Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Tetrakis[(4-cyanophenoxy)methyl]methane

Wei Xu,[a,b]* Yin-Xiang Lu,[a] Chun-Ming Liu,[a] Peng Guo,[a] Bi-Jian Lan[a] and Hui Zhou[a]

[a]Department of Materials Science, Fudan University, Shanghai 200433, People's Republic of China, and [b]Department of Chemistry, Fudan University, Shanghai 200433, People's Republic of China

Correspondence e-mail: wexu@fudan.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.062
wR factor = 0.145
Data-to-parameter ratio = 15.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

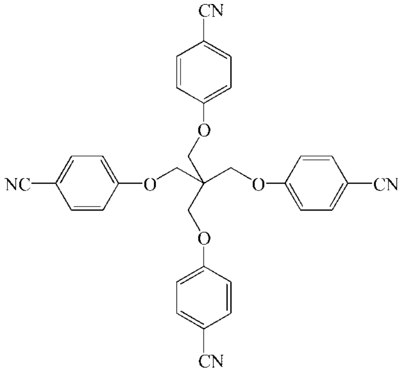
Received 12 March 2004
Accepted 27 April 2004
Online 22 May 2004

The title compound, $C_{33}H_{24}N_4O_4$, is a symmetric molecule with four chemically identical substituents bonded to a central C atom. In the crystal structure, there are C—H···N, C—H···O, C—H···C, C—H···π(C≡N) and edge-to-face aromatic interactions. The four chemically identical substituents have different conformations.

Comment

In an earlier publication of the structure of tetrakis[(4-phenylpiperazinyl)methyl]methane (Xu *et al.*, 2004), we have shown that the four identical substituents attached to the central C atom have different conformations. This paper continues our study of such chemically symmetric molecules.

The title compound, (I), is a tetrahedral molecule with four chemically identical substituents bonded to a central C atom. The molecular structure of (I), with the atom-labeling scheme, is shown in Fig. 1. Selected geometric parameters are given in Table 1.



(I)

In the crystal structure, there are some short non-covalent interactions, such as C—H···N, C—H···O and C—H···C (Table 2). Furthermore, many other interactions have also been observed. The C7—H7···π(C26≡N3)^{iv} interaction has an H7···N3^{iv} separation of 2.77 Å and a C7—H7···N3^{iv} angle of 133° [symmetry code: (iv) $x - 1, y, z$]; the H7···N3^{iv}—C26^{iv} angle of 92° may be indicative of a significant contribution of the N≡C π electrons to the interaction (Kumar *et al.*, 1998). The C8—H8···π(C19≡N2)^{v} interaction has H8···N2^{v} and H8···C19^{v} separations of 2.79 and 2.77 Å, respectively; the C8—H8···N2^{v} and C8—H8···C19^{v} angles are 162 and 139°, respectively [symmetry code: (v) $x, y - 1, z$]. The C3—H3B···π(C12≡N1)^{i} interaction has a short H3B···N1^{i} separation of 2.62 Å and a near right angle H3B···N1^{i}—C12^{i} of 98° [symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$].

In addition, there is an edge-to-face aromatic interaction (Jennings *et al.*, 2001) involving C10/H10 and the centroid of

DOI: 10.1107/S160053680401027X

the (C20–C25)^{vi} benzene ring; the H10···centroid distance is 2.74 Å and the C10—H10···centroid angle is 144° [symmetry code: (vi) $2 - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$]. A similar interaction exists involving C24/H24 and the centroid of the (C6–C11)^{vii} benzene ring; the H24···centroid distance is 3.05 Å and the C24—H24···centroid angle is 168° [symmetry code: (vii) $1 + x$, y, z].

The geometry around atom C1 is very slightly distorted from tetrahedral; the six C—C—C angles range from 107.47 (18) to 112.5 (2)°. As can be seen in Table 1, the torsion angles about C2—O1, C3—O2, C4—O3 and C5—O4 are different, and the differences are far larger than their uncertainties. These different conformations are further confirmed by the six N···N distances in the range 13.110 (4)–15.422 (4) Å, and the six O···O distances in the range 3.348 (2)–4.179 (2) Å.

