

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,2',2'',2'''-[Pyridine-2,6-diylbis(methyl-enenitrilo)]tetraethanol

Shou-Ying Cao and Cheng-He Zhou*

School of Chemistry and Chemical Engineering, Southwest University, Chongqing, 400715, People's Republic of China

Correspondence e-mail: zhouch@swu.edu.cn

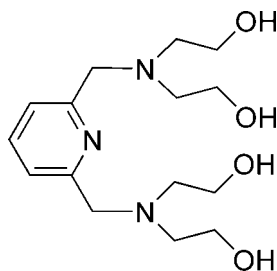
Received 10 March 2011; accepted 23 March 2011

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 14.5.

In the crystal structure of the title compound $\text{C}_{15}\text{H}_{27}\text{N}_3\text{O}_4$, the molecule is located on a twofold axis and the asymmetric unit contains one half-molecule, with one N and one C atom lying on the rotation axis. The pyridine ring is the hydrogen-bond acceptor, while two hydroxyl O atoms act as hydrogen-bond donors in intramolecular $\text{O}-\text{H}\cdots\text{N}$ and intermolecular $\text{O}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, thereby forming a closed hydrogen-bonded cage.

Related literature

For general background to pyridine, see: Klimesova *et al.* (1990); Rabasseda *et al.* (1999). For the synthesis, see: Fang *et al.* (2010).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{27}\text{N}_3\text{O}_4$ $M_r = 313.40$ Orthorhombic, $P2_12_12$ $a = 9.145$ (6) Å $b = 10.716$ (7) Å $c = 8.292$ (5) Å $V = 812.6$ (9) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 298$ K $0.45 \times 0.45 \times 0.45$ mm

Data collection

Bruker SMART APEX diffractometer
4239 measured reflections1511 independent reflections
1441 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$ $wR(F^2) = 0.098$ $S = 1.08$

1511 reflections

104 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2A}\cdots\text{O1}^1$	0.82	1.94	2.762 (2)	175
$\text{O1}-\text{H1A}\cdots\text{N2}^1$	0.82	2.74	3.248 (2)	122
$\text{O1}-\text{H1A}\cdots\text{N2}$	0.82	2.54	2.935 (2)	111
$\text{O1}-\text{H1A}\cdots\text{N1}$	0.82	2.25	3.004 (2)	153

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; 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*.

The authors thank Southwest University (SWUB2006018, XSGX0602 and SWUF2007023) and the Natural Science Foundation of Chongqing (2007BB5369) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2270).

References

- Bruker (2000). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Fang, B., Zhou, C. H. & Rao, X. C. (2010). *Eur. J. Med. Chem.* **45**, 4388–4398.
- Klimesova, V., Svoboda, M., Waissner, K., Pour, M. & Kaustova, J. (1999). *Farmaco*, **54**, 666–672.
- Rabasseda, X., Silverstre, J. & Castaner, J. (1999). *Drug Future*, **24**, 375–380.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2011). E67, o1003 [doi:10.1107/S1600536811010919]

2,2',2'',2'''-[Pyridine-2,6-diylbis(methylenenitrilo)]tetraethanol

S.-Y. Cao and C.-H. Zhou

Comment

Pyridine is an important type of nitrogen-containing aromatic heterocycle which is widely used as building block for preparing a variety of drugs, insecticides and herbicides in the pharmaceutical industry (Klimesova *et al.*, 1999; Rabasseda *et al.*, 1999). Recently, our researches have been focused on the development of aromatic ring-derived nitrogen mustards as potential antitumor agents. As part of our work, herein we report the molecular and crystal structures of the title compound as an intermediate for the pyridine-derived nitrogen mustards.

The molecular structure of the title compound is shown in Fig. 1. In the crystal structure of the title compound, $C_{15}H_{27}N_3O_4$, reveals that the molecule placed on two-fold axis (C1, H1 and N1 atoms placed on twofold axis) and asymmetric unit contains a half molecule. In the molecule, pyridine ring is the hydrogen-bond acceptor, and two hydroxyls which move closer to the pyridine ring *via* O—H \cdots N intramolecular hydrogen bond perform as hydrogen-bond donor, thus the closed hydrogen bond cage has been formed.

Experimental

The title compound was prepared according to the procedure of Fang *et al.* (2010). Single crystals were grown by slow evaporation of a solution of the title compound in the mixture of dichloromethane and *n*-hexane at room temperature.

