Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS
ISSN 2056-9890

Crystal structures of 4-methoxybenzoic acid–1,3-bis(pyridin-4-yl)propane (2/1) and biphenyl-4,4′-dicarboxylic acid–4-methoxypyridine (1/2)

Kazuma Gotoh and Hiroyuki Ishida*

Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan. *Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

Received 29 June 2017
Accepted 8 July 2017

Edited by A. J. Lough, University of Toronto, Canada

Keywords: crystal structure; 4-methoxybenzoic acid; 1,3-bis(pyridin-4-yl)propane; biphenyl-4,4′-dicarboxylic acid; 4-methoxypyridine.

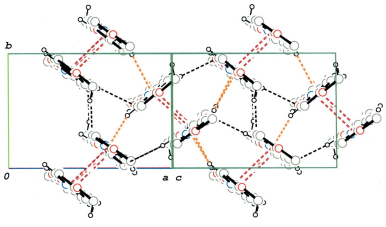
CCDC references: 1560979; 1560978

Supporting information: this article has supporting information at journals.iucr.org/e

The crystal structures of two hydrogen-bonded compounds, namely 4-methoxybenzoic acid–1,3-bis(pyridin-4-yl)propane (2/1), $C_{13}H_{14.59}N_2\cdot C_8H_{7.67}O_3\cdot C_8H_{7.74}O_3$, (I), and biphenyl-4,4′-dicarboxylic acid–4-methoxypyridine (1/2), $C_{14}H_{9.43}O_4\cdot C_6H_{7.32}NO\cdot C_6H_{7.25}NO$, (II), have been determined at 93 K. In (I), the asymmetric unit consists of two crystallographically independent 4-methoxybenzoic acid molecules and one 1,3-bis(pyridin-4-yl)propane molecule. The asymmetric unit of (II) comprises one biphenyl-4,4′-dicarboxylic acid molecule and two independent 4-methoxypyridine molecules. In each crystal, the acid and base molecules are linked by short O—H···N/N—H···O hydrogen bonds, in which H atoms are disordered over the acid O-atom and base N-atom sites, forming a linear hydrogen-bonded 2:1 or 1:2 unit of the acid and the base. The 2:1 units of (I) are linked *via* C—H···π, π–π and C—H···O interactions into a tape structure along [101], while the 1:2 units of (II) form a double-chain structure along [$\bar{1}$01] through π–π and C—H···O interactions.

1. Chemical context

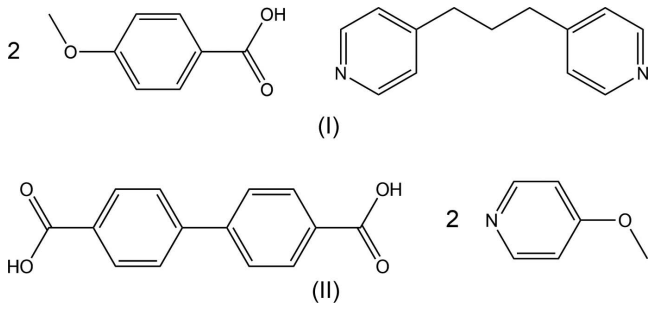
Co-crystals of the 4-alkoxybenzoic acid–4,4′-bipyridyl (2/1) and 4-alkoxybenzoic acid–(*E*)-1,2-bis(pyridin-4-yl)ethene (2/1) systems show thermotropic liquid crystallinity (Kato *et al.*, 1990, 1993; Grunert *et al.*, 1997). Of these co-crystals, the crystal structures of 4,4′-bipyridyl with 4-methoxybenzoic acid (Mukherjee & Desiraju, 2014; Ramon *et al.*, 2014), 4-ethoxy, 4-(*n*-propoxy)- and 4-(*n*-butoxy)benzoic acid (Tabuchi *et al.*, 2015*a*), and the crystal structures of (*E*)-1,2-bis(pyridin-4-yl)ethene with 4-methoxy-, 4-ethoxy-, 4-(*n*-propoxy)-, 4-(*n*-butoxy)-, 4-(*n*-pentyloxy)- and 4-(*n*-hexyloxy)benzoic acid (Tabuchi *et al.*, 2016*a*,*b*) have been reported. In these crystals, the two acids and the base are held together by short intermolecular O—H···N hydrogen bonds, forming linear 2:1 units of the acid and the base. As an expansion of our work on the structural characterization of hydrogen-bonded co-crystals with organic acid and base molecules, we have prepared 4-methoxybenzoic acid–1,3-bis(pyridin-4-yl)propane (2/1), (I), and biphenyl-4,4′-dicarboxylic acid–4-methoxypyridine (1/2), (II), and analyzed the crystal structures.



2. Structural commentary

The molecular structures of (I) and (II) are shown in Figs. 1 and 2, respectively. The asymmetric unit of (I) consists of two crystallographically independent 4-methoxybenzoic acid molecules and one 1,3-bis(pyridin-4-yl)propane molecule. The acid and base molecules are held together *via* short O—H···N/N—H···O hydrogen bonds between the carboxyl O

and pyridine N atoms (Table 1), forming a 2:1 unit. In the hydrogen bonds, the H atoms are disordered over O- and N-atom sites, with occupancy ratios of 0.67 (3):0.33 (3) between atoms O1 and N1, and 0.74 (3):0.26 (3) between atoms O4 and N2. The O1/C7/O2 and N1/C17–C21 planes in one hydrogen-bonded unit are approximately perpendicular to each other, with a dihedral angle of 85.97 (13)°. On the other hand, the O4/C15/O5 and N2/C22–C26 planes in the other hydrogen-bonded unit are approximately planar, with a dihedral angle of 10.18 (14)°, and a weak C—H···O hydrogen bond (C26—H26···O5) is observed in the hydrogen-bonded unit. The dihedral angles between the pyridine N1/C17–C21 and N2/C22–C26 rings, between the benzene C1–C6 and pyridine N1/C17–C21 planes, and between the benzene C9–C14 and pyridine N2/C22–C26 planes are 8.68 (6), 72.93 (6) and 9.05 (6)°, respectively.



The asymmetric unit of (II) is composed of one biphenyl-4,4′-dicarboxylic acid molecule and two crystallographically independent 4-methoxypyridine molecules, and the acid and the two bases are held together by short O—H···N/N—H···O hydrogen bonds (Table 2), forming a linear 1:2 aggregate with pseudo-inversion symmetry. Similar to compound (I), the H atoms in the hydrogen bonds are disordered over two sites, with occupancy ratios of 0.68 (3):0.32 (3) between atoms O1 and N1, and 0.75 (3):0.25 (3) between atoms O3 and N2. The hydrogen-bonded 1:2 unit is approximately planar and weak C—H···O hydrogen bonds (C19—H19···O2 and C25—H25···O4) are observed. The dihedral angle between the benzene rings of the biphenyl-4,4′-dicarboxylic acid is 3.87 (5)°. The N1/C15–C19 pyridine ring makes dihedral angles of 5.62 (12) and 2.55 (5)°, respectively, with the O1/C7/O2 and C1–C6 planes. The N2/C21–C25 pyridine ring makes dihedral angles of 6.84 (12) and 9.50 (5)°, respectively, with the O3/C14/O4 and C8–C13 planes.

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

*Cg*2, *Cg*3 and *Cg*4 are the centroids of the N2/C22–C26, C1–C6 and C9–C14 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.87 (2)	1.71 (2)	2.5730 (16)	170 (3)
O4—H4*A*···N2	0.88 (2)	1.80 (2)	2.6721 (17)	171 (2)
N1—H1*B*···O1	0.89 (2)	1.69 (4)	2.5730 (16)	171 (5)
N2—H4*B*···O4	0.87 (2)	1.80 (4)	2.6720 (16)	177 (7)
C17—H17···O6^{i}	0.95	2.48	3.342 (2)	151
C18—H18···O2^{ii}	0.95	2.60	3.515 (2)	162
C21—H21···O2^{iii}	0.95	2.43	3.2563 (19)	145
C26—H26···O5	0.95	2.36	3.0890 (18)	133
C3—H3···*Cg*4^{iv}	0.95	2.64	3.4265 (19)	140
C5—H5···*Cg*2^{v}	0.95	2.71	3.5440 (18)	146
C10—H10···*Cg*3^{vi}	0.95	2.89	3.5859 (19)	131
C28—H28*B*···*Cg*4^{vii}	0.99	2.91	3.7154 (19)	139

Symmetry codes: (i) $x+1, y, z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x, -y, -z+1$; (iv) $x+1, y-1, z+1$; (v) $-x, -y+1, -z+1$; (vi) $x, y+1, z-1$; (vii) $-x, -y+1, -z$.

