°
μ = 0.09 mm^{-1}
T = 93 K
Block, colorless
0.42 × 0.38 × 0.36 mm

Data collection

Rigaku R-AXIS RAPIDII diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.877, T_{max} = 0.967
6408 measured reflections
2908 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −9→8
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.113
S = 1.07
2908 reflections
177 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2 + 0.0636P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: mixed	$(\Delta/\sigma)_{max} < 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.25$ e Å^{-3}
	$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Reflections were merged by *SHELXL* according to the crystal class for the calculation of statistics and refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.08950 (10)	0.17801 (7)	0.27364 (6)	0.01773 (17)
O2	0.36783 (10)	0.14762 (7)	0.11743 (7)	0.02284 (18)
O3	0.30648 (10)	−0.53714 (7)	0.43129 (6)	0.01635 (17)
N1	0.08580 (11)	0.46999 (8)	0.17005 (7)	0.01554 (18)
C1	0.25803 (12)	−0.07314 (9)	0.26753 (8)	0.01328 (19)
C2	0.12341 (13)	−0.13188 (10)	0.38081 (8)	0.01439 (19)
H2	0.0175	−0.0645	0.4223	0.017*
C3	0.14273 (13)	−0.28698 (9)	0.43308 (8)	0.01467 (19)
H3	0.0511	−0.3258	0.5104	0.018*
C4	0.29770 (13)	−0.38638 (9)	0.37165 (8)	0.01337 (19)
C5	0.42960 (12)	−0.32990 (9)	0.25669 (8)	0.01389 (19)
H5	0.5326	−0.3976	0.2135	0.017*
C6	0.40911 (12)	−0.17344 (9)	0.20570 (8)	0.01382 (19)
H6	0.4994	−0.1346	0.1277	0.017*
C7	0.24541 (13)	0.09388 (10)	0.21156 (8)	0.01440 (19)
C8	0.46043 (13)	−0.64340 (10)	0.36946 (9)	0.0164 (2)
H8A	0.5947	−0.6172	0.3609	0.020*
H8B	0.4421	−0.6395	0.2808	0.020*
C9	0.44480 (14)	−0.80100 (10)	0.45311 (10)	0.0199 (2)
H9A	0.4609	−0.8032	0.5410	0.030*
H9B	0.5506	−0.8753	0.4136	0.030*
H9C	0.3128	−0.8269	0.4591	0.030*
C10	0.21117 (14)	0.51152 (10)	0.05431 (9)	0.0174 (2)
H10	0.3031	0.4343	0.0159	0.021*
C11	0.21180 (13)	0.66206 (10)	−0.01116 (8)	0.0168 (2)
H11	0.3038	0.6865	−0.0924	0.020*
C12	0.07739 (13)	0.77805 (9)	0.04216 (8)	0.01326 (19)
C13	−0.05253 (12)	0.73458 (9)	0.16120 (8)	0.01421 (19)
H13	−0.1479	0.8093	0.2010	0.017*
C14	−0.04226 (12)	0.58080 (9)	0.22195 (8)	0.01511 (19)
H14	−0.1305	0.5533	0.3043	0.018*
C15	0.08332 (12)	0.94192 (9)	−0.02916 (8)	0.01380 (19)

H15A	0.0760	0.9504	−0.1210	0.017*
H15B	0.2145	0.9686	−0.0308	0.017*
H1	0.091 (2)	0.285 (2)	0.2343 (17)	0.060 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0214 (3)	0.0103 (3)	0.0183 (3)	−0.0011 (2)	−0.0008 (2)	−0.0020 (2)
O2	0.0258 (4)	0.0134 (3)	0.0224 (4)	−0.0043 (3)	0.0035 (3)	0.0005 (2)
O3	0.0206 (3)	0.0087 (3)	0.0158 (3)	−0.0015 (2)	−0.0006 (2)	0.0005 (2)
N1	0.0183 (4)	0.0113 (3)	0.0182 (4)	−0.0028 (3)	−0.0076 (3)	−0.0011 (3)
C1	0.0161 (4)	0.0112 (4)	0.0135 (4)	−0.0030 (3)	−0.0053 (3)	−0.0014 (3)
C2	0.0165 (4)	0.0128 (4)	0.0139 (4)	−0.0016 (3)	−0.0027 (3)	−0.0039 (3)
C3	0.0171 (4)	0.0138 (4)	0.0119 (4)	−0.0037 (3)	−0.0008 (3)	−0.0018 (3)
C4	0.0160 (4)	0.0105 (4)	0.0139 (4)	−0.0026 (3)	−0.0053 (3)	−0.0007 (3)
C5	0.0141 (4)	0.0122 (4)	0.0150 (4)	−0.0006 (3)	−0.0035 (3)	−0.0030 (3)
C6	0.0138 (4)	0.0143 (4)	0.0125 (4)	−0.0037 (3)	−0.0022 (3)	−0.0008 (3)
C7	0.0168 (4)	0.0128 (4)	0.0144 (4)	−0.0027 (3)	−0.0048 (3)	−0.0025 (3)
C8	0.0167 (4)	0.0116 (4)	0.0189 (4)	0.0000 (3)	−0.0035 (3)	−0.0021 (3)
C9	0.0234 (4)	0.0114 (4)	0.0241 (5)	−0.0029 (3)	−0.0075 (4)	0.0004 (3)
C10	0.0220 (4)	0.0125 (4)	0.0175 (4)	−0.0009 (3)	−0.0050 (3)	−0.0037 (3)
C11	0.0205 (4)	0.0134 (4)	0.0145 (4)	−0.0024 (3)	−0.0020 (3)	−0.0016 (3)
C12	0.0147 (4)	0.0115 (4)	0.0151 (4)	−0.0029 (3)	−0.0070 (3)	−0.0009 (3)
C13	0.0132 (4)	0.0116 (4)	0.0169 (4)	−0.0010 (3)	−0.0035 (3)	−0.0019 (3)
C14	0.0152 (4)	0.0128 (4)	0.0163 (4)	−0.0036 (3)	−0.0042 (3)	0.0008 (3)
C15	0.0152 (4)	0.0106 (4)	0.0142 (4)	−0.0026 (3)	−0.0031 (3)	0.0002 (3)

Geometric parameters (Å, º)

O1—C7	1.3242 (11)	C8—C9	1.5076 (13)
O1—H1	0.970 (19)	C8—H8A	0.9900
O2—C7	1.2169 (12)	C8—H8B	0.9900
O3—C4	1.3653 (11)	C9—H9A	0.9800
O3—C8	1.4353 (11)	C9—H9B	0.9800
N1—C14	1.3365 (12)	C9—H9C	0.9800
N1—C10	1.3445 (13)	C10—C11	1.3831 (13)
C1—C6	1.3912 (12)	C10—H10	0.9500
C1—C2	1.3983 (13)	C11—C12	1.3954 (12)
C1—C7	1.4900 (13)	C11—H11	0.9500
C2—C3	1.3825 (13)	C12—C13	1.3874 (14)
C2—H2	0.9500	C12—C15	1.5091 (13)
C3—C4	1.3978 (12)	C13—C14	1.3920 (12)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.3936 (14)	C14—H14	0.9500
C5—C6	1.3913 (12)	C15—C15^{i}	1.5215 (16)
C5—H5	0.9500	C15—H15A	0.9900
C6—H6	0.9500	C15—H15B	0.9900

