Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# Crystal structures of three co-crystals of 4,4′-bipyridyl with 4-alkoxybenzoic acids: 4-ethoxybenzoic acid–4,4′-bipyridyl (2/1), 4-*n*-propoxybenzoic acid–4,4′-bipyridyl (2/1) and 4-*n*-butoxybenzoic acid–4,4′-bipyridyl (2/1)

**Yohei Tabuchi, 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 26 September 2015
Accepted 2 October 2015

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

**Keywords**: crystal structure; 4,4′-bipyridyl; 4-alkoxybenzoic acid; hydrogen-bonded liquid crystal

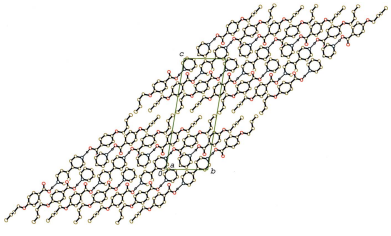
**CCDC references**: 1429203; 1429204; 1429205

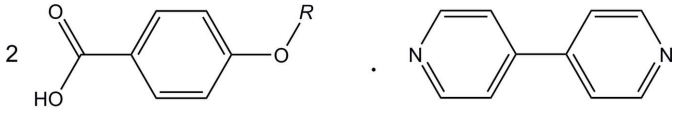
**Supporting information**: this article has supporting information at journals.iucr.org/e

The crystal structures of three hydrogen-bonded co-crystals of 4-alkoxybenzoic acid–4,4′-bipyridyl (2/1), namely, $2C_9H_{10}O_3\cdot C_{10}H_8N_2$, (I), $2C_{10}H_{12}O_3\cdot C_{10}H_8N_2$, (II) and $2C_{11}H_{14}O_3\cdot C_{10}H_8N_2$, (III), have been determined at 93 K. Although the structure of (I) has been determined in the space group $P2_1$ with $Z = 4$ [Lai *et al.* (2008). *J. Struct. Chem.* **49**, 1137–1140], the present study shows that the space group is $P2_1/n$ with $Z = 4$. In each crystal, the components are linked by O—H···N hydrogen bonds, forming a linear hydrogen-bonded 2:1 unit of the acid and the base. The 2:1 unit of (I) adopts nearly pseudo-$C_2$ symmetry, *viz*. twofold rotation around an axis passing through the mid-point of the central C—C bond of 4,4′-bipyridyl, while the units of (II) and (III), except for the terminal alkyl chains, have pseudo-inversion symmetry. The 2:1 units of (I), (II) and (III) are linked *via* C—H···O hydrogen bonds, forming sheet, double-tape and tape structures, respectively.

## 1. Chemical context

The 4-alkoxybenzoic acid–4,4′-bipyridyl (2/1) system, in which the two acids and the base are held together by intermolecular O—H···N hydrogen bonds, shows thermotropic liquid crystallinity (Kato *et al.*, 1990, 1993; Grunert *et al.*, 1997). The compounds of 4-methoxy-, 4-ethoxy- and 4-*n*-propoxybenzoic acid show nematic phases, while the compound of 4-*n*-butoxybenzoic acid exhibits a smectic A phase and then a nematic phase with increasing temperature (Kato *et al.*, 1990, 1993). The crystal structure of 4-methoxybenzoic acid–4,4′-bipyridyl (2/1) was reported recently (Mukherjee & Desiraju, 2014; Ramon *et al.*, 2014). Although the structure of 4-ethoxybenzoic acid–4,4′-bipyridyl (2/1) in space group $P2_1$ was also reported (Lai *et al.*, 2008), the molecular structure is distorted probably due to the wrong choice of space group. In the present study, we have analysed the structure of 4-ethoxybenzoic acid–4,4′-bipyridyl (2/1), (I), as well as the structures of 4-*n*-propoxybenzoic acid–4,4′-bipyridyl (2/1), (II), and 4-*n*-butoxybenzoic acid–4,4′-bipyridyl(2/1), (III).






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

OPEN ACCESS

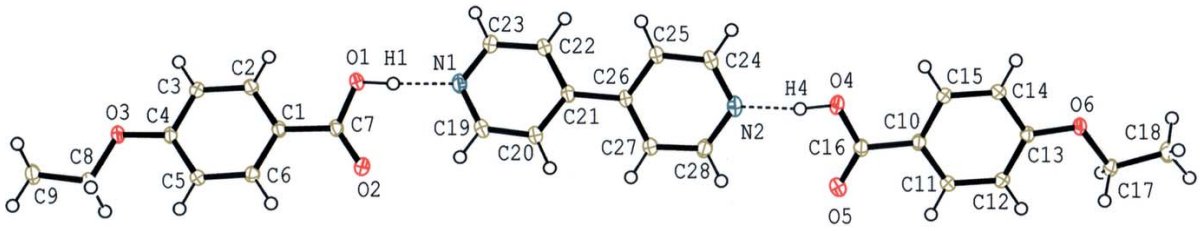



**Figure 1**
The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are drawn as circles of arbitrary size. The O—H···N hydrogen bonds are indicated by dashed lines.

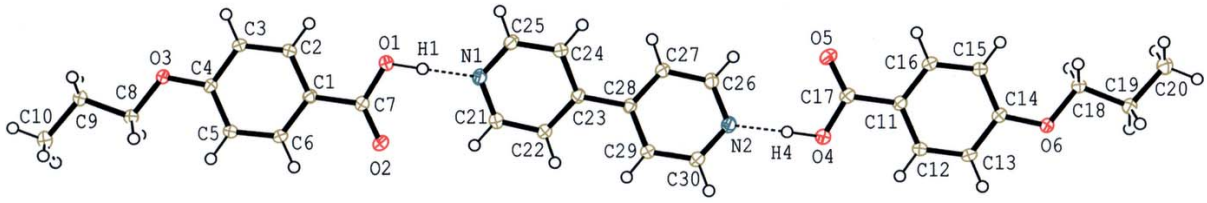



**Figure 2**
The molecular structure of compound (II), showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are drawn as circles of arbitrary size. The O—H···N hydrogen bonds are indicated by dashed lines.

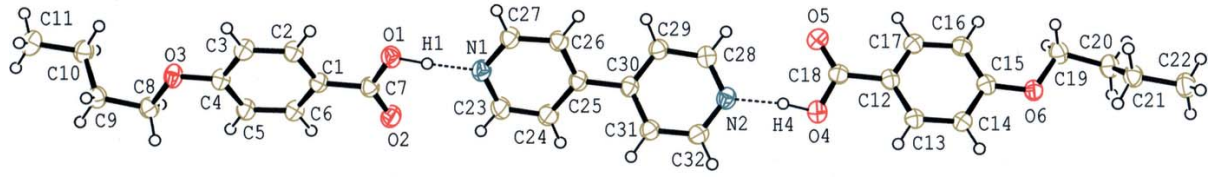



**Figure 3**
The molecular structure of compound (III), showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are drawn as circles of arbitrary size. The O—H···N hydrogen bonds are indicated by dashed lines.

## 2. Structural commentary

The molecular structure of (I) is shown in Fig. 1. Compound (I) crystallizes in the space group $P2_1/n$ with $Z = 4$. For the structure (space group $P2_1$) previously determined by Lai *et al.* (2008), *ADDSYM* in *PLATON* (Spek, 2009) detected missed symmetry elements, *viz.* a centre of inversion and a glide plane. The molecular structures of (II) and (III) are shown in Figs. 2 and 3, respectively. The asymmetric units each comprise two crystallographically independent 4-alkoxybenzoic acid molecules and one 4,4′-bipyridyl molecule, and the two acids and the base are held together by O—H···N hydrogen bonds (Tables 1, 2 and 3), forming a linear hydrogen-bonded 2:1 aggregate. Similar to the reported structure of the 2:1 unit of 4-methoxybenzoic acid–4,4′-bipyridyl (2/1) (Mukherjee & Desiraju, 2014; Ramon *et al.*, 2014), the 2:1 unit of (I) also adopts nearly pseudo-$C_2$ symmetry, *viz.* twofold rotation around an axis passing through the mid-point of the central C21—C26 bond of the 4,4′-bipyridyl molecule. On the other hand, the 2:1 units of (II) and (III), except for the terminal alkyl chains, have pseudo-inversion symmetry.

The dihedral angles between the pyridine rings of 4,4′-bipyridyl are 27.95 (5), 28.84 (4) and 38.76 (12)° for (I), (II) and (III), respectively. The pyridine ring and the carboxyl group hydrogen-bonded to it are twisted slightly to each other. The dihedral angles between the N1/C19–C23 and O1/O2/C7 planes, and the N2/C24–C28 and O4/O5/C16 planes are 6.54 (11) and 10.31 (11)°, respectively, in (I), those between the N1/C21–C25 and O1/O2/C7 planes, and the N2/C26–C30 and O4/O5/C17 planes are 12.13 (10) and 13.96 (10)°, respectively, in (II), and those between the N1/C23–C27 and O1/O2/C7 planes, and the N2/C28–C32 and O4/O5/C18 planes are 13.7 (3) and 8.5 (3)°, respectively, in (III).

The molecular structures of the ethoxy- and propoxybenzoic acids in (I) and (II) are approximately planar. The dihedral angles made by the benzene ring with the carboxyl group and the alkoxy group in each ethoxybenzoic acid in (I) are 9.60 (10), 1.13 (11), 4.48 (9) and 7.57 (9)°, respectively, between the C1–C6 and O1/O2/C7 planes, the C10–C15 and O4/O5/C16 planes, the C1–C6 and O3/C8/C9 planes, and the C10–C15 and O6/C17/C18 planes. The corresponding dihedral angles in (II) are 2.42 (10), 2.48 (10), 2.96 (7) and 5.82 (7)°, respectively, between the C1–C6 and O1/O2/C7 planes, the C11–C16 and O4/O5/C17 planes, the C1–C6 and O3/C8/C9/C10 planes, and the C11–C16 and O6/C18/C19/C20 planes. The butoxybenzoic acid molecules in (III) are also planar, except for the terminal ethyl groups which deviate from the molecular plane with dihedral angles of 66.6 (3) and 60.7 (3)°, respectively, between the C4/O3/C8 and C9/C10/C11planes, and the C15/O6/C19 and C20/C21/C22 planes. The dihedral angles made by the benzene ring with the carboxyl group and the alkoxy group are 5.6 (3), 5.4 (3), 5.2 (2) and 4.3 (2)°, respectively, between the C1–C6 and O1/O2/C7 planes, the

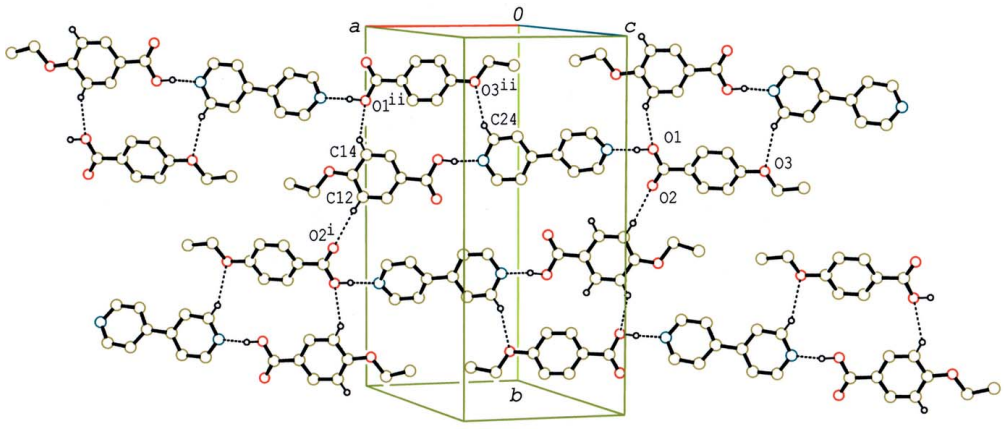



**Figure 4**
A partial packing diagram of compound (I), showing the sheet structure formed by O—H···N and C—H···O hydrogen bonds (dashed lines). H atoms not involved in the hydrogen bonds have been omitted. [Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+\frac{3}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.]

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C10–C15 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.942 (19) | 1.72 (2) | 2.6587 (11) | 177.2 (19) |
| O4—H4···N2 | 0.948 (19) | 1.690 (19) | 2.6312 (11) | 171.4 (19) |
| C12—H12···O2$^{i}$ | 0.95 | 2.43 | 3.3712 (11) | 172 |
| C14—H14···O1$^{ii}$ | 0.95 | 2.56 | 3.2288 (11) | 128 |
| C24—H24···O3$^{ii}$ | 0.95 | 2.57 | 3.4407 (12) | 153 |
| C9—H9*A*···*Cg*2$^{iii}$ | 0.98 | 2.68 | 3.6450 (11) | 169 |
| C18—H18*C*···*Cg*1$^{iv}$ | 0.98 | 2.67 | 3.6253 (11) | 164 |

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

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C11–C16 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 1.03 (2) | 1.61 (2) | 2.6407 (10) | 174.3 (19) |
| O4—H4···N2 | 1.01 (2) | 1.67 (2) | 2.6728 (11) | 173.9 (18) |
| C3—H3···O5$^{i}$ | 0.95 | 2.57 | 3.3981 (11) | 146 |
| C25—H25···O3$^{ii}$ | 0.95 | 2.57 | 3.4581 (11) | 156 |
| C9—H9*B*···*Cg*2$^{iii}$ | 0.99 | 2.84 | 3.6750 (1) | 142 |
| C19—H19*A*···*Cg*1$^{iv}$ | 0.99 | 2.72 | 3.5781 (1) | 146 |

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

**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 | 1.04 (3) | 1.56 (3) | 2.600 (3) | 173 (3) |
| O4—H4···N2 | 1.00 (4) | 1.64 (4) | 2.636 (3) | 172 (4) |
| C24—H24···O5$^{i}$ | 0.95 | 2.47 | 3.408 (3) | 171 |
| C29—H29···O2$^{ii}$ | 0.95 | 2.53 | 3.456 (3) | 164 |
| C2—H2···*Cg*2$^{iii}$ | 0.95 | 2.98 | 3.754 (3) | 139 |
| C8—H8*B*···*Cg*2$^{iv}$ | 0.99 | 2.68 | 3.518 (3) | 143 |
| C19—H19*B*···*Cg*1$^{v}$ | 0.99 | 2.77 | 3.586 (3) | 140 |

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

C12–C17 and O4/O5/C18 planes, the C1–C6 and O3/C8/C9 planes, and the C11–C16 and O6/C19/C20 planes.

