

2,3-Bis(prop-2-ynoxy)naphthalene

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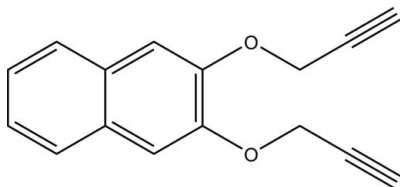
Received 30 September 2008; accepted 12 November 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 13.4.

In the crystal structure of the title compound, $\text{C}_{16}\text{H}_{12}\text{O}_2$, no classical hydrogen bonds or aromatic π - π stacking interactions were observed. The molecules are linked into a three-dimensional framework by a combination of $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi(\text{arene})$ hydrogen bonds.

Related literature

For related structures, see: Zhang *et al.* (2008); Ghosh *et al.* (2007); Wang & Kong (2007). For the synthesis, see: Burchell *et al.* (2006). For bond-length data, see: Allen *et al.* (1987). For π - π stacking interactions, see: Steed & Atwood (2000).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{O}_2$
 $M_r = 236.26$
 Orthorhombic, $Pbca$
 $a = 8.2921$ (12) Å
 $b = 9.0457$ (14) Å
 $c = 33.070$ (5) Å

$V = 2480.5$ (6) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 298$ (2) K
 $0.18 \times 0.16 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.984$, $T_{\max} = 0.989$

12468 measured reflections
 2182 independent reflections
 1782 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.101$
 $S = 1.04$
 2182 reflections

163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C4-C6/C11-C13 ring.

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C16}-\text{H16}\cdots\text{O2}^i$ | 0.93 | 2.48 | 3.409 (2) | 177 |
| $\text{C3}-\text{H3A}\cdots\text{Cg1}^{ii}$ | 0.97 | 2.95 | 3.634 (2) | 129 |

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2111).

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supplementary materials

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2,3-Bis(prop-2-ynyloxy)naphthalene

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Comment

The title compound has been characterized by *X*-ray methods (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.* 1987). Except for an ethynyl group [–C15≡C16–H16], all the remaining non–H atoms are almost coplanar, with a mean deviation from the least-square plane to be 0.0402 (14) Å. The angle between the ethynyl and the plane is 23.80 (9)°.

While π – π stacking interactions are often found in aromatics (Wang *et al.* 2007), in the title complex the minimal distance between ring centroids is 5.188 (1) Å indicating that there are no π – π stacking interactions present (Steed *et al.* 2000).

The molecules of the title complex are linked into a three-dimensional framework by a combination of C–H \cdots O and C–H \cdots π (arene) hydrogen bonds (Fig. 2, Fig. 3, Table 1). [Cg1 is the centroid of the C4–C6/C11–C13 ring. Symmetry codes: (i) $-x, y + 1/2, -z + 1/2$; (ii) $-x - 1/2, y - 1/2, z + 2$.]

Experimental

The title compound was obtained unintentionally during an attempted synthesis of a network complex (Burchell *et al.*, 2006) based on Co(II) and 2,3-bis(prop-2-ynyloxy)naphthalene, involving the evaporation of a methyl alcohol and acetone solution of CoCl $_2$ ·2H $_2$ O, NaN $_3$ and the title molecule, at 298 K.

Refinement

All the H atoms could be detected in the difference electron density maps. Nevertheless, they were situated into the idealized position and refined using a riding model. C–H = 0.97 Å for the methylene groups and C–H = 0.93 Å for the remaining H atoms. $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier C})$ for all the H atoms.

Figures

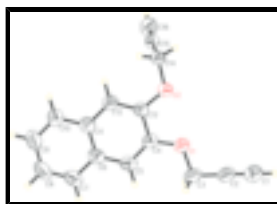


Fig. 1. A view of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

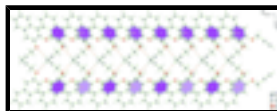


Fig. 2. The three-dimensional supramolecular framework of the title compound formed by C–H \cdots O and C–H \cdots π (arene) hydrogen bonds, viewed along the *a* axis.