C7—O1—H1	110.2 (10)	H8A—C8—H8B	108.4
C4—O3—C8	117.24 (7)	C8—C9—H9A	109.5
C14—N1—C10	117.48 (8)	C8—C9—H9B	109.5
C6—C1—C2	119.07 (8)	H9A—C9—H9B	109.5
C6—C1—C7	118.99 (8)	C8—C9—H9C	109.5
C2—C1—C7	121.94 (8)	H9A—C9—H9C	109.5
C3—C2—C1	120.72 (8)	H9B—C9—H9C	109.5
C3—C2—H2	119.6	N1—C10—C11	122.73 (8)
C1—C2—H2	119.6	N1—C10—H10	118.6
C2—C3—C4	119.69 (8)	C11—C10—H10	118.6
C2—C3—H3	120.2	C10—C11—C12	120.01 (9)
C4—C3—H3	120.2	C10—C11—H11	120.0
O3—C4—C5	124.27 (8)	C12—C11—H11	120.0
O3—C4—C3	115.52 (8)	C13—C12—C11	117.06 (8)
C5—C4—C3	120.21 (8)	C13—C12—C15	123.64 (8)
C6—C5—C4	119.43 (8)	C11—C12—C15	119.28 (8)
C6—C5—H5	120.3	C12—C13—C14	119.55 (8)
C4—C5—H5	120.3	C12—C13—H13	120.2
C1—C6—C5	120.84 (8)	C14—C13—H13	120.2
C1—C6—H6	119.6	N1—C14—C13	123.16 (8)
C5—C6—H6	119.6	N1—C14—H14	118.4
O2—C7—O1	123.25 (9)	C13—C14—H14	118.4
O2—C7—C1	122.84 (8)	C12—C15—C15^{i}	115.14 (9)
O1—C7—C1	113.92 (8)	C12—C15—H15A	108.5
O3—C8—C9	108.07 (8)	C15^{i}—C15—H15A	108.5
O3—C8—H8A	110.1	C12—C15—H15B	108.5
C9—C8—H8A	110.1	C15^{i}—C15—H15B	108.5
O3—C8—H8B	110.1	H15A—C15—H15B	107.5
C9—C8—H8B	110.1		

C6—C1—C2—C3	1.73 (13)	C6—C1—C7—O1	176.33 (7)
C7—C1—C2—C3	−177.82 (7)	C2—C1—C7—O1	−4.12 (12)
C1—C2—C3—C4	−0.40 (13)	C4—O3—C8—C9	178.74 (7)
C8—O3—C4—C5	−0.98 (12)	C14—N1—C10—C11	0.07 (13)
C8—O3—C4—C3	178.48 (7)	N1—C10—C11—C12	−0.53 (14)
C2—C3—C4—O3	179.15 (7)	C10—C11—C12—C13	0.12 (13)
C2—C3—C4—C5	−1.37 (13)	C10—C11—C12—C15	178.65 (8)
O3—C4—C5—C6	−178.79 (7)	C11—C12—C13—C14	0.71 (12)
C3—C4—C5—C6	1.78 (13)	C15—C12—C13—C14	−177.75 (7)
C2—C1—C6—C5	−1.31 (13)	C10—N1—C14—C13	0.83 (12)
C7—C1—C6—C5	178.25 (7)	C12—C13—C14—N1	−1.24 (13)
C4—C5—C6—C1	−0.43 (13)	C13—C12—C15—C15^{i}	−7.83 (14)
C6—C1—C7—O2	−3.77 (13)	C11—C12—C15—C15^{i}	173.74 (8)
C2—C1—C7—O2	175.77 (8)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.967 (18)	1.659 (18)	2.6247 (17)	178.0 (14)
C6—H6···O2^{ii}	0.95	2.60	3.406 (2)	144

Symmetry code: (ii) −*x*+1, −*y*, −*z*.

(II) 4-*n*-Propoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1)

Crystal data

$2C_{10}H_{12}O_3·C_{12}H_{12}N_2$
M_r = 544.63
Triclinic, $P\overline{1}$
a = 9.121 (3) Å
b = 12.552 (5) Å
c = 13.306 (6) Å
α = 71.328 (16)°
β = 75.076 (18)°
γ = 89.817 (16)°
V = 1389.2 (9) Å^3
Z = 2
$F(000)$ = 580.00
D_x = 1.302 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 12886 reflections
θ = 3.0–30.1°
μ = 0.09 mm^{-1}
T = 290 K
Block, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Rigaku R-AXIS RAPIDII diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.601, T_{max} = 0.982
14034 measured reflections
6347 independent reflections
4294 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −11→11
k = −16→16
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.122
S = 0.97
6347 reflections
371 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Reflections were merged by *SHELXL* according to the crystal class for the calculation of statistics and refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.86106 (9)	0.14285 (8)	0.69416 (7)	0.0513 (2)
O2	−0.85639 (9)	0.04844 (8)	0.57825 (7)	0.0572 (3)
O3	−1.54698 (8)	−0.05094 (7)	0.86644 (7)	0.0468 (2)
O4	0.77073 (9)	0.45876 (7)	0.19717 (7)	0.0492 (2)
O5	0.76146 (9)	0.54551 (8)	0.31997 (7)	0.0581 (3)
O6	1.46050 (9)	0.64460 (7)	0.05092 (6)	0.0470 (2)
N1	−0.57003 (10)	0.19071 (8)	0.59325 (8)	0.0432 (2)
N2	0.47841 (10)	0.40765 (8)	0.29105 (8)	0.0438 (2)
C1	−1.08880 (11)	0.04396 (9)	0.71057 (8)	0.0356 (2)
C2	−1.16087 (12)	0.06518 (9)	0.80729 (9)	0.0386 (2)
H2	−1.1057	0.1020	0.8382	0.046*
C3	−1.31324 (12)	0.03204 (9)	0.85748 (9)	0.0407 (3)
H3	−1.3601	0.0457	0.9225	0.049*
C4	−1.39693 (12)	−0.02169 (9)	0.81125 (9)	0.0380 (2)
C5	−1.32604 (12)	−0.04396 (10)	0.71520 (9)	0.0407 (3)
H5	−1.3811	−0.0807	0.6843	0.049*
C6	−1.17302 (12)	−0.01094 (9)	0.66615 (9)	0.0399 (2)
H6	−1.1256	−0.0259	0.6019	0.048*
C7	−0.92513 (12)	0.07793 (9)	0.65432 (9)	0.0384 (2)
C8	−1.63612 (12)	−0.10691 (10)	0.82118 (10)	0.0449 (3)
H8A	−1.5929	−0.1764	0.8164	0.054*
H8B	−1.6348	−0.0591	0.7474	0.054*
C9	−1.79706 (13)	−0.13286 (11)	0.89263 (10)	0.0484 (3)
H9A	−1.8427	−0.0631	0.8933	0.058*
H9B	−1.7979	−0.1763	0.9676	0.058*
C10	−1.88892 (14)	−0.19964 (12)	0.84880 (11)	0.0557 (3)
H10A	−1.8425	−0.2680	0.8471	0.084*
H10B	−1.8912	−0.1552	0.7756	0.084*
H10C	−1.9910	−0.2178	0.8960	0.084*
C11	0.99847 (12)	0.55101 (9)	0.19183 (9)	0.0372 (2)
C12	1.08279 (12)	0.51427 (9)	0.10802 (9)	0.0407 (3)
H12	1.0350	0.4672	0.0823	0.049*
C13	1.23598 (13)	0.54654 (10)	0.06260 (9)	0.0426 (3)
H13	1.2907	0.5217	0.0062	0.051*
C14	1.30898 (12)	0.61617 (9)	0.10086 (9)	0.0394 (2)
C15	1.22638 (12)	0.65343 (10)	0.18464 (9)	0.0436 (3)
H15	1.2744	0.6997	0.2111	0.052*
C16	1.07223 (13)	0.62128 (10)	0.22845 (9)	0.0437 (3)
H16	1.0169	0.6474	0.2837	0.052*
C17	0.83294 (12)	0.51878 (9)	0.24279 (9)	0.0400 (2)
C18	1.53914 (13)	0.70883 (10)	0.09605 (10)	0.0467 (3)
H18A	1.5325	0.6659	0.1722	0.056*
H18B	1.4910	0.7782	0.0944	0.056*
C19	1.70299 (13)	0.73646 (11)	0.03172 (10)	0.0467 (3)
H19A	1.7104	0.7790	−0.0447	0.056*

