Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N,*N*′-Bis(2-cyanoethyl)-4,4′-dimethyl-*N*,*N*′-(butane-1,4-diyl)dibenzenesulfonamide

Peng-Fei Cheng, Chao-Jie Wang and Yu-Xia Wang*

College of Chemistry and Chemical Engineering, Henan University, Kaifeng 475004, People's Republic of China
Correspondence e-mail: hgxywyx@163.com

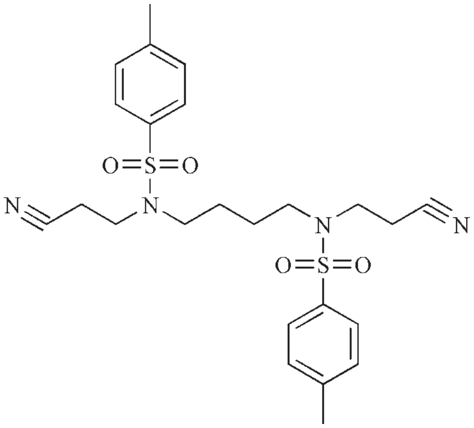
Received 6 September 2009; accepted 6 September 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.104; data-to-parameter ratio = 16.2.

The complete molecule of the title compound, $C_{24}H_{30}N_4O_4S_2$, is generated by a crystallographic inversion centre. In the crystal, weak C—H⋯O interactions link the molecules, forming infinite sheets.

Related literature

For background to polyamines, see: Thomas & Thomas (2003).



Experimental

Crystal data

$C_{24}H_{30}N_4O_4S_2$
M_r = 502.64
Monoclinic, $P2_1/c$
a = 16.688 (13) Å
b = 5.786 (5) Å
c = 13.675 (11) Å
β = 104.005 (13)°
V = 1281.3 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 296 K
0.34 × 0.26 × 0.21 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001) T_{min} = 0.921, T_{max} = 0.950
6684 measured reflections
2510 independent reflections
2114 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.104
S = 1.05
2510 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C5—H5A⋯O2^i	0.93	2.54	3.271 (4)	136

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5089).

References

Bruker (2001). *SAINT-Plus* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Thomas, T. & Thomas, T. J. (2003). *J. Cell. Mol. Med.* **7**, 113–126.

supplementary materials

Acta Cryst. (2009). E**65**, o2433 [**doi:10.1107/S1600536809036022**]

N,*N*'-Bis(2-cyanoethyl)-4,4'-dimethyl-*N*,*N*'-(butane-1,4-diyl)dibenzenesulfonamide

P.-F. Cheng, C.-J. Wang and Y.-X. Wang

Comment

Polyamines are natural products and have interesting biological activities. It present in the majority of cells. They play important roles in the synthesis of proteins, cell division, and bind to nucleic acids resulting in their condensation, thereby affecting gene expression. These effects might have implications in cancer treatment (e.g. Thomas & Thomas, 2003). We now report the crystal structure of the title compound, (I).

As shown in Fig.1, the title compound consists of two 4-methylbenzenesulfonyl groups anchoring to polyamine chain. In the structure of (I), the two phenyl ring of two 4-methylbenzenesulfonyl groups are antiparallel by symmetry.

In the crystal, the molecules are linked into infinite sheets by intermolecular C–H···O hydrogen bonds (Fig. 2).

Experimental

To a solution of 1,4-diaminobutane (8.8 g, 0.1 mol) in MeOH (300 ml), acrylonitrile (11.66 g, 0.22 mol) was added dropwise at room temperature during 1 h. After stirring for additional 7 h, the solvent was evaporated. The residue was fractionated in vacuum, yielding 17.27 g (89%) of *N*,*N*'-bis(2-cyanoethyl)-1,4-diaminobutane.

To a mixture of *N*,*N*'-bis(2-cyanoethyl)-1,4-diaminobutane (17.27 g, 89 mmol) and Et_3N (17.98 g, 178 mmol) in THF (120 ml), a solution of 4-methylbenzenesulfonyl chloride (TsCl, 34.02 g, 178 mmol) in THF (120 ml) was added dropwise at room temperature. The precipitate was filtered off and discarded. The filtrate was subsequently washed with 4 mol/*L* NaOH (in order to hydrolyze any unreacted TsCl) and NaCl solution. Evaporation gave the target compound (I) as colourless rods (43.34 g, 97%).

Refinement

The H atoms were geometrically placed (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Figures

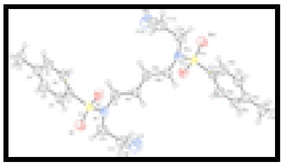


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Atoms with the suffix a are generated by (–x, 1–y, 1–z).