Refinement

All H atoms were placed in idealized positions and treated as riding, with C—H = 0.93 Å (CH) or 0.97 Å (CH₂), and $U_{iso}(H) = 1.2U_{eq}(C)$; O—H = 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

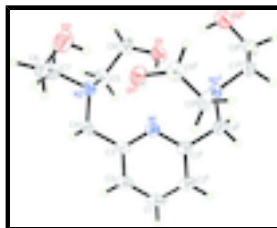


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

2,2',2'',2'''-[Pyridine-2,6-diylbis(methylenenitrilo)]tetraethanol

Crystal data

$C_{15}H_{27}N_3O_4$

$F(000) = 340$

supplementary materials

$M_r = 313.40$

Orthorhombic, $P2_12_12$

Hall symbol: P 2 2ab

$a = 9.145 (6) \text{ \AA}$

$b = 10.716 (7) \text{ \AA}$

$c = 8.292 (5) \text{ \AA}$

$V = 812.6 (9) \text{ \AA}^3$

$Z = 2$

$D_x = 1.281 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2637 reflections

$\theta = 2.5\text{--}27.1^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colourless

$0.45 \times 0.45 \times 0.45 \text{ mm}$

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ - and ω -scans

4239 measured reflections

1511 independent reflections

1441 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.5^\circ$

$h = -11 \rightarrow 8$

$k = -9 \rightarrow 12$

$l = -10 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.098$

$S = 1.08$

1511 reflections

104 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.1402P]$

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

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

$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Extinction correction: *SHELXL*,

$F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.105 (9)

Special details

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0000	1.0000	0.8502 (3)	0.0603 (7)
H1	0.0000	1.0000	0.9624	0.072*
C2	0.0769 (2)	0.91187 (18)	0.7667 (2)	0.0537 (5)
H2	0.1312	0.8520	0.8215	0.064*
C3	0.07333 (19)	0.91260 (16)	0.6004 (2)	0.0453 (4)
C4	0.1444 (2)	0.81022 (17)	0.5072 (2)	0.0564 (5)
H4A	0.2346	0.7873	0.5610	0.068*
H4B	0.0806	0.7379	0.5090	0.068*
C5	0.29756 (19)	0.93047 (18)	0.3315 (3)	0.0590 (5)
H5A	0.3889	0.8861	0.3158	0.071*
H5B	0.3040	0.9755	0.4328	0.071*
C6	0.2762 (2)	1.02106 (18)	0.1970 (2)	0.0567 (5)
H6A	0.3634	1.0719	0.1855	0.068*
H6B	0.2618	0.9757	0.0970	0.068*
C7	0.2098 (2)	0.72780 (17)	0.2461 (3)	0.0617 (5)
H7A	0.2738	0.6747	0.3093	0.074*
H7B	0.2628	0.7520	0.1497	0.074*
C8	0.0801 (3)	0.65392 (18)	0.1972 (3)	0.0671 (5)
H8A	0.1129	0.5729	0.1595	0.081*
H8B	0.0193	0.6403	0.2914	0.081*
N1	0.0000	1.0000	0.5180 (2)	0.0460 (5)
N2	0.17740 (14)	0.84064 (12)	0.34005 (17)	0.0443 (4)
O1	0.15472 (15)	1.09903 (12)	0.22517 (18)	0.0608 (4)
H1A	0.0944	1.0617	0.2802	0.091*
O2	-0.0051 (2)	0.70862 (16)	0.0769 (2)	0.0867 (5)
H2A	-0.0531	0.7659	0.1157	0.130*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0685 (17)	0.0814 (19)	0.0309 (11)	-0.0226 (16)	0.000	0.000
C2	0.0567 (10)	0.0636 (11)	0.0408 (9)	-0.0147 (9)	-0.0090 (8)	0.0123 (8)
C3	0.0449 (9)	0.0501 (9)	0.0410 (8)	-0.0046 (8)	-0.0020 (7)	0.0090 (7)
C4	0.0620 (11)	0.0540 (10)	0.0533 (11)	0.0119 (9)	-0.0016 (9)	0.0147 (8)
C5	0.0383 (9)	0.0654 (11)	0.0734 (12)	0.0017 (8)	-0.0028 (9)	0.0111 (10)
C6	0.0450 (9)	0.0621 (11)	0.0631 (11)	-0.0064 (8)	0.0069 (9)	0.0084 (10)
C7	0.0612 (11)	0.0542 (10)	0.0696 (12)	0.0205 (10)	0.0148 (10)	0.0044 (9)
C8	0.0867 (14)	0.0460 (10)	0.0687 (12)	0.0053 (10)	0.0155 (10)	-0.0064 (9)
N1	0.0536 (11)	0.0513 (11)	0.0333 (9)	0.0073 (10)	0.000	0.000
N2	0.0406 (7)	0.0433 (7)	0.0491 (8)	0.0064 (6)	0.0038 (6)	0.0053 (6)
O1	0.0550 (8)	0.0577 (7)	0.0697 (9)	0.0026 (6)	0.0056 (6)	0.0142 (7)
O2	0.0996 (12)	0.0797 (11)	0.0807 (10)	0.0185 (9)	-0.0140 (10)	-0.0251 (8)