H19B	1.7522	0.6674	0.0342	0.056*
C20	1.78187 (14)	0.80588 (11)	0.08115 (11)	0.0557 (3)
H20A	1.7796	0.7617	0.1553	0.084*
H20B	1.7300	0.8725	0.0813	0.084*
H20C	1.8857	0.8271	0.0379	0.084*
C21	−0.50433 (13)	0.14613 (10)	0.51637 (10)	0.0448 (3)
H21	−0.5642	0.0987	0.4987	0.054*
C22	−0.35180 (12)	0.16703 (10)	0.46180 (10)	0.0443 (3)
H22	−0.3108	0.1336	0.4088	0.053*
C23	−0.25902 (12)	0.23753 (9)	0.48532 (9)	0.0369 (2)
C24	−0.32816 (13)	0.28490 (11)	0.56375 (10)	0.0494 (3)
H24	−0.2715	0.3339	0.5819	0.059*
C25	−0.48225 (13)	0.25931 (12)	0.61548 (10)	0.0543 (3)
H25	−0.5265	0.2919	0.6685	0.065*
C26	0.41740 (13)	0.43030 (11)	0.38378 (10)	0.0474 (3)
H26	0.4803	0.4649	0.4114	0.057*
C27	0.26572 (13)	0.40488 (10)	0.44034 (10)	0.0459 (3)
H27	0.2284	0.4220	0.5048	0.055*
C28	0.16865 (11)	0.35392 (9)	0.40132 (9)	0.0376 (2)
C29	0.23253 (12)	0.32884 (10)	0.30641 (9)	0.0424 (3)
H29	0.1728	0.2926	0.2780	0.051*
C30	0.38599 (13)	0.35788 (10)	0.25382 (10)	0.0453 (3)
H30	0.4264	0.3417	0.1892	0.054*
C31	−0.09183 (12)	0.25894 (10)	0.42528 (9)	0.0401 (3)
H31A	−0.0818	0.2946	0.3472	0.048*
H31B	−0.0489	0.1867	0.4347	0.048*
C32	0.00142 (12)	0.33167 (10)	0.46139 (9)	0.0406 (3)
H32A	−0.0420	0.4037	0.4524	0.049*
H32B	−0.0084	0.2958	0.5394	0.049*
H1	−0.755 (2)	0.1609 (15)	0.6535 (14)	0.098 (6)*
H4	0.658 (2)	0.4399 (16)	0.2335 (15)	0.106 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0343 (4)	0.0726 (6)	0.0542 (5)	−0.0057 (4)	−0.0016 (4)	−0.0386 (5)
O2	0.0386 (4)	0.0833 (6)	0.0575 (5)	−0.0022 (4)	0.0016 (4)	−0.0451 (5)
O3	0.0326 (4)	0.0641 (5)	0.0447 (5)	−0.0036 (4)	−0.0004 (3)	−0.0273 (4)
O4	0.0359 (4)	0.0656 (5)	0.0546 (5)	−0.0020 (4)	−0.0107 (4)	−0.0324 (4)
O5	0.0434 (5)	0.0806 (6)	0.0562 (5)	−0.0068 (4)	−0.0011 (4)	−0.0401 (5)
O6	0.0368 (4)	0.0590 (5)	0.0484 (5)	−0.0050 (4)	−0.0037 (3)	−0.0281 (4)
N1	0.0339 (5)	0.0533 (6)	0.0421 (5)	−0.0010 (4)	−0.0052 (4)	−0.0194 (4)
N2	0.0337 (5)	0.0465 (5)	0.0497 (6)	0.0016 (4)	−0.0118 (4)	−0.0135 (5)
C1	0.0344 (5)	0.0373 (5)	0.0354 (6)	0.0066 (4)	−0.0080 (4)	−0.0137 (5)
C2	0.0375 (5)	0.0444 (6)	0.0388 (6)	0.0037 (5)	−0.0096 (4)	−0.0210 (5)
C3	0.0398 (6)	0.0493 (6)	0.0350 (6)	0.0048 (5)	−0.0038 (4)	−0.0217 (5)
C4	0.0341 (5)	0.0408 (6)	0.0371 (6)	0.0033 (4)	−0.0051 (4)	−0.0139 (5)
C5	0.0367 (5)	0.0497 (6)	0.0397 (6)	0.0000 (5)	−0.0079 (4)	−0.0222 (5)

C6	0.0386 (6)	0.0479 (6)	0.0354 (6)	0.0043 (5)	−0.0048 (4)	−0.0209 (5)
C7	0.0344 (5)	0.0447 (6)	0.0374 (6)	0.0045 (5)	−0.0071 (4)	−0.0171 (5)
C8	0.0373 (6)	0.0527 (7)	0.0468 (7)	−0.0009 (5)	−0.0063 (5)	−0.0230 (5)
C9	0.0389 (6)	0.0548 (7)	0.0466 (7)	−0.0028 (5)	−0.0011 (5)	−0.0184 (6)
C10	0.0407 (6)	0.0621 (8)	0.0621 (8)	−0.0084 (6)	−0.0018 (6)	−0.0270 (7)
C11	0.0370 (5)	0.0407 (6)	0.0357 (6)	0.0032 (4)	−0.0124 (4)	−0.0130 (5)
C12	0.0423 (6)	0.0460 (6)	0.0399 (6)	0.0002 (5)	−0.0151 (5)	−0.0194 (5)
C13	0.0438 (6)	0.0503 (6)	0.0383 (6)	0.0030 (5)	−0.0088 (5)	−0.0224 (5)
C14	0.0380 (5)	0.0427 (6)	0.0378 (6)	0.0026 (5)	−0.0096 (4)	−0.0141 (5)
C15	0.0401 (6)	0.0494 (6)	0.0469 (6)	−0.0026 (5)	−0.0100 (5)	−0.0251 (5)
C16	0.0429 (6)	0.0510 (7)	0.0413 (6)	0.0032 (5)	−0.0073 (5)	−0.0240 (5)
C17	0.0390 (6)	0.0432 (6)	0.0395 (6)	0.0040 (5)	−0.0123 (5)	−0.0149 (5)
C18	0.0424 (6)	0.0506 (7)	0.0512 (7)	−0.0017 (5)	−0.0083 (5)	−0.0256 (6)
C19	0.0397 (6)	0.0505 (7)	0.0487 (7)	−0.0024 (5)	−0.0066 (5)	−0.0191 (5)
C20	0.0434 (6)	0.0641 (8)	0.0609 (8)	−0.0070 (6)	−0.0073 (6)	−0.0276 (7)
C21	0.0378 (6)	0.0512 (7)	0.0484 (7)	−0.0026 (5)	−0.0084 (5)	−0.0229 (5)
C22	0.0381 (6)	0.0529 (7)	0.0457 (6)	0.0014 (5)	−0.0045 (5)	−0.0266 (5)
C23	0.0332 (5)	0.0411 (6)	0.0350 (5)	0.0034 (4)	−0.0078 (4)	−0.0119 (5)
C24	0.0387 (6)	0.0653 (8)	0.0511 (7)	−0.0061 (5)	−0.0058 (5)	−0.0332 (6)
C25	0.0408 (6)	0.0771 (9)	0.0513 (7)	−0.0030 (6)	−0.0006 (5)	−0.0388 (7)
C26	0.0393 (6)	0.0556 (7)	0.0502 (7)	−0.0022 (5)	−0.0164 (5)	−0.0177 (6)
C27	0.0408 (6)	0.0571 (7)	0.0428 (6)	0.0000 (5)	−0.0119 (5)	−0.0202 (5)
C28	0.0334 (5)	0.0388 (6)	0.0388 (6)	0.0033 (4)	−0.0103 (4)	−0.0101 (5)
C29	0.0356 (5)	0.0474 (6)	0.0486 (7)	0.0004 (5)	−0.0120 (5)	−0.0210 (5)
C30	0.0376 (6)	0.0494 (7)	0.0489 (7)	0.0036 (5)	−0.0072 (5)	−0.0200 (5)
C31	0.0322 (5)	0.0482 (6)	0.0397 (6)	0.0003 (5)	−0.0050 (4)	−0.0178 (5)
C32	0.0342 (5)	0.0483 (6)	0.0398 (6)	0.0008 (5)	−0.0079 (4)	−0.0169 (5)