## 3. Supramolecular features

In the crystal of (I), the 2:1 units are linked by C—H···O hydrogen bonds (Table 1), forming a sheet structure parallel to (103) (Fig. 4). In addition, the units are stacked in a column through $\pi$–$\pi$ interactions between the acid and base rings along the *a* axis (Fig. 5). The centroid–centroid distances between the C1–C6 and N1/C19–C23($x-1, y, z$) rings, and between the C10–C15 and N2/C24–C28 ($x+1, y, z$) rings are

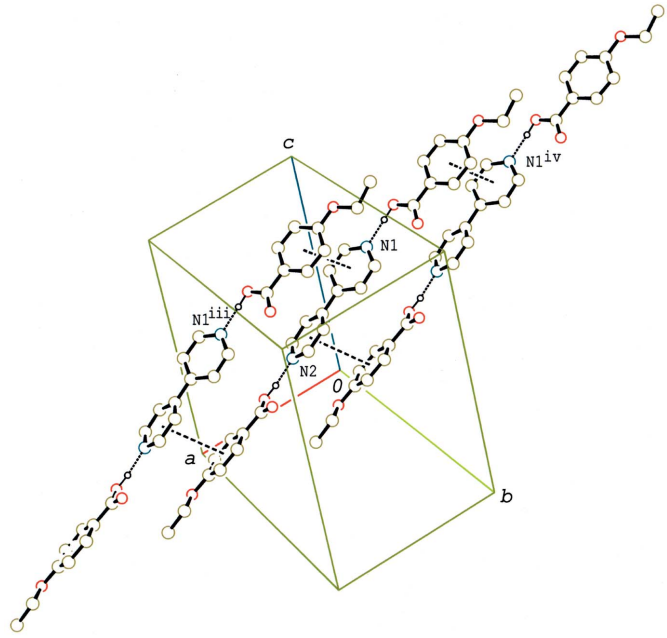



**Figure 5**
A partial packing diagram of compound (I), showing the column structure formed by $\pi$–$\pi$ stacking interactions (dashed lines). H atoms not involved in the O—H···N hydrogen bonds have been omitted. [Symmetry codes: (iii) $x+1, y, z$; (iv) $x-1, y, z$.]

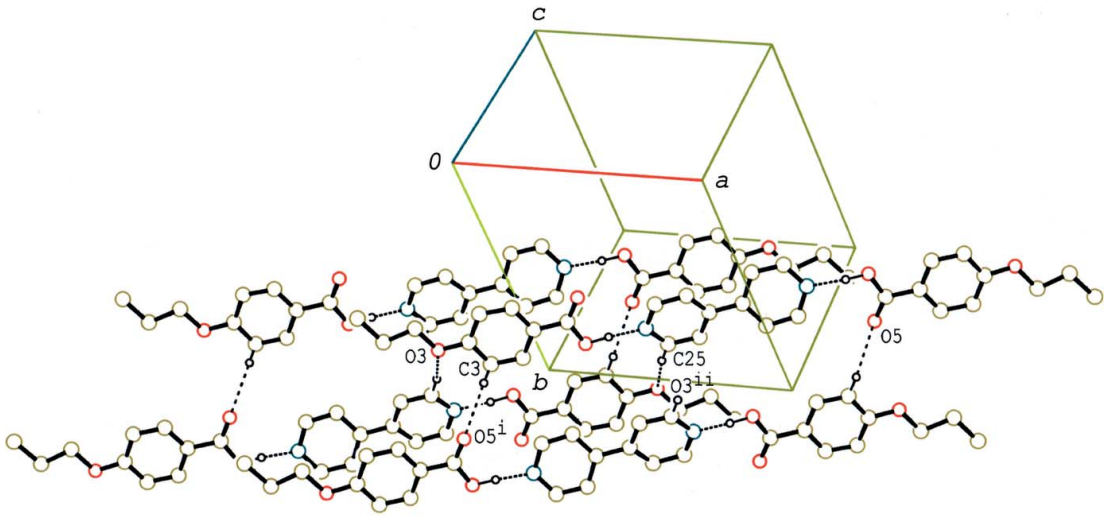



**Figure 6**
A partial packing diagram of compound (II), showing the double-tape structure formed by C—H···O interactions. H atoms not involved in the C—H···O and O—H···N hydrogen bonds (dashed lines) have been omitted. [Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $-x, -y+2, -z$.]

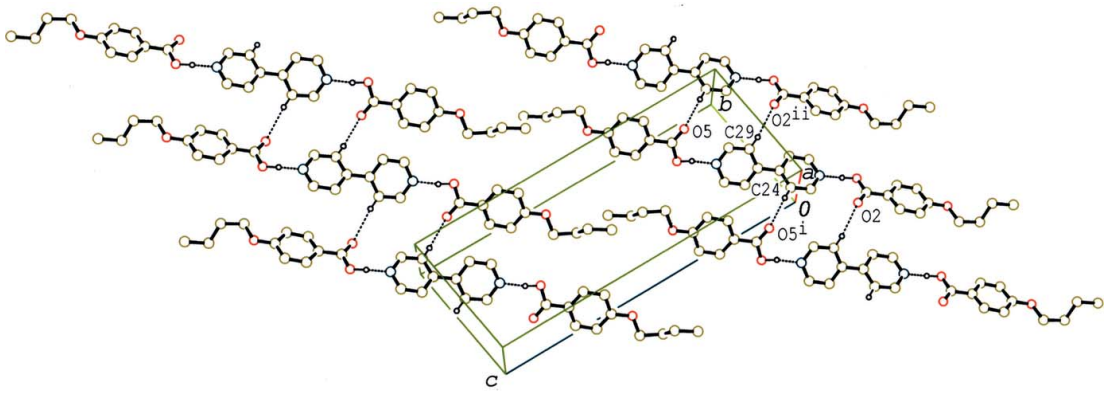



**Figure 7**
A partial packing diagram of compound (III), showing the tape structure formed by C—H···O interactions. H atoms not involved in the C—H···O and O—H···N hydrogen bonds (dashed lines) have been omitted. [Symmetry codes: (i) $x, y-1, z$; (ii) $x, y+1, z$.]

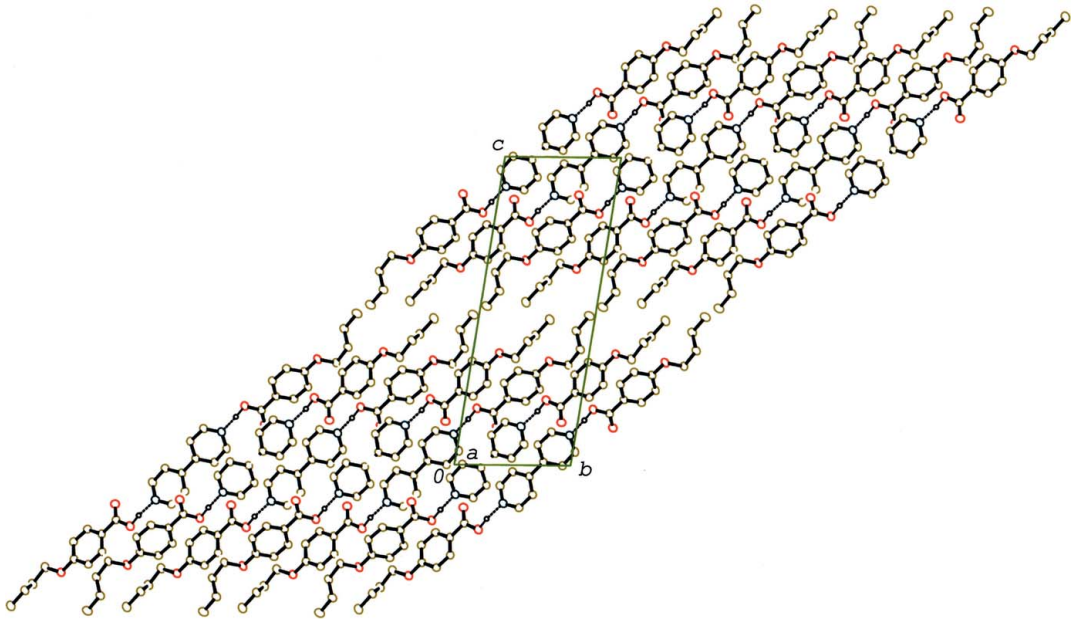



**Figure 8**
A packing diagram of compound (III) viewed along the *a* axis, showing a layer aggregate. H atoms not involved in the O—H···N hydrogen bonds (dashed lines) have been omitted.

**Table 4**
Experimental details.

| | (I) | (II) | (III) |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $2C_9H_{10}O_3{\cdot}C_{10}H_8N_2$ | $2C_{10}H_{12}O_3{\cdot}C_{10}H_8N_2$ | $2C_{11}H_{14}O_3{\cdot}C_{10}H_8N_2$ |
| $M_r$ | 488.52 | 516.57 | 544.63 |
| Crystal system, space group | Monoclinic, $P2_1/n$ | Triclinic, $P\overline{1}$ | Triclinic, $P\overline{1}$ |
| Temperature (K) | 93 | 93 | 93 |
| $a, b, c$ (Å) | 9.1090 (2), 20.9348 (5), 12.8738 (4) | 10.7592 (4), 10.8838 (3), 11.6462 (4) | 7.6645 (10), 8.5087 (13), 22.606 (3) |
| $\alpha, \beta, \gamma$ (°) | 90, 102.9429 (10), 90 | 86.6411 (11), 89.2313 (13), 73.8867 (12) | 80.498 (3), 86.486 (3), 80.082 (3) |
| $V$ (Å$^3$) | 2392.60 (11) | 1307.95 (8) | 1431.5 (4) |
| $Z$ | 4 | 2 | 2 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.10 | 0.09 | 0.09 |
| Crystal size (mm) | 0.28 × 0.25 × 0.10 | 0.50 × 0.40 × 0.10 | 0.53 × 0.41 × 0.11 |
| Data collection | | | |
| Diffractometer | Rigaku R-AXIS RAPIDII | Rigaku R-AXIS RAPIDII | Rigaku R-AXIS RAPIDIIr |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 28629, 6941, 6004 | 15909, 7507, 5980 | 12433, 5612, 3432 |
| $R_{int}$ | 0.035 | 0.069 | 0.075 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.703 | 0.703 | 0.617 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.045, 0.127, 1.04 | 0.047, 0.135, 1.04 | 0.069, 0.193, 1.01 |
| No. of reflections | 6941 | 7507 | 5610 |
| No. of parameters | 335 | 354 | 371 |
| 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$^{-3}$) | 0.39, −0.37 | 0.35, −0.32 | 0.24, −0.41 |

Computer programs: *RAPID-AUTO* (Rigaku, 2006), *SIR92* (Altomare *et al.*, 1994), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *CrystalStructure* (Rigaku, 2010) and *PLATON* (Spek, 2009).

3.7052 (5) and 3.7752 (6) Å, respectively. C—H···$\pi$ interactions (Table 1) are also observed between the columns and between the sheets.

In the crystal of (II) and (III), the 2:1 units are linked by C—H···O interactions (Tables 2 and 3), forming a double-tape structure along the *a* axis (Fig. 6) and a tape structure along the *b* axis (Fig. 7), respectively. Between the tapes in (II) and (III) C—H···$\pi$ interactions are observed (Tables 2 and 3). A packing diagram of (III) viewed along the *a* axis, which is approximately perpendicular to the mean plane of the 2:1 unit, is shown in Fig. 8. The units are arranged into a layer parallel to the *bc* plane, which leads to a smectic structure. On the other hand, no such a layer structure is observed in compounds (I) and (II), which form nematic liquid phases.

## 4. Database survey

A search of the Cambridge Structural Database (Version 5.36, last update February 2015; Groom & Allen, 2014) for co-crystals of 4,4′-bipyridyl with 4-alkoxybenzoic acid gave five structures (refcodes: NOPXIZ, ORASAC, RIRGUV, YAKVAI and YANCUM), except for 4-methoxybenzoic acid–4,4′-bipyridyl (2/1) and 4-ethoxybenzoic acid–4,4′-bipyridyl (2/1). Of these compounds, NOPXIZ, 4-[(*S*)-(−)-2-methylbutoxy]benzoic acid–4,4′-bipyridyl (2/10, shows smectic A and nematic phases (Grunert *et al.*, 1997).

## 5. Synthesis and crystallization

Single crystals of compound (I) were obtained by slow evaporation from an acetone solution (150 ml) of 4,4′-bipyridyl (70 mg) with 4-ethoxybenzoic acid (150 mg) at room temperature. Crystals of compounds (II) and (III) were obtained from ethanol solutions of 4,4′-bipyridyl with 4-*n*-propoxybenzoic acid and 4-*n*-butoxybenzoic acid, respectively, at room temperature [ethanol solution (150 ml) of 4,4′-bipyridyl (65 mg) and 4-*n*-propoxybenzoic acid (150 mg) for (II), and ethanol solution (150 ml) of 4,4′-bipyridyl (60 mg) and 4-*n*-butoxybenzoic acid (150 mg) for (III)].