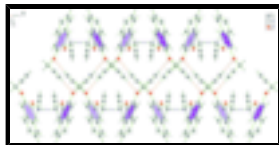


Fig. 3. The three-dimensional supramolecular framework of the title compound formed by C—H···O and C—H··· π (arene) hydrogen bonds, viewed along the *b* axis.

2,3-bis(prop-2-ynyloxy)naphthalene

Crystal data

$C_{16}H_{12}O_2$

$M_r = 236.26$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 8.2921$ (12) Å

$b = 9.0457$ (14) Å

$c = 33.070$ (5) Å

$V = 2480.5$ (6) Å³

$Z = 8$

$F_{000} = 992$

$D_x = 1.265$ Mg m⁻³

Mo *K* α radiation

$\lambda = 0.71073$ Å

Cell parameters from 3821 reflections

$\theta = 2.8$ – 25.7°

$\mu = 0.08$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.18 \times 0.16 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.984$, $T_{\max} = 0.989$

12468 measured reflections

2182 independent reflections

1782 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.5^\circ$

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -39 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.101$

$S = 1.04$

2182 reflections

163 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.338P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.17$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|--------------|-------------|----------------------------------|
| C1 | -0.1360 (2) | -0.0848 (2) | 0.32753 (6) | 0.0752 (6) |
| H1 | -0.1425 | -0.1672 | 0.3108 | 0.090* |
| C2 | -0.1277 (2) | 0.01867 (18) | 0.34856 (5) | 0.0574 (4) |
| C3 | -0.1181 (2) | 0.14633 (16) | 0.37505 (5) | 0.0554 (4) |
| H3A | -0.2249 | 0.1865 | 0.3798 | 0.066* |
| H3B | -0.0720 | 0.1182 | 0.4009 | 0.066* |
| C4 | 0.00170 (15) | 0.38601 (15) | 0.37523 (4) | 0.0400 (3) |
| C5 | -0.05860 (17) | 0.42250 (16) | 0.41224 (4) | 0.0462 (4) |
| H5 | -0.1182 | 0.3532 | 0.4267 | 0.055* |
| C6 | -0.03174 (17) | 0.56457 (16) | 0.42903 (4) | 0.0451 (4) |
| C7 | -0.0984 (2) | 0.60874 (19) | 0.46649 (4) | 0.0586 (4) |
| H7 | -0.1609 | 0.5422 | 0.4811 | 0.070* |
| C8 | -0.0727 (3) | 0.7468 (2) | 0.48143 (5) | 0.0703 (5) |
| H8 | -0.1182 | 0.7741 | 0.5060 | 0.084* |
| C9 | 0.0218 (2) | 0.8480 (2) | 0.45997 (5) | 0.0696 (5) |
| H9 | 0.0401 | 0.9417 | 0.4706 | 0.084* |
| C10 | 0.0875 (2) | 0.80997 (18) | 0.42353 (5) | 0.0577 (4) |
| H10 | 0.1500 | 0.8782 | 0.4095 | 0.069* |
| C11 | 0.06147 (17) | 0.66812 (16) | 0.40699 (4) | 0.0443 (3) |
| C12 | 0.12274 (17) | 0.62803 (15) | 0.36856 (4) | 0.0436 (3) |
| H12 | 0.1830 | 0.6959 | 0.3538 | 0.052* |
| C13 | 0.09452 (16) | 0.49128 (15) | 0.35298 (4) | 0.0391 (3) |
| C14 | 0.24207 (18) | 0.53826 (15) | 0.29190 (4) | 0.0447 (3) |
| H14A | 0.3080 | 0.4809 | 0.2735 | 0.054* |
| H14B | 0.3139 | 0.5947 | 0.3092 | 0.054* |
| C15 | 0.14206 (18) | 0.64031 (16) | 0.26860 (4) | 0.0448 (4) |
| C16 | 0.0667 (2) | 0.71916 (18) | 0.24743 (5) | 0.0575 (4) |
| H16 | 0.0073 | 0.7814 | 0.2307 | 0.069* |
