Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N,*N*,*N*′,*N*′-Tetrakis(2-hydroxyethyl)-terephthalamide

Zhi-Qiang Wang,[a]* Chen Xu,[a] Ying-Fei Li,[b] Fei-Fei Cen[b] and Yu-Qing Zhang[b]

[a]College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang 471022, People's Republic of China, and [b]Chemical Engineering and Pharmaceutics School, Henan University of Science and Technology, Luoyang 471003, People's Republic of China
Correspondence e-mail: wzq197811@sohu.com

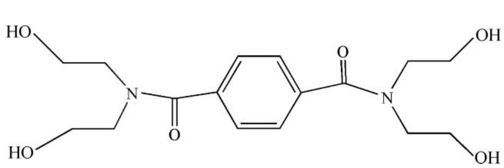
Received 1 November 2008; accepted 8 December 2008

Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.002 Å; disorder in main residue; *R* factor = 0.042; *wR* factor = 0.126; data-to-parameter ratio = 14.0.

The molecule of the title compound, $C_{16}H_{24}N_2O_6$, which lies on a crystallographic inversion centre in the centre of the benzene ring, adopts an *anti* conformation in terms of the relative orientation of two amide carbonyl groups. One pair of the 2-hydroxyethyl groups is partially disordered with site occupancy factors of 0.811 (2) and 0.189 (2). The dihedral angle between the amide group and central benzene ring is 67.0 (2)°. Two O—H···O and one bifurcated O—H···(O,O) hydrogen bonds are present, resulting in a three-dimensional network.

Related literature

For bond-length data, see: Allen *et al.* (1987). For general background, see: Katoono *et al.* (2006); Tosin *et al.* (2005); Yin *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{24}N_2O_6$
M_r = 340.37
Orthorhombic, *Pbca*
a = 10.3244 (12) Å
b = 12.5378 (14) Å
c = 12.8384 (15) Å
V = 1661.9 (3) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.29 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEXII detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.976
11505 measured reflections
1550 independent reflections
1273 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.126
S = 1.06
1550 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3^{i}	0.82	1.91	2.723 (9)	169
O2—H2*A*···O3′^{i}	0.82	1.90	2.675 (10)	157
O3′—H3′···O2^{ii}	0.82	2.31	2.675 (3)	108
O3—H3*D*···O1^{iii}	0.82	2.00	2.810 (2)	170

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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*.

This work was supported by the Doctoral Foundation of Luoyang Normal University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2054).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Katoono, R., Kawai, H., Fujiwara, K. & Suzuki, T. (2006). *Tetrahedron Lett.* **47**, 1513–1518.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tosin, M., Gouin, S. G. & Murphy, P. V. (2005). *Org. Lett.* **7**, 211–214.
Yin, H., Lee, G., Sedey, K. A., Rodriguez, J. M., Wang, H. G., Sebti, S. M. & Hamilton, A. D. (2005). *J. Am. Chem. Soc.* **127**, 5463–5468.

supplementary materials

Acta Cryst. (2009). E**65**, o166 [**doi:10.1107/S1600536808041573**]

N,*N*,*N'*,*N'*-Tetrakis(2-hydroxyethyl)terephthalamide

Z.-Q. Wang, C. Xu, Y.-F. Li, F.-F. Cen and Y.-Q. Zhang

Comment

Terephthalamide derivatives are important compounds in molecular recognition and supramolecular chemistry (Yin *et al.*, 2005; Tosin *et al.*,2005; Katoono *et al.*,2006). Although numerous tetrasubstituted terephthalamides have been investigated, only a few tetrakis(alkyl)terephthalamides are known. In order to further the study of such compounds, we report the crystal structure of the title compound.

A view of the molecular structure of the title compound is given in Fig.1. Molecules of the title compound lie across crystallographic inversion centres and adopt the anti-conformation. The bond distances and angles are normal (Allen *et al.*, 1987). One set of the 2-hydroxyethyl groups is disordered with site occupancy factors of *ca* 0.811 (2) and 0.189 (2). The dihedral angle between the amide plane (C4,O1,N1) and phenyl planes (C1—C3,C1A—C3A) is 67.0 (2)°. The structural study shows the presence of four different intermolecular O—H···O hydrogen bonds (Table 1), resulting in a three-dimensional supramolecular architecture (Fig. 2).

Experimental

To a solution of diethanolamine (2 mmol) in dry chloroform (5 ml), at 273 K, was added dropwise a solution of terephthalyl chloride (2 mmol) in dry chloroform (25 ml). Then, the mixture stirred at room temperature for 24hr, removal of solvent resulted in a yellow powder that was recrystallized from methanol-DMF solution at room temperature to give the desired product as colourless crystals suitable for single-crystal X-ray diffraction.

