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[Butane-1,4-diylbis(nitrilodiethylidene)]-tetraacetonitrile

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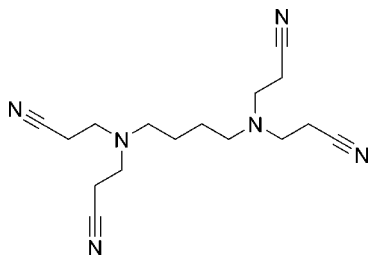
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.037; wR factor = 0.092; data-to-parameter ratio = 20.3.

In the centrosymmetric title compound, $\text{C}_{16}\text{H}_{24}\text{N}_6$, close intermolecular $\text{C}-\text{H}\cdots\text{N}$ interactions are observed. All bond distances and angles are unremarkable.

Related literature

For related literature on dendrimers, see: Meijboom *et al.* (2003); Meijboom, Hutton & Moss (2004); Meijboom, Moss *et al.* (2004); Harder *et al.* (2004); Overett *et al.* (2005); Blom *et al.* (2005). For the synthesis of the current dendritic starting material, see: De Brabander-van den Berg & Meijer (1994).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{24}\text{N}_6$
 $M_r = 300.41$
 Monoclinic, $P2_1/c$
 $a = 5.1149$ (1) Å
 $b = 11.4001$ (3) Å
 $c = 14.4864$ (4) Å
 $\beta = 98.970$ (1)°

$V = 834.38$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 100$ (2) K
 $0.24 \times 0.17 \times 0.15$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.982$, $T_{\max} = 0.989$

8480 measured reflections
 2034 independent reflections
 1753 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.092$
 $S = 1.05$
 2034 reflections

100 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4A}\cdots\text{N2}^i$	0.99	2.47	3.4546 (13)	170
$\text{C7}-\text{H7B}\cdots\text{N3}^i$	0.99	2.57	3.5561 (14)	175

Symmetry code: (i) $x + 1, y, z$.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT-Plus (Bruker, 2004); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2046).

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supplementary materials

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[Butane-1,4-diylbis(nitrilodiethylidene)]tetraacetonitrile

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Comment

Dendrimers are a unique class of synthetic polymers in which growth emanates from a central core molecule. In the divergent synthesis method, polymer growth emanates in an outward direction from the initiator core by a series of stepwise polymerization reactions that attach layers to form the final tree-like structure.

From our interest in organometallic dendrimers (Meijboom *et al.*, 2003; Meijboom, Hutton & Moss, 2004; Harder *et al.*, 2004; Meijboom, Moss *et al.*, 2004; Overett *et al.*, 2005; Blom *et al.*, 2005), we decided to synthesize the well known poly-propylene amine dendrimers (De Brabander-van den Berg & Meijer, 1994).

Simple Michael addition of acrylonitrile to the primary amine groups of butylenediamine afforded compound (I) in quantitative yield.

Experimental

The compound was synthesized according to previously reported procedures. The analytical data was identical to those previously reported (De Brabander-van den Berg & Meijer, 1994).

Refinement

H atoms were positioned geometrically and refined using a riding model, with fixed C—H distances of 0.99 Å [$U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$]. The highest residual peak is 0.30 e located 0.76 Å from atom C1 and the deepest hole -0.18 e, 0.62 Å from C4. A minor disorder on C4 and C6 afforded a Hirshfeld warning. Refining the structure disordered gave a disorder on C4 of 99.3/0.7% and a disorder on C6 of 99.4/0.6%. It was decided that the disorder was too small to be significant and the molecule was refined without these disorders.

Figures

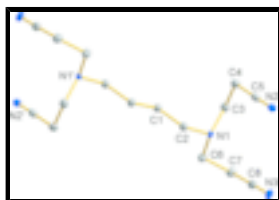


Fig. 1. The structure (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. Primed atoms were generated by symmetry.

[Butane-1,4-diylbis(nitrilodiethylidene)]tetraacetonitrile

Crystal data

$C_{16}H_{24}N_6$	$F_{000} = 324$
$M_r = 300.41$	$D_x = 1.196 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 5.1149 (1) \text{ \AA}$	Cell parameters from 2763 reflections
$b = 11.4001 (3) \text{ \AA}$	$\theta = 2.9\text{--}28.1^\circ$
$c = 14.4864 (4) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\beta = 98.970 (1)^\circ$	$T = 100 (2) \text{ K}$
$V = 834.38 (4) \text{ \AA}^3$	Cuboid, colourless
$Z = 2$	$0.24 \times 0.17 \times 0.15 \text{ mm}$