| O1 | -0.01832 (12) | 0.25415 (10) | 0.35571 (3) | 0.0479 (3) |
| O2 | 0.14871 (12) | 0.43928 (10) | 0.31646 (3) | 0.0471 (3) |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0882 (14) | 0.0598 (11) | 0.0778 (12) | -0.0163 (10) | 0.0254 (11) | -0.0032 (10) |
| C2 | 0.0584 (10) | 0.0467 (9) | 0.0672 (10) | -0.0065 (7) | 0.0180 (8) | 0.0088 (8) |
| C3 | 0.0586 (10) | 0.0444 (8) | 0.0631 (9) | -0.0032 (7) | 0.0164 (8) | 0.0104 (7) |
| C4 | 0.0354 (7) | 0.0398 (7) | 0.0448 (8) | 0.0031 (6) | 0.0005 (6) | 0.0047 (6) |
| C5 | 0.0454 (8) | 0.0499 (8) | 0.0433 (8) | 0.0030 (7) | 0.0040 (6) | 0.0117 (7) |
| C6 | 0.0448 (8) | 0.0544 (9) | 0.0361 (7) | 0.0091 (7) | -0.0050 (6) | 0.0042 (6) |
| C7 | 0.0660 (10) | 0.0696 (11) | 0.0403 (8) | 0.0089 (8) | 0.0054 (8) | 0.0055 (8) |
| C8 | 0.0896 (14) | 0.0803 (13) | 0.0410 (8) | 0.0154 (11) | 0.0053 (9) | -0.0092 (8) |
| C9 | 0.0884 (13) | 0.0660 (11) | 0.0545 (10) | 0.0014 (10) | -0.0006 (9) | -0.0156 (9) |
| C10 | 0.0643 (10) | 0.0572 (9) | 0.0517 (9) | -0.0036 (8) | -0.0007 (8) | -0.0081 (7) |
| C11 | 0.0418 (8) | 0.0508 (8) | 0.0403 (7) | 0.0042 (6) | -0.0051 (6) | -0.0007 (6) |
| C12 | 0.0418 (8) | 0.0453 (8) | 0.0438 (8) | -0.0042 (6) | 0.0031 (6) | 0.0029 (6) |
| C13 | 0.0345 (7) | 0.0440 (7) | 0.0388 (7) | 0.0025 (6) | 0.0021 (6) | 0.0022 (6) |
| C14 | 0.0452 (8) | 0.0438 (7) | 0.0453 (7) | -0.0025 (6) | 0.0115 (7) | 0.0018 (6) |
| C15 | 0.0497 (8) | 0.0442 (8) | 0.0405 (7) | -0.0073 (7) | 0.0029 (7) | -0.0073 (6) |
| C16 | 0.0616 (10) | 0.0558 (9) | 0.0552 (9) | -0.0044 (8) | -0.0124 (8) | 0.0009 (8) |
| O1 | 0.0499 (6) | 0.0404 (5) | 0.0535 (6) | -0.0050 (4) | 0.0135 (5) | 0.0027 (4) |
| O2 | 0.0535 (6) | 0.0423 (5) | 0.0454 (5) | -0.0060 (5) | 0.0140 (5) | -0.0010 (4) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-------------|-------------|-------------|
| C1—C2 | 1.168 (2) | C8—H8 | 0.9300 |
| C1—H1 | 0.9300 | C9—C10 | 1.367 (2) |
| C2—C3 | 1.452 (2) | C9—H9 | 0.9300 |
| C3—O1 | 1.4299 (16) | C10—C11 | 1.412 (2) |
| C3—H3A | 0.9700 | C10—H10 | 0.9300 |
| C3—H3B | 0.9700 | C11—C12 | 1.416 (2) |
| C4—C5 | 1.363 (2) | C12—C13 | 1.3603 (19) |
| C4—O1 | 1.3663 (16) | C12—H12 | 0.9300 |
| C4—C13 | 1.4285 (18) | C13—O2 | 1.3718 (15) |
| C5—C6 | 1.418 (2) | C14—O2 | 1.4354 (16) |
| C5—H5 | 0.9300 | C14—C15 | 1.461 (2) |
| C6—C7 | 1.414 (2) | C14—H14A | 0.9700 |
| C6—C11 | 1.416 (2) | C14—H14B | 0.9700 |
| C7—C8 | 1.360 (2) | C15—C16 | 1.179 (2) |
| C7—H7 | 0.9300 | C16—H16 | 0.9300 |
| C8—C9 | 1.399 (3) | | |
| C2—C1—H1 | 180.0 | C10—C9—H9 | 119.8 |
| C1—C2—C3 | 179.39 (17) | C8—C9—H9 | 119.8 |
| O1—C3—C2 | 107.72 (12) | C9—C10—C11 | 120.64 (16) |
| O1—C3—H3A | 110.2 | C9—C10—H10 | 119.7 |
| C2—C3—H3A | 110.2 | C11—C10—H10 | 119.7 |
| O1—C3—H3B | 110.2 | C10—C11—C12 | 121.72 (14) |
| C2—C3—H3B | 110.2 | C10—C11—C6 | 119.03 (13) |

