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trans-4-[(2,6-Dimethylphenoxy)methyl]-cyclohexanecarboxylic acid

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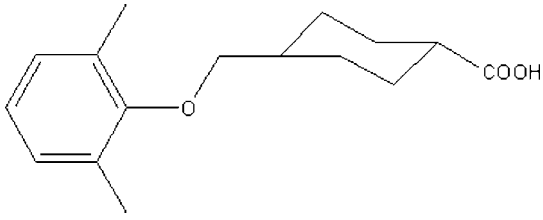
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; R factor = 0.066; wR factor = 0.196; data-to-parameter ratio = 15.5.

The title compound, $C_{16}H_{22}O_3$, is a useful intermediate in the synthesis of poly(amidoamine) dendrimers. The cyclohexane ring adopts a chair conformation. In the crystal structure, molecules are linked into centrosymmetric dimers by pairs of O—H···O hydrogen bonds.

Related literature

For general background on poly(amidoamine) dendrimers, see: Ahmed *et al.* (2001); Grabchev *et al.* (2003); Wang *et al.* (2004). For related structures, see: Bucourt & Hainaut (1965); Dunitz & Strickler (1966); Luger *et al.* (1972).



Experimental

Crystal data

$C_{16}H_{22}O_3$
M_r = 262.34
Triclinic, $P\overline{1}$
a = 7.162 (3) Å
b = 7.680 (4) Å
c = 14.451 (4) Å
α = 95.26 (4)°
β = 98.35 (4)°
γ = 106.44 (3)°
V = 746.9 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 292 (2) K
0.60 × 0.52 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2886 measured reflections
2715 independent reflections
1461 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
3 standard reflections every 250 reflections
intensity decay: 2.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.196
S = 1.16
2715 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.82	1.86	2.658 (3)	166

Symmetry code: (i) $-x+3, -y-1, -z+2$.

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2698).

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supporting information

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trans-4-[(2,6-Dimethylphenoxy)methyl]cyclohexanecarboxylic acid

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S1. Comment

Poly(amidoamine) [PAMAM] dendrimers have attracted much interest for their symmetry, high degree of branching and high density of terminal functional groups, and with different structures they could be used in different fields. Various modifications of periphery of PAMAM dendrimers to change its physical or chemical properties have been reported recently (Wang *et al.*, 2004; Grabchev *et al.*, 2003; Ahmed *et al.*, 2001). To change the lipophilicity of PAMAM dendrimers and provide a new type of linker with special stereostructure, a series of cyclohexanic acid derivatives were synthesized. In our synthetic work the title compound was obtained and here we report its crystal structure.

The cyclohexane ring of the title compound (Fig. 1) adopts a chair conformation. The average C—C bond length of the cyclohexane ring is 1.517 (4) Å, which is close to that of *trans*-1,4-cyclohexane dicarboxylic acid (1.523 (3) Å, Luger *et al.*, 1972). The mean endocyclic angle of the cyclohexane is 111.1 (3)°, which is close to that observed for cyclohexane rings (111.1°, Bucourt & Hainaut, 1965; 111.4 (4)°, Dunitz & Strickler, 1966; Luger *et al.*, 1972).

In the crystal structure, the molecules are linked into centrosymmetric dimers by O—H···O hydrogen bonds (Table 1).

S2. Experimental

Methyl *trans*-4-(tosylmethyl)cyclohexanecarboxylate (3.26 g, 10 mmol), 2,6-dimethylphenol (3.66 g, 30 mmol) and potassium phosphate (10.6 g, 50 mmol) were suspended in dry DMF (20 ml) and heated at 368 K for 8 h, and then water (30 ml) and toluene (30 ml) were added. After agitation, the water layer separated was washed twice with toluene and the organic layer combined was washed with water and then dried with sodium sulfate. After filtration and distillation under vaccum, the crude product obtained was further purified by column chromatography to give pure methyl ester. The ester was then hydrolyzed in a ethanol (15 ml)–1 N NaOH (15 ml) solution for 5 h at 313 K. After cooling and acidification with hydrochloride, the white solid precipitated was collected. Colourless crystals were obtained by slow evaporation in chloroform at room temperature.