supplementary materials

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

C1—C2 ⁱ	1.366 (2)	C6—O1	1.410 (2)
C1—C2	1.366 (2)	C6—H6A	0.9700
C1—H1	0.9300	C6—H6B	0.9700
C2—C3	1.380 (3)	C7—N2	1.469 (2)
C2—H2	0.9300	C7—C8	1.482 (3)
C3—N1	1.339 (2)	C7—H7A	0.9700
C3—C4	1.491 (3)	C7—H7B	0.9700
C4—N2	1.455 (2)	C8—O2	1.395 (3)
C4—H4A	0.9700	C8—H8A	0.9700
C4—H4B	0.9700	C8—H8B	0.9700
C5—N2	1.463 (2)	N1—C3 ⁱ	1.339 (2)
C5—C6	1.491 (3)	O1—H1A	0.8200
C5—H5A	0.9700	O2—H2A	0.8200
C5—H5B	0.9700		
C2 ⁱ —C1—C2	119.1 (2)	C5—C6—H6A	109.3
C2 ⁱ —C1—H1	120.5	O1—C6—H6B	109.3
C2—C1—H1	120.5	C5—C6—H6B	109.3
C1—C2—C3	119.4 (2)	H6A—C6—H6B	108.0
C1—C2—H2	120.3	N2—C7—C8	115.04 (16)
C3—C2—H2	120.3	N2—C7—H7A	108.5
N1—C3—C2	121.73 (18)	C8—C7—H7A	108.5
N1—C3—C4	117.91 (15)	N2—C7—H7B	108.5
C2—C3—C4	120.25 (18)	C8—C7—H7B	108.5
N2—C4—C3	114.79 (14)	H7A—C7—H7B	107.5
N2—C4—H4A	108.6	O2—C8—C7	114.72 (18)
C3—C4—H4A	108.6	O2—C8—H8A	108.6
N2—C4—H4B	108.6	C7—C8—H8A	108.6
C3—C4—H4B	108.6	O2—C8—H8B	108.6
H4A—C4—H4B	107.5	C7—C8—H8B	108.6
N2—C5—C6	111.48 (16)	H8A—C8—H8B	107.6
N2—C5—H5A	109.3	C3 ⁱ —N1—C3	118.6 (2)
C6—C5—H5A	109.3	C4—N2—C5	110.45 (16)
N2—C5—H5B	109.3	C4—N2—C7	111.27 (14)
C6—C5—H5B	109.3	C5—N2—C7	111.38 (15)
H5A—C5—H5B	108.0	C6—O1—H1A	109.5
O1—C6—C5	111.42 (17)	C8—O2—H2A	109.5
O1—C6—H6A	109.3		
C2 ⁱ —C1—C2—C3	-1.04 (13)	C4—C3—N1—C3 ⁱ	175.19 (18)
C1—C2—C3—N1	2.2 (3)	C3—C4—N2—C5	70.9 (2)
C1—C2—C3—C4	-174.02 (14)	C3—C4—N2—C7	-164.87 (16)
N1—C3—C4—N2	23.7 (2)	C6—C5—N2—C4	-143.71 (17)
C2—C3—C4—N2	-160.01 (17)	C6—C5—N2—C7	92.11 (19)
N2—C5—C6—O1	66.6 (2)	C8—C7—N2—C4	77.8 (2)
N2—C7—C8—O2	71.9 (2)	C8—C7—N2—C5	-158.52 (17)

C2—C3—N1—C3ⁱ -1.09 (13)
 Symmetry codes: (i) $-x, -y+2, z$.

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>
O2—H2A \cdots O1 ⁱ	0.82	1.94	2.762 (2)	175
O1—H1A \cdots N2 ⁱ	0.82	2.74	3.248 (2)	122
O1—H1A \cdots N2	0.82	2.54	2.935 (2)	111
O1—H1A \cdots N1	0.82	2.25	3.004 (2)	153

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

Fig. 1