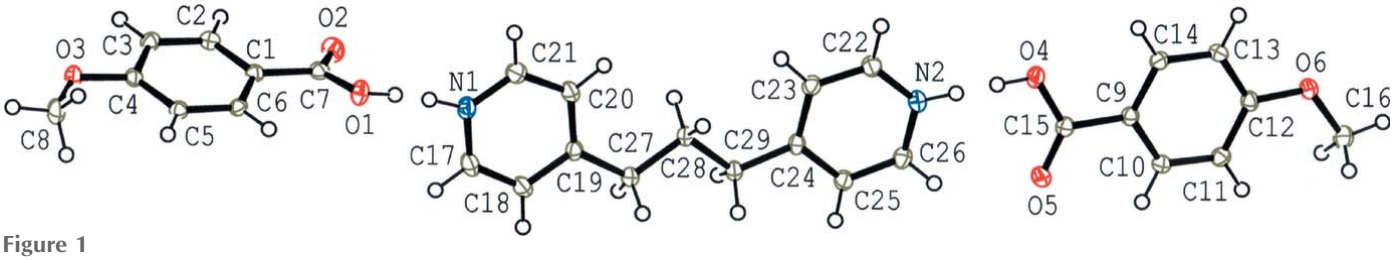


Figure 1
The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are drawn as circles of arbitrary size.

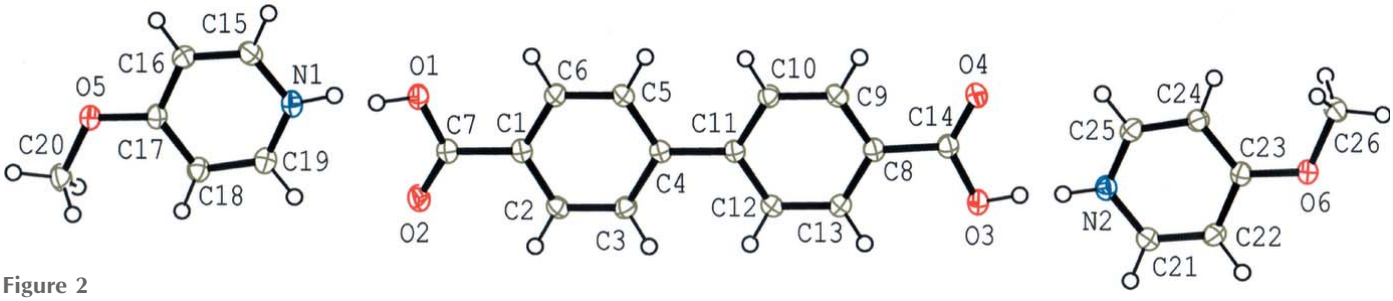


Figure 2
The molecular structure of compound (II), showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are drawn as circles of arbitrary size.

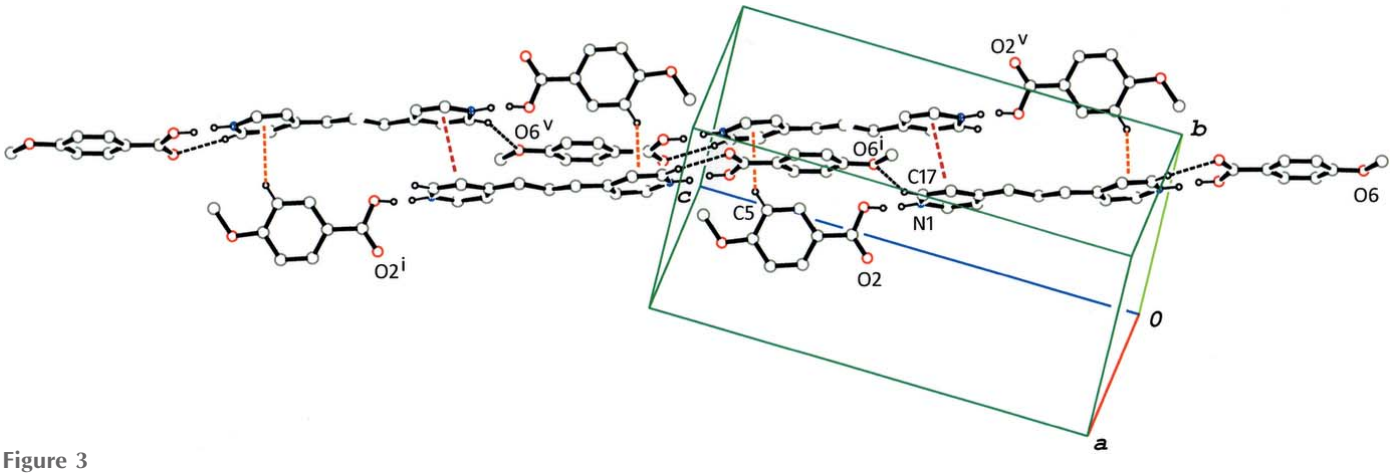


Figure 3
A partial packing diagram of compound (I), showing inversion dimers formed by C—H···π interactions (orange–red dashed lines) and π–π stacking interactions (brown dashed lines), and a tape structure formed by C—H···O hydrogen bonds (black dashed lines) between the dimers. H atoms not involved in the above interactions and O—H···N/N—H···O hydrogen bonds have been omitted. [Symmetry codes: (i) $x+1, y, z+1$; (v) $-x, -y+1, -z+1$.]

3. Supramolecular features

In the crystal of (I), the 2:1 units are linked by a pair of C—H···π interactions (C5—H5···*Cg*2^{v}; *Cg*2 in the centroid of the pyridine N2/C22–C26 ring; symmetry code as given in Table 1), and a π–π interaction [*Cg*1···*Cg*1^{v} = 3.6588 (16) Å; *Cg*1 is the centroid of the pyridine N1/C17–C21 ring], forming an inversion dimer. The dimers are linked *via* C—H···O interactions (C17—H17···O6^{i}; Table 1) into a tape structure running along [101] (Fig. 3). The tapes running along the same direction are further linked *via* the rest of the C—H···O and C—H···π interactions (Table 1), forming a three-dimensional network (Fig. 4).

In the crystal of (II), the 1:2 units are linked by a C—H···O interaction (C20—H20*A*···O6^{ii}; symmetry code as given in Table 2) into a chain structure along [$\bar{1}$01]. Ajacent chains, which are related by an inversion centre, are further linked *via* π–π interactions between pyridine N2/C21–C25 rings [centroid-centroid distance = 3.8113 (13) Å] and between the benzene C1–C6 and pyridine N1/C15–C19 rings [centroid-centroid distance = 3.6613 (12) Å], forming a double-chain structure (Fig. 5). Weak C—H···O and C—H···π interactions are observed between the double chains (Table 2) and the 1:2 units are arranged in the crystals with their long axes parallel to each other (Fig. 6).

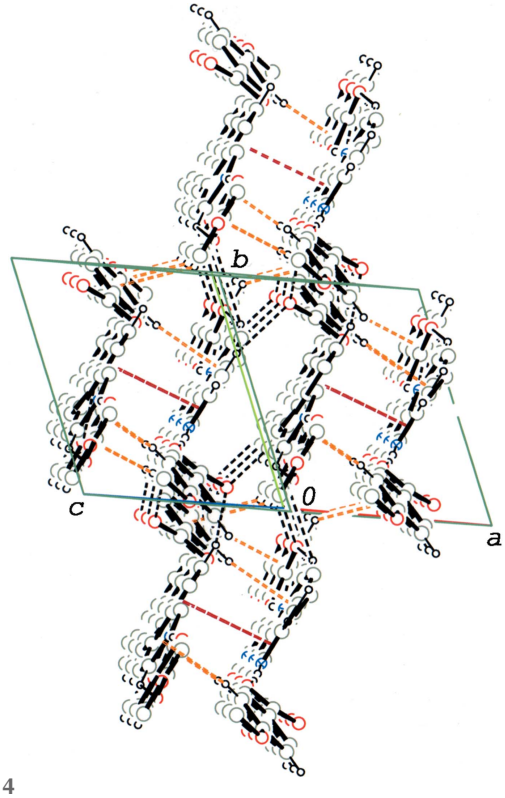


Figure 4
A packing diagram of compound (I), viewed approximately along [101], showing C—H···O hydrogen bonds (black dashed lines). C—H···π interactions (orange–red dashed lines) and π–π stacking interactions (brown dashed lines) formed between molecular tapes. H atoms not involved in the above interactions have been omitted.