Liquid crystalline phases of these compounds were confirmed by measurements of DSC (differential scanning calorimetry) and polarizing microscope. DSC measurements were performed by using Perkin Elmer 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:

(I) 373 (2) [5.4 (4)] $K_1 \rightarrow K_2$, 424 (1) [50 (3)] $K_2 \rightarrow N$, 442 (1) [7.2 (6)] $N \rightarrow I$;

(II) 365 (1) [2.9 (6)] $K_1 \rightarrow K_2$, 369 (1) [3.9 (2)] $K_2 \rightarrow K_3$, 417 (1) [39 (1)] $K_3 \rightarrow N$, 430 (1) [5.7 (2)] $N \rightarrow I$;

(III) 358 (1) [2.5 (2)] $K_1 \rightarrow K_2$, 386 (1) [0.30 (3)] $K_2 \rightarrow K_3$, 403 (1) [11.1 (5)] $K_3 \rightarrow K_4$, 407 (1) [24.5 (6)] $K_4 \rightarrow S_A$, 425 (1) [2.2 (6)] $S_A \rightarrow N$, 432 (1) [6.4 (1)] $N \rightarrow I$.

$K_i$, $S_A$, N and I denote crystal, smectic A, nematic and isotropic phases, respectively. The observed transition temperatures and enthalpies from the solid phase to the liquid crystalline phase are in good agreement with those reported Kato *et al.* (1990, 1993). Some unreported thermal anomalies, 373 (2) K for (I), 365 (1) and 369 (1) K for (II), and 358 (1) and 386 (1) K for (III), were also observed.

## 6. 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 = 0.95–0.99 Å and were refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C). The O-bound H atoms were located in a difference Fourier map and refined freely [refined O—H = 0.942 (19)–1.04 (3) Å].

## References


Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Grunert, M., Howie, A., Kaeding, A. & Imrie, C. T. (1997). *J. Mater. Chem.* **7**, 211–214.
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.
Lai, L.-L., Lee, L.-J., Luo, D.-W., Liu, Y.-H. & Wang, Y. (2008). *J. Struct. Chem.* **49**, 1137–1140.
Mukherjee, A. & Desiraju, G. R. (2014). *Cryst. Growth Des.* **14**, 1375–1385.
Ramon, G., Davies, K. & Nassimbeni, L. R. (2014). *CrystEngComm*, **16**, 5802–5810.
Rigaku (2006). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2010). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2015). E**71**, 1290-1295 [https://doi.org/10.1107/S2056989015018435]

# Crystal structures of three co-crystals of 4,4′-bipyridyl with 4-alkoxybenzoic acids: 4-ethoxybenzoic acid–4,4′-bipyridyl (2/1), 4-*n*-propoxybenzoic acid–4,4′-bipyridyl (2/1) and 4-*n*-butoxybenzoic acid–4,4′-bipyridyl (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: *SIR92* (Altomare *et al.*, 1994) for (I); *SHELXS97* (Sheldrick, 2008) for (II), (III). For all compounds, 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–4,4′-bipyridyl (2/1)

*Crystal data*

$2C_9H_{10}O_3{\cdot}C_{10}H_8N_2$
$M_r$ = 488.52
Monoclinic, $P2_1/n$
$a$ = 9.1090 (2) Å
$b$ = 20.9348 (5) Å
$c$ = 12.8738 (4) Å
$\beta$ = 102.9429 (10)°
$V$ = 2392.60 (11) Å$^3$
$Z$ = 4
$F(000)$ = 1032.00
$D_x$ = 1.356 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71075 Å
Cell parameters from 23666 reflections
$\theta$ = 3.0–30.0°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 93 K
Needle, colorless
0.28 × 0.25 × 0.10 mm

*Data collection*

Rigaku R-AXIS RAPIDII diffractometer
Detector resolution: 10.000 pixels mm$^{-1}$
$\omega$ scans
28629 measured reflections
6941 independent reflections
6004 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035
$\theta_{max}$ = 30.0°
$h$ = −12→12
$k$ = −28→29
$l$ = −18→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
$S$ = 1.04
6941 reflections
335 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

$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3767P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.37$ 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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.33398 (8) | 0.30866 (3) | 0.77777 (6) | 0.02333 (16) |
| O2 | −0.37344 (8) | 0.40959 (3) | 0.72097 (6) | 0.02177 (15) |
| O3 | −0.92113 (7) | 0.36336 (3) | 0.94659 (6) | 0.01856 (14) |
| O4 | 0.74748 (8) | 0.35124 (3) | 0.34692 (6) | 0.02249 (16) |
| O5 | 0.77838 (8) | 0.45585 (3) | 0.38227 (6) | 0.02363 (16) |
| O6 | 1.33565 (7) | 0.38813 (3) | 0.17479 (6) | 0.01967 (15) |
| N1 | −0.10882 (9) | 0.31350 (4) | 0.67751 (7) | 0.02016 (17) |
| N2 | 0.51947 (9) | 0.35033 (4) | 0.44136 (7) | 0.02191 (17) |
| C1 | −0.54350 (10) | 0.36269 (4) | 0.81521 (7) | 0.01557 (17) |
| C2 | −0.57223 (10) | 0.31282 (4) | 0.88043 (7) | 0.01700 (17) |
| H2 | −0.5048 | 0.2777 | 0.8950 | 0.020* |
| C3 | −0.69860 (10) | 0.31438 (4) | 0.92393 (8) | 0.01764 (17) |
| H3 | −0.7169 | 0.2806 | 0.9686 | 0.021* |
| C4 | −0.79903 (10) | 0.36588 (4) | 0.90180 (7) | 0.01553 (17) |
| C5 | −0.77115 (10) | 0.41585 (4) | 0.83675 (7) | 0.01658 (17) |
| H5 | −0.8388 | 0.4509 | 0.8216 | 0.020* |
| C6 | −0.64371 (10) | 0.41374 (4) | 0.79445 (7) | 0.01672 (17) |
| H6 | −0.6246 | 0.4478 | 0.7505 | 0.020* |
| C7 | −0.40997 (10) | 0.36318 (4) | 0.76677 (7) | 0.01667 (17) |
| C8 | −1.02432 (10) | 0.41622 (4) | 0.92520 (8) | 0.01880 (18) |
| H8A | −1.0723 | 0.4179 | 0.8482 | 0.023* |
| H8B | −0.9696 | 0.4568 | 0.9453 | 0.023* |
| C9 | −1.14267 (11) | 0.40745 (5) | 0.98921 (8) | 0.02217 (19) |
| H9A | −1.0945 | 0.4070 | 1.0653 | 0.033* |
| H9B | −1.1954 | 0.3669 | 0.9695 | 0.033* |
| H9C | −1.2150 | 0.4427 | 0.9744 | 0.033* |
| C10 | 0.95579 (10) | 0.40171 (4) | 0.30216 (7) | 0.01621 (17) |
| C11 | 1.04428 (10) | 0.45571 (4) | 0.30003 (8) | 0.01774 (17) |
| H11 | 1.0160 | 0.4949 | 0.3273 | 0.021* |
| C12 | 1.17334 (10) | 0.45373 (4) | 0.25886 (8) | 0.01782 (18) |
| H12 | 1.2328 | 0.4910 | 0.2585 | 0.021* |
| C13 | 1.21379 (10) | 0.39607 (4) | 0.21808 (7) | 0.01639 (17) |
| C14 | 1.12647 (10) | 0.34139 (4) | 0.22033 (8) | 0.01837 (18) |
| H14 | 1.1544 | 0.3022 | 0.1928 | 0.022* |

| | | | | |
|---|---|---|---|---|
| C15 | 0.99956 (10) | 0.34406 (4) | 0.26245 (8) | 0.01753 (17) |
| H15 | 0.9416 | 0.3065 | 0.2645 | 0.021* |
| C16 | 0.81942 (10) | 0.40630 (5) | 0.34748 (7) | 0.01747 (17) |
| C17 | 1.43238 (10) | 0.44212 (5) | 0.17318 (8) | 0.02026 (19) |
| H17A | 1.4830 | 0.4543 | 0.2468 | 0.024* |
| H17B | 1.3729 | 0.4791 | 0.1388 | 0.024* |
| C18 | 1.54764 (11) | 0.42313 (5) | 0.11099 (8) | 0.0229 (2) |
| H18A | 1.5998 | 0.3843 | 0.1421 | 0.034* |
| H18B | 1.6209 | 0.4578 | 0.1139 | 0.034* |
| H18C | 1.4970 | 0.4150 | 0.0366 | 0.034* |
| C19 | −0.09136 (10) | 0.36667 (5) | 0.62319 (8) | 0.01935 (18) |
| H19 | −0.1662 | 0.3990 | 0.6159 | 0.023* |
| C20 | 0.03056 (10) | 0.37669 (5) | 0.57728 (7) | 0.01765 (17) |
| H20 | 0.0388 | 0.4153 | 0.5402 | 0.021* |
| C21 | 0.14130 (10) | 0.32950 (4) | 0.58603 (7) | 0.01586 (17) |
| C22 | 0.12277 (10) | 0.27418 (5) | 0.64248 (8) | 0.01962 (18) |
| H22 | 0.1954 | 0.2409 | 0.6508 | 0.024* |
| C23 | −0.00263 (11) | 0.26821 (5) | 0.68641 (8) | 0.02095 (19) |
| H23 | −0.0137 | 0.2303 | 0.7245 | 0.025* |
| C24 | 0.46423 (11) | 0.29264 (5) | 0.45714 (9) | 0.0242 (2) |
| H24 | 0.5105 | 0.2559 | 0.4352 | 0.029* |
| C25 | 0.34301 (11) | 0.28424 (5) | 0.50393 (9) | 0.0228 (2) |
| H25 | 0.3080 | 0.2424 | 0.5140 | 0.027* |
| C26 | 0.27224 (10) | 0.33746 (4) | 0.53636 (7) | 0.01618 (17) |
| C27 | 0.32923 (10) | 0.39752 (4) | 0.51958 (8) | 0.01849 (18) |
| H27 | 0.2847 | 0.4352 | 0.5400 | 0.022* |
| C28 | 0.45223 (11) | 0.40137 (5) | 0.47250 (8) | 0.02086 (19) |
| H28 | 0.4906 | 0.4425 | 0.4619 | 0.025* |
| H1 | −0.254 (2) | 0.3118 (9) | 0.7426 (17) | 0.059 (5)* |
| H4 | 0.663 (2) | 0.3549 (10) | 0.3782 (17) | 0.066 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0216 (3) | 0.0188 (3) | 0.0345 (4) | 0.0023 (3) | 0.0167 (3) | 0.0000 (3) |
| O2 | 0.0200 (3) | 0.0207 (3) | 0.0274 (4) | −0.0013 (3) | 0.0114 (3) | 0.0013 (3) |
| O3 | 0.0148 (3) | 0.0197 (3) | 0.0238 (3) | 0.0017 (2) | 0.0099 (2) | 0.0022 (2) |
| O4 | 0.0194 (3) | 0.0226 (3) | 0.0293 (4) | −0.0033 (3) | 0.0136 (3) | −0.0007 (3) |
| O5 | 0.0217 (3) | 0.0227 (3) | 0.0293 (4) | 0.0038 (3) | 0.0117 (3) | −0.0003 (3) |
| O6 | 0.0165 (3) | 0.0182 (3) | 0.0276 (4) | −0.0019 (2) | 0.0120 (3) | −0.0021 (3) |
| N1 | 0.0170 (3) | 0.0235 (4) | 0.0220 (4) | −0.0013 (3) | 0.0086 (3) | −0.0038 (3) |
| N2 | 0.0168 (3) | 0.0281 (4) | 0.0228 (4) | 0.0005 (3) | 0.0088 (3) | 0.0003 (3) |
| C1 | 0.0141 (4) | 0.0167 (4) | 0.0167 (4) | −0.0013 (3) | 0.0053 (3) | −0.0030 (3) |
| C2 | 0.0152 (4) | 0.0166 (4) | 0.0198 (4) | 0.0006 (3) | 0.0052 (3) | −0.0012 (3) |
| C3 | 0.0163 (4) | 0.0169 (4) | 0.0207 (4) | −0.0003 (3) | 0.0062 (3) | 0.0019 (3) |
| C4 | 0.0134 (4) | 0.0176 (4) | 0.0165 (4) | −0.0009 (3) | 0.0054 (3) | −0.0019 (3) |
| C5 | 0.0161 (4) | 0.0157 (4) | 0.0189 (4) | 0.0009 (3) | 0.0059 (3) | −0.0003 (3) |
| C6 | 0.0173 (4) | 0.0165 (4) | 0.0175 (4) | −0.0010 (3) | 0.0061 (3) | −0.0007 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.0150 (4) | 0.0179 (4) | 0.0181 (4) | −0.0015 (3) | 0.0058 (3) | −0.0043 (3) |
| C8 | 0.0169 (4) | 0.0185 (4) | 0.0228 (4) | 0.0022 (3) | 0.0082 (3) | −0.0003 (3) |
| C9 | 0.0175 (4) | 0.0280 (5) | 0.0230 (5) | 0.0027 (4) | 0.0087 (3) | 0.0021 (4) |
| C10 | 0.0148 (4) | 0.0179 (4) | 0.0168 (4) | 0.0010 (3) | 0.0051 (3) | 0.0023 (3) |
| C11 | 0.0177 (4) | 0.0159 (4) | 0.0209 (4) | 0.0019 (3) | 0.0070 (3) | 0.0005 (3) |
| C12 | 0.0164 (4) | 0.0156 (4) | 0.0227 (4) | −0.0010 (3) | 0.0070 (3) | 0.0012 (3) |
| C13 | 0.0142 (4) | 0.0176 (4) | 0.0183 (4) | 0.0008 (3) | 0.0056 (3) | 0.0018 (3) |
| C14 | 0.0179 (4) | 0.0150 (4) | 0.0240 (4) | 0.0009 (3) | 0.0084 (3) | −0.0001 (3) |
| C15 | 0.0171 (4) | 0.0159 (4) | 0.0209 (4) | −0.0009 (3) | 0.0069 (3) | 0.0016 (3) |
| C16 | 0.0153 (4) | 0.0207 (4) | 0.0172 (4) | 0.0015 (3) | 0.0053 (3) | 0.0026 (3) |
| C17 | 0.0181 (4) | 0.0192 (4) | 0.0260 (5) | −0.0034 (3) | 0.0104 (4) | −0.0015 (3) |
| C18 | 0.0187 (4) | 0.0272 (5) | 0.0255 (5) | −0.0047 (4) | 0.0108 (4) | −0.0055 (4) |
| C19 | 0.0159 (4) | 0.0230 (4) | 0.0201 (4) | 0.0026 (3) | 0.0061 (3) | −0.0022 (3) |
| C20 | 0.0159 (4) | 0.0205 (4) | 0.0174 (4) | 0.0009 (3) | 0.0057 (3) | −0.0007 (3) |
| C21 | 0.0134 (4) | 0.0196 (4) | 0.0153 (4) | −0.0008 (3) | 0.0047 (3) | −0.0034 (3) |
| C22 | 0.0173 (4) | 0.0200 (4) | 0.0233 (4) | 0.0022 (3) | 0.0084 (3) | 0.0000 (3) |
| C23 | 0.0204 (4) | 0.0212 (4) | 0.0237 (4) | −0.0006 (3) | 0.0102 (4) | 0.0007 (3) |
| C24 | 0.0212 (4) | 0.0241 (4) | 0.0313 (5) | 0.0035 (4) | 0.0141 (4) | −0.0014 (4) |
| C25 | 0.0218 (4) | 0.0194 (4) | 0.0313 (5) | 0.0000 (4) | 0.0147 (4) | −0.0019 (4) |
| C26 | 0.0139 (4) | 0.0197 (4) | 0.0160 (4) | 0.0009 (3) | 0.0055 (3) | −0.0010 (3) |
| C27 | 0.0186 (4) | 0.0194 (4) | 0.0191 (4) | 0.0006 (3) | 0.0077 (3) | −0.0007 (3) |
| C28 | 0.0201 (4) | 0.0227 (4) | 0.0218 (4) | −0.0028 (3) | 0.0090 (3) | 0.0000 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.3259 (11) | C10—C16 | 1.4891 (12) |
| O1—H1 | 0.942 (19) | C11—C12 | 1.3941 (12) |
| O2—C7 | 1.2208 (12) | C11—H11 | 0.9500 |
| O3—C4 | 1.3637 (10) | C12—C13 | 1.3984 (13) |
| O3—C8 | 1.4380 (11) | C12—H12 | 0.9500 |
| O4—C16 | 1.3252 (12) | C13—C14 | 1.3979 (12) |
| O4—H4 | 0.948 (19) | C14—C15 | 1.3836 (12) |
| O5—C16 | 1.2213 (12) | C14—H14 | 0.9500 |
| O6—C13 | 1.3592 (10) | C15—H15 | 0.9500 |
| O6—C17 | 1.4361 (11) | C17—C18 | 1.5092 (13) |
| N1—C23 | 1.3409 (13) | C17—H17A | 0.9900 |
| N1—C19 | 1.3429 (13) | C17—H17B | 0.9900 |
| N2—C28 | 1.3370 (13) | C18—H18A | 0.9800 |
| N2—C24 | 1.3412 (14) | C18—H18B | 0.9800 |
| C1—C6 | 1.3920 (12) | C18—H18C | 0.9800 |
| C1—C2 | 1.4006 (13) | C19—C20 | 1.3859 (12) |
| C1—C7 | 1.4860 (12) | C19—H19 | 0.9500 |
| C2—C3 | 1.3887 (12) | C20—C21 | 1.3983 (12) |
| C2—H2 | 0.9500 | C20—H20 | 0.9500 |
| C3—C4 | 1.4012 (12) | C21—C22 | 1.3974 (13) |
| C3—H3 | 0.9500 | C21—C26 | 1.4835 (12) |
| C4—C5 | 1.3984 (12) | C22—C23 | 1.3892 (12) |
| C5—C6 | 1.3890 (12) | C22—H22 | 0.9500 |