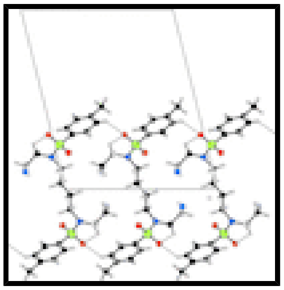


Fig. 2. One-dimensional structure of (I) along *c* axis, Hydrogen bonds are shown as dashed lines.

N,*N*'-Bis(2-cyanoethyl)-4,4'-dimethyl-*N*,*N*'- (butane-1,4-diyl)dibenzenesulfonamide

Crystal data

$C_{24}H_{30}N_4O_4S_2$

$M_r = 502.64$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 16.688$ (13) Å

$b = 5.786$ (5) Å

$c = 13.675$ (11) Å

$\beta = 104.005$ (13)°

$V = 1281.3$ (17) Å^3

$Z = 2$

$F_{000} = 532$

$D_x = 1.303$ Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2757 reflections

θ = 3.0–27.6°

$\mu = 0.25$ mm^{-1}

$T = 296$ K

Rod, colorless

0.34 × 0.26 × 0.21 mm

Data collection

Bruker SMART CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296$ K

ω scans

Absorption correction: Multi-scan (SADABS; Sheldrick, 2001)

$T_{min} = 0.921$, $T_{max} = 0.950$

6684 measured reflections

2510 independent reflections

2114 reflections with $I > 2\sigma(I)$

$R_{int} = 0.019$

$\theta_{max} = 26.0$°

$\theta_{min} = 2.5$°

$h = -16\rightarrow20$

$k = -7\rightarrow7$

$l = -16\rightarrow15$

Refinement

Refinement on F^2

Least-squares matrix: full

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

$wR(F^2) = 0.104$

$S = 1.05$

2510 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.2525P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.20$ e Å^{-3}

155 parameters	$\Delta\rho_{\min} = -0.28$ 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*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.24666 (2)	0.32320 (7)	0.53380 (3)	0.03878 (15)
O1	0.20256 (7)	0.1573 (2)	0.57929 (10)	0.0506 (3)
O2	0.30224 (8)	0.2415 (2)	0.47571 (10)	0.0558 (3)
N1	0.17969 (8)	0.4914 (2)	0.46127 (10)	0.0392 (3)
N2	0.09678 (12)	0.0954 (3)	0.26259 (14)	0.0718 (5)
C1	0.30449 (9)	0.4988 (3)	0.63262 (12)	0.0371 (4)
C2	0.33821 (10)	0.7065 (3)	0.61032 (13)	0.0469 (4)
H2A	0.3275	0.7592	0.5442	0.056*
C3	0.38797 (11)	0.8344 (3)	0.68757 (15)	0.0532 (5)
H3A	0.4108	0.9727	0.6725	0.064*
C4	0.40431 (10)	0.7595 (4)	0.78710 (14)	0.0496 (4)
C5	0.36879 (11)	0.5538 (4)	0.80728 (13)	0.0541 (5)
H5A	0.3787	0.5020	0.8735	0.065*
C6	0.31883 (11)	0.4232 (3)	0.73143 (13)	0.0498 (4)
H6A	0.2952	0.2865	0.7468	0.060*
C7	0.45863 (13)	0.8996 (4)	0.87086 (16)	0.0720 (6)
H7A	0.4839	1.0233	0.8423	0.108*
H7B	0.5008	0.8020	0.9103	0.108*
H7C	0.4257	0.9625	0.9131	0.108*
C8	0.03371 (9)	0.4433 (3)	0.47933 (13)	0.0450 (4)
H8A	0.0144	0.4254	0.4069	0.054*
H8B	0.0455	0.2908	0.5087	0.054*
C9	0.11282 (9)	0.5880 (3)	0.50313 (12)	0.0429 (4)
H9A	0.0999	0.7425	0.4765	0.052*
H9B	0.1326	0.6008	0.5757	0.052*
C10	0.19632 (11)	0.5953 (3)	0.37044 (13)	0.0469 (4)
H10A	0.2493	0.5407	0.3622	0.056*
H10B	0.1997	0.7618	0.3785	0.056*
C11	0.12903 (12)	0.5356 (3)	0.27550 (12)	0.0497 (4)
H11A	0.0788	0.6183	0.2772	0.060*