S3. Refinement

H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93–0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2–1.5U_{eq}(C). A rotating group model was used for methyl and hydroxyl groups.

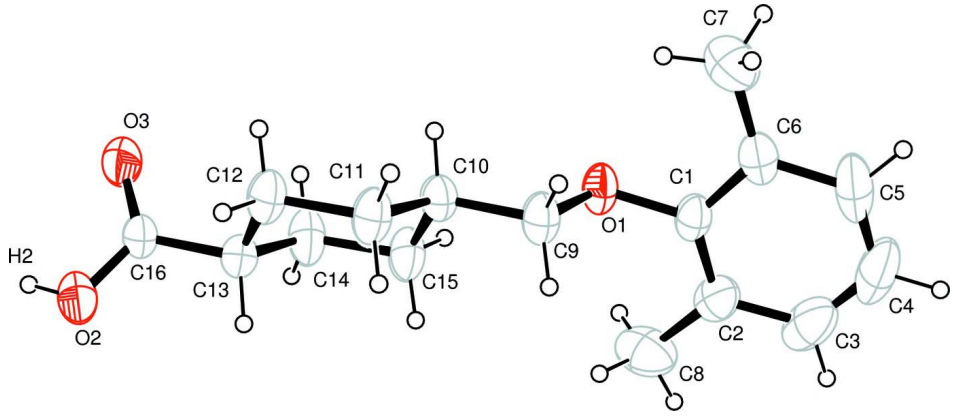

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

$C_{16}H_{22}O_3$
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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.162$ (3) Å
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$c = 14.451$ (4) Å
$\alpha = 95.26$ (4)°
$\beta = 98.35$ (4)°
$\gamma = 106.44$ (3)°
$V = 746.9$ (6) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.167$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 26 reflections
$\theta = 4.3$–7.4°
$\mu = 0.08$ mm^{-1}
$T = 292$ K
Block, colourless
0.60 × 0.52 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
2886 measured reflections
2715 independent reflections
1461 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.4°$
$h = -8\rightarrow8$
$k = -5\rightarrow9$
$l = -17\rightarrow17$
3 standard reflections every 250 reflections
intensity decay: 2.3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.196$
$S = 1.16$
2715 reflections
175 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 0.0605P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.8686 (2)	0.1188 (2)	0.70598 (13)	0.0678 (5)
O2	1.2419 (3)	−0.5645 (3)	0.96803 (18)	0.0922 (7)
H2	1.3381	−0.5780	1.0015	0.138*
O3	1.4736 (3)	−0.3312 (3)	0.93470 (18)	0.0964 (8)
C1	0.7116 (4)	0.1817 (3)	0.6701 (2)	0.0628 (7)
C2	0.5908 (4)	0.2148 (4)	0.7308 (2)	0.0769 (8)
C3	0.4398 (5)	0.2823 (5)	0.6941 (4)	0.1122 (14)
H3	0.3518	0.3017	0.7324	0.135*
C4	0.4162 (6)	0.3209 (5)	0.6047 (5)	0.1232 (17)
H4	0.3148	0.3684	0.5828	0.148*
C5	0.5403 (6)	0.2907 (4)	0.5465 (3)	0.1052 (13)
H5	0.5235	0.3191	0.4853	0.126*
C6	0.6931 (4)	0.2174 (4)	0.5772 (2)	0.0728 (8)
C7	0.8312 (6)	0.1882 (5)	0.5141 (2)	0.1050 (11)
H7A	0.9250	0.1368	0.5466	0.157*
H7B	0.9000	0.3034	0.4965	0.157*
H7C	0.7576	0.1056	0.4584	0.157*
C8	0.6272 (6)	0.1818 (5)	0.8314 (3)	0.1143 (13)
H8A	0.6137	0.0542	0.8333	0.171*
H8B	0.5326	0.2158	0.8642	0.171*
H8C	0.7585	0.2541	0.8612	0.171*
C9	0.8238 (4)	−0.0765 (3)	0.6916 (2)	0.0710 (8)
H9A	0.8080	−0.1196	0.6249	0.085*
H9B	0.7005	−0.1320	0.7125	0.085*
C10	0.9881 (4)	−0.1320 (3)	0.74612 (18)	0.0610 (7)
H10	1.1123	−0.0654	0.7276	0.073*
C11	0.9560 (4)	−0.3349 (4)	0.7197 (2)	0.0764 (8)
H11A	0.9530	−0.3606	0.6524	0.092*
H11B	0.8287	−0.4032	0.7329	0.092*
C12	1.1171 (4)	−0.3994 (4)	0.7733 (2)	0.0746 (8)
H12A	1.2426	−0.3407	0.7551	0.089*
H12B	1.0871	−0.5308	0.7564	0.089*
C13	1.1352 (4)	−0.3548 (4)	0.8800 (2)	0.0712 (8)
H13	1.0096	−0.4209	0.8974	0.085*
C14	1.1699 (5)	−0.1513 (4)	0.9070 (2)	0.0838 (9)