| | | | |
|---|---|---|---|
| C5—H5 | 0.9500 | C23—H23 | 0.9500 |
| C6—H6 | 0.9500 | C24—C25 | 1.3825 (13) |
| C8—C9 | 1.5077 (13) | C24—H24 | 0.9500 |
| C8—H8A | 0.9900 | C25—C26 | 1.3971 (13) |
| C8—H8B | 0.9900 | C25—H25 | 0.9500 |
| C9—H9A | 0.9800 | C26—C27 | 1.3956 (13) |
| C9—H9B | 0.9800 | C27—C28 | 1.3905 (12) |
| C9—H9C | 0.9800 | C27—H27 | 0.9500 |
| C10—C11 | 1.3923 (12) | C28—H28 | 0.9500 |
| C10—C15 | 1.4030 (12) | | |

| | | | |
|---|---|---|---|
| C7—O1—H1 | 109.2 (12) | C15—C14—C13 | 120.28 (8) |
| C4—O3—C8 | 116.70 (7) | C15—C14—H14 | 119.9 |
| C16—O4—H4 | 112.0 (13) | C13—C14—H14 | 119.9 |
| C13—O6—C17 | 118.02 (7) | C14—C15—C10 | 120.54 (8) |
| C23—N1—C19 | 117.60 (8) | C14—C15—H15 | 119.7 |
| C28—N2—C24 | 117.50 (8) | C10—C15—H15 | 119.7 |
| C6—C1—C2 | 119.00 (8) | O5—C16—O4 | 123.31 (8) |
| C6—C1—C7 | 118.51 (8) | O5—C16—C10 | 123.32 (8) |
| C2—C1—C7 | 122.49 (8) | O4—C16—C10 | 113.37 (8) |
| C3—C2—C1 | 120.48 (8) | O6—C17—C18 | 107.60 (8) |
| C3—C2—H2 | 119.8 | O6—C17—H17A | 110.2 |
| C1—C2—H2 | 119.8 | C18—C17—H17A | 110.2 |
| C2—C3—C4 | 119.90 (8) | O6—C17—H17B | 110.2 |
| C2—C3—H3 | 120.0 | C18—C17—H17B | 110.2 |
| C4—C3—H3 | 120.0 | H17A—C17—H17B | 108.5 |
| O3—C4—C5 | 123.84 (8) | C17—C18—H18A | 109.5 |
| O3—C4—C3 | 116.20 (8) | C17—C18—H18B | 109.5 |
| C5—C4—C3 | 119.96 (8) | H18A—C18—H18B | 109.5 |
| C6—C5—C4 | 119.40 (8) | C17—C18—H18C | 109.5 |
| C6—C5—H5 | 120.3 | H18A—C18—H18C | 109.5 |
| C4—C5—H5 | 120.3 | H18B—C18—H18C | 109.5 |
| C5—C6—C1 | 121.25 (8) | N1—C19—C20 | 123.18 (8) |
| C5—C6—H6 | 119.4 | N1—C19—H19 | 118.4 |
| C1—C6—H6 | 119.4 | C20—C19—H19 | 118.4 |
| O2—C7—O1 | 123.00 (8) | C19—C20—C21 | 119.42 (9) |
| O2—C7—C1 | 123.05 (8) | C19—C20—H20 | 120.3 |
| O1—C7—C1 | 113.95 (8) | C21—C20—H20 | 120.3 |
| O3—C8—C9 | 108.56 (8) | C22—C21—C20 | 117.29 (8) |
| O3—C8—H8A | 110.0 | C22—C21—C26 | 121.30 (8) |
| C9—C8—H8A | 110.0 | C20—C21—C26 | 121.41 (8) |
| O3—C8—H8B | 110.0 | C23—C22—C21 | 119.50 (8) |
| C9—C8—H8B | 110.0 | C23—C22—H22 | 120.2 |
| H8A—C8—H8B | 108.4 | C21—C22—H22 | 120.2 |
| C8—C9—H9A | 109.5 | N1—C23—C22 | 123.01 (9) |
| C8—C9—H9B | 109.5 | N1—C23—H23 | 118.5 |
| H9A—C9—H9B | 109.5 | C22—C23—H23 | 118.5 |
| C8—C9—H9C | 109.5 | N2—C24—C25 | 122.95 (9) |

| | | | |
|---|---|---|---|
| H9A—C9—H9C | 109.5 | N2—C24—H24 | 118.5 |
| H9B—C9—H9C | 109.5 | C25—C24—H24 | 118.5 |
| C11—C10—C15 | 118.58 (8) | C24—C25—C26 | 119.73 (9) |
| C11—C10—C16 | 119.52 (8) | C24—C25—H25 | 120.1 |
| C15—C10—C16 | 121.89 (8) | C26—C25—H25 | 120.1 |
| C10—C11—C12 | 121.64 (8) | C27—C26—C25 | 117.37 (8) |
| C10—C11—H11 | 119.2 | C27—C26—C21 | 122.05 (8) |
| C12—C11—H11 | 119.2 | C25—C26—C21 | 120.57 (8) |
| C11—C12—C13 | 118.94 (8) | C28—C27—C26 | 118.92 (8) |
| C11—C12—H12 | 120.5 | C28—C27—H27 | 120.5 |
| C13—C12—H12 | 120.5 | C26—C27—H27 | 120.5 |
| O6—C13—C14 | 115.41 (8) | N2—C28—C27 | 123.53 (9) |
| O6—C13—C12 | 124.58 (8) | N2—C28—H28 | 118.2 |
| C14—C13—C12 | 120.01 (8) | C27—C28—H28 | 118.2 |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −0.16 (13) | C11—C10—C15—C14 | −1.18 (14) |
| C7—C1—C2—C3 | −179.95 (8) | C16—C10—C15—C14 | 179.68 (8) |
| C1—C2—C3—C4 | 0.57 (14) | C11—C10—C16—O5 | 1.11 (14) |
| C8—O3—C4—C5 | −0.72 (13) | C15—C10—C16—O5 | −179.75 (9) |
| C8—O3—C4—C3 | 179.48 (8) | C11—C10—C16—O4 | −178.52 (8) |
| C2—C3—C4—O3 | 179.25 (8) | C15—C10—C16—O4 | 0.62 (13) |
| C2—C3—C4—C5 | −0.56 (14) | C13—O6—C17—C18 | −173.89 (8) |
| O3—C4—C5—C6 | −179.65 (8) | C23—N1—C19—C20 | −0.45 (14) |
| C3—C4—C5—C6 | 0.14 (14) | N1—C19—C20—C21 | 0.69 (14) |
| C4—C5—C6—C1 | 0.28 (14) | C19—C20—C21—C22 | −0.57 (13) |
| C2—C1—C6—C5 | −0.27 (14) | C19—C20—C21—C26 | 178.73 (8) |
| C7—C1—C6—C5 | 179.53 (8) | C20—C21—C22—C23 | 0.28 (14) |
| C6—C1—C7—O2 | 9.58 (13) | C26—C21—C22—C23 | −179.03 (9) |
| C2—C1—C7—O2 | −170.62 (9) | C19—N1—C23—C22 | 0.13 (15) |
| C6—C1—C7—O1 | −170.43 (8) | C21—C22—C23—N1 | −0.05 (15) |
| C2—C1—C7—O1 | 9.37 (13) | C28—N2—C24—C25 | −0.27 (16) |
| C4—O3—C8—C9 | −174.93 (8) | N2—C24—C25—C26 | 0.41 (17) |
| C15—C10—C11—C12 | 0.53 (14) | C24—C25—C26—C27 | −0.13 (15) |
| C16—C10—C11—C12 | 179.70 (8) | C24—C25—C26—C21 | 179.56 (9) |
| C10—C11—C12—C13 | 0.48 (14) | C22—C21—C26—C27 | −152.77 (9) |
| C17—O6—C13—C14 | −178.14 (8) | C20—C21—C26—C27 | 27.95 (13) |
| C17—O6—C13—C12 | 1.77 (13) | C22—C21—C26—C25 | 27.55 (13) |
| C11—C12—C13—O6 | 179.23 (9) | C20—C21—C26—C25 | −151.72 (10) |
| C11—C12—C13—C14 | −0.87 (14) | C25—C26—C27—C28 | −0.26 (14) |
| O6—C13—C14—C15 | −179.85 (8) | C21—C26—C27—C28 | −179.94 (9) |
| C12—C13—C14—C15 | 0.24 (14) | C24—N2—C28—C27 | −0.16 (15) |
| C13—C14—C15—C10 | 0.80 (14) | C26—C27—C28—N2 | 0.43 (15) |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C10–C15 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.942 (19) | 1.72 (2) | 2.6587 (11) | 177.2 (19) |

| | | | | |
|---|---|---|---|---|
| O4—H4···N2 | 0.948 (19) | 1.690 (19) | 2.6312 (11) | 171.4 (19) |
| C12—H12···O2$^{i}$ | 0.95 | 2.43 | 3.3712 (11) | 172 |
| C14—H14···O1$^{ii}$ | 0.95 | 2.56 | 3.2288 (11) | 128 |
| C24—H24···O3$^{ii}$ | 0.95 | 2.57 | 3.4407 (12) | 153 |
| C9—H9*A*···*Cg*2$^{iii}$ | 0.98 | 2.68 | 3.6450 (11) | 169 |
| C18—H18*C*···*Cg*1$^{iv}$ | 0.98 | 2.67 | 3.6253 (11) | 164 |