Geometric parameters (Å, º)

O1—C7	1.3136 (14)	C13—C14	1.3891 (15)
O1—H1	0.963 (19)	C13—H13	0.9300
O2—C7	1.2108 (14)	C14—C15	1.3885 (16)
O3—C4	1.3638 (13)	C15—C16	1.3821 (16)
O3—C8	1.4297 (13)	C15—H15	0.9300
O4—C17	1.3157 (14)	C16—H16	0.9300
O4—H4	1.01 (2)	C18—C19	1.4969 (16)
O5—C17	1.2110 (14)	C18—H18A	0.9700
O6—C14	1.3628 (13)	C18—H18B	0.9700
O6—C18	1.4355 (13)	C19—C20	1.5245 (16)
N1—C25	1.3264 (15)	C19—H19A	0.9700
N1—C21	1.3294 (15)	C19—H19B	0.9700
N2—C30	1.3277 (15)	C20—H20A	0.9600
N2—C26	1.3352 (16)	C20—H20B	0.9600
C1—C6	1.3828 (15)	C20—H20C	0.9600
C1—C2	1.3935 (15)	C21—C22	1.3754 (16)
C1—C7	1.4815 (15)	C21—H21	0.9300
C2—C3	1.3778 (15)	C22—C23	1.3841 (15)

C2—H2	0.9300	C22—H22	0.9300
C3—C4	1.3881 (15)	C23—C24	1.3770 (16)
C3—H3	0.9300	C23—C31	1.5079 (15)
C4—C5	1.3897 (16)	C24—C25	1.3840 (16)
C5—C6	1.3810 (15)	C24—H24	0.9300
C5—H5	0.9300	C25—H25	0.9300
C6—H6	0.9300	C26—C27	1.3766 (17)
C8—C9	1.5002 (16)	C26—H26	0.9300
C8—H8A	0.9700	C27—C28	1.3841 (15)
C8—H8B	0.9700	C27—H27	0.9300
C9—C10	1.5211 (16)	C28—C29	1.3812 (16)
C9—H9A	0.9700	C28—C32	1.5084 (15)
C9—H9B	0.9700	C29—C30	1.3846 (16)
C10—H10A	0.9600	C29—H29	0.9300
C10—H10B	0.9600	C30—H30	0.9300
C10—H10C	0.9600	C31—C32	1.5113 (16)
C11—C16	1.3842 (15)	C31—H31A	0.9700
C11—C12	1.3903 (16)	C31—H31B	0.9700
C11—C17	1.4846 (16)	C32—H32A	0.9700
C12—C13	1.3761 (16)	C32—H32B	0.9700
C12—H12	0.9300		

C7—O1—H1	109.0 (11)	O5—C17—O4	122.94 (10)
C4—O3—C8	117.37 (9)	O5—C17—C11	122.69 (10)
C17—O4—H4	111.7 (11)	O4—C17—C11	114.38 (10)
C14—O6—C18	116.48 (9)	O6—C18—C19	110.74 (10)
C25—N1—C21	117.15 (10)	O6—C18—H18A	109.5
C30—N2—C26	117.12 (10)	C19—C18—H18A	109.5
C6—C1—C2	118.76 (10)	O6—C18—H18B	109.5
C6—C1—C7	119.13 (10)	C19—C18—H18B	109.5
C2—C1—C7	122.11 (10)	H18A—C18—H18B	108.1
C3—C2—C1	120.48 (10)	C18—C19—C20	109.14 (10)
C3—C2—H2	119.8	C18—C19—H19A	109.9
C1—C2—H2	119.8	C20—C19—H19A	109.9
C2—C3—C4	120.17 (10)	C18—C19—H19B	109.9
C2—C3—H3	119.9	C20—C19—H19B	109.9
C4—C3—H3	119.9	H19A—C19—H19B	108.3
O3—C4—C3	116.02 (10)	C19—C20—H20A	109.5
O3—C4—C5	124.12 (10)	C19—C20—H20B	109.5
C3—C4—C5	119.85 (10)	H20A—C20—H20B	109.5
C6—C5—C4	119.36 (10)	C19—C20—H20C	109.5
C6—C5—H5	120.3	H20A—C20—H20C	109.5
C4—C5—H5	120.3	H20B—C20—H20C	109.5
C5—C6—C1	121.37 (10)	N1—C21—C22	122.90 (10)
C5—C6—H6	119.3	N1—C21—H21	118.6
C1—C6—H6	119.3	C22—C21—H21	118.6
O2—C7—O1	122.92 (10)	C21—C22—C23	120.34 (11)
O2—C7—C1	122.94 (10)	C21—C22—H22	119.8

O1—C7—C1	114.14 (10)	C23—C22—H22	119.8
O3—C8—C9	109.94 (10)	C24—C23—C22	116.51 (10)
O3—C8—H8A	109.7	C24—C23—C31	123.83 (10)
C9—C8—H8A	109.7	C22—C23—C31	119.66 (10)
O3—C8—H8B	109.7	C23—C24—C25	119.76 (11)
C9—C8—H8B	109.7	C23—C24—H24	120.1
H8A—C8—H8B	108.2	C25—C24—H24	120.1
C8—C9—C10	109.84 (10)	N1—C25—C24	123.33 (11)
C8—C9—H9A	109.7	N1—C25—H25	118.3
C10—C9—H9A	109.7	C24—C25—H25	118.3
C8—C9—H9B	109.7	N2—C26—C27	123.04 (11)
C10—C9—H9B	109.7	N2—C26—H26	118.5
H9A—C9—H9B	108.2	C27—C26—H26	118.5
C9—C10—H10A	109.5	C26—C27—C28	120.05 (11)
C9—C10—H10B	109.5	C26—C27—H27	120.0
H10A—C10—H10B	109.5	C28—C27—H27	120.0
C9—C10—H10C	109.5	C29—C28—C27	116.80 (10)
H10A—C10—H10C	109.5	C29—C28—C32	123.87 (10)
H10B—C10—H10C	109.5	C27—C28—C32	119.32 (10)
C16—C11—C12	118.39 (10)	C28—C29—C30	119.67 (10)
C16—C11—C17	118.83 (10)	C28—C29—H29	120.2
C12—C11—C17	122.78 (10)	C30—C29—H29	120.2
C13—C12—C11	120.97 (10)	N2—C30—C29	123.30 (11)
C13—C12—H12	119.5	N2—C30—H30	118.3
C11—C12—H12	119.5	C29—C30—H30	118.3
C12—C13—C14	120.07 (10)	C23—C31—C32	115.61 (10)
C12—C13—H13	120.0	C23—C31—H31A	108.4
C14—C13—H13	120.0	C32—C31—H31A	108.4
O6—C14—C15	124.08 (10)	C23—C31—H31B	108.4
O6—C14—C13	116.27 (10)	C32—C31—H31B	108.4
C15—C14—C13	119.64 (10)	H31A—C31—H31B	107.4
C16—C15—C14	119.53 (10)	C28—C32—C31	115.92 (10)
C16—C15—H15	120.2	C28—C32—H32A	108.3
C14—C15—H15	120.2	C31—C32—H32A	108.3
C15—C16—C11	121.38 (11)	C28—C32—H32B	108.3
C15—C16—H16	119.3	C31—C32—H32B	108.3
C11—C16—H16	119.3	H32A—C32—H32B	107.4