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

*Cg*2 and *Cg*4 are the centroids of the C8–C13 and N2/C21–C25 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.87 (2)	1.73 (2)	2.5882 (15)	173 (2)
O3—H3*A*···N2	0.87 (2)	1.74 (2)	2.6078 (15)	175 (2)
N1—H1*B*···O1	0.88 (2)	1.73 (5)	2.5882 (16)	167 (5)
N2—H3*B*···O3	0.88 (2)	1.74 (6)	2.6077 (15)	169 (5)
C10—H10···O2^{i}	0.95	2.57	3.4146 (17)	148
C19—H19···O2	0.95	2.52	3.1901 (17)	128
C20—H20*A*···O6^{ii}	0.98	2.60	3.3210 (18)	131
C25—H25···O4	0.95	2.54	3.2035 (17)	127
C26—H26*B*···O4^{iii}	0.98	2.43	3.3874 (17)	167
C12—H12···*Cg*4^{iv}	0.95	2.90	3.6968 (16)	142
C21—H21···*Cg*2^{iv}	0.95	2.64	3.5284 (16)	155

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

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{13}H_{14.59}N_2{\cdot}C_8H_{7.67}O_3{\cdot}C_8H_{7.74}O_3$	$C_{14}H_{9.43}O_4{\cdot}C_6H_{7.32}NO{\cdot}C_6H_{7.25}NO$
M_r	502.55	460.47
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
Temperature (K)	93	93
a, b, c (Å)	7.759 (3), 8.733 (4), 19.904 (7)	18.354 (6), 7.4166 (16), 16.674 (5)
α, β, γ (°)	91.087 (16), 90.593 (17), 113.241 (15)	90, 104.943 (12), 90
V (Å^3)	1238.7 (8)	2192.9 (10)
Z	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.10	0.10
Crystal size (mm)	0.54 × 0.50 × 0.19	0.45 × 0.40 × 0.35
Data collection		
Diffractometer	Rigaku R-AXIS RAPID II	Rigaku R-AXIS RAPID II
Absorption correction	Numerical (*NUMABS*; Higashi, 1999)	Numerical (*NUMABS*; Higashi, 1999)
T_{min}, T_{max}	0.970, 0.982	0.963, 0.966
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12378, 5653, 4561	20851, 5026, 4085
R_{int}	0.033	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.649
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.106, 1.07	0.038, 0.108, 1.06
No. of reflections	5653	5026
No. of parameters	350	323
No. of restraints	4	4
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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.30	0.35, −0.26

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

4. Database survey

The crystal structures of co-crystals similar to compound (I), namely 4-methoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) (Mukherjee & Desiraju, 2014), 4-ethoxybenzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1), 4-(*n*-propoxy)benzoic acid–bis(pyridin-4-yl)ethane (2/1) and 4-(*n*-butoxy)benzoic acid–1,2-bis(pyridin-4-yl)ethane (2/1) (Tabuchi *et al.*, 2015*b*) have been reported. These compounds also show thermotropic liquid crystallinity (Tabuchi *et al.*, 2015*b*). A search of the Cambridge Structural Database (CSD, Version 5.38, last update February 2017; Groom *et al.*, 2016) for organic co-crystals or salts similar to compound (II), namely 4,4′-biphenyldicarboxylic acid with pyridine derivatives, gave three structures, with CSD refcodes ATOJEZ (Gong *et al.*, 2011), BIKFUX (Cruz *et al.*, 2004) and MAZYUI (Du *et al.*, 2006).

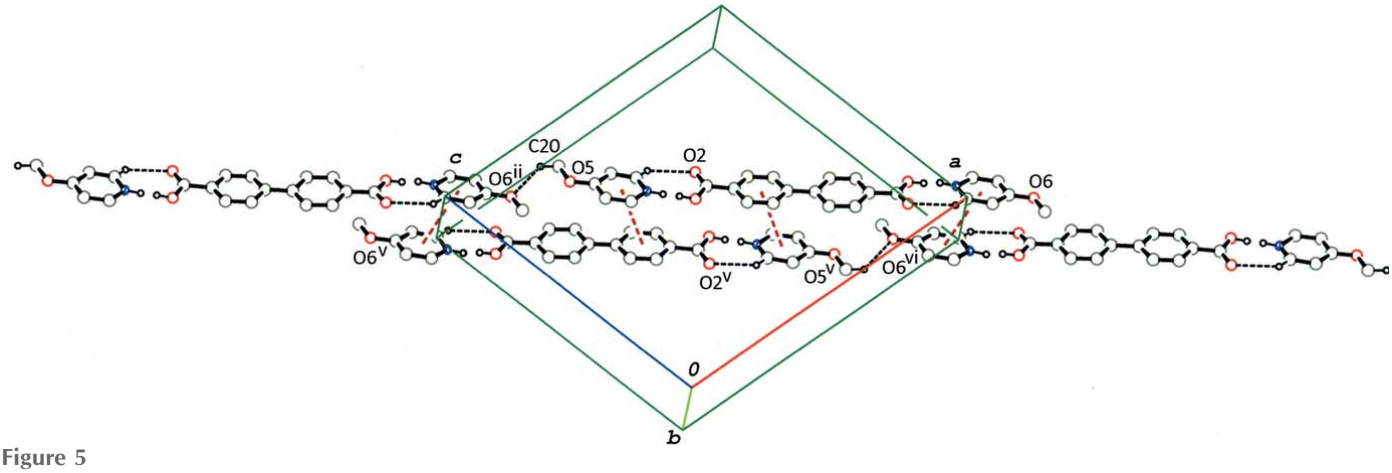


Figure 5
A partial packing diagram of compound (II), showing a double-chain structure formed by O—H···N/N—H···O hydrogen bonds, C—H···O interactions (black dashed lines) and π–π stacking interactions (brown dashed lines). H atoms not involved in the above interactions have been omitted. [Symmetry codes: (ii) $x-1, y, z+1$; (v) $-x+1, -y+1, -z+1$: (vi) $-x+2, -y+1, -z$.]

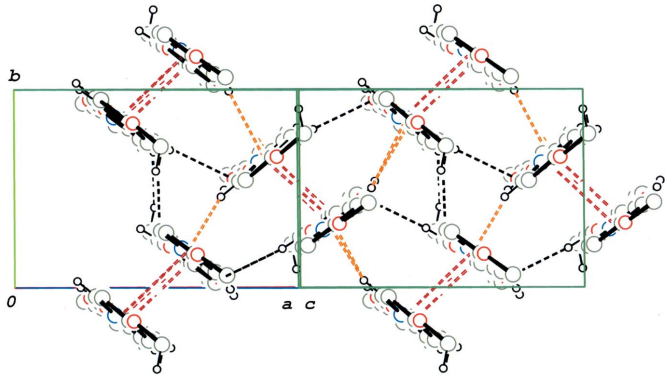


Figure 6
A partial packing diagram of compound (II), viewed along [$\bar{1}$01], showing the arrangement of the molecular chains. C—H···O hydrogen bonds, C—H···π interactions and π–π stacking interactions are shown by black, orange–red and brown dashed lines, respectively. H atoms not involved in the above interactions have been omitted.

5. Synthesis and crystallization

Single crystals of compound (I) were obtained by slow evaporation from an ethanol solution (200 ml) of 1,3-bis-(pyridin-4-yl)propane (100 mg) with 4-methoxybenzoic acid (155 mg) at room temperature. Crystals of compound (I) were obtained by slow evaporation from a 4-methoxypyridine solution (5 ml) of biphenyl-4,4′-dicarboxylic acid (100 mg) at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms in compounds (I) and (II) were found in difference Fourier maps. The H atoms in both compounds which are involved in the O—H···N/N—H···O hydrogen bonds were found to be disordered over two positions in difference Fourier maps. The positional parameters and the occupancy factors were refined with bond-length restraints of O—H = 0.84 (2) Å and N—H = 0.88 (2) Å, and with U_{iso}(H) = 1.5U_{eq}(O,N). Other H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and were treated as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). For compound (I), six reflections were omitted in the final refinement owing to poor agreement between the measured and calculated intensities.

References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G., Siliqi, D. & Spagna, R. (2007). *J. Appl. Cryst.* **40**, 609–613.
Cruz, C., Delgado, R., Drew, M. G. B. & Felix, V. (2004). *Org. Biomol. Chem.* **2**, 2911–2918.
Du, M., You, Y.-P. & Zhang, Z.-H. (2006). *Acta Cryst.* C**62**, o33–o35.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Gong, H.-Y., Rambo, B. M., Karnas, E., Lynch, V. M., Keller, K. M. & Sessler, J. L. (2011). *J. Am. Chem. Soc.* **133**, 1526–1533.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Grunert, M., Howie, A., Kaeding, A. & Imrie, C. T. (1997). *J. Mater. Chem.* **7**, 211–214.
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Kato, T., Fréchet, J. M. J., Wilson, P. G., Saito, T., Uryu, T., Fujishima, A., Jin, C. & Kaneuchi, F. (1993). *Chem. Mater.* **5**, 1094–1100.
Kato, T., Wilson, P. G., Fujishima, A. & Fréchet, J. M. J. (1990). *Chem. Lett.* pp. 2003–2006.
Mukherjee, A. & Desiraju, G. R. (2014). *Cryst. Growth Des.* **14**, 1375–1385.
Ramon, G., Davies, K. & Nassimbeni, L. R. (2014). *CrstEngComm*, **16**, 5802–5810.
Rigaku (2006). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2010). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tabuchi, Y., Gotoh, K. & Ishida, H. (2015*a*). *Acta Cryst.* E**71**, 1290–1295.
Tabuchi, Y., Gotoh, K. & Ishida, H. (2015*b*). *Acta Cryst.* E**71**, 1340–1344.
Tabuchi, Y., Gotoh, K. & Ishida, H. (2016*a*). *Acta Cryst.* E**72**, 1666–1671.
Tabuchi, Y., Gotoh, K. & Ishida, H. (2016*b*). *Acta Cryst.* E**72**, 1771–1775.

supporting information

Acta Cryst. (2017). E**73**, 1192-1196 [https://doi.org/10.1107/S2056989017010167]

Crystal structures of 4-methoxybenzoic acid–1,3-bis(pyridin-4-yl)propane (2/1) and biphenyl-4,4′-dicarboxylic acid–4-methoxypyridine (1/2)

Kazuma Gotoh and Hiroyuki Ishida

Computing details

For both structures, 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* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2010) and *PLATON* (Spek, 2009).