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

### (II) 4-*n*-Propoxybenzoic acid–4,4′-bipyridyl (2/1)

*Crystal data*

$2C_{10}H_{12}O_3·C_{10}H_8N_2$
$M_r$ = 516.57
Triclinic, $P\overline{1}$
*a* = 10.7592 (4) Å
*b* = 10.8838 (3) Å
*c* = 11.6462 (4) Å
*α* = 86.6411 (11)°
*β* = 89.2313 (13)°
*γ* = 73.8867 (12)°
*V* = 1307.95 (8) Å$^3$
*Z* = 2
*F*(000) = 548.00
$D_x$ = 1.312 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71075 Å
Cell parameters from 13551 reflections
*θ* = 3.0–30.0°
*μ* = 0.09 mm$^{-1}$
*T* = 93 K
Block, colorless
0.50 × 0.40 × 0.10 mm

*Data collection*

Rigaku R-AXIS RAPIDII diffractometer
Detector resolution: 10.000 pixels mm$^{-1}$
*ω* scans
15909 measured reflections
7507 independent reflections
5980 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069
$\theta_{max}$ = 30.0°, $\theta_{min}$ = 3.0°
*h* = −15→15
*k* = −13→15
*l* = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.135
*S* = 1.04
7507 reflections
354 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$
Extinction correction: SHELXL-2014/7, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (4)

*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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.21741 (6) | 0.84171 (6) | −0.00993 (6) | 0.02370 (16) |
| O2 | 0.26015 (7) | 0.62711 (6) | 0.01082 (6) | 0.02497 (16) |
| O3 | −0.31475 (6) | 0.79522 (6) | −0.19102 (6) | 0.02084 (15) |
| O4 | 1.32849 (7) | 0.66282 (6) | 0.36794 (6) | 0.02574 (16) |
| O5 | 1.28863 (7) | 0.87599 (6) | 0.33722 (6) | 0.02456 (15) |
| O6 | 1.86048 (6) | 0.70388 (6) | 0.55057 (5) | 0.01982 (14) |
| N1 | 0.44816 (7) | 0.80971 (7) | 0.08178 (6) | 0.01957 (16) |
| N2 | 1.08835 (7) | 0.69161 (7) | 0.29159 (6) | 0.01975 (16) |
| C1 | 0.05619 (8) | 0.74854 (8) | −0.06643 (7) | 0.01587 (16) |
| C2 | −0.02538 (8) | 0.86942 (8) | −0.09987 (7) | 0.01713 (16) |
| H2 | 0.0038 | 0.9435 | −0.0934 | 0.021* |
| C3 | −0.14819 (8) | 0.88217 (8) | −0.14234 (7) | 0.01796 (17) |
| H3 | −0.2024 | 0.9644 | −0.1660 | 0.022* |
| C4 | −0.19207 (8) | 0.77306 (8) | −0.15022 (7) | 0.01676 (17) |
| C5 | −0.11202 (8) | 0.65191 (8) | −0.11692 (7) | 0.01732 (16) |
| H5 | −0.1416 | 0.5779 | −0.1222 | 0.021* |
| C6 | 0.01143 (8) | 0.64099 (8) | −0.07598 (7) | 0.01671 (16) |
| H6 | 0.0665 | 0.5586 | −0.0541 | 0.020* |
| C7 | 0.18746 (8) | 0.73163 (8) | −0.01851 (7) | 0.01780 (17) |
| C8 | −0.36390 (8) | 0.68649 (8) | −0.20224 (7) | 0.01802 (17) |
| H8A | −0.3609 | 0.6385 | −0.1270 | 0.022* |
| H8B | −0.3108 | 0.6283 | −0.2576 | 0.022* |
| C9 | −0.50170 (9) | 0.73524 (9) | −0.24460 (8) | 0.02173 (18) |
| H9B | −0.5034 | 0.7767 | −0.3228 | 0.026* |
| H9A | −0.5522 | 0.7999 | −0.1931 | 0.026* |
| C10 | −0.56236 (9) | 0.62384 (9) | −0.24724 (10) | 0.0281 (2) |
| H10A | −0.6465 | 0.6532 | −0.2862 | 0.042* |
| H10B | −0.5742 | 0.5925 | −0.1684 | 0.042* |
| H10C | −0.5053 | 0.5544 | −0.2889 | 0.042* |
| C11 | 1.49360 (8) | 0.75522 (8) | 0.41394 (7) | 0.01674 (16) |
| C12 | 1.57341 (9) | 0.63400 (8) | 0.44879 (7) | 0.01858 (17) |
| H12 | 1.5434 | 0.5605 | 0.4418 | 0.022* |
| C13 | 1.69517 (9) | 0.62010 (8) | 0.49316 (7) | 0.01892 (17) |
| H13 | 1.7485 | 0.5373 | 0.5164 | 0.023* |
| C14 | 1.74023 (8) | 0.72791 (8) | 0.50403 (7) | 0.01712 (17) |
| C15 | 1.66235 (9) | 0.84947 (8) | 0.46796 (7) | 0.01804 (17) |
| H15 | 1.6927 | 0.9229 | 0.4740 | 0.022* |
| C16 | 1.54008 (8) | 0.86179 (8) | 0.42315 (7) | 0.01771 (17) |
| H16 | 1.4873 | 0.9443 | 0.3984 | 0.021* |
| C17 | 1.36100 (9) | 0.77237 (8) | 0.36890 (7) | 0.01841 (17) |
| C18 | 1.91667 (8) | 0.80929 (8) | 0.55338 (7) | 0.01815 (17) |
| H18A | 1.8635 | 0.8761 | 0.6016 | 0.022* |
| H18B | 1.9206 | 0.8477 | 0.4747 | 0.022* |
| C19 | 2.05137 (9) | 0.75818 (9) | 0.60313 (8) | 0.02037 (18) |
| H19B | 2.1028 | 0.6888 | 0.5564 | 0.024* |

| | | | | |
|---|---|---|---|---|
| H19A | 2.0465 | 0.7220 | 0.6825 | 0.024* |
| C20 | 2.11746 (9) | 0.86578 (9) | 0.60422 (9) | 0.0277 (2) |
| H20A | 2.0643 | 0.9360 | 0.6475 | 0.042* |
| H20B | 2.1277 | 0.8972 | 0.5251 | 0.042* |
| H20C | 2.2027 | 0.8330 | 0.6408 | 0.042* |
| C21 | 0.52853 (9) | 0.69053 (9) | 0.08870 (8) | 0.02069 (18) |
| H21 | 0.4970 | 0.6220 | 0.0666 | 0.025* |
| C22 | 0.65531 (8) | 0.66285 (8) | 0.12658 (8) | 0.01907 (17) |
| H22 | 0.7088 | 0.5771 | 0.1302 | 0.023* |
| C23 | 0.70378 (8) | 0.76194 (8) | 0.15928 (7) | 0.01608 (16) |
| C24 | 0.61977 (8) | 0.88583 (8) | 0.15197 (7) | 0.01863 (17) |
| H24 | 0.6488 | 0.9563 | 0.1730 | 0.022* |
| C25 | 0.49411 (8) | 0.90534 (8) | 0.11387 (7) | 0.01989 (17) |
| H25 | 0.4380 | 0.9900 | 0.1103 | 0.024* |
| C26 | 1.02841 (8) | 0.80376 (8) | 0.23616 (7) | 0.02020 (18) |
| H26 | 1.0730 | 0.8678 | 0.2273 | 0.024* |
| C27 | 0.90438 (8) | 0.83033 (8) | 0.19124 (7) | 0.01839 (17) |
| H27 | 0.8655 | 0.9110 | 0.1527 | 0.022* |
| C28 | 0.83731 (8) | 0.73745 (8) | 0.20317 (7) | 0.01565 (16) |
| C29 | 0.90043 (8) | 0.62059 (8) | 0.26043 (7) | 0.01836 (17) |
| H29 | 0.8586 | 0.5544 | 0.2701 | 0.022* |
| C30 | 1.02416 (9) | 0.60203 (8) | 0.30285 (7) | 0.02023 (18) |
| H30 | 1.0656 | 0.5222 | 0.3417 | 0.024* |
| H1 | 0.305 (2) | 0.8292 (19) | 0.0308 (18) | 0.088 (6)* |
| H4 | 1.240 (2) | 0.6740 (19) | 0.3337 (16) | 0.082 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0188 (3) | 0.0171 (3) | 0.0356 (4) | −0.0057 (3) | −0.0076 (3) | 0.0000 (3) |
| O2 | 0.0195 (3) | 0.0176 (3) | 0.0355 (4) | −0.0016 (2) | −0.0077 (3) | 0.0014 (3) |
| O3 | 0.0148 (3) | 0.0155 (3) | 0.0318 (3) | −0.0040 (2) | −0.0071 (2) | 0.0017 (2) |
| O4 | 0.0198 (3) | 0.0180 (3) | 0.0396 (4) | −0.0060 (3) | −0.0084 (3) | 0.0027 (3) |
| O5 | 0.0202 (3) | 0.0193 (3) | 0.0314 (3) | −0.0018 (3) | −0.0051 (3) | 0.0043 (3) |
| O6 | 0.0175 (3) | 0.0163 (3) | 0.0255 (3) | −0.0048 (2) | −0.0064 (2) | 0.0022 (2) |
| N1 | 0.0156 (3) | 0.0211 (4) | 0.0216 (3) | −0.0048 (3) | −0.0021 (3) | 0.0009 (3) |
| N2 | 0.0166 (3) | 0.0199 (3) | 0.0219 (3) | −0.0035 (3) | −0.0022 (3) | −0.0019 (3) |
| C1 | 0.0154 (4) | 0.0154 (4) | 0.0163 (3) | −0.0036 (3) | −0.0007 (3) | 0.0006 (3) |
| C2 | 0.0171 (4) | 0.0139 (3) | 0.0207 (4) | −0.0049 (3) | −0.0016 (3) | 0.0000 (3) |
| C3 | 0.0165 (4) | 0.0132 (3) | 0.0229 (4) | −0.0025 (3) | −0.0028 (3) | 0.0018 (3) |
| C4 | 0.0143 (4) | 0.0161 (4) | 0.0188 (4) | −0.0028 (3) | −0.0019 (3) | 0.0007 (3) |
| C5 | 0.0178 (4) | 0.0139 (4) | 0.0205 (4) | −0.0049 (3) | −0.0018 (3) | 0.0003 (3) |
| C6 | 0.0168 (4) | 0.0134 (3) | 0.0186 (3) | −0.0024 (3) | −0.0017 (3) | 0.0012 (3) |
| C7 | 0.0167 (4) | 0.0173 (4) | 0.0190 (4) | −0.0041 (3) | −0.0011 (3) | −0.0008 (3) |
| C8 | 0.0172 (4) | 0.0164 (4) | 0.0206 (4) | −0.0051 (3) | −0.0024 (3) | 0.0003 (3) |
| C9 | 0.0169 (4) | 0.0194 (4) | 0.0287 (4) | −0.0052 (3) | −0.0052 (3) | 0.0013 (3) |
| C10 | 0.0205 (4) | 0.0241 (4) | 0.0409 (5) | −0.0080 (4) | −0.0064 (4) | −0.0010 (4) |
| C11 | 0.0161 (4) | 0.0166 (4) | 0.0168 (3) | −0.0036 (3) | −0.0004 (3) | 0.0003 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.0196 (4) | 0.0153 (4) | 0.0209 (4) | −0.0052 (3) | −0.0017 (3) | 0.0005 (3) |
| C13 | 0.0196 (4) | 0.0136 (4) | 0.0220 (4) | −0.0024 (3) | −0.0031 (3) | 0.0017 (3) |
| C14 | 0.0163 (4) | 0.0168 (4) | 0.0170 (3) | −0.0027 (3) | −0.0011 (3) | 0.0005 (3) |
| C15 | 0.0189 (4) | 0.0146 (4) | 0.0203 (4) | −0.0042 (3) | −0.0012 (3) | 0.0000 (3) |
| C16 | 0.0177 (4) | 0.0148 (4) | 0.0187 (4) | −0.0016 (3) | −0.0006 (3) | 0.0010 (3) |
| C17 | 0.0176 (4) | 0.0185 (4) | 0.0179 (4) | −0.0034 (3) | −0.0003 (3) | 0.0006 (3) |
| C18 | 0.0178 (4) | 0.0161 (4) | 0.0206 (4) | −0.0051 (3) | −0.0021 (3) | 0.0010 (3) |
| C19 | 0.0177 (4) | 0.0193 (4) | 0.0239 (4) | −0.0052 (3) | −0.0035 (3) | 0.0017 (3) |
| C20 | 0.0213 (4) | 0.0252 (4) | 0.0376 (5) | −0.0091 (4) | −0.0056 (4) | 0.0043 (4) |
| C21 | 0.0184 (4) | 0.0204 (4) | 0.0240 (4) | −0.0064 (3) | −0.0024 (3) | −0.0019 (3) |
| C22 | 0.0163 (4) | 0.0164 (4) | 0.0238 (4) | −0.0031 (3) | −0.0019 (3) | −0.0010 (3) |
| C23 | 0.0142 (4) | 0.0172 (4) | 0.0160 (3) | −0.0035 (3) | 0.0002 (3) | 0.0010 (3) |
| C24 | 0.0174 (4) | 0.0158 (4) | 0.0223 (4) | −0.0042 (3) | −0.0012 (3) | −0.0003 (3) |
| C25 | 0.0164 (4) | 0.0180 (4) | 0.0235 (4) | −0.0025 (3) | −0.0016 (3) | 0.0025 (3) |
| C26 | 0.0171 (4) | 0.0214 (4) | 0.0227 (4) | −0.0066 (3) | −0.0014 (3) | 0.0006 (3) |
| C27 | 0.0167 (4) | 0.0167 (4) | 0.0213 (4) | −0.0043 (3) | −0.0016 (3) | 0.0018 (3) |
| C28 | 0.0138 (4) | 0.0164 (4) | 0.0161 (3) | −0.0030 (3) | 0.0000 (3) | −0.0017 (3) |
| C29 | 0.0169 (4) | 0.0157 (4) | 0.0219 (4) | −0.0036 (3) | −0.0018 (3) | −0.0002 (3) |
| C30 | 0.0180 (4) | 0.0175 (4) | 0.0235 (4) | −0.0021 (3) | −0.0037 (3) | −0.0002 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.3335 (10) | C11—C17 | 1.4853 (12) |
| O1—H1 | 1.03 (2) | C12—C13 | 1.3802 (12) |
| O2—C7 | 1.2208 (11) | C12—H12 | 0.9500 |
| O3—C4 | 1.3612 (10) | C13—C14 | 1.4010 (11) |
| O3—C8 | 1.4368 (10) | C13—H13 | 0.9500 |
| O4—C17 | 1.3331 (11) | C14—C15 | 1.3993 (12) |
| O4—H4 | 1.01 (2) | C15—C16 | 1.3904 (12) |
| O5—C17 | 1.2190 (11) | C15—H15 | 0.9500 |
| O6—C14 | 1.3603 (10) | C16—H16 | 0.9500 |
| O6—C18 | 1.4397 (10) | C18—C19 | 1.5109 (11) |
| N1—C25 | 1.3430 (11) | C18—H18A | 0.9900 |
| N1—C21 | 1.3430 (12) | C18—H18B | 0.9900 |
| N2—C30 | 1.3420 (11) | C19—C20 | 1.5301 (12) |
| N2—C26 | 1.3433 (12) | C19—H19B | 0.9900 |
| C1—C6 | 1.3944 (11) | C19—H19A | 0.9900 |
| C1—C2 | 1.4004 (12) | C20—H20A | 0.9800 |
| C1—C7 | 1.4848 (11) | C20—H20B | 0.9800 |
| C2—C3 | 1.3848 (11) | C20—H20C | 0.9800 |
| C2—H2 | 0.9500 | C21—C22 | 1.3851 (12) |
| C3—C4 | 1.4023 (11) | C21—H21 | 0.9500 |
| C3—H3 | 0.9500 | C22—C23 | 1.3950 (11) |
| C4—C5 | 1.3963 (12) | C22—H22 | 0.9500 |
| C5—C6 | 1.3886 (11) | C23—C24 | 1.3981 (11) |
| C5—H5 | 0.9500 | C23—C28 | 1.4790 (11) |
| C6—H6 | 0.9500 | C24—C25 | 1.3840 (12) |
| C8—C9 | 1.5084 (11) | C24—H24 | 0.9500 |