C6—C1—C2—C3	−0.05 (16)	C17—C11—C16—C15	179.52 (10)
C7—C1—C2—C3	−179.81 (10)	C16—C11—C17—O5	−4.84 (17)
C1—C2—C3—C4	−0.79 (17)	C12—C11—C17—O5	175.67 (11)
C8—O3—C4—C3	−179.71 (10)	C16—C11—C17—O4	175.24 (9)
C8—O3—C4—C5	−0.69 (16)	C12—C11—C17—O4	−4.25 (16)
C2—C3—C4—O3	−179.73 (10)	C14—O6—C18—C19	−178.58 (9)
C2—C3—C4—C5	1.21 (17)	O6—C18—C19—C20	179.25 (10)
O3—C4—C5—C6	−179.77 (10)	C25—N1—C21—C22	0.98 (18)
C3—C4—C5—C6	−0.78 (17)	N1—C21—C22—C23	−0.30 (19)
C4—C5—C6—C1	−0.06 (17)	C21—C22—C23—C24	−0.73 (18)

C2—C1—C6—C5	0.48 (17)	C21—C22—C23—C31	179.59 (10)
C7—C1—C6—C5	−179.76 (10)	C22—C23—C24—C25	1.05 (18)
C6—C1—C7—O2	−8.98 (17)	C31—C23—C24—C25	−179.28 (11)
C2—C1—C7—O2	170.78 (11)	C21—N1—C25—C24	−0.6 (2)
C6—C1—C7—O1	171.02 (10)	C23—C24—C25—N1	−0.4 (2)
C2—C1—C7—O1	−9.22 (15)	C30—N2—C26—C27	−0.28 (18)
C4—O3—C8—C9	179.75 (10)	N2—C26—C27—C28	−0.30 (19)
O3—C8—C9—C10	−176.05 (10)	C26—C27—C28—C29	1.34 (17)
C16—C11—C12—C13	0.19 (17)	C26—C27—C28—C32	−177.13 (10)
C17—C11—C12—C13	179.68 (10)	C27—C28—C29—C30	−1.82 (17)
C11—C12—C13—C14	0.47 (17)	C32—C28—C29—C30	176.57 (10)
C18—O6—C14—C15	5.71 (16)	C26—N2—C30—C29	−0.25 (17)
C18—O6—C14—C13	−175.08 (10)	C28—C29—C30—N2	1.35 (18)
C12—C13—C14—O6	−179.62 (10)	C24—C23—C31—C32	3.71 (17)
C12—C13—C14—C15	−0.37 (17)	C22—C23—C31—C32	−176.64 (10)
O6—C14—C15—C16	178.80 (10)	C29—C28—C32—C31	11.42 (17)
C13—C14—C15—C16	−0.39 (17)	C27—C28—C32—C31	−170.22 (10)
C14—C15—C16—C11	1.07 (18)	C23—C31—C32—C28	−179.72 (9)
C12—C11—C16—C15	−0.96 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.966 (18)	1.657 (19)	2.6207 (17)	175.4 (16)
O4—H4···N2	1.010 (19)	1.610 (19)	2.6198 (17)	179 (2)
C6—H6···O2^{i}	0.93	2.55	3.376 (2)	149
C27—H27···O5^{ii}	0.93	2.52	3.389 (2)	156

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

(III) 4-*n*-Butoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1)

Crystal data

$2C_{11}H_{14}O_3·C_{12}H_{12}N_2$	Z = 2
M_r = 572.68	F(000) = 612.00
Triclinic, $P\overline{1}$	D_x = 1.289 Mg m^{-3}
a = 7.702 (2) Å	Mo $K\alpha$ radiation, λ = 0.71075 Å
b = 10.726 (4) Å	Cell parameters from 15221 reflections
c = 19.010 (7) Å	θ = 3.0–30.2°
α = 83.861 (17)°	μ = 0.09 mm^{-1}
β = 78.794 (16)°	T = 93 K
γ = 73.612 (15)°	Block, colorless
V = 1475.5 (9) Å^3	0.40 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPIDII diffractometer	T_{min} = 0.768, T_{max} = 0.991
Detector resolution: 10.000 pixels mm^{-1}	14444 measured reflections
ω scans	6670 independent reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	5219 reflections with $I > 2\sigma(I)$
	R_{int} = 0.021
	θ_{max} = 27.5°, θ_{min} = 3.0°

$h = -9 \rightarrow 10$
$k = -12 \rightarrow 13$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.129$
$S = 1.06$
6670 reflections
389 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.390P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Reflections were merged by *SHELXL* according to the crystal class for the calculation of statistics and refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.51653 (13)	0.27771 (9)	0.61192 (5)	0.0218 (2)
O2	0.64354 (14)	0.06698 (9)	0.59129 (5)	0.0236 (2)
O3	0.68036 (13)	0.09242 (9)	0.92200 (5)	0.0209 (2)
O4	0.22564 (13)	0.47777 (10)	−0.13269 (5)	0.0230 (2)
O5	0.11681 (15)	0.69204 (10)	−0.11701 (5)	0.0309 (3)
O6	0.08262 (14)	0.65267 (9)	−0.44591 (5)	0.0225 (2)
N1	0.45623 (15)	0.30647 (11)	0.47936 (6)	0.0192 (2)
N2	0.28494 (16)	0.44868 (12)	−0.00061 (6)	0.0218 (3)
C1	0.62039 (17)	0.13761 (13)	0.70801 (7)	0.0163 (3)
C2	0.56165 (17)	0.24210 (13)	0.75274 (7)	0.0182 (3)
H2	0.5050	0.3265	0.7343	0.022*
C3	0.58528 (18)	0.22352 (13)	0.82341 (7)	0.0193 (3)
H3	0.5460	0.2953	0.8533	0.023*
C4	0.66680 (17)	0.09943 (13)	0.85130 (7)	0.0178 (3)
C5	0.72677 (18)	−0.00590 (13)	0.80742 (7)	0.0182 (3)
H5	0.7830	−0.0904	0.8260	0.022*
C6	0.70292 (17)	0.01481 (13)	0.73615 (7)	0.0176 (3)
H6	0.7439	−0.0565	0.7059	0.021*
C7	0.59569 (17)	0.15588 (13)	0.63141 (7)	0.0171 (3)
C8	0.76407 (18)	−0.03082 (13)	0.95543 (7)	0.0191 (3)
H8A	0.8937	−0.0635	0.9321	0.023*
H8B	0.6977	−0.0957	0.9514	0.023*
C9	0.75313 (18)	−0.00767 (14)	1.03313 (7)	0.0201 (3)
H9A	0.6224	0.0241	1.0554	0.024*