4-Methoxybenzoic acid–1,3-bis(pyridin-4-yl)propane (2/1) (I)

Crystal data

$C_{13}H_{14.59}N_2{\cdot}C_8H_{7.67}O_3{\cdot}C_8H_{7.74}O_3$
M_r = 502.55
Triclinic, $P\overline{1}$
a = 7.759 (3) Å
b = 8.733 (4) Å
c = 19.904 (7) Å
α = 91.087 (16)°
β = 90.593 (17)°
γ = 113.241 (15)°
V = 1238.7 (8) Å^3
Z = 2
$F(000)$ = 532.00
D_x = 1.347 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 13611 reflections
θ = 3.0–30.1°
μ = 0.10 mm^{-1}
T = 93 K
Platelet, colorless
0.54 × 0.50 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID II diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: numerical (NUMABS; Higashi, 1999)
T_{min} = 0.970, T_{max} = 0.982
12378 measured reflections
5653 independent reflections
4561 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −10→10
k = −11→11
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.106
S = 1.07
5653 reflections
350 parameters
4 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

$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

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

Refinement. Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.
_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.13329 (12)	0.23348 (10)	0.63470 (4)	0.02284 (19)	
H1A	0.132 (4)	0.258 (3)	0.5927 (8)	0.034*	0.67 (3)
O2	0.29365 (12)	0.08921 (10)	0.59389 (4)	0.0251 (2)	
O3	0.39277 (11)	0.01871 (10)	0.90676 (4)	0.02030 (18)	
O4	−0.32465 (11)	0.58373 (10)	−0.08794 (4)	0.02144 (18)	
H4A	−0.280 (3)	0.580 (3)	−0.0475 (8)	0.032*	0.74 (3)
O5	−0.10967 (13)	0.84664 (10)	−0.08750 (4)	0.0271 (2)	
O6	−0.44746 (12)	0.77002 (10)	−0.38406 (4)	0.02324 (19)	
N1	0.12203 (14)	0.33437 (12)	0.51474 (5)	0.0211 (2)	
H1B	0.126 (7)	0.290 (5)	0.5542 (14)	0.032*	0.33 (3)
N2	−0.15522 (13)	0.57544 (12)	0.02868 (5)	0.0190 (2)	
H4B	−0.208 (7)	0.577 (7)	−0.0101 (16)	0.028*	0.26 (3)
C1	0.27123 (14)	0.11283 (13)	0.71215 (5)	0.0159 (2)	
C2	0.34417 (15)	−0.00596 (13)	0.72592 (5)	0.0181 (2)	
H2	0.3669 (19)	−0.0701 (16)	0.6893 (7)	0.020 (3)*	
C3	0.38150 (15)	−0.03646 (13)	0.79103 (6)	0.0185 (2)	
H3	0.429441	−0.118989	0.799698	0.022*	
C4	0.34844 (14)	0.05478 (13)	0.84434 (5)	0.0159 (2)	
C5	0.27662 (14)	0.17456 (13)	0.83148 (5)	0.0163 (2)	
H5	0.254784	0.237297	0.867450	0.020*	
C6	0.23720 (14)	0.20155 (12)	0.76565 (5)	0.0157 (2)	
H6	0.186115	0.281924	0.756937	0.019*	
C7	0.23409 (15)	0.14359 (13)	0.64133 (5)	0.0176 (2)	
C8	0.35498 (18)	0.10742 (15)	0.96211 (5)	0.0244 (3)	
H8A	0.428596	0.227192	0.957834	0.037*	
H8B	0.389443	0.069884	1.004341	0.037*	
H8C	0.221074	0.085851	0.962013	0.037*	
C9	−0.29525 (14)	0.73995 (13)	−0.18604 (5)	0.0159 (2)	
C10	−0.19209 (15)	0.88110 (13)	−0.22204 (5)	0.0175 (2)	
H10	−0.088827	0.968468	−0.200711	0.021*	
C11	−0.23745 (15)	0.89642 (13)	−0.28853 (5)	0.0180 (2)	
H11	−0.165531	0.992937	−0.312675	0.022*	

C12	−0.38967 (15)	0.76852 (13)	−0.31932 (5)	0.0174 (2)
C13	−0.49534 (15)	0.62658 (13)	−0.28356 (5)	0.0184 (2)
H13	−0.599812	0.539892	−0.304622	0.022*
C14	−0.44744 (15)	0.61277 (13)	−0.21759 (5)	0.0173 (2)
H14	−0.518706	0.515903	−0.193521	0.021*
C15	−0.23371 (15)	0.73027 (13)	−0.11581 (5)	0.0172 (2)
C16	−0.34828 (18)	0.91561 (15)	−0.42219 (6)	0.0263 (3)
H16A	−0.365021	1.011781	−0.401850	0.039*
H16B	−0.397633	0.897616	−0.468488	0.039*
H16C	−0.214467	0.936813	−0.422242	0.039*
C17	0.26553 (17)	0.48170 (15)	0.50821 (6)	0.0252 (3)
H17	0.347776	0.528136	0.545692	0.030*
C18	0.29914 (17)	0.56891 (15)	0.44953 (6)	0.0238 (3)
H18	0.403164	0.672865	0.446964	0.029*
C19	0.18023 (15)	0.50428 (13)	0.39398 (5)	0.0168 (2)
C20	0.03222 (17)	0.35114 (14)	0.40097 (6)	0.0226 (2)
H20	−0.052170	0.301547	0.364326	0.027*
C21	0.00822 (17)	0.27103 (14)	0.46161 (6)	0.0241 (3)
H21	−0.093865	0.166285	0.465557	0.029*
C22	−0.20434 (16)	0.45762 (14)	0.07493 (5)	0.0203 (2)
H22	−0.314048	0.359357	0.066800	0.024*
C23	−0.10326 (16)	0.47160 (13)	0.13413 (5)	0.0193 (2)
H23	−0.143513	0.384225	0.165454	0.023*
C24	0.05757 (15)	0.61441 (13)	0.14743 (5)	0.0165 (2)
C25	0.11057 (15)	0.73538 (13)	0.09840 (5)	0.0189 (2)
H25	0.220969	0.833944	0.104789	0.023*
C26	0.00299 (16)	0.71201 (14)	0.04075 (5)	0.0198 (2)
H26	0.042030	0.795985	0.007956	0.024*
C27	0.21952 (16)	0.60443 (13)	0.33100 (5)	0.0203 (2)
H27A	0.350222	0.627564	0.318157	0.024*
H27B	0.213336	0.713050	0.342222	0.024*
C28	0.09391 (15)	0.53060 (13)	0.26962 (5)	0.0171 (2)
H28A	−0.035274	0.520828	0.278537	0.021*
H28B	0.089535	0.418013	0.258752	0.021*
C29	0.17452 (15)	0.64592 (13)	0.21094 (5)	0.0188 (2)
H29A	0.202096	0.761447	0.226854	0.023*
H29B	0.295721	0.640333	0.199359	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0278 (4)	0.0288 (4)	0.0157 (4)	0.0149 (3)	0.0010 (3)	0.0051 (3)
O2	0.0313 (4)	0.0287 (4)	0.0168 (4)	0.0133 (4)	0.0017 (3)	−0.0015 (3)
O3	0.0236 (4)	0.0241 (4)	0.0163 (4)	0.0128 (3)	−0.0020 (3)	0.0023 (3)
O4	0.0218 (4)	0.0214 (4)	0.0188 (4)	0.0059 (3)	−0.0023 (3)	0.0059 (3)
O5	0.0331 (5)	0.0214 (4)	0.0199 (4)	0.0034 (4)	−0.0091 (4)	0.0014 (3)
O6	0.0308 (4)	0.0205 (4)	0.0151 (4)	0.0068 (3)	−0.0054 (3)	0.0003 (3)
N1	0.0261 (5)	0.0240 (5)	0.0157 (4)	0.0126 (4)	−0.0012 (4)	0.0013 (4)