| | | | |
|---|---|---|---|
| C8—H8A | 0.9900 | C25—H25 | 0.9500 |
| C8—H8B | 0.9900 | C26—C27 | 1.3875 (11) |
| C9—C10 | 1.5301 (12) | C26—H26 | 0.9500 |
| C9—H9B | 0.9900 | C27—C28 | 1.3955 (11) |
| C9—H9A | 0.9900 | C27—H27 | 0.9500 |
| C10—H10A | 0.9800 | C28—C29 | 1.3990 (12) |
| C10—H10B | 0.9800 | C29—C30 | 1.3834 (11) |
| C10—H10C | 0.9800 | C29—H29 | 0.9500 |
| C11—C16 | 1.3946 (11) | C30—H30 | 0.9500 |
| C11—C12 | 1.4002 (12) | | |

| | | | |
|---|---|---|---|
| C7—O1—H1 | 112.5 (11) | C16—C15—C14 | 119.37 (8) |
| C4—O3—C8 | 117.72 (6) | C16—C15—H15 | 120.3 |
| C17—O4—H4 | 113.0 (11) | C14—C15—H15 | 120.3 |
| C14—O6—C18 | 117.65 (6) | C15—C16—C11 | 121.16 (8) |
| C25—N1—C21 | 117.73 (7) | C15—C16—H16 | 119.4 |
| C30—N2—C26 | 117.69 (7) | C11—C16—H16 | 119.4 |
| C6—C1—C2 | 118.87 (7) | O5—C17—O4 | 123.34 (8) |
| C6—C1—C7 | 119.01 (7) | O5—C17—C11 | 123.60 (8) |
| C2—C1—C7 | 122.11 (7) | O4—C17—C11 | 113.06 (7) |
| C3—C2—C1 | 120.70 (7) | O6—C18—C19 | 107.89 (7) |
| C3—C2—H2 | 119.6 | O6—C18—H18A | 110.1 |
| C1—C2—H2 | 119.6 | C19—C18—H18A | 110.1 |
| C2—C3—C4 | 119.63 (7) | O6—C18—H18B | 110.1 |
| C2—C3—H3 | 120.2 | C19—C18—H18B | 110.1 |
| C4—C3—H3 | 120.2 | H18A—C18—H18B | 108.4 |
| O3—C4—C5 | 124.21 (8) | C18—C19—C20 | 110.05 (7) |
| O3—C4—C3 | 115.45 (7) | C18—C19—H19B | 109.6 |
| C5—C4—C3 | 120.34 (8) | C20—C19—H19B | 109.6 |
| C6—C5—C4 | 119.14 (8) | C18—C19—H19A | 109.6 |
| C6—C5—H5 | 120.4 | C20—C19—H19A | 109.6 |
| C4—C5—H5 | 120.4 | H19B—C19—H19A | 108.2 |
| C5—C6—C1 | 121.31 (8) | C19—C20—H20A | 109.5 |
| C5—C6—H6 | 119.3 | C19—C20—H20B | 109.5 |
| C1—C6—H6 | 119.3 | H20A—C20—H20B | 109.5 |
| O2—C7—O1 | 123.50 (8) | C19—C20—H20C | 109.5 |
| O2—C7—C1 | 123.11 (8) | H20A—C20—H20C | 109.5 |
| O1—C7—C1 | 113.39 (7) | H20B—C20—H20C | 109.5 |
| O3—C8—C9 | 107.90 (7) | N1—C21—C22 | 123.03 (8) |
| O3—C8—H8A | 110.1 | N1—C21—H21 | 118.5 |
| C9—C8—H8A | 110.1 | C22—C21—H21 | 118.5 |
| O3—C8—H8B | 110.1 | C21—C22—C23 | 119.47 (8) |
| C9—C8—H8B | 110.1 | C21—C22—H22 | 120.3 |
| H8A—C8—H8B | 108.4 | C23—C22—H22 | 120.3 |
| C8—C9—C10 | 109.79 (7) | C22—C23—C24 | 117.30 (8) |
| C8—C9—H9B | 109.7 | C22—C23—C28 | 121.74 (8) |
| C10—C9—H9B | 109.7 | C24—C23—C28 | 120.94 (8) |
| C8—C9—H9A | 109.7 | C25—C24—C23 | 119.67 (8) |

| | | | |
|---|---|---|---|
| C10—C9—H9A | 109.7 | C25—C24—H24 | 120.2 |
| H9B—C9—H9A | 108.2 | C23—C24—H24 | 120.2 |
| C9—C10—H10A | 109.5 | N1—C25—C24 | 122.80 (8) |
| C9—C10—H10B | 109.5 | N1—C25—H25 | 118.6 |
| H10A—C10—H10B | 109.5 | C24—C25—H25 | 118.6 |
| C9—C10—H10C | 109.5 | N2—C26—C27 | 123.00 (8) |
| H10A—C10—H10C | 109.5 | N2—C26—H26 | 118.5 |
| H10B—C10—H10C | 109.5 | C27—C26—H26 | 118.5 |
| C16—C11—C12 | 118.80 (8) | C26—C27—C28 | 119.40 (8) |
| C16—C11—C17 | 119.71 (7) | C26—C27—H27 | 120.3 |
| C12—C11—C17 | 121.49 (8) | C28—C27—H27 | 120.3 |
| C13—C12—C11 | 120.75 (8) | C27—C28—C29 | 117.37 (7) |
| C13—C12—H12 | 119.6 | C27—C28—C23 | 121.57 (8) |
| C11—C12—H12 | 119.6 | C29—C28—C23 | 121.05 (8) |
| C12—C13—C14 | 120.08 (8) | C30—C29—C28 | 119.54 (8) |
| C12—C13—H13 | 120.0 | C30—C29—H29 | 120.2 |
| C14—C13—H13 | 120.0 | C28—C29—H29 | 120.2 |
| O6—C14—C15 | 124.82 (7) | N2—C30—C29 | 123.00 (8) |
| O6—C14—C13 | 115.36 (7) | N2—C30—H30 | 118.5 |
| C15—C14—C13 | 119.82 (8) | C29—C30—H30 | 118.5 |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −0.36 (13) | C12—C11—C16—C15 | −1.18 (13) |
| C7—C1—C2—C3 | −178.83 (7) | C17—C11—C16—C15 | 178.18 (7) |
| C1—C2—C3—C4 | 1.02 (13) | C16—C11—C17—O5 | 0.98 (13) |
| C8—O3—C4—C5 | −1.09 (12) | C12—C11—C17—O5 | −179.69 (8) |
| C8—O3—C4—C3 | 179.21 (7) | C16—C11—C17—O4 | −178.26 (7) |
| C2—C3—C4—O3 | 178.88 (7) | C12—C11—C17—O4 | 1.08 (12) |
| C2—C3—C4—C5 | −0.84 (13) | C14—O6—C18—C19 | −177.34 (7) |
| O3—C4—C5—C6 | −179.70 (7) | O6—C18—C19—C20 | 178.05 (7) |
| C3—C4—C5—C6 | −0.01 (13) | C25—N1—C21—C22 | −0.30 (13) |
| C4—C5—C6—C1 | 0.69 (13) | N1—C21—C22—C23 | −0.11 (14) |
| C2—C1—C6—C5 | −0.51 (13) | C21—C22—C23—C24 | 0.12 (12) |
| C7—C1—C6—C5 | 178.01 (7) | C21—C22—C23—C28 | 178.42 (8) |
| C6—C1—C7—O2 | 1.75 (13) | C22—C23—C24—C25 | 0.27 (12) |
| C2—C1—C7—O2 | −179.78 (8) | C28—C23—C24—C25 | −178.04 (8) |
| C6—C1—C7—O1 | −177.75 (7) | C21—N1—C25—C24 | 0.72 (13) |
| C2—C1—C7—O1 | 0.72 (12) | C23—C24—C25—N1 | −0.72 (13) |
| C4—O3—C8—C9 | 178.04 (7) | C30—N2—C26—C27 | 0.30 (13) |
| O3—C8—C9—C10 | −174.77 (7) | N2—C26—C27—C28 | −0.11 (13) |
| C16—C11—C12—C13 | 0.99 (13) | C26—C27—C28—C29 | −0.23 (12) |
| C17—C11—C12—C13 | −178.35 (8) | C26—C27—C28—C23 | 178.56 (8) |
| C11—C12—C13—C14 | 0.13 (13) | C22—C23—C28—C27 | 152.95 (9) |
| C18—O6—C14—C15 | −5.96 (12) | C24—C23—C28—C27 | −28.82 (12) |
| C18—O6—C14—C13 | 174.06 (7) | C22—C23—C28—C29 | −28.31 (12) |
| C12—C13—C14—O6 | 178.89 (7) | C24—C23—C28—C29 | 149.93 (8) |
| C12—C13—C14—C15 | −1.09 (13) | C27—C28—C29—C30 | 0.37 (12) |
| O6—C14—C15—C16 | −179.08 (7) | C23—C28—C29—C30 | −178.42 (8) |
| C13—C14—C15—C16 | 0.91 (13) | C26—N2—C30—C29 | −0.15 (13) |

| | | | |
|---|---|---|---|
| C14—C15—C16—C11 | 0.23 (13) | C28—C29—C30—N2 | −0.19 (13) |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C11–C16 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 1.03 (2) | 1.61 (2) | 2.6407 (10) | 174.3 (19) |
| O4—H4···N2 | 1.01 (2) | 1.67 (2) | 2.6728 (11) | 173.9 (18) |
| C3—H3···O5$^{i}$ | 0.95 | 2.57 | 3.3981 (11) | 146 |
| C25—H25···O3$^{ii}$ | 0.95 | 2.57 | 3.4581 (11) | 156 |
| C9—H9*B*···*Cg*2$^{iii}$ | 0.99 | 2.84 | 3.6750 (1) | 142 |
| C19—H19*A*···*Cg*1$^{iv}$ | 0.99 | 2.72 | 3.5781 (1) | 146 |

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

### (III) 4-*n*-Butoxybenzoic acid–4,4′-bipyridyl (2/1)

*Crystal data*

$2C_{11}H_{14}O_3·C_{10}H_8N_2$
$M_r$ = 544.63
Triclinic, $P\overline{1}$
*a* = 7.6645 (10) Å
*b* = 8.5087 (13) Å
*c* = 22.606 (3) Å
*α* = 80.498 (3)°
*β* = 86.486 (3)°
*γ* = 80.082 (3)°
*V* = 1431.5 (4) Å$^3$
*Z* = 2
*F*(000) = 580.00
$D_x$ = 1.264 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71075 Å
Cell parameters from 9788 reflections
*θ* = 3.2–30.1°
*μ* = 0.09 mm$^{-1}$
*T* = 93 K
Platelet, colorless
0.53 × 0.41 × 0.11 mm