H9B	0.8140	0.0612	1.0358	0.024*
C10	0.84284 (19)	−0.12942 (14)	1.07582 (7)	0.0234 (3)
H10A	0.7823	−0.1985	1.0733	0.028*
H10B	0.9738	−0.1611	1.0539	0.028*
C11	0.8294 (2)	−0.10349 (16)	1.15397 (7)	0.0275 (3)
H11A	0.7000	−0.0701	1.1755	0.041*
H11B	0.8958	−0.0390	1.1568	0.041*
H11C	0.8836	−0.1846	1.1800	0.041*
C12	0.13736 (17)	0.61449 (13)	−0.23165 (7)	0.0186 (3)
C13	0.14716 (18)	0.50757 (13)	−0.26965 (7)	0.0200 (3)
H13	0.1674	0.4232	−0.2462	0.024*
C14	0.12777 (18)	0.52337 (13)	−0.34095 (7)	0.0206 (3)
H14	0.1331	0.4502	−0.3662	0.025*
C15	0.10025 (17)	0.64686 (13)	−0.37591 (7)	0.0182 (3)
C16	0.09327 (18)	0.75389 (13)	−0.33906 (7)	0.0192 (3)
H16	0.0764	0.8379	−0.3629	0.023*
C17	0.11117 (17)	0.73674 (13)	−0.26720 (7)	0.0189 (3)
H17	0.1054	0.8099	−0.2418	0.023*
C18	0.15721 (18)	0.60009 (14)	−0.15474 (7)	0.0205 (3)
C19	0.02570 (19)	0.77902 (13)	−0.48132 (7)	0.0196 (3)
H19A	0.1233	0.8246	−0.4871	0.024*
H19B	−0.0871	0.8324	−0.4528	0.024*
C20	−0.01042 (18)	0.75828 (13)	−0.55364 (7)	0.0199 (3)
H20A	−0.0972	0.7036	−0.5473	0.024*
H20B	0.1059	0.7114	−0.5831	0.024*
C21	−0.0906 (2)	0.88721 (14)	−0.59280 (7)	0.0244 (3)
H21A	−0.2101	0.9317	−0.5644	0.029*
H21B	−0.0070	0.9437	−0.5964	0.029*
C22	−0.1187 (2)	0.86963 (16)	−0.66777 (8)	0.0279 (3)
H22A	0.0000	0.8288	−0.6968	0.042*
H22B	−0.1723	0.9547	−0.6903	0.042*
H22C	−0.2017	0.8141	−0.6645	0.042*
C23	0.48692 (18)	0.19855 (13)	0.44432 (7)	0.0200 (3)
H23	0.5283	0.1166	0.4688	0.024*
C24	0.46126 (18)	0.20069 (13)	0.37437 (7)	0.0197 (3)
H24	0.4851	0.1214	0.3517	0.024*
C25	0.40048 (17)	0.31899 (13)	0.33696 (7)	0.0176 (3)
C26	0.3688 (2)	0.43079 (13)	0.37340 (7)	0.0224 (3)
H26	0.3279	0.5140	0.3502	0.027*
C27	0.3969 (2)	0.42053 (14)	0.44380 (7)	0.0227 (3)
H27	0.3729	0.4982	0.4680	0.027*
C28	0.18349 (19)	0.54309 (15)	0.04192 (7)	0.0249 (3)
H28	0.0936	0.6116	0.0230	0.030*
C29	0.20326 (19)	0.54573 (14)	0.11249 (7)	0.0224 (3)
H29	0.1278	0.6151	0.1409	0.027*
C30	0.33331 (17)	0.44704 (13)	0.14174 (7)	0.0176 (3)
C31	0.43955 (19)	0.34872 (13)	0.09681 (7)	0.0213 (3)
H31	0.5310	0.2792	0.1141	0.026*

C32	0.41101 (19)	0.35308 (14)	0.02703 (7)	0.0221 (3)
H32	0.4842	0.2851	−0.0028	0.027*
C33	0.37029 (19)	0.32021 (13)	0.26098 (7)	0.0203 (3)
H33A	0.2547	0.2962	0.2622	0.024*
H33B	0.4717	0.2525	0.2355	0.024*
C34	0.35958 (18)	0.44973 (12)	0.21788 (7)	0.0174 (3)
H34A	0.2562	0.5173	0.2425	0.021*
H34B	0.4741	0.4749	0.2172	0.021*
H1	0.497 (3)	0.288 (2)	0.5597 (13)	0.060 (7)*
H4	0.247 (3)	0.468 (2)	−0.0805 (14)	0.069 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0304 (5)	0.0184 (5)	0.0149 (5)	−0.0016 (4)	−0.0083 (4)	0.0015 (4)
O2	0.0355 (6)	0.0196 (5)	0.0155 (5)	−0.0052 (4)	−0.0069 (4)	−0.0011 (4)
O3	0.0293 (5)	0.0204 (5)	0.0122 (5)	−0.0023 (4)	−0.0087 (4)	0.0002 (4)
O4	0.0268 (5)	0.0238 (5)	0.0158 (5)	−0.0011 (4)	−0.0076 (4)	0.0015 (4)
O5	0.0420 (6)	0.0285 (6)	0.0196 (5)	−0.0005 (5)	−0.0105 (4)	−0.0044 (4)
O6	0.0329 (5)	0.0204 (5)	0.0124 (5)	−0.0031 (4)	−0.0067 (4)	0.0009 (4)
N1	0.0218 (6)	0.0222 (6)	0.0131 (5)	−0.0046 (5)	−0.0048 (4)	0.0013 (4)
N2	0.0261 (6)	0.0261 (6)	0.0139 (5)	−0.0077 (5)	−0.0062 (4)	0.0020 (4)
C1	0.0165 (6)	0.0193 (6)	0.0134 (6)	−0.0050 (5)	−0.0039 (4)	0.0012 (5)
C2	0.0203 (6)	0.0167 (6)	0.0166 (6)	−0.0032 (5)	−0.0052 (5)	0.0022 (5)
C3	0.0231 (7)	0.0183 (6)	0.0160 (6)	−0.0037 (5)	−0.0044 (5)	−0.0023 (5)
C4	0.0174 (6)	0.0231 (7)	0.0131 (6)	−0.0053 (5)	−0.0044 (4)	0.0006 (5)
C5	0.0197 (6)	0.0169 (6)	0.0167 (6)	−0.0020 (5)	−0.0055 (5)	0.0017 (5)
C6	0.0185 (6)	0.0177 (6)	0.0157 (6)	−0.0030 (5)	−0.0028 (5)	−0.0026 (5)
C7	0.0181 (6)	0.0188 (6)	0.0151 (6)	−0.0063 (5)	−0.0036 (4)	0.0013 (5)
C8	0.0204 (6)	0.0201 (7)	0.0163 (6)	−0.0028 (5)	−0.0069 (5)	0.0015 (5)
C9	0.0196 (6)	0.0257 (7)	0.0142 (6)	−0.0044 (5)	−0.0046 (5)	0.0017 (5)
C10	0.0245 (7)	0.0277 (7)	0.0158 (7)	−0.0040 (6)	−0.0055 (5)	0.0036 (5)
C11	0.0292 (8)	0.0367 (8)	0.0166 (7)	−0.0087 (6)	−0.0079 (5)	0.0049 (6)
C12	0.0151 (6)	0.0233 (7)	0.0152 (6)	−0.0017 (5)	−0.0037 (5)	0.0008 (5)
C13	0.0213 (7)	0.0196 (6)	0.0172 (6)	−0.0026 (5)	−0.0051 (5)	0.0025 (5)
C14	0.0253 (7)	0.0176 (6)	0.0184 (7)	−0.0036 (5)	−0.0053 (5)	−0.0018 (5)
C15	0.0180 (6)	0.0216 (7)	0.0129 (6)	−0.0024 (5)	−0.0029 (4)	0.0003 (5)
C16	0.0196 (6)	0.0191 (6)	0.0170 (6)	−0.0031 (5)	−0.0034 (5)	0.0020 (5)
C17	0.0186 (6)	0.0195 (6)	0.0182 (6)	−0.0033 (5)	−0.0042 (5)	−0.0026 (5)
C18	0.0181 (6)	0.0263 (7)	0.0155 (6)	−0.0029 (5)	−0.0043 (5)	0.0007 (5)
C19	0.0244 (7)	0.0187 (6)	0.0143 (6)	−0.0036 (5)	−0.0050 (5)	0.0020 (5)
C20	0.0223 (7)	0.0227 (7)	0.0141 (6)	−0.0049 (5)	−0.0044 (5)	0.0004 (5)
C21	0.0274 (7)	0.0260 (7)	0.0171 (7)	−0.0017 (6)	−0.0069 (5)	0.0011 (5)
C22	0.0291 (8)	0.0355 (8)	0.0200 (7)	−0.0086 (6)	−0.0102 (6)	0.0053 (6)
C23	0.0227 (7)	0.0197 (6)	0.0171 (6)	−0.0044 (5)	−0.0058 (5)	0.0026 (5)
C24	0.0239 (7)	0.0182 (6)	0.0174 (6)	−0.0054 (5)	−0.0051 (5)	−0.0005 (5)
C25	0.0169 (6)	0.0220 (7)	0.0137 (6)	−0.0055 (5)	−0.0029 (4)	0.0009 (5)
C26	0.0312 (7)	0.0177 (6)	0.0167 (7)	−0.0020 (6)	−0.0086 (5)	0.0015 (5)