| | | | |
|----------------|--------------|-----------------|--------------|
| H3A—C3—H3B | 108.5 | C12—C11—C6 | 119.23 (13) |
| C5—C4—O1 | 126.24 (12) | C13—C12—C11 | 120.74 (13) |
| C5—C4—C13 | 119.93 (13) | C13—C12—H12 | 119.6 |
| O1—C4—C13 | 113.83 (11) | C11—C12—H12 | 119.6 |
| C4—C5—C6 | 120.91 (13) | C12—C13—O2 | 126.08 (12) |
| C4—C5—H5 | 119.5 | C12—C13—C4 | 120.25 (12) |
| C6—C5—H5 | 119.5 | O2—C13—C4 | 113.67 (11) |
| C7—C6—C11 | 118.51 (14) | O2—C14—C15 | 112.73 (12) |
| C7—C6—C5 | 122.53 (14) | O2—C14—H14A | 109.0 |
| C11—C6—C5 | 118.93 (12) | C15—C14—H14A | 109.0 |
| C8—C7—C6 | 121.10 (16) | O2—C14—H14B | 109.0 |
| C8—C7—H7 | 119.4 | C15—C14—H14B | 109.0 |
| C6—C7—H7 | 119.4 | H14A—C14—H14B | 107.8 |
| C7—C8—C9 | 120.29 (16) | C16—C15—C14 | 175.37 (15) |
| C7—C8—H8 | 119.9 | C15—C16—H16 | 180.0 |
| C9—C8—H8 | 119.9 | C4—O1—C3 | 117.03 (11) |
| C10—C9—C8 | 120.41 (17) | C13—O2—C14 | 117.45 (10) |
| O1—C4—C5—C6 | -178.85 (12) | C10—C11—C12—C13 | -179.18 (14) |
| C13—C4—C5—C6 | 0.5 (2) | C6—C11—C12—C13 | -0.7 (2) |
| C4—C5—C6—C7 | 177.07 (13) | C11—C12—C13—O2 | -179.26 (12) |
| C4—C5—C6—C11 | -1.0 (2) | C11—C12—C13—C4 | 0.2 (2) |
| C11—C6—C7—C8 | -0.7 (2) | C5—C4—C13—C12 | -0.1 (2) |
| C5—C6—C7—C8 | -178.81 (15) | O1—C4—C13—C12 | 179.33 (12) |
| C6—C7—C8—C9 | -0.5 (3) | C5—C4—C13—O2 | 179.43 (12) |
| C7—C8—C9—C10 | 0.9 (3) | O1—C4—C13—O2 | -1.12 (16) |
| C8—C9—C10—C11 | -0.2 (3) | C5—C4—O1—C3 | 3.1 (2) |
| C9—C10—C11—C12 | 177.43 (15) | C13—C4—O1—C3 | -176.35 (12) |
| C9—C10—C11—C6 | -1.0 (2) | C2—C3—O1—C4 | 177.75 (12) |
| C7—C6—C11—C10 | 1.4 (2) | C12—C13—O2—C14 | -1.0 (2) |
| C5—C6—C11—C10 | 179.60 (13) | C4—C13—O2—C14 | 179.51 (11) |
| C7—C6—C11—C12 | -177.05 (13) | C15—C14—O2—C13 | -81.84 (15) |
| C5—C6—C11—C12 | 1.1 (2) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C16—H16 \cdots O2 ⁱ | 0.93 | 2.48 | 3.409 (2) | 177 |
| C3—H3A \cdots Cg1 ⁱⁱ | 0.97 | 2.95 | 3.634 (2) | 129 |