N2	0.0197 (4)	0.0230 (5)	0.0156 (4)	0.0100 (4)	−0.0013 (4)	0.0008 (4)
C1	0.0125 (4)	0.0152 (5)	0.0169 (5)	0.0021 (4)	−0.0002 (4)	0.0011 (4)
C2	0.0172 (5)	0.0170 (5)	0.0186 (5)	0.0053 (4)	0.0014 (4)	−0.0009 (4)
C3	0.0176 (5)	0.0170 (5)	0.0228 (5)	0.0087 (4)	0.0004 (4)	0.0023 (4)
C4	0.0129 (4)	0.0168 (5)	0.0158 (5)	0.0033 (4)	−0.0004 (4)	0.0027 (4)
C5	0.0162 (5)	0.0158 (5)	0.0161 (5)	0.0053 (4)	0.0010 (4)	0.0001 (4)
C6	0.0148 (5)	0.0137 (5)	0.0178 (5)	0.0046 (4)	0.0009 (4)	0.0027 (4)
C7	0.0161 (5)	0.0158 (5)	0.0171 (5)	0.0023 (4)	0.0012 (4)	0.0016 (4)
C8	0.0321 (6)	0.0304 (6)	0.0151 (5)	0.0169 (5)	−0.0011 (5)	0.0019 (4)
C9	0.0165 (5)	0.0168 (5)	0.0155 (5)	0.0077 (4)	0.0002 (4)	0.0004 (4)
C10	0.0166 (5)	0.0161 (5)	0.0182 (5)	0.0047 (4)	−0.0005 (4)	0.0003 (4)
C11	0.0182 (5)	0.0172 (5)	0.0175 (5)	0.0057 (4)	0.0018 (4)	0.0023 (4)
C12	0.0215 (5)	0.0197 (5)	0.0140 (5)	0.0113 (4)	−0.0013 (4)	−0.0010 (4)
C13	0.0184 (5)	0.0158 (5)	0.0192 (5)	0.0050 (4)	−0.0034 (4)	−0.0025 (4)
C14	0.0168 (5)	0.0154 (5)	0.0196 (5)	0.0062 (4)	0.0006 (4)	0.0015 (4)
C15	0.0182 (5)	0.0185 (5)	0.0166 (5)	0.0090 (4)	0.0004 (4)	0.0013 (4)
C16	0.0360 (7)	0.0253 (6)	0.0153 (5)	0.0095 (5)	−0.0002 (5)	0.0034 (4)
C17	0.0271 (6)	0.0292 (6)	0.0171 (5)	0.0092 (5)	−0.0063 (5)	−0.0007 (5)
C18	0.0238 (6)	0.0237 (6)	0.0190 (5)	0.0042 (5)	−0.0049 (5)	−0.0003 (4)
C19	0.0195 (5)	0.0188 (5)	0.0149 (5)	0.0108 (4)	−0.0011 (4)	−0.0003 (4)
C20	0.0242 (6)	0.0236 (6)	0.0166 (5)	0.0061 (5)	−0.0047 (4)	0.0002 (4)
C21	0.0265 (6)	0.0219 (5)	0.0198 (5)	0.0052 (5)	−0.0009 (5)	0.0025 (4)
C22	0.0190 (5)	0.0217 (5)	0.0192 (5)	0.0071 (4)	−0.0004 (4)	0.0013 (4)
C23	0.0203 (5)	0.0192 (5)	0.0179 (5)	0.0072 (4)	0.0016 (4)	0.0038 (4)
C24	0.0173 (5)	0.0206 (5)	0.0147 (5)	0.0109 (4)	0.0008 (4)	0.0015 (4)
C25	0.0175 (5)	0.0194 (5)	0.0190 (5)	0.0063 (4)	−0.0002 (4)	0.0026 (4)
C26	0.0209 (5)	0.0216 (5)	0.0175 (5)	0.0088 (4)	0.0017 (4)	0.0043 (4)
C27	0.0238 (5)	0.0185 (5)	0.0165 (5)	0.0061 (4)	−0.0032 (4)	0.0020 (4)
C28	0.0174 (5)	0.0182 (5)	0.0166 (5)	0.0078 (4)	−0.0015 (4)	0.0014 (4)
C29	0.0188 (5)	0.0195 (5)	0.0168 (5)	0.0062 (4)	−0.0027 (4)	0.0025 (4)

Geometric parameters (Å, º)

O1—C7	1.3158 (15)	C11—C12	1.3914 (16)
O1—H1A	0.869 (17)	C11—H11	0.9500
O2—C7	1.2229 (13)	C12—C13	1.3995 (15)
O3—C4	1.3610 (14)	C13—C14	1.3814 (16)
O3—C8	1.4330 (13)	C13—H13	0.9500
O4—C15	1.3289 (13)	C14—H14	0.9500
O4—H4A	0.879 (16)	C16—H16A	0.9800
O5—C15	1.2132 (14)	C16—H16B	0.9800
O6—C12	1.3620 (14)	C16—H16C	0.9800
O6—C16	1.4356 (14)	C17—C18	1.3769 (17)
N1—C21	1.3330 (16)	C17—H17	0.9500
N1—C17	1.3382 (16)	C18—C19	1.3915 (16)
N1—H1B	0.889 (19)	C18—H18	0.9500
N2—C22	1.3360 (15)	C19—C20	1.3874 (16)
N2—C26	1.3470 (15)	C19—C27	1.5061 (15)

N2—H4B	0.87 (2)	C20—C21	1.3838 (17)
C1—C6	1.3928 (14)	C20—H20	0.9500
C1—C2	1.3938 (17)	C21—H21	0.9500
C1—C7	1.4864 (16)	C22—C23	1.3850 (17)
C2—C3	1.3786 (16)	C22—H22	0.9500
C2—H2	0.971 (13)	C23—C24	1.3910 (16)
C3—C4	1.4005 (15)	C23—H23	0.9500
C3—H3	0.9500	C24—C25	1.3936 (15)
C4—C5	1.3919 (16)	C24—C29	1.5043 (16)
C5—C6	1.3878 (15)	C25—C26	1.3755 (17)
C5—H5	0.9500	C25—H25	0.9500
C6—H6	0.9500	C26—H26	0.9500
C8—H8A	0.9800	C27—C28	1.5190 (16)
C8—H8B	0.9800	C27—H27A	0.9900
C8—H8C	0.9800	C27—H27B	0.9900
C9—C10	1.3930 (14)	C28—C29	1.5271 (15)
C9—C14	1.3947 (16)	C28—H28A	0.9900
C9—C15	1.4871 (16)	C28—H28B	0.9900
C10—C11	1.3882 (16)	C29—H29A	0.9900
C10—H10	0.9500	C29—H29B	0.9900

C7—O1—H1A	108.6 (17)	O5—C15—C9	122.12 (10)
C4—O3—C8	116.51 (9)	O4—C15—C9	114.09 (9)
C15—O4—H4A	111.9 (14)	O6—C16—H16A	109.5
C12—O6—C16	117.68 (9)	O6—C16—H16B	109.5
C21—N1—C17	117.75 (10)	H16A—C16—H16B	109.5
C21—N1—H1B	130 (3)	O6—C16—H16C	109.5
C17—N1—H1B	112 (3)	H16A—C16—H16C	109.5
C22—N2—C26	117.21 (10)	H16B—C16—H16C	109.5
C22—N2—H4B	130 (4)	N1—C17—C18	122.84 (11)
C26—N2—H4B	113 (4)	N1—C17—H17	118.6
C6—C1—C2	118.66 (10)	C18—C17—H17	118.6
C6—C1—C7	121.65 (10)	C17—C18—C19	119.78 (11)
C2—C1—C7	119.69 (9)	C17—C18—H18	120.1
C3—C2—C1	121.08 (10)	C19—C18—H18	120.1
C3—C2—H2	119.2 (9)	C20—C19—C18	117.10 (10)
C1—C2—H2	119.7 (9)	C20—C19—C27	124.59 (10)
C2—C3—C4	119.70 (11)	C18—C19—C27	118.31 (10)
C2—C3—H3	120.1	C21—C20—C19	119.63 (11)
C4—C3—H3	120.1	C21—C20—H20	120.2
O3—C4—C5	124.33 (9)	C19—C20—H20	120.2
O3—C4—C3	115.68 (10)	N1—C21—C20	122.90 (11)
C5—C4—C3	119.98 (10)	N1—C21—H21	118.6
C6—C5—C4	119.44 (10)	C20—C21—H21	118.6
C6—C5—H5	120.3	N2—C22—C23	123.27 (11)
C4—C5—H5	120.3	N2—C22—H22	118.4
C5—C6—C1	121.13 (10)	C23—C22—H22	118.4
C5—C6—H6	119.4	C22—C23—C24	119.58 (11)