Experimental

Under a nitrogen atmosphere, potassium 4-cyanophenoxide was prepared in methanol by reacting a mixture of KOH (82%; 2.0 g, 0.03 mol) and 4-cyanophenol (3.6 g, 0.03 mol) at 323 K for 1 h. The solvent was removed under reduced pressure at 323 K. The phenol salt was dissolved in dimethylformamide (60 ml), and the reaction mixture was then cooled in ice and stirred as $C(CH_2Br)_4$ (1.94 g, 0.005 mol) in dimethylformamide (30 ml) was added dropwise. The mixture was warmed to 398 K and left to react for 24 h. The mixture was then cooled to room temperature and poured into ice water (200 ml). The precipitate was filtered off, washed with water and ethanol, and purified by recrystallization from dimethylformamide, resulting in white crystals of (I) in 82% yield (m.p. 485–487 K). IR (KBr): ν 3105, 2935, 2892, 2223, 1604, 1508, 1463, 1420, 1302, 835, 716 cm^{-1}; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 4.41 (*s*, 8H), 7.14 (*d*, J = 8.91 Hz, 8H), 7.74 (*d*, J = 8.88 Hz, 8H).

Crystal data

$C_{33}H_{24}N_4O_4$
$M_r = 540.56$
Monoclinic, $P2_1/c$
$a = 11.2069$ (17) Å
$b = 12.703$ (2) Å
$c = 20.750$ (3) Å
$\beta = 101.003$ (2)°
$V = 2899.7$ (8) Å^3
$Z = 4$

$D_x = 1.238$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 2967 reflections
$\theta = 2.4$–22.2°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Parallelepiped, colorless
0.60 × 0.50 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SABABS*; Sheldrick, 1997*a*)
$T_{min} = 0.952$, $T_{max} = 0.976$
13 056 measured reflections

5683 independent reflections
3685 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 26.0°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 15$
$l = -15 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.145$
$S = 1.04$
5683 reflections
371 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.3945P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0011 (6)

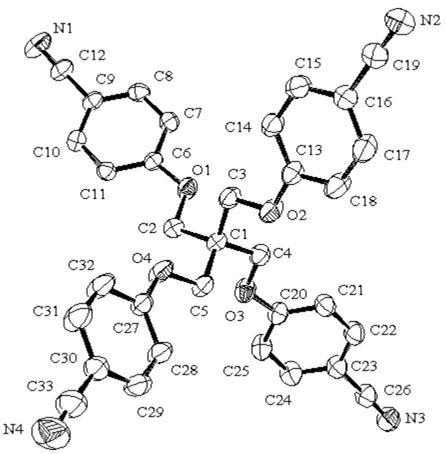


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Table 1
Selected torsion angles (°).

C1—C2—O1—C6	−158.68 (18)	C1—C4—O3—C20	−166.3 (2)
C1—C3—O2—C13	175.16 (19)	C1—C5—O4—C27	−173.9 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···N1^i	0.97	2.77	3.529 (3)	136
C11—H11···N4^{ii}	0.93	2.60	3.518 (4)	169
C28—H28···O1^{iii}	0.93	2.72	3.312 (3)	123
C28—H28···C6^{iii}	0.93	2.70	3.533 (4)	150

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

H atoms were included using a riding model, with C—H = 0.95 Å and $U_{iso} = 1.2U_{eq}$(C).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997*b*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support from the Ministry of Education, China, and through project No. 60171008 supported by the NSFC.

References

Bruker (1999). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Jennings, W. B., Farrell, B. M. & Malone, J. F. (2001). *Acc. Chem. Res.* **34**, 885–894.

Kumar, S., Subramanian, K., Srinivasan, R., Rajagopalan, K. & Steiner, T. (1998). *J. Mol. Struct.* **471**, 251–255.

Sheldrick, G. M. (1997*a*). *SADABS* and *SHELXS*97. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Xu, W., Lu, Y.-X., Guo, P., Zhou, H. & Lan, B.-J. (2004). *Acta Cryst.* E**60**, o428–o430.