H14A	1.2980	−0.0839	0.8942	0.101*
H14B	1.1718	−0.1259	0.9742	0.101*
C15	1.0103 (5)	−0.0858 (4)	0.8526 (2)	0.0789 (8)
H15A	1.0425	0.0460	0.8689	0.095*
H15B	0.8849	−0.1419	0.8716	0.095*
C16	1.2952 (4)	−0.4194 (4)	0.9309 (2)	0.0679 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0677 (12)	0.0521 (10)	0.0835 (12)	0.0263 (9)	−0.0042 (9)	0.0107 (8)
O2	0.0857 (14)	0.0824 (15)	0.1224 (19)	0.0393 (12)	0.0165 (13)	0.0434 (13)
O3	0.0695 (14)	0.0986 (16)	0.136 (2)	0.0373 (12)	0.0181 (12)	0.0551 (14)
C1	0.0573 (15)	0.0486 (14)	0.0804 (18)	0.0191 (12)	−0.0007 (13)	0.0088 (12)
C2	0.0742 (19)	0.0565 (17)	0.104 (2)	0.0234 (15)	0.0221 (17)	0.0078 (15)
C3	0.083 (3)	0.071 (2)	0.193 (5)	0.0338 (19)	0.041 (3)	0.013 (3)
C4	0.074 (3)	0.079 (3)	0.216 (6)	0.037 (2)	−0.010 (3)	0.029 (3)
C5	0.103 (3)	0.074 (2)	0.122 (3)	0.024 (2)	−0.038 (2)	0.027 (2)
C6	0.0754 (19)	0.0592 (17)	0.0762 (19)	0.0179 (14)	−0.0078 (15)	0.0131 (14)
C7	0.130 (3)	0.105 (3)	0.085 (2)	0.038 (2)	0.024 (2)	0.0224 (19)
C8	0.154 (3)	0.100 (3)	0.102 (3)	0.040 (2)	0.062 (3)	0.012 (2)
C9	0.0734 (18)	0.0548 (16)	0.0841 (19)	0.0268 (13)	−0.0026 (14)	0.0088 (13)
C10	0.0652 (16)	0.0495 (14)	0.0686 (16)	0.0217 (12)	0.0039 (12)	0.0087 (12)
C11	0.0809 (19)	0.0603 (17)	0.090 (2)	0.0345 (14)	−0.0016 (15)	0.0030 (14)
C12	0.0780 (19)	0.0602 (17)	0.090 (2)	0.0348 (14)	0.0035 (15)	0.0051 (14)
C13	0.0676 (17)	0.0716 (18)	0.090 (2)	0.0353 (14)	0.0230 (14)	0.0304 (15)
C14	0.112 (2)	0.081 (2)	0.0727 (19)	0.0588 (19)	0.0024 (16)	0.0080 (15)
C15	0.098 (2)	0.0747 (19)	0.079 (2)	0.0534 (17)	0.0075 (15)	0.0093 (15)
C16	0.0757 (19)	0.0644 (17)	0.0816 (19)	0.0397 (15)	0.0238 (15)	0.0268 (15)

Geometric parameters (Å, º)