Refinement

H atoms attached to C atoms of the title compound were placed in geometrically idealized positions and treated as riding with C—H distances constrained to 0.93–0.97 Å, with *U*iso~(H) = 1.2 or 1.5 times *U*~eq~(C). H atoms bonded to O atoms were located in a difference map and refined independently with isotropic displacement parameters.

Figures

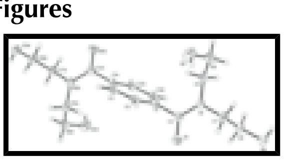


Fig. 1. The molecular structure of the title compound with displacement ellipsoids at the 30% probability level (suffix A denotes the symmetry code: -*x* + 2, -*y*, -*z* + 1).

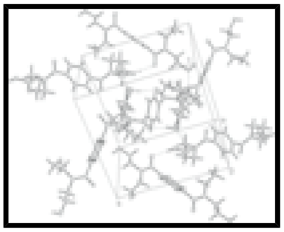


Fig. 2. Partial view of the crystal packing showing the intermolecular O—H···O hydrogen bonds.

N,*N*,*N*',*N*'-Tetrakis(2-hydroxyethyl)terephthalamide

Crystal data

$C_{16}H_{24}N_2O_6$	D_x = 1.360 Mg m^{-3}
M_r = 340.37	Mo *K*α radiation λ = 0.71073 Å
Orthorhombic, *Pbca*	Cell parameters from 3593 reflections
a = 10.3244 (12) Å	θ = 3.0–23.6º
b = 12.5378 (14) Å	μ = 0.10 mm^{-1}
c = 12.8384 (15) Å	*T* = 296 (2) K
V = 1661.9 (3) Å^3	Block, colourless
Z = 4	0.29 × 0.24 × 0.23 mm
F_{000} = 728	

Data collection

Bruker SMART APEXII detector diffractometer	1550 independent reflections
Radiation source: fine-focus sealed tube	1273 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 296(2) K	θ_{max} = 25.5º
phi and ω scans	θ_{min} = 3.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −12→12
T_{min} = 0.961, T_{max} = 0.976	*k* = −15→15
11505 measured reflections	*l* = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.6392P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ < 0.001
1550 reflections	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
111 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Primary atom site location: structure-invariant direct methods	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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C7	0.78238 (19)	0.31459 (17)	0.30476 (16)	0.0460 (6)	0.811 (2)
H7A	0.8162	0.3857	0.3168	0.055*	0.811 (2)
H7B	0.8130	0.2906	0.2373	0.055*	0.811 (2)
C8	0.63782 (13)	0.31829 (14)	0.30437 (14)	0.0498 (6)	0.811 (2)
H8A	0.6033	0.2481	0.2887	0.060*	0.811 (2)
H8B	0.6064	0.3396	0.3725	0.060*	0.811 (2)
O3	0.59529 (19)	0.39348 (12)	0.22709 (12)	0.0569 (6)	0.811 (2)
H3D	0.6203	0.4534	0.2429	0.085*	0.811 (2)
C7'	0.70527 (19)	0.28471 (18)	0.34485 (17)	0.0460 (6)	0.189 (2)
H7'1	0.6561	0.3216	0.3982	0.055*	0.189 (2)
H7'2	0.6523	0.2288	0.3147	0.055*	0.189 (2)
C8'	0.75511 (17)	0.36199 (17)	0.26151 (16)	0.0498 (6)	0.189 (2)
H8'1	0.8070	0.4170	0.2944	0.060*	0.189 (2)
H8'2	0.8102	0.3236	0.2131	0.060*	0.189 (2)
O3'	0.6539 (2)	0.40926 (16)	0.20721 (15)	0.0569 (6)	0.189 (2)
H3'	0.5952	0.3659	0.1996	0.085*	0.189 (2)
C1	0.93464 (15)	0.07120 (12)	0.43518 (12)	0.0362 (4)	
C2	1.06224 (16)	0.04283 (12)	0.41398 (13)	0.0392 (4)	

