

2,4-Bis[2-(benzylaminocarbonyl)phenoxy-
methyl]-1,3,5-trimethylbenzeneYu Tang,* Kuan-Zhen Tang and
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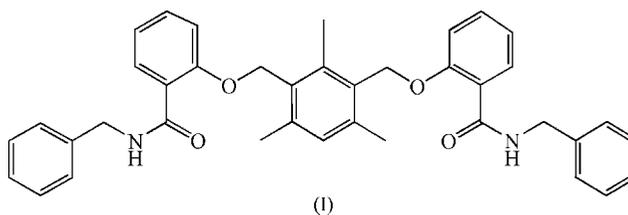
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
Disorder in main residue
 R factor = 0.054
 wR factor = 0.151
Data-to-parameter ratio = 12.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{39}\text{H}_{38}\text{N}_2\text{O}_4$, possesses crystallographically imposed C_2 symmetry, with the twofold axis bisecting the central benzene ring. There is an intramolecular hydrogen bond between the phenoxy O atom and the amide N atom.

Comment

Podand-type ligands have drawn much attention in recent years, mainly due to their selective coordinating capacity, spheroidal cavities and hard binding sites, thereby stabilizing their complexes, acquiring novel coordination structure and shielding the encapsulated ion from interaction with the surroundings (Renaud *et al.*, 1999). Among numerous podands which have demonstrated their potential use in functional supramolecular chemistry (Fujita *et al.*, 1998; Su *et al.*, 2002, 2003), amide-type podands are important for preparing rare earth complexes possessing strong luminescent properties. It is expected that the amide-type podand ligands, which are flexible in structure and have 'terminal group effects' (Tümmler *et al.*, 1979), will shield the encapsulated rare-earth ion from interaction with the surroundings, and thus achieve strong luminescent properties. We report here the synthesis and structure of a new amide-type podand ligand, namely 2,4-bis[2-(benzylaminocarbonyl)phenoxy-methyl]-1,3,5-trimethylbenzene, (I).



As shown in Fig. 1, the molecule of (I) possesses crystallographically imposed C_2 symmetry, with the twofold axis bisecting the central benzene ring. There is an intramolecular hydrogen bond between the phenoxy O atom and the amide N atom (Table 1). In addition, the terminal benzene ring of the benzyl group interacts with the central benzene ring through very weak π - π stacking, with a centroid-centroid distance of 4.152 (3) Å and a dihedral angle of 26.49 (2)°.

Experimental

N-Benzylsalicylamide (0.58 g, 2.57 mmol), potassium carbonate (0.5 g, 3.6 mmol) and dimethylformamide (10 ml) were heated to *ca* 353 K and 1,3,5-trimethyl-2,4-bis(bromomethyl)benzene (0.31 g, 0.85 mmol) was added. The reaction mixture was stirred at 353–363 K for 5 h. After cooling, the mixture was poured into water (100 ml).

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The resulting solid was subjected to column chromatography on silica gel [petroleum ether–ethyl acetate 1:1 (v/v)], producing the title compound, (I) (yield 70%, m.p. 457–459 K). Analysis calculated for $C_{39}H_{38}N_2O_4$: C 78.26, H 6.35, N 4.68%; found: C 78.37, H 6.30, N 4.40%. The crystal used for the data collection was obtained by slow evaporation of a saturated ethanol solution of (I) at room temperature.

Crystal data

$C_{39}H_{38}N_2O_4$
 $M_r = 598.71$
 Monoclinic, $C2/c$
 $a = 20.5494$ (10) Å
 $b = 12.1369$ (10) Å
 $c = 16.3862$ (14) Å
 $\beta = 126.097$ (10)°
 $V = 3302.2$ (4) Å³
 $Z = 4$

$D_x = 1.204$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 2.5$ – 26.1 °
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
 Block, colorless
 $0.43 \times 0.35 \times 0.30$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{min} = 0.905$, $T_{max} = 0.986$
 3668 measured reflections
 3247 independent reflections
 1596 reflections with $I > 2\sigma(I)$

$R_{int} = 0.019$
 $\theta_{max} = 26.0$ °
 $h = -25 \rightarrow 0$
 $k = -14 \rightarrow 1$
 $l = -16 \rightarrow 20$
 2 standard reflections every 800 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.151$
 $S = 1.00$
 3247 reflections
 256 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 1.5772P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.002$
 $\Delta\rho_{max} = 0.27$ e Å⁻³
 $\Delta\rho_{min} = -0.17$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0045 (5)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O2$	0.90 (3)	1.93 (3)	2.628 (3)	133 (3)

All H atoms except those on C7, C19 and C20 were initially located in a difference Fourier map and refined freely with isotropic displacement parameters. The H atoms on C7, C19 and C20 were included as riding atoms ($C7-H = 0.97$ Å, and $C19-H$ and $C20-H = 0.96$ Å) with $U_{iso}(H) = 1.2$ or 1.5 times $U_{eq}(C)$. The three H atoms attached to C20 are disordered; the refined occupancies were all 0.50.

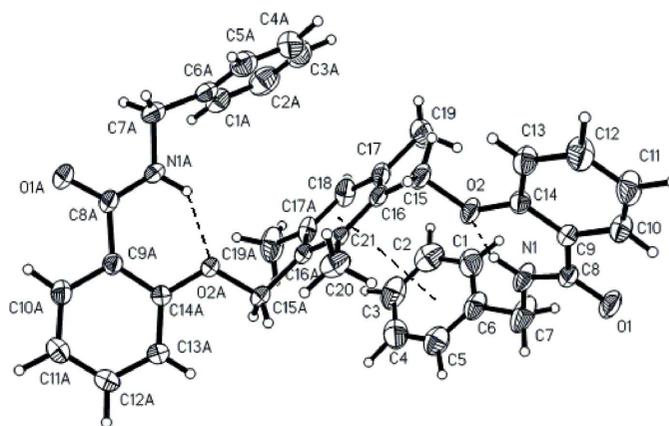


Figure 1

A view of the molecular structure of (I), showing the atom-numbering scheme and 30% displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii. The intramolecular hydrogen bond and weak π – π interactions are shown as dashed lines.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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