C27	0.0312 (7)	0.0196 (7)	0.0166 (7)	−0.0032 (6)	−0.0077 (5)	−0.0018 (5)
C28	0.0250 (7)	0.0280 (7)	0.0189 (7)	−0.0005 (6)	−0.0079 (5)	0.0010 (5)
C29	0.0234 (7)	0.0246 (7)	0.0162 (6)	−0.0011 (6)	−0.0037 (5)	−0.0017 (5)
C30	0.0195 (6)	0.0215 (6)	0.0133 (6)	−0.0078 (5)	−0.0038 (5)	0.0010 (5)
C31	0.0246 (7)	0.0208 (7)	0.0170 (7)	−0.0020 (5)	−0.0069 (5)	0.0007 (5)
C32	0.0282 (7)	0.0224 (7)	0.0153 (6)	−0.0056 (6)	−0.0040 (5)	−0.0015 (5)
C33	0.0286 (7)	0.0202 (7)	0.0136 (6)	−0.0074 (5)	−0.0070 (5)	0.0004 (5)
C34	0.0220 (6)	0.0186 (6)	0.0123 (6)	−0.0049 (5)	−0.0051 (5)	−0.0010 (5)

Geometric parameters (Å, º)

O1—C7	1.3234 (16)	C14—C15	1.3962 (19)
O1—H1	1.02 (2)	C14—H14	0.9500
O2—C7	1.2144 (17)	C15—C16	1.391 (2)
O3—C4	1.3606 (16)	C16—C17	1.3871 (19)
O3—C8	1.4372 (16)	C16—H16	0.9500
O4—C18	1.3240 (17)	C17—H17	0.9500
O4—H4	1.03 (3)	C19—C20	1.5053 (18)
O5—C18	1.2146 (18)	C19—H19A	0.9900
O6—C15	1.3568 (16)	C19—H19B	0.9900
O6—C19	1.4374 (16)	C20—C21	1.5246 (19)
N1—C23	1.3374 (18)	C20—H20A	0.9900
N1—C27	1.3380 (18)	C20—H20B	0.9900
N2—C28	1.3314 (18)	C21—C22	1.521 (2)
N2—C32	1.3384 (18)	C21—H21A	0.9900
C1—C6	1.3906 (18)	C21—H21B	0.9900
C1—C2	1.3971 (18)	C22—H22A	0.9800
C1—C7	1.4903 (18)	C22—H22B	0.9800
C2—C3	1.3764 (18)	C22—H22C	0.9800
C2—H2	0.9500	C23—C24	1.3788 (19)
C3—C4	1.3977 (18)	C23—H23	0.9500
C3—H3	0.9500	C24—C25	1.3915 (18)
C4—C5	1.3939 (19)	C24—H24	0.9500
C5—C6	1.3887 (18)	C25—C26	1.3871 (19)
C5—H5	0.9500	C25—C33	1.5051 (18)
C6—H6	0.9500	C26—C27	1.3849 (19)
C8—C9	1.5062 (19)	C26—H26	0.9500
C8—H8A	0.9900	C27—H27	0.9500
C8—H8B	0.9900	C28—C29	1.3833 (19)
C9—C10	1.5243 (19)	C28—H28	0.9500
C9—H9A	0.9900	C29—C30	1.3875 (19)
C9—H9B	0.9900	C29—H29	0.9500
C10—C11	1.519 (2)	C30—C31	1.3946 (19)
C10—H10A	0.9900	C30—C34	1.5044 (18)
C10—H10B	0.9900	C31—C32	1.3799 (19)
C11—H11A	0.9800	C31—H31	0.9500
C11—H11B	0.9800	C32—H32	0.9500
C11—H11C	0.9800	C33—C34	1.5251 (19)

C12—C17	1.3895 (19)	C33—H33A	0.9900
C12—C13	1.396 (2)	C33—H33B	0.9900
C12—C18	1.4865 (18)	C34—H34A	0.9900
C13—C14	1.3796 (19)	C34—H34B	0.9900
C13—H13	0.9500		