H2	1.1041	0.0714	0.3562	0.047*
C3	1.12701 (16)	−0.02751 (13)	0.47837 (14)	0.0405 (4)
H3	1.2124	−0.0458	0.4638	0.049*
C4	0.86624 (17)	0.14222 (13)	0.35884 (14)	0.0436 (4)
C5	0.84524 (17)	0.28318 (13)	0.49384 (14)	0.0440 (4)
H5A	0.8715	0.2257	0.5398	0.053*
H5B	0.7615	0.3087	0.5175	0.053*
C6	0.94175 (18)	0.37226 (15)	0.50233 (16)	0.0492 (5)
H6A	0.9164	0.4296	0.4559	0.059*
H6B	0.9410	0.3999	0.5729	0.059*
N1	0.83155 (14)	0.24090 (12)	0.38745 (12)	0.0496 (4)
O1	0.84678 (17)	0.10814 (11)	0.26939 (11)	0.0669 (5)
O2	1.06824 (13)	0.33898 (13)	0.47707 (12)	0.0645 (5)
H2A	1.0808	0.3474	0.4145	0.097*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0484 (13)	0.0439 (12)	0.0455 (12)	0.0069 (9)	−0.0011 (9)	0.0099 (9)
C8	0.0524 (13)	0.0459 (12)	0.0510 (13)	0.0052 (10)	−0.0096 (10)	0.0017 (10)
O3	0.0677 (14)	0.0500 (9)	0.0530 (11)	0.0135 (9)	−0.0240 (9)	−0.0022 (7)
C7'	0.0484 (13)	0.0439 (12)	0.0455 (12)	0.0069 (9)	−0.0011 (9)	0.0099 (9)
C8'	0.0524 (13)	0.0459 (12)	0.0510 (13)	0.0052 (10)	−0.0096 (10)	0.0017 (10)
O3'	0.0677 (14)	0.0500 (9)	0.0530 (11)	0.0135 (9)	−0.0240 (9)	−0.0022 (7)
C1	0.0407 (9)	0.0283 (8)	0.0397 (9)	0.0003 (6)	−0.0054 (7)	−0.0037 (7)
C2	0.0428 (9)	0.0343 (8)	0.0405 (9)	−0.0014 (7)	0.0026 (7)	0.0014 (7)
C3	0.0340 (8)	0.0362 (8)	0.0514 (10)	0.0022 (7)	0.0006 (7)	−0.0013 (7)
C4	0.0482 (10)	0.0382 (9)	0.0445 (9)	0.0042 (7)	−0.0087 (8)	−0.0018 (7)
C5	0.0449 (9)	0.0377 (9)	0.0494 (10)	0.0065 (7)	0.0000 (7)	−0.0032 (8)
C6	0.0548 (11)	0.0423 (10)	0.0505 (10)	−0.0013 (8)	0.0010 (9)	−0.0028 (8)
N1	0.0605 (10)	0.0382 (8)	0.0500 (9)	0.0135 (7)	−0.0178 (7)	−0.0031 (7)
O1	0.1004 (12)	0.0534 (8)	0.0468 (8)	0.0175 (7)	−0.0234 (8)	−0.0089 (6)
O2	0.0476 (8)	0.0816 (11)	0.0641 (9)	−0.0001 (7)	0.0030 (7)	0.0111 (8)

Geometric parameters (Å, °)

C7—C8	1.4933 (14)	C1—C3^{i}	1.392 (2)
C7—N1	1.496 (2)	C1—C4	1.501 (2)
C7—H7A	0.9700	C2—C3	1.382 (2)
C7—H7B	0.9700	C2—H2	0.9300
C8—O3	1.4372 (14)	C3—C1^{i}	1.392 (2)
C8—H8A	0.9700	C3—H3	0.9300
C8—H8B	0.9700	C4—O1	1.242 (2)
O3—H3D	0.8200	C4—N1	1.339 (2)
C7'—N1	1.517 (2)	C5—N1	1.472 (2)
C7'—C8'	1.5324 (15)	C5—C6	1.501 (3)
C7'—H7'1	0.9700	C5—H5A	0.9700
C7'—H7'2	0.9700	C5—H5B	0.9700

C8'—O3'	1.3890 (13)	C6—O2	1.409 (2)
C8'—H8'1	0.9700	C6—H6A	0.9700
C8'—H8'2	0.9700	C6—H6B	0.9700
O3'—H3'	0.8200	O2—H2A	0.8200
C1—C2	1.391 (2)		