C1—C6—H6	119.4	C22—C23—H23	120.2
O2—C7—O1	123.73 (11)	C24—C23—H23	120.2
O2—C7—C1	122.00 (11)	C23—C24—C25	116.96 (11)
O1—C7—C1	114.28 (9)	C23—C24—C29	124.26 (10)
O3—C8—H8A	109.5	C25—C24—C29	118.78 (10)
O3—C8—H8B	109.5	C26—C25—C24	119.98 (10)
H8A—C8—H8B	109.5	C26—C25—H25	120.0
O3—C8—H8C	109.5	C24—C25—H25	120.0
H8A—C8—H8C	109.5	N2—C26—C25	122.97 (10)
H8B—C8—H8C	109.5	N2—C26—H26	118.5
C10—C9—C14	119.04 (10)	C25—C26—H26	118.5
C10—C9—C15	117.96 (10)	C19—C27—C28	118.18 (9)
C14—C9—C15	122.98 (10)	C19—C27—H27A	107.8
C11—C10—C9	121.19 (10)	C28—C27—H27A	107.8
C11—C10—H10	119.4	C19—C27—H27B	107.8
C9—C10—H10	119.4	C28—C27—H27B	107.8
C10—C11—C12	119.11 (10)	H27A—C27—H27B	107.1
C10—C11—H11	120.4	C27—C28—C29	108.07 (9)
C12—C11—H11	120.4	C27—C28—H28A	110.1
O6—C12—C11	124.28 (10)	C29—C28—H28A	110.1
O6—C12—C13	115.45 (10)	C27—C28—H28B	110.1
C11—C12—C13	120.27 (10)	C29—C28—H28B	110.1
C14—C13—C12	119.88 (10)	H28A—C28—H28B	108.4
C14—C13—H13	120.1	C24—C29—C28	117.92 (9)
C12—C13—H13	120.1	C24—C29—H29A	107.8
C13—C14—C9	120.51 (10)	C28—C29—H29A	107.8
C13—C14—H14	119.7	C24—C29—H29B	107.8
C9—C14—H14	119.7	C28—C29—H29B	107.8
O5—C15—O4	123.78 (11)	H29A—C29—H29B	107.2

C6—C1—C2—C3	0.30 (16)	C15—C9—C14—C13	−178.28 (10)
C7—C1—C2—C3	179.49 (9)	C10—C9—C15—O5	7.14 (16)
C1—C2—C3—C4	−0.95 (16)	C14—C9—C15—O5	−174.62 (10)
C8—O3—C4—C5	2.42 (14)	C10—C9—C15—O4	−172.09 (9)
C8—O3—C4—C3	−178.29 (9)	C14—C9—C15—O4	6.15 (15)
C2—C3—C4—O3	−178.75 (9)	C21—N1—C17—C18	0.08 (18)
C2—C3—C4—C5	0.57 (15)	N1—C17—C18—C19	0.39 (19)
O3—C4—C5—C6	179.70 (9)	C17—C18—C19—C20	−0.65 (17)
C3—C4—C5—C6	0.44 (15)	C17—C18—C19—C27	179.03 (11)
C4—C5—C6—C1	−1.10 (15)	C18—C19—C20—C21	0.48 (17)
C2—C1—C6—C5	0.73 (15)	C27—C19—C20—C21	−179.17 (11)
C7—C1—C6—C5	−178.44 (9)	C17—N1—C21—C20	−0.26 (18)
C6—C1—C7—O2	166.69 (10)	C19—C20—C21—N1	−0.03 (19)
C2—C1—C7—O2	−12.48 (15)	C26—N2—C22—C23	1.34 (16)
C6—C1—C7—O1	−13.18 (14)	N2—C22—C23—C24	0.23 (17)
C2—C1—C7—O1	167.66 (9)	C22—C23—C24—C25	−1.59 (15)
C14—C9—C10—C11	−0.44 (16)	C22—C23—C24—C29	177.80 (10)
C15—C9—C10—C11	177.87 (10)	C23—C24—C25—C26	1.39 (15)

C9—C10—C11—C12	0.44 (16)	C29—C24—C25—C26	−178.03 (10)
C16—O6—C12—C11	−2.11 (16)	C22—N2—C26—C25	−1.55 (16)
C16—O6—C12—C13	177.66 (9)	C24—C25—C26—N2	0.18 (17)
C10—C11—C12—O6	179.81 (10)	C20—C19—C27—C28	−2.62 (17)
C10—C11—C12—C13	0.04 (16)	C18—C19—C27—C28	177.73 (10)
O6—C12—C13—C14	179.69 (10)	C19—C27—C28—C29	−173.98 (9)
C11—C12—C13—C14	−0.52 (16)	C23—C24—C29—C28	−11.68 (16)
C12—C13—C14—C9	0.53 (16)	C25—C24—C29—C28	167.70 (9)
C10—C9—C14—C13	−0.05 (16)	C27—C28—C29—C24	−170.16 (9)

Hydrogen-bond geometry (Å, º)

*Cg*2, *Cg*3 and *Cg*4 are the centroids of the N2/C22–C26, C1–C6 and C9–C14 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.87 (2)	1.71 (2)	2.5730 (16)	170 (3)
O4—H4*A*···N2	0.88 (2)	1.80 (2)	2.6721 (17)	171 (2)
N1—H1*B*···O1	0.89 (2)	1.69 (4)	2.5730 (16)	171 (5)
N2—H4*B*···O4	0.87 (2)	1.80 (4)	2.6720 (16)	177 (7)
C17—H17···O6^{i}	0.95	2.48	3.342 (2)	151
C18—H18···O2^{ii}	0.95	2.60	3.515 (2)	162
C21—H21···O2^{iii}	0.95	2.43	3.2563 (19)	145
C26—H26···O5	0.95	2.36	3.0890 (18)	133
C3—H3···*Cg*4^{iv}	0.95	2.64	3.4265 (19)	140
C5—H5···*Cg*2^{v}	0.95	2.71	3.5440 (18)	146
C10—H10···*Cg*3^{vi}	0.95	2.89	3.5859 (19)	131
C28—H28*B*···*Cg*4^{vii}	0.99	2.91	3.7154 (19)	139

Symmetry codes: (i) *x*+1, *y*, *z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*, −*y*, −*z*+1; (iv) *x*+1, *y*−1, *z*+1; (v) −*x*, −*y*+1, −*z*+1; (vi) *x*, *y*+1, *z*−1; (vii) −*x*, −*y*+1, −*z*.

Biphenyl-4,4′-dicarboxylic acid–4-methoxypyridine (1/2) (II)

Crystal data

$C_{14}H_{9.43}O_4{\cdot}C_6H_{7.32}NO{\cdot}C_6H_{7.25}NO$
M_r = 460.47
Monoclinic, $P2_1/c$
a = 18.354 (6) Å
b = 7.4166 (16) Å
c = 16.674 (5) Å
β = 104.943 (12)°
V = 2192.9 (10) Å^3
Z = 4
$F(000)$ = 968.00
D_x = 1.395 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 20884 reflections
θ = 3.0–33.0°
μ = 0.10 mm^{-1}
T = 93 K
Block, colorless
0.45 × 0.40 × 0.35 mm

Data collection

Rigaku R-AXIS RAPID II diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: numerical (NUMABS; Higashi, 1999)
T_{min} = 0.963, T_{max} = 0.966
20851 measured reflections
5026 independent reflections
4085 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −23→23
k = −8→9
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.06$
5026 reflections
323 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.1824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

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

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

_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.50808 (4)	0.28798 (11)	0.54421 (5)	0.02581 (19)	
H1A	0.4857 (13)	0.300 (3)	0.5837 (12)	0.039*	0.68 (3)
O2	0.59317 (5)	0.45187 (11)	0.63520 (5)	0.02892 (19)	
O3	0.97286 (4)	0.40830 (10)	0.19753 (5)	0.02486 (18)	
H3A	0.9981 (11)	0.387 (3)	0.1612 (11)	0.037*	0.75 (3)
O4	0.89523 (5)	0.21022 (11)	0.11695 (5)	0.02858 (19)	
O5	0.31541 (4)	0.32860 (10)	0.83543 (5)	0.02399 (18)	
O6	1.16611 (4)	0.33625 (10)	−0.09371 (5)	0.02332 (18)	
N1	0.43554 (5)	0.29753 (12)	0.65825 (6)	0.0224 (2)	
H1B	0.457 (2)	0.279 (6)	0.618 (2)	0.034*	0.32 (3)
N2	1.05080 (5)	0.36571 (12)	0.08839 (6)	0.0214 (2)	
H3B	1.029 (3)	0.375 (8)	0.130 (3)	0.032*	0.25 (3)
C1	0.61880 (6)	0.37495 (13)	0.50687 (6)	0.0189 (2)	
C2	0.68583 (6)	0.47177 (14)	0.52414 (6)	0.0209 (2)	
H2	0.6997	0.5436	0.5729	0.025*	
C3	0.73242 (6)	0.46464 (14)	0.47108 (6)	0.0217 (2)	
H3	0.7783	0.5308	0.4844	0.026*	
C4	0.71359 (6)	0.36215 (13)	0.39803 (6)	0.0174 (2)	
C5	0.64529 (6)	0.26812 (15)	0.38096 (7)	0.0234 (2)	
H5	0.6305	0.1986	0.3316	0.028*	
C6	0.59875 (6)	0.27387 (15)	0.43431 (7)	0.0244 (2)	
H6	0.5527	0.2082	0.4212	0.029*	
C7	0.57184 (6)	0.37705 (13)	0.56804 (6)	0.0200 (2)	
C8	0.86113 (6)	0.32919 (13)	0.23465 (6)	0.0194 (2)	
C9	0.79105 (6)	0.24541 (14)	0.21431 (6)	0.0214 (2)	