O1—C1	1.397 (3)	C8—H8C	0.96
O1—C9	1.431 (3)	C9—C10	1.505 (3)
O2—C16	1.266 (3)	C9—H9A	0.97
O2—H2	0.82	C9—H9B	0.97
O3—C16	1.256 (3)	C10—C11	1.513 (4)
C1—C2	1.373 (4)	C10—C15	1.522 (4)
C1—C6	1.390 (4)	C10—H10	0.98
C2—C3	1.386 (5)	C11—C12	1.521 (4)
C2—C8	1.496 (5)	C11—H11A	0.97
C3—C4	1.350 (6)	C11—H11B	0.97
C3—H3	0.93	C12—C13	1.526 (4)
C4—C5	1.359 (6)	C12—H12A	0.97
C4—H4	0.93	C12—H12B	0.97
C5—C6	1.402 (5)	C13—C16	1.498 (4)
C5—H5	0.93	C13—C14	1.516 (4)
C6—C7	1.487 (5)	C13—H13	0.98

C7—H7A	0.96	C14—C15	1.521 (4)
C7—H7B	0.96	C14—H14A	0.97
C7—H7C	0.96	C14—H14B	0.97
C8—H8A	0.96	C15—H15A	0.97
C8—H8B	0.96	C15—H15B	0.97
C1—O1—C9	113.91 (18)	C9—C10—C15	113.2 (2)
C16—O2—H2	109.5	C11—C10—C15	110.1 (2)
C2—C1—C6	123.7 (3)	C9—C10—H10	107.8
C2—C1—O1	117.7 (3)	C11—C10—H10	107.8
C6—C1—O1	118.5 (3)	C15—C10—H10	107.8
C1—C2—C3	116.5 (3)	C10—C11—C12	112.2 (2)
C1—C2—C8	120.4 (3)	C10—C11—H11A	109.2
C3—C2—C8	123.1 (3)	C12—C11—H11A	109.2
C4—C3—C2	122.2 (4)	C10—C11—H11B	109.2
C4—C3—H3	118.9	C12—C11—H11B	109.2
C2—C3—H3	118.9	H11A—C11—H11B	107.9
C3—C4—C5	120.2 (4)	C11—C12—C13	111.6 (2)
C3—C4—H4	119.9	C11—C12—H12A	109.3
C5—C4—H4	119.9	C13—C12—H12A	109.3
C4—C5—C6	121.2 (4)	C11—C12—H12B	109.3
C4—C5—H5	119.4	C13—C12—H12B	109.3
C6—C5—H5	119.4	H12A—C12—H12B	108.0
C1—C6—C5	116.2 (3)	C16—C13—C14	112.1 (2)
C1—C6—C7	122.8 (3)	C16—C13—C12	110.4 (2)
C5—C6—C7	121.0 (3)	C14—C13—C12	110.0 (2)
C6—C7—H7A	109.5	C16—C13—H13	108.1
C6—C7—H7B	109.5	C14—C13—H13	108.1
H7A—C7—H7B	109.5	C12—C13—H13	108.1
C6—C7—H7C	109.5	C13—C14—C15	111.7 (2)
H7A—C7—H7C	109.5	C13—C14—H14A	109.3
H7B—C7—H7C	109.5	C15—C14—H14A	109.3
C2—C8—H8A	109.5	C13—C14—H14B	109.3
C2—C8—H8B	109.5	C15—C14—H14B	109.3
H8A—C8—H8B	109.5	H14A—C14—H14B	107.9
C2—C8—H8C	109.5	C14—C15—C10	112.6 (2)
H8A—C8—H8C	109.5	C14—C15—H15A	109.1
H8B—C8—H8C	109.5	C10—C15—H15A	109.1
O1—C9—C10	109.9 (2)	C14—C15—H15B	109.1
O1—C9—H9A	109.7	C10—C15—H15B	109.1
C10—C9—H9A	109.7	H15A—C15—H15B	107.8
O1—C9—H9B	109.7	O3—C16—O2	122.8 (2)
C10—C9—H9B	109.7	O3—C16—C13	119.9 (2)
H9A—C9—H9B	108.2	O2—C16—C13	117.3 (3)
C9—C10—C11	109.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.82	1.86	2.658 (3)	166

Symmetry code: (i) −*x*+3, −*y*−1, −*z*+2.