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

Fig. 1

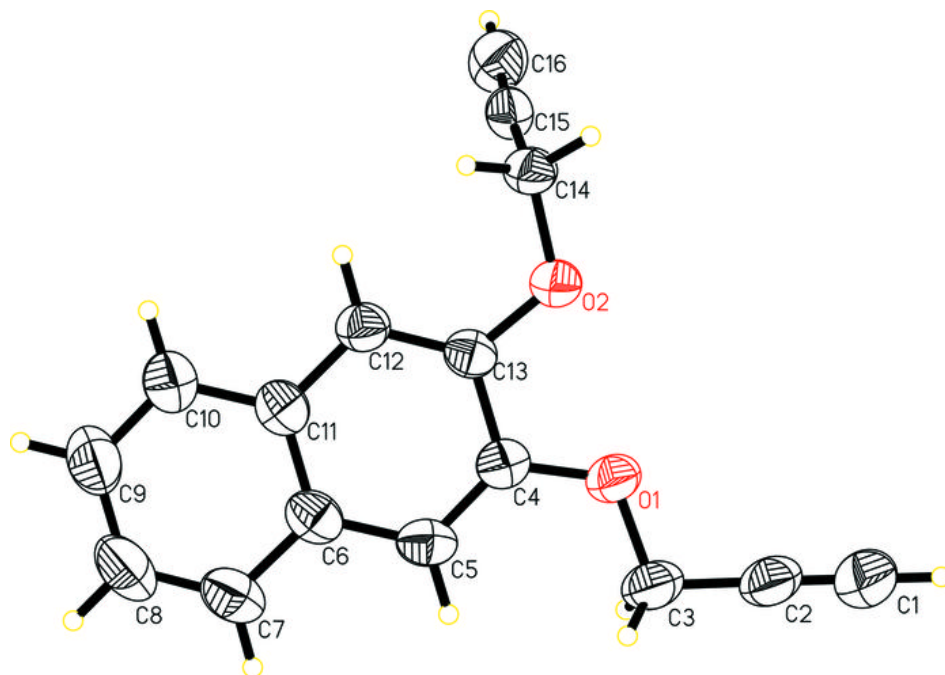


Fig. 2

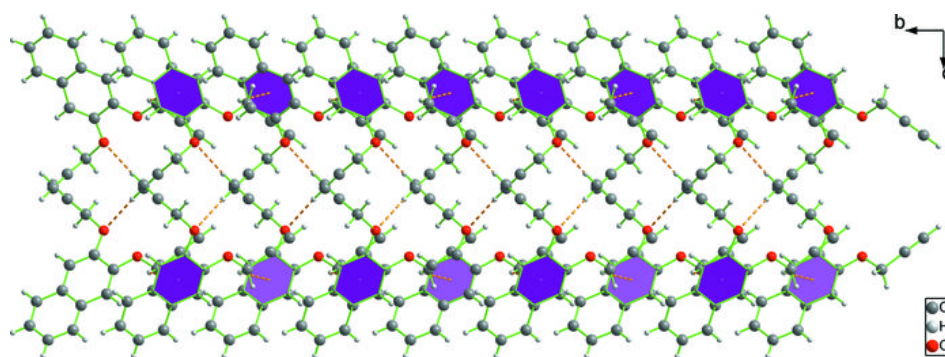


Fig. 3