H9	0.7757	0.1788	0.1641	0.026*
C10	0.74320 (6)	0.25758 (14)	0.26609 (6)	0.0206 (2)
H10	0.6954	0.2000	0.2505	0.025*
C11	0.76402 (6)	0.35335 (13)	0.34110 (6)	0.0178 (2)
C12	0.83442 (6)	0.43886 (14)	0.36029 (7)	0.0224 (2)
H12	0.8499	0.5057	0.4104	0.027*
C13	0.88208 (6)	0.42841 (14)	0.30799 (7)	0.0222 (2)
H13	0.9292	0.4892	0.3222	0.027*
C14	0.91111 (6)	0.30991 (13)	0.17715 (7)	0.0204 (2)
C15	0.37013 (6)	0.21013 (14)	0.65102 (7)	0.0233 (2)
H15	0.3508	0.1389	0.6029	0.028*
C16	0.32982 (6)	0.21856 (14)	0.70967 (7)	0.0232 (2)
H16	0.2839	0.1538	0.7023	0.028*
C17	0.35741 (6)	0.32408 (13)	0.78043 (6)	0.0198 (2)
C18	0.42461 (6)	0.41775 (14)	0.78814 (6)	0.0222 (2)
H18	0.4448	0.4921	0.8350	0.027*
C19	0.46117 (6)	0.39954 (15)	0.72562 (7)	0.0240 (2)
H19	0.5071	0.4632	0.7309	0.029*
C20	0.34345 (7)	0.43321 (17)	0.90933 (7)	0.0282 (2)
H20A	0.3074	0.4280	0.9436	0.042*
H20B	0.3500	0.5587	0.8942	0.042*
H20C	0.3921	0.3843	0.9406	0.042*
C21	1.11352 (6)	0.46206 (14)	0.09146 (7)	0.0240 (2)
H21	1.1321	0.5391	0.1377	0.029*
C22	1.15210 (6)	0.45467 (14)	0.03100 (7)	0.0236 (2)
H22	1.1959	0.5262	0.0353	0.028*
C23	1.12592 (6)	0.34022 (13)	−0.03684 (6)	0.0192 (2)
C24	1.06118 (6)	0.23911 (14)	−0.04050 (7)	0.0210 (2)
H24	1.0418	0.1591	−0.0855	0.025*
C25	1.02579 (6)	0.25802 (14)	0.02294 (7)	0.0221 (2)
H25	0.9811	0.1905	0.0197	0.027*
C26	1.13950 (7)	0.21987 (15)	−0.16463 (7)	0.0261 (2)
H26A	1.0887	0.2570	−0.1952	0.039*
H26B	1.1382	0.0950	−0.1458	0.039*
H26C	1.1736	0.2285	−0.2011	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0224 (4)	0.0336 (4)	0.0239 (4)	−0.0043 (3)	0.0104 (3)	−0.0059 (3)
O2	0.0271 (4)	0.0399 (5)	0.0216 (4)	−0.0053 (3)	0.0095 (3)	−0.0067 (3)
O3	0.0229 (4)	0.0294 (4)	0.0249 (4)	−0.0019 (3)	0.0108 (3)	−0.0029 (3)
O4	0.0351 (5)	0.0281 (4)	0.0277 (4)	−0.0069 (3)	0.0176 (4)	−0.0069 (3)
O5	0.0257 (4)	0.0266 (4)	0.0228 (4)	−0.0030 (3)	0.0119 (3)	−0.0018 (3)
O6	0.0227 (4)	0.0254 (4)	0.0245 (4)	−0.0023 (3)	0.0109 (3)	−0.0028 (3)
N1	0.0239 (5)	0.0245 (4)	0.0211 (4)	0.0038 (4)	0.0096 (4)	0.0032 (4)
N2	0.0214 (4)	0.0209 (4)	0.0237 (5)	0.0025 (3)	0.0092 (4)	0.0015 (3)
C1	0.0203 (5)	0.0185 (5)	0.0182 (5)	0.0041 (4)	0.0057 (4)	0.0032 (4)

C2	0.0238 (5)	0.0215 (5)	0.0170 (4)	0.0001 (4)	0.0047 (4)	−0.0017 (4)
C3	0.0202 (5)	0.0240 (5)	0.0207 (5)	−0.0034 (4)	0.0051 (4)	−0.0012 (4)
C4	0.0187 (5)	0.0161 (4)	0.0172 (5)	0.0037 (4)	0.0044 (4)	0.0027 (4)
C5	0.0243 (5)	0.0260 (5)	0.0209 (5)	−0.0037 (4)	0.0076 (4)	−0.0057 (4)
C6	0.0212 (5)	0.0290 (5)	0.0245 (5)	−0.0053 (4)	0.0083 (4)	−0.0051 (4)
C7	0.0198 (5)	0.0209 (5)	0.0196 (5)	0.0037 (4)	0.0055 (4)	0.0025 (4)
C8	0.0222 (5)	0.0178 (4)	0.0194 (5)	0.0029 (4)	0.0075 (4)	0.0030 (4)
C9	0.0238 (5)	0.0227 (5)	0.0172 (5)	0.0002 (4)	0.0047 (4)	−0.0021 (4)
C10	0.0186 (5)	0.0229 (5)	0.0201 (5)	−0.0010 (4)	0.0047 (4)	−0.0004 (4)
C11	0.0191 (5)	0.0160 (4)	0.0183 (5)	0.0033 (4)	0.0051 (4)	0.0024 (4)
C12	0.0239 (5)	0.0231 (5)	0.0209 (5)	−0.0020 (4)	0.0069 (4)	−0.0043 (4)
C13	0.0205 (5)	0.0234 (5)	0.0233 (5)	−0.0026 (4)	0.0072 (4)	−0.0014 (4)
C14	0.0232 (5)	0.0182 (5)	0.0210 (5)	0.0020 (4)	0.0081 (4)	0.0031 (4)
C15	0.0288 (6)	0.0205 (5)	0.0209 (5)	0.0009 (4)	0.0071 (4)	−0.0004 (4)
C16	0.0246 (5)	0.0206 (5)	0.0255 (5)	−0.0026 (4)	0.0087 (4)	0.0003 (4)
C17	0.0215 (5)	0.0191 (5)	0.0203 (5)	0.0037 (4)	0.0082 (4)	0.0048 (4)
C18	0.0211 (5)	0.0254 (5)	0.0199 (5)	0.0004 (4)	0.0051 (4)	0.0008 (4)
C19	0.0213 (5)	0.0279 (5)	0.0234 (5)	0.0007 (4)	0.0070 (4)	0.0022 (4)
C20	0.0280 (6)	0.0383 (6)	0.0206 (5)	−0.0024 (5)	0.0103 (4)	−0.0039 (5)
C21	0.0244 (5)	0.0237 (5)	0.0249 (5)	−0.0014 (4)	0.0077 (4)	−0.0042 (4)
C22	0.0204 (5)	0.0230 (5)	0.0280 (5)	−0.0031 (4)	0.0075 (4)	−0.0021 (4)
C23	0.0184 (5)	0.0181 (5)	0.0222 (5)	0.0042 (4)	0.0070 (4)	0.0038 (4)
C24	0.0204 (5)	0.0201 (5)	0.0226 (5)	−0.0006 (4)	0.0057 (4)	−0.0003 (4)
C25	0.0198 (5)	0.0200 (5)	0.0270 (5)	−0.0002 (4)	0.0069 (4)	0.0032 (4)
C26	0.0307 (6)	0.0246 (5)	0.0255 (5)	−0.0005 (4)	0.0114 (5)	−0.0036 (4)

Geometric parameters (Å, º)