C7—O1—H1	112.6 (13)	O5—C18—O4	124.19 (13)
C4—O3—C8	119.21 (10)	O5—C18—C12	122.87 (13)
C18—O4—H4	113.1 (13)	O4—C18—C12	112.93 (12)
C15—O6—C19	117.94 (10)	O6—C19—C20	107.13 (11)
C23—N1—C27	117.21 (12)	O6—C19—H19A	110.3
C28—N2—C32	117.67 (12)	C20—C19—H19A	110.3
C6—C1—C2	118.85 (12)	O6—C19—H19B	110.3
C6—C1—C7	119.97 (12)	C20—C19—H19B	110.3
C2—C1—C7	121.18 (12)	H19A—C19—H19B	108.5
C3—C2—C1	120.44 (12)	C19—C20—C21	111.41 (11)
C3—C2—H2	119.8	C19—C20—H20A	109.3
C1—C2—H2	119.8	C21—C20—H20A	109.3
C2—C3—C4	120.22 (12)	C19—C20—H20B	109.3
C2—C3—H3	119.9	C21—C20—H20B	109.3
C4—C3—H3	119.9	H20A—C20—H20B	108.0
O3—C4—C5	124.82 (12)	C22—C21—C20	112.54 (12)
O3—C4—C3	115.04 (11)	C22—C21—H21A	109.1
C5—C4—C3	120.13 (12)	C20—C21—H21A	109.1
C6—C5—C4	118.85 (12)	C22—C21—H21B	109.1
C6—C5—H5	120.6	C20—C21—H21B	109.1
C4—C5—H5	120.6	H21A—C21—H21B	107.8
C5—C6—C1	121.50 (12)	C21—C22—H22A	109.5
C5—C6—H6	119.3	C21—C22—H22B	109.5
C1—C6—H6	119.3	H22A—C22—H22B	109.5
O2—C7—O1	123.76 (12)	C21—C22—H22C	109.5
O2—C7—C1	122.85 (12)	H22A—C22—H22C	109.5
O1—C7—C1	113.38 (11)	H22B—C22—H22C	109.5
O3—C8—C9	106.52 (11)	N1—C23—C24	123.07 (12)
O3—C8—H8A	110.4	N1—C23—H23	118.5
C9—C8—H8A	110.4	C24—C23—H23	118.5
O3—C8—H8B	110.4	C23—C24—C25	120.01 (12)
C9—C8—H8B	110.4	C23—C24—H24	120.0
H8A—C8—H8B	108.6	C25—C24—H24	120.0
C8—C9—C10	112.97 (12)	C26—C25—C24	116.82 (12)
C8—C9—H9A	109.0	C26—C25—C33	123.58 (12)
C10—C9—H9A	109.0	C24—C25—C33	119.59 (12)
C8—C9—H9B	109.0	C27—C26—C25	119.72 (13)
C10—C9—H9B	109.0	C27—C26—H26	120.1
H9A—C9—H9B	107.8	C25—C26—H26	120.1
C11—C10—C9	111.88 (12)	N1—C27—C26	123.16 (13)
C11—C10—H10A	109.2	N1—C27—H27	118.4
C9—C10—H10A	109.2	C26—C27—H27	118.4

C11—C10—H10B	109.2	N2—C28—C29	122.87 (13)
C9—C10—H10B	109.2	N2—C28—H28	118.6
H10A—C10—H10B	107.9	C29—C28—H28	118.6
C10—C11—H11A	109.5	C28—C29—C30	119.95 (13)
C10—C11—H11B	109.5	C28—C29—H29	120.0
H11A—C11—H11B	109.5	C30—C29—H29	120.0
C10—C11—H11C	109.5	C29—C30—C31	116.87 (12)
H11A—C11—H11C	109.5	C29—C30—C34	120.72 (12)
H11B—C11—H11C	109.5	C31—C30—C34	122.39 (12)
C17—C12—C13	118.89 (12)	C32—C31—C30	119.61 (12)
C17—C12—C18	119.51 (12)	C32—C31—H31	120.2
C13—C12—C18	121.60 (12)	C30—C31—H31	120.2
C14—C13—C12	120.56 (13)	N2—C32—C31	123.03 (13)
C14—C13—H13	119.7	N2—C32—H32	118.5
C12—C13—H13	119.7	C31—C32—H32	118.5
C13—C14—C15	119.97 (13)	C25—C33—C34	115.09 (11)
C13—C14—H14	120.0	C25—C33—H33A	108.5
C15—C14—H14	120.0	C34—C33—H33A	108.5
O6—C15—C16	124.24 (12)	C25—C33—H33B	108.5
O6—C15—C14	115.68 (12)	C34—C33—H33B	108.5
C16—C15—C14	120.08 (12)	H33A—C33—H33B	107.5
C17—C16—C15	119.31 (12)	C30—C34—C33	113.82 (11)
C17—C16—H16	120.3	C30—C34—H34A	108.8
C15—C16—H16	120.3	C33—C34—H34A	108.8
C16—C17—C12	121.17 (12)	C30—C34—H34B	108.8
C16—C17—H17	119.4	C33—C34—H34B	108.8
C12—C17—H17	119.4	H34A—C34—H34B	107.7

C6—C1—C2—C3	0.01 (19)	C18—C12—C17—C16	179.58 (11)
C7—C1—C2—C3	179.91 (12)	C17—C12—C18—O5	14.7 (2)
C1—C2—C3—C4	−0.6 (2)	C13—C12—C18—O5	−166.26 (14)
C8—O3—C4—C5	0.84 (19)	C17—C12—C18—O4	−164.26 (12)
C8—O3—C4—C3	−179.33 (11)	C13—C12—C18—O4	14.75 (18)
C2—C3—C4—O3	−179.01 (12)	C15—O6—C19—C20	−171.63 (10)
C2—C3—C4—C5	0.8 (2)	O6—C19—C20—C21	174.20 (11)
O3—C4—C5—C6	179.42 (12)	C19—C20—C21—C22	176.83 (12)
C3—C4—C5—C6	−0.40 (19)	C27—N1—C23—C24	0.4 (2)
C4—C5—C6—C1	−0.23 (19)	N1—C23—C24—C25	−0.1 (2)
C2—C1—C6—C5	0.43 (19)	C23—C24—C25—C26	0.1 (2)
C7—C1—C6—C5	−179.47 (12)	C23—C24—C25—C33	−179.12 (12)
C6—C1—C7—O2	0.6 (2)	C24—C25—C26—C27	−0.4 (2)
C2—C1—C7—O2	−179.28 (13)	C33—C25—C26—C27	178.76 (13)
C6—C1—C7—O1	−179.56 (11)	C23—N1—C27—C26	−0.7 (2)
C2—C1—C7—O1	0.54 (17)	C25—C26—C27—N1	0.8 (2)
C4—O3—C8—C9	−178.27 (11)	C32—N2—C28—C29	−0.1 (2)
O3—C8—C9—C10	−178.06 (11)	N2—C28—C29—C30	0.0 (2)
C8—C9—C10—C11	−179.82 (12)	C28—C29—C30—C31	0.2 (2)
C17—C12—C13—C14	−1.19 (19)	C28—C29—C30—C34	178.69 (13)

C18—C12—C13—C14	179.80 (12)	C29—C30—C31—C32	−0.4 (2)
C12—C13—C14—C15	0.8 (2)	C34—C30—C31—C32	−178.80 (13)
C19—O6—C15—C16	−9.84 (19)	C28—N2—C32—C31	−0.1 (2)
C19—O6—C15—C14	170.46 (11)	C30—C31—C32—N2	0.3 (2)
C13—C14—C15—O6	−179.97 (11)	C26—C25—C33—C34	18.14 (19)
C13—C14—C15—C16	0.3 (2)	C24—C25—C33—C34	−162.70 (12)
O6—C15—C16—C17	179.36 (12)	C29—C30—C34—C33	133.61 (14)
C14—C15—C16—C17	−0.95 (19)	C31—C30—C34—C33	−48.02 (17)
C15—C16—C17—C12	0.52 (19)	C25—C33—C34—C30	178.79 (10)
C13—C12—C17—C16	0.54 (19)		

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C12–C17 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	1.02 (2)	1.60 (2)	2.6209 (18)	177 (2)
O4—H4···N2	1.03 (3)	1.58 (3)	2.6092 (18)	178.1 (19)
C32—H32···O3^{i}	0.95	2.57	3.524 (2)	177
C11—H11*A*···*Cg*1^{ii}	0.98	2.80	3.662 (2)	148
C33—H33*A*···*Cg*2^{iii}	0.99	2.74	3.598 (2)	145

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