H11B	0.1466	0.5868	0.2164	0.060*
C12	0.11145 (12)	0.2864 (4)	0.26701 (13)	0.0499 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0360 (2)	0.0370 (3)	0.0428 (2)	0.00198 (15)	0.00858 (17)	−0.00311 (16)
O1	0.0517 (7)	0.0410 (7)	0.0564 (7)	−0.0076 (5)	0.0077 (6)	0.0045 (5)
O2	0.0506 (7)	0.0601 (8)	0.0597 (7)	0.0117 (6)	0.0191 (6)	−0.0116 (7)
N1	0.0341 (7)	0.0462 (8)	0.0371 (7)	0.0025 (6)	0.0086 (5)	−0.0004 (6)
N2	0.0825 (13)	0.0529 (11)	0.0736 (12)	−0.0051 (10)	0.0062 (10)	−0.0087 (9)
C1	0.0296 (7)	0.0394 (9)	0.0420 (8)	0.0018 (6)	0.0080 (6)	−0.0010 (7)
C2	0.0475 (9)	0.0444 (10)	0.0470 (9)	−0.0020 (8)	0.0075 (8)	0.0057 (8)
C3	0.0520 (10)	0.0429 (10)	0.0627 (12)	−0.0099 (8)	0.0100 (9)	−0.0004 (8)
C4	0.0377 (9)	0.0570 (11)	0.0531 (10)	−0.0020 (8)	0.0092 (8)	−0.0103 (9)
C5	0.0539 (10)	0.0667 (13)	0.0400 (9)	−0.0098 (9)	0.0078 (8)	−0.0006 (9)
C6	0.0490 (10)	0.0542 (11)	0.0464 (9)	−0.0109 (8)	0.0118 (8)	0.0035 (8)
C7	0.0594 (12)	0.0813 (16)	0.0688 (14)	−0.0156 (11)	0.0033 (10)	−0.0215 (12)
C8	0.0369 (8)	0.0532 (11)	0.0440 (9)	0.0028 (8)	0.0078 (7)	−0.0079 (8)
C9	0.0371 (8)	0.0465 (10)	0.0441 (9)	0.0045 (7)	0.0077 (7)	−0.0060 (8)
C10	0.0493 (10)	0.0462 (10)	0.0465 (9)	−0.0050 (8)	0.0140 (8)	0.0028 (8)
C11	0.0633 (11)	0.0454 (10)	0.0388 (9)	0.0042 (8)	0.0092 (8)	0.0056 (7)
C12	0.0549 (11)	0.0527 (12)	0.0395 (9)	0.0032 (9)	0.0062 (8)	−0.0036 (8)

Geometric parameters (Å, °)

S1—O1	1.4392 (14)	C6—H6A	0.9300
S1—O2	1.4393 (14)	C7—H7A	0.9600
S1—N1	1.6259 (15)	C7—H7B	0.9600
S1—C1	1.7772 (18)	C7—H7C	0.9600
N1—C10	1.466 (2)	C8—C8^{i}	1.524 (3)
N1—C9	1.481 (2)	C8—C9	1.530 (2)
N2—C12	1.130 (3)	C8—H8A	0.9700
C1—C6	1.385 (2)	C8—H8B	0.9700
C1—C2	1.392 (2)	C9—H9A	0.9700
C2—C3	1.389 (3)	C9—H9B	0.9700
C2—H2A	0.9300	C10—C11	1.536 (2)
C3—C4	1.391 (3)	C10—H10A	0.9700
C3—H3A	0.9300	C10—H10B	0.9700
C4—C5	1.387 (3)	C11—C12	1.471 (3)
C4—C7	1.513 (3)	C11—H11A	0.9700
C5—C6	1.387 (3)	C11—H11B	0.9700
C5—H5A	0.9300		
O1—S1—O2	118.99 (9)	H7A—C7—H7B	109.5
O1—S1—N1	108.36 (9)	C4—C7—H7C	109.5
O2—S1—N1	107.44 (9)	H7A—C7—H7C	109.5
O1—S1—C1	107.02 (9)	H7B—C7—H7C	109.5
O2—S1—C1	107.68 (9)	C8^{i}—C8—C9	111.23 (18)