*Data collection*

Rigaku R-AXIS RAPIDII diffractometer
Detector resolution: 10.000 pixels mm$^{-1}$
*ω* scans
12433 measured reflections
5612 independent reflections
3432 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.075
$\theta_{max}$ = 26.0°
*h* = −8→9
*k* = −10→10
*l* = −27→27

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.193
*S* = 1.01
5610 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
$w = 1/[\sigma^2(F_o^2) + (0.0965P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.1390 (2) | −0.1143 (2) | −0.18237 (8) | 0.0358 (4) |
| O2 | 0.3054 (2) | −0.3129 (2) | −0.12317 (8) | 0.0399 (5) |
| O3 | 0.1632 (2) | −0.6711 (2) | −0.33646 (8) | 0.0347 (4) |
| O4 | 0.2363 (2) | 0.7005 (2) | 0.21058 (8) | 0.0380 (5) |
| O5 | 0.3659 (3) | 0.8609 (3) | 0.13954 (8) | 0.0433 (5) |
| O6 | 0.3442 (2) | 1.2321 (2) | 0.36266 (7) | 0.0336 (4) |
| N1 | 0.1708 (3) | 0.0497 (3) | −0.09736 (10) | 0.0375 (5) |
| N2 | 0.2511 (3) | 0.5220 (3) | 0.12499 (9) | 0.0343 (5) |
| C1 | 0.2120 (3) | −0.3677 (3) | −0.21459 (10) | 0.0288 (5) |
| C2 | 0.1061 (3) | −0.3154 (3) | −0.26457 (11) | 0.0318 (6) |
| H2 | 0.0429 | −0.2080 | −0.2710 | 0.038* |
| C3 | 0.0926 (3) | −0.4186 (3) | −0.30463 (11) | 0.0325 (6) |
| H3 | 0.0206 | −0.3821 | −0.3385 | 0.039* |
| C4 | 0.1843 (3) | −0.5760 (3) | −0.29530 (11) | 0.0306 (5) |
| C5 | 0.2917 (3) | −0.6282 (3) | −0.24626 (11) | 0.0329 (6) |
| H5 | 0.3555 | −0.7354 | −0.2399 | 0.040* |
| C6 | 0.3055 (3) | −0.5231 (3) | −0.20661 (11) | 0.0323 (6) |
| H6 | 0.3804 | −0.5586 | −0.1734 | 0.039* |
| C7 | 0.2243 (3) | −0.2636 (3) | −0.16939 (11) | 0.0323 (6) |
| C8 | 0.2479 (3) | −0.8367 (3) | −0.32529 (12) | 0.0338 (6) |
| H8A | 0.2005 | −0.8918 | −0.2872 | 0.041* |
| H8B | 0.3771 | −0.8427 | −0.3219 | 0.041* |
| C9 | 0.2121 (3) | −0.9175 (4) | −0.37668 (11) | 0.0352 (6) |
| H9A | 0.2542 | −1.0350 | −0.3663 | 0.042* |
| H9B | 0.0824 | −0.9016 | −0.3814 | 0.042* |
| C10 | 0.2976 (3) | −0.8574 (4) | −0.43586 (12) | 0.0381 (6) |
| H10A | 0.4271 | −0.8701 | −0.4313 | 0.046* |
| H10B | 0.2521 | −0.7409 | −0.4475 | 0.046* |
| C11 | 0.2622 (4) | −0.9474 (4) | −0.48561 (13) | 0.0474 (7) |
| H11A | 0.3074 | −1.0629 | −0.4745 | 0.071* |
| H11B | 0.3221 | −0.9055 | −0.5230 | 0.071* |
| H11C | 0.1343 | −0.9319 | −0.4914 | 0.071* |
| C12 | 0.3175 (3) | 0.9341 (3) | 0.23705 (11) | 0.0300 (5) |
| C13 | 0.2420 (3) | 0.9052 (3) | 0.29435 (11) | 0.0300 (5) |
| H13 | 0.1816 | 0.8155 | 0.3053 | 0.036* |
| C14 | 0.2538 (3) | 1.0058 (3) | 0.33557 (11) | 0.0301 (5) |
| H14 | 0.2019 | 0.9852 | 0.3747 | 0.036* |

| | | | | |
|---|---|---|---|---|
| C15 | 0.3423 (3) | 1.1382 (3) | 0.31945 (11) | 0.0305 (5) |
| C16 | 0.4185 (3) | 1.1671 (3) | 0.26282 (11) | 0.0325 (6) |
| H16 | 0.4781 | 1.2572 | 0.2518 | 0.039* |
| C17 | 0.4079 (3) | 1.0642 (3) | 0.22178 (11) | 0.0320 (6) |
| H17 | 0.4629 | 1.0829 | 0.1830 | 0.038* |
| C18 | 0.3088 (3) | 0.8303 (3) | 0.19068 (11) | 0.0323 (6) |
| C19 | 0.4228 (3) | 1.3756 (3) | 0.34655 (12) | 0.0375 (6) |
| H19A | 0.3595 | 1.4478 | 0.3129 | 0.045* |
| H19B | 0.5485 | 1.3469 | 0.3337 | 0.045* |
| C20 | 0.4099 (3) | 1.4595 (4) | 0.40073 (12) | 0.0376 (6) |
| H20A | 0.4719 | 1.5539 | 0.3912 | 0.045* |
| H20B | 0.4720 | 1.3845 | 0.4340 | 0.045* |
| C21 | 0.2211 (3) | 1.5162 (4) | 0.42172 (12) | 0.0372 (6) |
| H21A | 0.1552 | 1.5829 | 0.3875 | 0.045* |
| H21B | 0.1624 | 1.4210 | 0.4354 | 0.045* |
| C22 | 0.2123 (4) | 1.6142 (4) | 0.47241 (13) | 0.0452 (7) |
| H22A | 0.2700 | 1.5464 | 0.5075 | 0.068* |
| H22B | 0.0881 | 1.6524 | 0.4830 | 0.068* |
| H22C | 0.2731 | 1.7070 | 0.4596 | 0.068* |
| C23 | 0.2365 (4) | −0.0184 (4) | −0.04412 (13) | 0.0472 (7) |
| H23 | 0.2711 | −0.1324 | −0.0368 | 0.057* |
| C24 | 0.2571 (4) | 0.0681 (4) | 0.00093 (12) | 0.0421 (7) |
| H24 | 0.3000 | 0.0144 | 0.0388 | 0.051* |
| C25 | 0.2135 (3) | 0.2354 (3) | −0.01037 (11) | 0.0307 (5) |
| C26 | 0.1489 (3) | 0.3070 (3) | −0.06606 (11) | 0.0332 (6) |
| H26 | 0.1191 | 0.4211 | −0.0756 | 0.040* |
| C27 | 0.1286 (3) | 0.2094 (3) | −0.10754 (12) | 0.0347 (6) |
| H27 | 0.0819 | 0.2593 | −0.1453 | 0.042* |
| C28 | 0.2950 (3) | 0.5728 (4) | 0.06786 (11) | 0.0365 (6) |
| H28 | 0.3359 | 0.6735 | 0.0581 | 0.044* |
| C29 | 0.2834 (3) | 0.4853 (3) | 0.02218 (11) | 0.0329 (6) |
| H29 | 0.3116 | 0.5271 | −0.0182 | 0.040* |
| C30 | 0.2296 (3) | 0.3348 (3) | 0.03651 (11) | 0.0298 (5) |
| C31 | 0.1889 (3) | 0.2808 (3) | 0.09596 (11) | 0.0334 (6) |
| H31 | 0.1551 | 0.1775 | 0.1075 | 0.040* |
| C32 | 0.1978 (3) | 0.3784 (3) | 0.13857 (11) | 0.0332 (6) |
| H32 | 0.1648 | 0.3420 | 0.1790 | 0.040* |
| H1 | 0.160 (4) | −0.054 (4) | −0.1475 (15) | 0.060 (9)* |
| H4 | 0.242 (5) | 0.642 (5) | 0.1754 (16) | 0.065 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0412 (10) | 0.0304 (11) | 0.0354 (10) | −0.0006 (8) | −0.0033 (8) | −0.0091 (8) |
| O2 | 0.0485 (11) | 0.0369 (12) | 0.0331 (10) | 0.0009 (9) | −0.0080 (8) | −0.0085 (8) |
| O3 | 0.0326 (9) | 0.0333 (11) | 0.0377 (10) | 0.0069 (8) | −0.0063 (7) | −0.0156 (8) |
| O4 | 0.0436 (10) | 0.0351 (12) | 0.0371 (10) | −0.0067 (8) | −0.0014 (8) | −0.0110 (9) |
| O5 | 0.0581 (12) | 0.0399 (13) | 0.0319 (10) | −0.0076 (10) | 0.0004 (9) | −0.0064 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O6 | 0.0371 (9) | 0.0322 (11) | 0.0333 (9) | −0.0080 (8) | 0.0011 (7) | −0.0089 (8) |
| N1 | 0.0455 (12) | 0.0327 (14) | 0.0337 (12) | −0.0007 (10) | −0.0019 (9) | −0.0092 (10) |
| N2 | 0.0328 (10) | 0.0357 (14) | 0.0340 (11) | 0.0000 (9) | −0.0018 (9) | −0.0097 (10) |
| C1 | 0.0261 (11) | 0.0307 (15) | 0.0292 (12) | −0.0020 (10) | 0.0019 (9) | −0.0074 (10) |
| C2 | 0.0290 (11) | 0.0315 (15) | 0.0338 (13) | 0.0009 (10) | −0.0018 (10) | −0.0079 (11) |
| C3 | 0.0280 (11) | 0.0358 (16) | 0.0321 (13) | 0.0023 (11) | −0.0042 (10) | −0.0071 (11) |
| C4 | 0.0250 (11) | 0.0340 (15) | 0.0327 (13) | 0.0001 (10) | −0.0016 (9) | −0.0100 (11) |
| C5 | 0.0329 (12) | 0.0283 (15) | 0.0357 (13) | 0.0050 (10) | −0.0041 (10) | −0.0088 (11) |
| C6 | 0.0293 (11) | 0.0369 (16) | 0.0304 (12) | −0.0021 (11) | −0.0018 (10) | −0.0078 (11) |
| C7 | 0.0324 (12) | 0.0317 (15) | 0.0328 (13) | −0.0040 (11) | 0.0008 (10) | −0.0074 (11) |
| C8 | 0.0357 (12) | 0.0288 (15) | 0.0360 (14) | 0.0005 (11) | 0.0002 (10) | −0.0088 (11) |
| C9 | 0.0351 (12) | 0.0359 (16) | 0.0360 (13) | −0.0052 (11) | 0.0022 (10) | −0.0115 (12) |
| C10 | 0.0383 (13) | 0.0402 (17) | 0.0365 (14) | −0.0050 (12) | 0.0019 (11) | −0.0111 (12) |
| C11 | 0.0518 (16) | 0.054 (2) | 0.0385 (15) | −0.0066 (15) | 0.0001 (13) | −0.0161 (14) |
| C12 | 0.0268 (11) | 0.0271 (14) | 0.0351 (13) | 0.0014 (10) | −0.0069 (10) | −0.0060 (11) |
| C13 | 0.0261 (11) | 0.0291 (14) | 0.0331 (13) | 0.0000 (10) | −0.0030 (10) | −0.0043 (11) |
| C14 | 0.0280 (11) | 0.0326 (15) | 0.0296 (12) | −0.0038 (10) | −0.0021 (10) | −0.0053 (11) |
| C15 | 0.0265 (11) | 0.0289 (15) | 0.0353 (13) | −0.0004 (10) | −0.0035 (10) | −0.0058 (11) |
| C16 | 0.0312 (12) | 0.0315 (15) | 0.0338 (13) | −0.0040 (11) | −0.0019 (10) | −0.0035 (11) |
| C17 | 0.0290 (11) | 0.0341 (15) | 0.0309 (13) | −0.0009 (10) | −0.0023 (10) | −0.0037 (11) |
| C18 | 0.0333 (12) | 0.0297 (15) | 0.0317 (13) | 0.0019 (11) | −0.0065 (10) | −0.0035 (11) |
| C19 | 0.0354 (13) | 0.0357 (17) | 0.0442 (15) | −0.0095 (12) | 0.0020 (11) | −0.0120 (12) |
| C20 | 0.0351 (13) | 0.0387 (17) | 0.0419 (15) | −0.0062 (12) | −0.0037 (11) | −0.0135 (12) |
| C21 | 0.0377 (13) | 0.0335 (16) | 0.0397 (14) | 0.0009 (11) | −0.0032 (11) | −0.0101 (12) |
| C22 | 0.0491 (16) | 0.0396 (18) | 0.0474 (16) | −0.0021 (13) | 0.0005 (13) | −0.0149 (14) |
| C23 | 0.071 (2) | 0.0278 (16) | 0.0404 (15) | 0.0043 (14) | −0.0095 (14) | −0.0083 (13) |
| C24 | 0.0577 (17) | 0.0306 (16) | 0.0344 (14) | 0.0049 (13) | −0.0063 (12) | −0.0055 (12) |
| C25 | 0.0292 (11) | 0.0310 (15) | 0.0314 (13) | −0.0004 (10) | 0.0003 (10) | −0.0086 (11) |
| C26 | 0.0368 (13) | 0.0280 (15) | 0.0337 (13) | 0.0002 (11) | −0.0044 (10) | −0.0061 (11) |
| C27 | 0.0351 (13) | 0.0339 (16) | 0.0340 (13) | 0.0014 (11) | −0.0027 (10) | −0.0087 (11) |
| C28 | 0.0382 (13) | 0.0349 (16) | 0.0377 (14) | −0.0058 (12) | −0.0014 (11) | −0.0093 (12) |
| C29 | 0.0327 (12) | 0.0326 (15) | 0.0327 (13) | −0.0018 (11) | 0.0012 (10) | −0.0071 (11) |
| C30 | 0.0273 (11) | 0.0295 (14) | 0.0314 (12) | 0.0026 (10) | −0.0020 (9) | −0.0086 (11) |
| C31 | 0.0346 (12) | 0.0316 (15) | 0.0326 (13) | −0.0004 (11) | −0.0004 (10) | −0.0064 (11) |
| C32 | 0.0323 (12) | 0.0324 (15) | 0.0312 (13) | 0.0036 (11) | 0.0003 (10) | −0.0042 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.319 (3) | C12—C18 | 1.489 (3) |
| O1—H1 | 1.04 (3) | C13—C14 | 1.382 (3) |
| O2—C7 | 1.225 (3) | C13—H13 | 0.9500 |
| O3—C4 | 1.363 (3) | C14—C15 | 1.399 (3) |
| O3—C8 | 1.435 (3) | C14—H14 | 0.9500 |
| O4—C18 | 1.322 (3) | C15—C16 | 1.376 (3) |
| O4—H4 | 1.00 (4) | C16—C17 | 1.392 (3) |
| O5—C18 | 1.214 (3) | C16—H16 | 0.9500 |
| O6—C15 | 1.362 (3) | C17—H17 | 0.9500 |
| O6—C19 | 1.438 (3) | C19—C20 | 1.506 (3) |