C8—C7—N1	111.14 (14)	C3—C2—C1	120.30 (16)
C8—C7—H7A	109.4	C3—C2—H2	119.8
N1—C7—H7A	109.4	C1—C2—H2	119.8
C8—C7—H7B	109.4	C2—C3—C1^{i}	120.47 (15)
N1—C7—H7B	109.4	C2—C3—H3	119.8
H7A—C7—H7B	108.0	C1^{i}—C3—H3	119.8
O3—C8—C7	109.1	O1—C4—N1	121.89 (16)
O3—C8—H8A	109.9	O1—C4—C1	118.42 (15)
C7—C8—H8A	109.9	N1—C4—C1	119.66 (15)
O3—C8—H8B	109.9	N1—C5—C6	113.53 (15)
C7—C8—H8B	109.9	N1—C5—H5A	108.9
H8A—C8—H8B	108.3	C6—C5—H5A	108.9
N1—C7'—C8'	101.08 (14)	N1—C5—H5B	108.9
N1—C7'—H7'1	111.6	C6—C5—H5B	108.9
C8'—C7'—H7'1	111.6	H5A—C5—H5B	107.7
N1—C7'—H7'2	111.6	O2—C6—C5	112.23 (15)
C8'—C7'—H7'2	111.6	O2—C6—H6A	109.2
H7'1—C7'—H7'2	109.4	C5—C6—H6A	109.2
O3'—C8'—C7'	111.6	O2—C6—H6B	109.2
O3'—C8'—H8'1	109.3	C5—C6—H6B	109.2
C7'—C8'—H8'1	109.3	H6A—C6—H6B	107.9
O3'—C8'—H8'2	109.3	C4—N1—C5	124.16 (14)
C7'—C8'—H8'2	109.3	C4—N1—C7	117.81 (15)
H8'1—C8'—H8'2	108.0	C5—N1—C7	117.94 (14)
C8'—O3'—H3'	109.5	C4—N1—C7'	117.74 (16)
C2—C1—C3^{i}	119.23 (15)	C5—N1—C7'	106.66 (15)
C2—C1—C4	118.00 (15)	C7—N1—C7'	39.54 (8)
C3^{i}—C1—C4	122.62 (15)	C6—O2—H2A	109.5

N1—C7—C8—O3	177.43 (15)	C1—C4—N1—C7	170.75 (15)
N1—C7'—C8'—O3'	177.19 (14)	O1—C4—N1—C7'	37.8 (3)
C3^{i}—C1—C2—C3	−0.3 (3)	C1—C4—N1—C7'	−144.29 (16)
C4—C1—C2—C3	−176.06 (15)	C6—C5—N1—C4	113.8 (2)
C1—C2—C3—C1^{i}	0.3 (3)	C6—C5—N1—C7	−62.8 (2)
C2—C1—C4—O1	64.1 (2)	C6—C5—N1—C7'	−104.03 (17)
C3^{i}—C1—C4—O1	−111.5 (2)	C8—C7—N1—C4	98.8 (2)
C2—C1—C4—N1	−113.88 (19)	C8—C7—N1—C5	−84.3 (2)
C3^{i}—C1—C4—N1	70.5 (2)	C8—C7—N1—C7'	−1.98 (10)
N1—C5—C6—O2	−63.2 (2)	C8'—C7'—N1—C4	−104.2 (2)
O1—C4—N1—C5	176.24 (18)	C8'—C7'—N1—C5	110.73 (19)
C1—C4—N1—C5	−5.8 (3)	C8'—C7'—N1—C7	−3.21 (8)
O1—C4—N1—C7	−7.2 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O3^{ii}	0.82	1.91	2.723 (9)	169
O2—H2A···O3'^{ii}	0.82	1.90	2.675 (10)	157
O3'—H3'···O2^{iii}	0.82	2.31	2.675 (3)	108
O3—H3D···O1^{iv}	0.82	2.00	2.810 (2)	170

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

Fig. 1

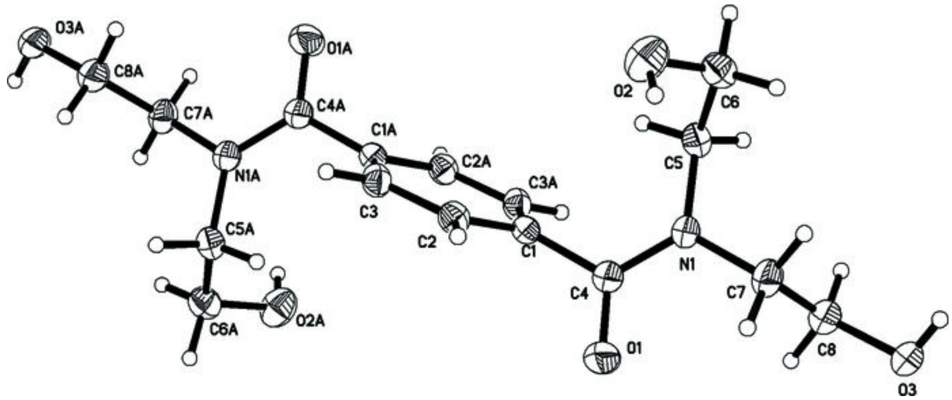

O3A
C8A
C7A
N1A
C5A
C6A
O2A
O1A
C4A
C1A
C2A
C3
C2
C3A
C1
C4
O1
N1
O2
C6
C5
C7
C8
O3

Fig. 2