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	2034 independent reflections
Monochromator: graphite	1753 reflections with $I > 2\sigma(I)$
Detector resolution: $8.6 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.031$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 28.3^\circ$
φ scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -6 \rightarrow 4$
$T_{\text{min}} = 0.982, T_{\text{max}} = 0.989$	$k = -14 \rightarrow 14$
8480 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 0.2519P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.092$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
2034 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
100 parameters	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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.53485 (18)	0.46941 (9)	0.45673 (6)	0.0140 (2)
H1A	0.7011	0.4245	0.4738	0.017*
H1B	0.5641	0.5289	0.4096	0.017*
C2	0.31330 (18)	0.38621 (9)	0.41476 (7)	0.0137 (2)
H2A	0.2869	0.328	0.4632	0.016*
H2B	0.1479	0.4324	0.4006	0.016*
C3	0.59406 (19)	0.25324 (9)	0.33950 (7)	0.0152 (2)
H3A	0.7476	0.3071	0.3505	0.018*
H3B	0.6016	0.2108	0.2804	0.018*
C4	0.61514 (19)	0.16494 (9)	0.41946 (7)	0.0168 (2)
H4A	0.7768	0.1171	0.4199	0.02*
H4B	0.6321	0.2077	0.4796	0.02*
C5	0.38407 (19)	0.08709 (9)	0.41123 (7)	0.0163 (2)
C6	0.3261 (2)	0.39962 (9)	0.24859 (7)	0.0170 (2)
H6A	0.4889	0.4474	0.252	0.02*
H6B	0.1756	0.4538	0.2502	0.02*
C7	0.2825 (2)	0.33178 (9)	0.15604 (7)	0.0174 (2)
H7A	0.2466	0.3881	0.1036	0.021*
H7B	0.4463	0.2885	0.1493	0.021*
C8	0.06207 (19)	0.24867 (9)	0.15039 (7)	0.0168 (2)
N1	0.35032 (15)	0.32200 (7)	0.32974 (5)	0.01323 (19)
N2	0.20659 (18)	0.02511 (8)	0.40503 (6)	0.0219 (2)
N3	-0.11103 (19)	0.18428 (9)	0.14412 (6)	0.0253 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0134 (4)	0.0145 (5)	0.0138 (5)	-0.0001 (4)	0.0015 (3)	-0.0019 (4)
C2	0.0137 (4)	0.0140 (5)	0.0136 (4)	-0.0002 (4)	0.0026 (3)	-0.0017 (3)
C3	0.0145 (4)	0.0156 (5)	0.0160 (5)	-0.0007 (4)	0.0038 (3)	-0.0013 (4)
C4	0.0150 (4)	0.0174 (5)	0.0173 (5)	0.0017 (4)	0.0000 (3)	-0.0004 (4)
C5	0.0189 (5)	0.0158 (5)	0.0142 (5)	0.0049 (4)	0.0025 (3)	0.0017 (4)
C6	0.0216 (5)	0.0148 (5)	0.0138 (5)	-0.0032 (4)	0.0002 (4)	0.0012 (4)
C7	0.0182 (5)	0.0207 (6)	0.0129 (5)	-0.0019 (4)	0.0018 (3)	0.0012 (4)
C8	0.0188 (5)	0.0190 (6)	0.0123 (4)	0.0032 (4)	0.0014 (3)	-0.0026 (4)
N1	0.0150 (4)	0.0131 (4)	0.0112 (4)	0.0002 (3)	0.0011 (3)	-0.0010 (3)
N2	0.0208 (4)	0.0199 (5)	0.0249 (5)	0.0008 (4)	0.0034 (3)	0.0031 (4)
N3	0.0254 (5)	0.0281 (6)	0.0222 (5)	-0.0059 (4)	0.0032 (4)	-0.0078 (4)

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

C1—C1 ⁱ	1.5246 (18)	C4—H4A	0.99
C1—C2	1.5292 (13)	C4—H4B	0.99
C1—H1A	0.99	C5—N2	1.1428 (14)

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C1—H1B	0.99	C6—N1	1.4611 (12)
C2—N1	1.4702 (12)	C6—C7	1.5336 (14)
C2—H2A	0.99	C6—H6A	0.99
C2—H2B	0.99	C6—H6B	0.99
C3—N1	1.4607 (12)	C7—C8	1.4654 (14)
C3—C4	1.5257 (14)	C7—H7A	0.99
C3—H3A	0.99	C7—H7B	0.99
C3—H3B	0.99	C8—N3	1.1427 (14)
C4—C5	1.4680 (14)		
C1 ⁱ —C1—C2	110.91 (9)	C5—C4—H4B	109.2
C1 ⁱ —C1—H1A	109.5	C3—C4—H4B	109.2
C2—C1—H1A	109.5	H4A—C4—H4B	107.9
C1 ⁱ —C1—H1B	109.5	N2—C5—C4	179.01 (11)
C2—C1—H1B	109.5	N1—C6—C7	112.36 (8)
H1A—C1—H1B	108	N1—C6—H6A	109.1
N1—C2—C1	117.23 (8)	C7—C6—H6A	109.1
N1—C2—H2A	108	N1—C6—H6B	109.1
C1—C2—H2A	108	C7—C6—H6B	109.1
N1—C2—H2B	108	H6A—C6—H6B	107.9
C1—C2—H2B	108	C8—C7—C6	112.36 (8)
H2A—C2—H2B	107.2	C8—C7—H7A	109.1
N1—C3—C4	112.69 (8)	C6—C7—H7A	109.1
N1—C3—H3A	109.1	C8—C7—H7B	109.1
C4—C3—H3A	109.1	C6—C7—H7B	109.1
N1—C3—H3B	109.1	H7A—C7—H7B	107.9
C4—C3—H3B	109.1	N3—C8—C7	178.64 (11)
H3A—C3—H3B	107.8	C3—N1—C6	111.53 (8)
C5—C4—C3	112.12 (8)	C3—N1—C2	113.92 (7)
C5—C4—H4A	109.2	C6—N1—C2	111.51 (8)
C3—C4—H4A	109.2		
C1 ⁱ —C1—C2—N1	179.07 (10)	C7—C6—N1—C3	68.15 (10)
N1—C3—C4—C5	54.79 (11)	C7—C6—N1—C2	-163.24 (8)
N1—C6—C7—C8	52.27 (11)	C1—C2—N1—C3	55.57 (11)
C4—C3—N1—C6	-175.81 (8)	C1—C2—N1—C6	-71.76 (10)
C4—C3—N1—C2	56.87 (11)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4A \cdots N2 ⁱⁱ	0.99	2.47	3.4546 (13)	170
C7—H7B \cdots N3 ⁱⁱ	0.99	2.57	3.5561 (14)	175

Symmetry codes: (ii) $x+1, y, z$.

Fig. 1