| | | | |
|---|---|---|---|
| N1—C27 | 1.325 (4) | C19—H19A | 0.9900 |
| N1—C23 | 1.335 (4) | C19—H19B | 0.9900 |
| N2—C28 | 1.334 (3) | C20—C21 | 1.518 (4) |
| N2—C32 | 1.337 (3) | C20—H20A | 0.9900 |
| C1—C6 | 1.381 (4) | C20—H20B | 0.9900 |
| C1—C2 | 1.398 (3) | C21—C22 | 1.516 (4) |
| C1—C7 | 1.475 (3) | C21—H21A | 0.9900 |
| C2—C3 | 1.381 (3) | C21—H21B | 0.9900 |
| C2—H2 | 0.9500 | C22—H22A | 0.9800 |
| C3—C4 | 1.390 (4) | C22—H22B | 0.9800 |
| C3—H3 | 0.9500 | C22—H22C | 0.9800 |
| C4—C5 | 1.389 (3) | C23—C24 | 1.382 (4) |
| C5—C6 | 1.387 (3) | C23—H23 | 0.9500 |
| C5—H5 | 0.9500 | C24—C25 | 1.389 (4) |
| C6—H6 | 0.9500 | C24—H24 | 0.9500 |
| C8—C9 | 1.505 (3) | C25—C26 | 1.386 (3) |
| C8—H8A | 0.9900 | C25—C30 | 1.483 (3) |
| C8—H8B | 0.9900 | C26—C27 | 1.383 (3) |
| C9—C10 | 1.504 (4) | C26—H26 | 0.9500 |
| C9—H9A | 0.9900 | C27—H27 | 0.9500 |
| C9—H9B | 0.9900 | C28—C29 | 1.384 (3) |
| C10—C11 | 1.523 (3) | C28—H28 | 0.9500 |
| C10—H10A | 0.9900 | C29—C30 | 1.394 (4) |
| C10—H10B | 0.9900 | C29—H29 | 0.9500 |
| C11—H11A | 0.9800 | C30—C31 | 1.382 (3) |
| C11—H11B | 0.9800 | C31—C32 | 1.383 (3) |
| C11—H11C | 0.9800 | C31—H31 | 0.9500 |
| C12—C13 | 1.387 (3) | C32—H32 | 0.9500 |
| C12—C17 | 1.390 (3) | | |

| | | | |
|---|---|---|---|
| C7—O1—H1 | 107 (2) | C15—C16—C17 | 119.9 (2) |
| C4—O3—C8 | 117.16 (19) | C15—C16—H16 | 120.1 |
| C18—O4—H4 | 105 (2) | C17—C16—H16 | 120.1 |
| C15—O6—C19 | 117.34 (19) | C12—C17—C16 | 120.5 (2) |
| C27—N1—C23 | 117.5 (2) | C12—C17—H17 | 119.7 |
| C28—N2—C32 | 118.2 (2) | C16—C17—H17 | 119.7 |
| C6—C1—C2 | 118.9 (2) | O5—C18—O4 | 123.1 (2) |
| C6—C1—C7 | 118.8 (2) | O5—C18—C12 | 123.0 (2) |
| C2—C1—C7 | 122.3 (2) | O4—C18—C12 | 113.9 (2) |
| C3—C2—C1 | 120.5 (2) | O6—C19—C20 | 108.0 (2) |
| C3—C2—H2 | 119.7 | O6—C19—H19A | 110.1 |
| C1—C2—H2 | 119.7 | C20—C19—H19A | 110.1 |
| C2—C3—C4 | 120.0 (2) | O6—C19—H19B | 110.1 |
| C2—C3—H3 | 120.0 | C20—C19—H19B | 110.1 |
| C4—C3—H3 | 120.0 | H19A—C19—H19B | 108.4 |
| O3—C4—C5 | 123.5 (2) | C19—C20—C21 | 113.9 (2) |
| O3—C4—C3 | 116.6 (2) | C19—C20—H20A | 108.8 |
| C5—C4—C3 | 119.9 (2) | C21—C20—H20A | 108.8 |

| | | | |
|---|---|---|---|
| C6—C5—C4 | 119.6 (2) | C19—C20—H20B | 108.8 |
| C6—C5—H5 | 120.2 | C21—C20—H20B | 108.8 |
| C4—C5—H5 | 120.2 | H20A—C20—H20B | 107.7 |
| C1—C6—C5 | 121.1 (2) | C22—C21—C20 | 112.6 (2) |
| C1—C6—H6 | 119.5 | C22—C21—H21A | 109.1 |
| C5—C6—H6 | 119.5 | C20—C21—H21A | 109.1 |
| O2—C7—O1 | 122.9 (2) | C22—C21—H21B | 109.1 |
| O2—C7—C1 | 122.4 (2) | C20—C21—H21B | 109.1 |
| O1—C7—C1 | 114.7 (2) | H21A—C21—H21B | 107.8 |
| O3—C8—C9 | 108.3 (2) | C21—C22—H22A | 109.5 |
| O3—C8—H8A | 110.0 | C21—C22—H22B | 109.5 |
| C9—C8—H8A | 110.0 | H22A—C22—H22B | 109.5 |
| O3—C8—H8B | 110.0 | C21—C22—H22C | 109.5 |
| C9—C8—H8B | 110.0 | H22A—C22—H22C | 109.5 |
| H8A—C8—H8B | 108.4 | H22B—C22—H22C | 109.5 |
| C10—C9—C8 | 114.8 (2) | N1—C23—C24 | 123.5 (3) |
| C10—C9—H9A | 108.6 | N1—C23—H23 | 118.3 |
| C8—C9—H9A | 108.6 | C24—C23—H23 | 118.3 |
| C10—C9—H9B | 108.6 | C23—C24—C25 | 118.5 (3) |
| C8—C9—H9B | 108.6 | C23—C24—H24 | 120.7 |
| H9A—C9—H9B | 107.5 | C25—C24—H24 | 120.7 |
| C9—C10—C11 | 112.5 (2) | C26—C25—C24 | 118.2 (2) |
| C9—C10—H10A | 109.1 | C26—C25—C30 | 120.8 (2) |
| C11—C10—H10A | 109.1 | C24—C25—C30 | 121.0 (2) |
| C9—C10—H10B | 109.1 | C27—C26—C25 | 118.9 (3) |
| C11—C10—H10B | 109.1 | C27—C26—H26 | 120.6 |
| H10A—C10—H10B | 107.8 | C25—C26—H26 | 120.6 |
| C10—C11—H11A | 109.5 | N1—C27—C26 | 123.4 (2) |
| C10—C11—H11B | 109.5 | N1—C27—H27 | 118.3 |
| H11A—C11—H11B | 109.5 | C26—C27—H27 | 118.3 |
| C10—C11—H11C | 109.5 | N2—C28—C29 | 122.9 (2) |
| H11A—C11—H11C | 109.5 | N2—C28—H28 | 118.5 |
| H11B—C11—H11C | 109.5 | C29—C28—H28 | 118.5 |
| C13—C12—C17 | 119.2 (2) | C28—C29—C30 | 118.8 (2) |
| C13—C12—C18 | 123.0 (2) | C28—C29—H29 | 120.6 |
| C17—C12—C18 | 117.7 (2) | C30—C29—H29 | 120.6 |
| C14—C13—C12 | 120.5 (2) | C31—C30—C29 | 118.0 (2) |
| C14—C13—H13 | 119.7 | C31—C30—C25 | 120.5 (2) |
| C12—C13—H13 | 119.7 | C29—C30—C25 | 121.5 (2) |
| C13—C14—C15 | 119.8 (2) | C30—C31—C32 | 119.5 (2) |
| C13—C14—H14 | 120.1 | C30—C31—H31 | 120.3 |
| C15—C14—H14 | 120.1 | C32—C31—H31 | 120.3 |
| O6—C15—C16 | 124.4 (2) | N2—C32—C31 | 122.5 (2) |
| O6—C15—C14 | 115.6 (2) | N2—C32—H32 | 118.7 |
| C16—C15—C14 | 120.0 (2) | C31—C32—H32 | 118.7 |
| | | | |
| C6—C1—C2—C3 | 1.2 (3) | C18—C12—C17—C16 | 179.0 (2) |
| C7—C1—C2—C3 | −177.4 (2) | C15—C16—C17—C12 | 1.4 (4) |

| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | 0.2 (4) | C13—C12—C18—O5 | 175.6 (2) |
| C8—O3—C4—C5 | 4.5 (3) | C17—C12—C18—O5 | −5.2 (4) |
| C8—O3—C4—C3 | −176.1 (2) | C13—C12—C18—O4 | −5.6 (3) |
| C2—C3—C4—O3 | 179.5 (2) | C17—C12—C18—O4 | 173.5 (2) |
| C2—C3—C4—C5 | −1.1 (4) | C15—O6—C19—C20 | 179.9 (2) |
| O3—C4—C5—C6 | 180.0 (2) | O6—C19—C20—C21 | −62.8 (3) |
| C3—C4—C5—C6 | 0.5 (4) | C19—C20—C21—C22 | −174.4 (2) |
| C2—C1—C6—C5 | −1.8 (3) | C27—N1—C23—C24 | 2.1 (4) |
| C7—C1—C6—C5 | 176.9 (2) | N1—C23—C24—C25 | −2.6 (5) |
| C4—C5—C6—C1 | 0.9 (4) | C23—C24—C25—C26 | 1.0 (4) |
| C6—C1—C7—O2 | −4.7 (4) | C23—C24—C25—C30 | 178.9 (3) |
| C2—C1—C7—O2 | 174.0 (2) | C24—C25—C26—C27 | 0.8 (4) |
| C6—C1—C7—O1 | 176.3 (2) | C30—C25—C26—C27 | −177.1 (2) |
| C2—C1—C7—O1 | −5.1 (3) | C23—N1—C27—C26 | −0.2 (4) |
| C4—O3—C8—C9 | −178.40 (19) | C25—C26—C27—N1 | −1.3 (4) |
| O3—C8—C9—C10 | 66.9 (3) | C32—N2—C28—C29 | 1.6 (4) |
| C8—C9—C10—C11 | 178.0 (2) | N2—C28—C29—C30 | −2.3 (4) |
| C17—C12—C13—C14 | 1.0 (4) | C28—C29—C30—C31 | 0.5 (4) |
| C18—C12—C13—C14 | −179.8 (2) | C28—C29—C30—C25 | 179.6 (2) |
| C12—C13—C14—C15 | 0.1 (4) | C26—C25—C30—C31 | 140.9 (3) |
| C19—O6—C15—C16 | 3.6 (4) | C24—C25—C30—C31 | −37.0 (3) |
| C19—O6—C15—C14 | −175.7 (2) | C26—C25—C30—C29 | −38.2 (3) |
| C13—C14—C15—O6 | 178.8 (2) | C24—C25—C30—C29 | 144.0 (3) |
| C13—C14—C15—C16 | −0.5 (4) | C29—C30—C31—C32 | 1.8 (4) |
| O6—C15—C16—C17 | −179.5 (2) | C25—C30—C31—C32 | −177.3 (2) |
| C14—C15—C16—C17 | −0.3 (4) | C28—N2—C32—C31 | 0.9 (4) |
| C13—C12—C17—C16 | −1.8 (4) | C30—C31—C32—N2 | −2.6 (4) |

*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 | 1.04 (3) | 1.56 (3) | 2.600 (3) | 173 (3) |
| O4—H4···N2 | 1.00 (4) | 1.64 (4) | 2.636 (3) | 172 (4) |
| C24—H24···O5$^{i}$ | 0.95 | 2.47 | 3.408 (3) | 171 |
| C29—H29···O2$^{ii}$ | 0.95 | 2.53 | 3.456 (3) | 164 |
| C2—H2···*Cg*2$^{iii}$ | 0.95 | 2.98 | 3.754 (3) | 139 |
| C8—H8*B*···*Cg*2$^{iv}$ | 0.99 | 2.68 | 3.518 (3) | 143 |
| C19—H19*B*···*Cg*1$^{v}$ | 0.99 | 2.77 | 3.586 (3) | 140 |

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