N1—S1—C1	106.75 (9)	C8^{i}—C8—H8A	109.4
C10—N1—C9	119.19 (14)	C9—C8—H8A	109.4
C10—N1—S1	121.28 (12)	C8^{i}—C8—H8B	109.4
C9—N1—S1	117.45 (11)	C9—C8—H8B	109.4
C6—C1—C2	120.19 (16)	H8A—C8—H8B	108.0
C6—C1—S1	119.69 (14)	N1—C9—C8	113.80 (14)
C2—C1—S1	120.06 (13)	N1—C9—H9A	108.8
C3—C2—C1	119.52 (17)	C8—C9—H9A	108.8
C3—C2—H2A	120.2	N1—C9—H9B	108.8
C1—C2—H2A	120.2	C8—C9—H9B	108.8
C2—C3—C4	121.16 (18)	H9A—C9—H9B	107.7
C2—C3—H3A	119.4	N1—C10—C11	111.99 (15)
C4—C3—H3A	119.4	N1—C10—H10A	109.2
C5—C4—C3	118.05 (17)	C11—C10—H10A	109.2
C5—C4—C7	121.07 (18)	N1—C10—H10B	109.2
C3—C4—C7	120.88 (19)	C11—C10—H10B	109.2
C4—C5—C6	121.83 (17)	H10A—C10—H10B	107.9
C4—C5—H5A	119.1	C12—C11—C10	112.22 (15)
C6—C5—H5A	119.1	C12—C11—H11A	109.2
C1—C6—C5	119.23 (18)	C10—C11—H11A	109.2
C1—C6—H6A	120.4	C12—C11—H11B	109.2
C5—C6—H6A	120.4	C10—C11—H11B	109.2
C4—C7—H7A	109.5	H11A—C11—H11B	107.9
C4—C7—H7B	109.5	N2—C12—C11	178.0 (2)

O1—S1—N1—C10	−149.35 (13)	C1—C2—C3—C4	−0.4 (3)
O2—S1—N1—C10	−19.60 (15)	C2—C3—C4—C5	−0.6 (3)
C1—S1—N1—C10	95.68 (14)	C2—C3—C4—C7	179.95 (17)
O1—S1—N1—C9	47.17 (14)	C3—C4—C5—C6	0.5 (3)
O2—S1—N1—C9	176.93 (11)	C7—C4—C5—C6	179.98 (18)
C1—S1—N1—C9	−67.79 (13)	C2—C1—C6—C5	−1.6 (3)
O1—S1—C1—C6	17.62 (16)	S1—C1—C6—C5	175.56 (13)
O2—S1—C1—C6	−111.40 (15)	C4—C5—C6—C1	0.6 (3)
N1—S1—C1—C6	133.48 (14)	C10—N1—C9—C8	102.87 (18)
O1—S1—C1—C2	−165.17 (13)	S1—N1—C9—C8	−93.29 (15)
O2—S1—C1—C2	65.80 (15)	C8^{i}—C8—C9—N1	−177.68 (17)
N1—S1—C1—C2	−49.32 (15)	C9—N1—C10—C11	−73.0 (2)
C6—C1—C2—C3	1.6 (3)	S1—N1—C10—C11	123.77 (15)
S1—C1—C2—C3	−175.62 (13)	N1—C10—C11—C12	−50.0 (2)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···O2^{ii}	0.93	2.54	3.271 (4)	136

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

Fig. 1

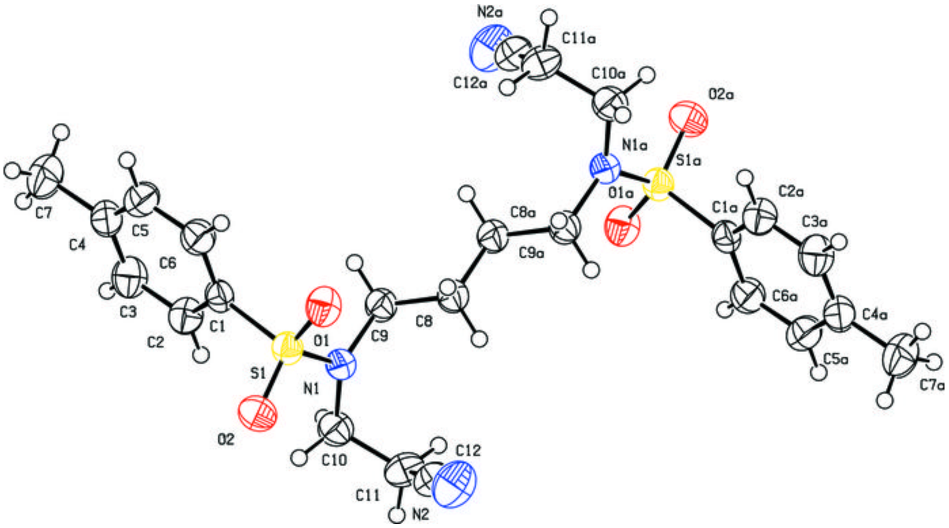

N2a
C11a
C10a
C12a
O2a
N1a
S1a
O1a
C1a
C2a
C3a
C8a
C9a
C6a
C4a
C5a
C7a
C7
C4
C5
C6
C3
C2
C1
O1
C9
C8
S1
N1
O2
C10
C11
C12
N2

Fig. 2