O1—C7	1.3130 (13)	C9—C10	1.3841 (15)
O1—H1A	0.866 (17)	C9—H9	0.9500
O2—C7	1.2202 (13)	C10—C11	1.4029 (14)
O3—C14	1.3168 (13)	C10—H10	0.9500
O3—H3A	0.868 (16)	C11—C12	1.4003 (15)
O4—C14	1.2198 (13)	C12—C13	1.3875 (15)
O5—C17	1.3420 (13)	C12—H12	0.9500
O5—C20	1.4345 (13)	C13—H13	0.9500
O6—C23	1.3435 (13)	C15—C16	1.3715 (16)
O6—C26	1.4436 (13)	C15—H15	0.9500
N1—C19	1.3353 (15)	C16—C17	1.3974 (15)
N1—C15	1.3422 (15)	C16—H16	0.9500
N1—H1B	0.88 (2)	C17—C18	1.3924 (15)
N2—C25	1.3347 (14)	C18—C19	1.3841 (16)
N2—C21	1.3446 (15)	C18—H18	0.9500
N2—H3B	0.88 (2)	C19—H19	0.9500
C1—C2	1.3890 (15)	C20—H20A	0.9800
C1—C6	1.3899 (15)	C20—H20B	0.9800
C1—C7	1.4955 (15)	C20—H20C	0.9800
C2—C3	1.3809 (15)	C21—C22	1.3743 (16)

C2—H2	0.9500	C21—H21	0.9500
C3—C4	1.4013 (14)	C22—C23	1.3967 (15)
C3—H3	0.9500	C22—H22	0.9500
C4—C5	1.3981 (15)	C23—C24	1.3931 (15)
C4—C11	1.4885 (15)	C24—C25	1.3838 (16)
C5—C6	1.3837 (15)	C24—H24	0.9500
C5—H5	0.9500	C25—H25	0.9500
C6—H6	0.9500	C26—H26A	0.9800
C8—C9	1.3894 (15)	C26—H26B	0.9800
C8—C13	1.3937 (15)	C26—H26C	0.9800
C8—C14	1.4949 (15)		

C7—O1—H1A	106.0 (15)	C12—C13—H13	119.8
C14—O3—H3A	107.3 (14)	C8—C13—H13	119.8
C17—O5—C20	117.29 (9)	O4—C14—O3	123.73 (10)
C23—O6—C26	117.38 (8)	O4—C14—C8	121.99 (10)
C19—N1—C15	117.52 (10)	O3—C14—C8	114.27 (9)
C19—N1—H1B	127 (3)	N1—C15—C16	123.26 (10)
C15—N1—H1B	115 (3)	N1—C15—H15	118.4
C25—N2—C21	117.36 (10)	C16—C15—H15	118.4
C25—N2—H3B	124 (4)	C15—C16—C17	118.88 (10)
C21—N2—H3B	119 (4)	C15—C16—H16	120.6
C2—C1—C6	118.75 (10)	C17—C16—H16	120.6
C2—C1—C7	119.25 (9)	O5—C17—C18	125.15 (10)
C6—C1—C7	121.97 (9)	O5—C17—C16	116.36 (9)
C3—C2—C1	120.60 (9)	C18—C17—C16	118.48 (10)
C3—C2—H2	119.7	C19—C18—C17	118.15 (10)
C1—C2—H2	119.7	C19—C18—H18	120.9
C2—C3—C4	121.53 (10)	C17—C18—H18	120.9
C2—C3—H3	119.2	N1—C19—C18	123.71 (10)
C4—C3—H3	119.2	N1—C19—H19	118.1
C5—C4—C3	117.05 (9)	C18—C19—H19	118.1
C5—C4—C11	121.38 (9)	O5—C20—H20A	109.5
C3—C4—C11	121.57 (9)	O5—C20—H20B	109.5
C6—C5—C4	121.57 (10)	H20A—C20—H20B	109.5
C6—C5—H5	119.2	O5—C20—H20C	109.5
C4—C5—H5	119.2	H20A—C20—H20C	109.5
C5—C6—C1	120.49 (10)	H20B—C20—H20C	109.5
C5—C6—H6	119.8	N2—C21—C22	123.29 (10)
C1—C6—H6	119.8	N2—C21—H21	118.4
O2—C7—O1	123.81 (10)	C22—C21—H21	118.4
O2—C7—C1	121.71 (10)	C21—C22—C23	118.92 (10)
O1—C7—C1	114.45 (9)	C21—C22—H22	120.5
C9—C8—C13	118.61 (10)	C23—C22—H22	120.5
C9—C8—C14	118.90 (9)	O6—C23—C24	125.08 (9)
C13—C8—C14	122.50 (9)	O6—C23—C22	116.62 (9)
C10—C9—C8	121.00 (10)	C24—C23—C22	118.30 (10)
C10—C9—H9	119.5	C25—C24—C23	118.37 (10)

C8—C9—H9	119.5	C25—C24—H24	120.8
C9—C10—C11	121.21 (10)	C23—C24—H24	120.8
C9—C10—H10	119.4	N2—C25—C24	123.75 (10)
C11—C10—H10	119.4	N2—C25—H25	118.1
C12—C11—C10	117.15 (10)	C24—C25—H25	118.1
C12—C11—C4	121.55 (9)	O6—C26—H26A	109.5
C10—C11—C4	121.30 (9)	O6—C26—H26B	109.5
C13—C12—C11	121.68 (10)	H26A—C26—H26B	109.5
C13—C12—H12	119.2	O6—C26—H26C	109.5
C11—C12—H12	119.2	H26A—C26—H26C	109.5
C12—C13—C8	120.33 (10)	H26B—C26—H26C	109.5

C6—C1—C2—C3	−1.35 (15)	C9—C8—C13—C12	1.60 (15)
C7—C1—C2—C3	176.47 (9)	C14—C8—C13—C12	−178.50 (9)
C1—C2—C3—C4	0.75 (15)	C9—C8—C14—O4	−6.65 (15)
C2—C3—C4—C5	0.35 (15)	C13—C8—C14—O4	173.45 (10)
C2—C3—C4—C11	−179.56 (9)	C9—C8—C14—O3	173.64 (9)
C3—C4—C5—C6	−0.85 (15)	C13—C8—C14—O3	−6.26 (14)
C11—C4—C5—C6	179.06 (10)	C19—N1—C15—C16	−1.16 (15)
C4—C5—C6—C1	0.25 (17)	N1—C15—C16—C17	0.51 (16)
C2—C1—C6—C5	0.86 (16)	C20—O5—C17—C18	−2.33 (14)
C7—C1—C6—C5	−176.90 (9)	C20—O5—C17—C16	178.78 (9)
C2—C1—C7—O2	−4.87 (15)	C15—C16—C17—O5	179.48 (9)
C6—C1—C7—O2	172.88 (10)	C15—C16—C17—C18	0.51 (15)
C2—C1—C7—O1	177.12 (9)	O5—C17—C18—C19	−179.69 (9)
C6—C1—C7—O1	−5.13 (14)	C16—C17—C18—C19	−0.81 (15)
C13—C8—C9—C10	−0.89 (15)	C15—N1—C19—C18	0.82 (15)
C14—C8—C9—C10	179.21 (9)	C17—C18—C19—N1	0.15 (16)
C8—C9—C10—C11	−0.58 (15)	C25—N2—C21—C22	0.17 (15)
C9—C10—C11—C12	1.30 (15)	N2—C21—C22—C23	−0.80 (16)
C9—C10—C11—C4	−178.20 (9)	C26—O6—C23—C24	0.86 (14)
C5—C4—C11—C12	−176.28 (10)	C26—O6—C23—C22	−179.47 (9)
C3—C4—C11—C12	3.63 (14)	C21—C22—C23—O6	−179.22 (9)
C5—C4—C11—C10	3.20 (14)	C21—C22—C23—C24	0.47 (15)
C3—C4—C11—C10	−176.89 (9)	O6—C23—C24—C25	−179.92 (9)
C10—C11—C12—C13	−0.58 (15)	C22—C23—C24—C25	0.42 (14)
C4—C11—C12—C13	178.92 (9)	C21—N2—C25—C24	0.82 (15)
C11—C12—C13—C8	−0.87 (16)	C23—C24—C25—N2	−1.12 (15)

Hydrogen-bond geometry (Å, º)

*Cg*2 and *Cg*4 are the centroids of the C8–C13 and N2/C21–C25 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.87 (2)	1.73 (2)	2.5882 (15)	173 (2)
O3—H3*A*···N2	0.87 (2)	1.74 (2)	2.6078 (15)	175 (2)
N1—H1*B*···O1	0.88 (2)	1.73 (5)	2.5882 (16)	167 (5)
N2—H3*B*···O3	0.88 (2)	1.74 (6)	2.6077 (15)	169 (5)
C10—H10···O2^{i}	0.95	2.57	3.4146 (17)	148

C19—H19···O2	0.95	2.52	3.1901 (17)	128
C20—H20*A*···O6^{ii}	0.98	2.60	3.3210 (18)	131
C25—H25···O4	0.95	2.54	3.2035 (17)	127
C26—H26*B*···O4^{iii}	0.98	2.43	3.3874 (17)	167
C12—H12···*Cg*4^{iv}	0.95	2.90	3.6968 (16)	142
C21—H21···*Cg*2^{iv}	0.95	2.64	3.5284 (16)	155

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