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Key indicators

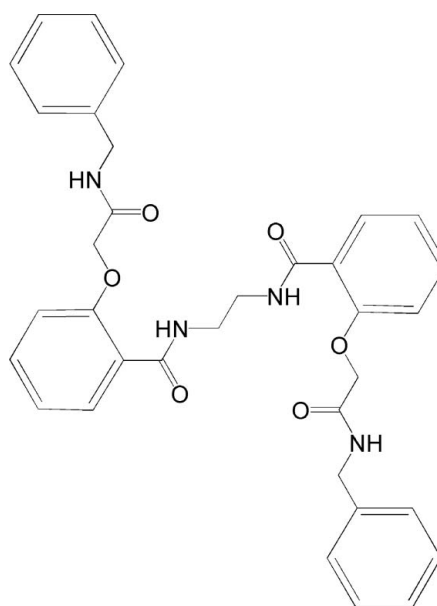
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(C-C) = 0.003$ Å
 R factor = 0.041
 wR factor = 0.118
Data-to-parameter ratio = 14.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N,N'*-1,2-Ethanediybis{2-[(*N*-benzylcarbamoyl)-
methoxy]benzamide}**

The title compound, $C_{34}H_{34}N_4O_6$, has been synthesized by the reaction of *N,N'*-1,2-ethanediybis(2-hydroxybenzamide) with *N*-benzyl-2-chloroacetamide and crystallized from DMF. Individual molecules possess crystallographically imposed inversion symmetry. There are intramolecular hydrogen bonds between the phenoxy O atoms and the benzamide N atoms. Intermolecular hydrogen bonds between amide N and amide O atoms of neighbouring molecules allow the construction of a two-dimensional network.

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Comment

Open-chain crown ether ligands have attracted increasing attention in recent years due to certain properties, such as their high capacity to encapsulate metal ions (Zeckert *et al.*, 2004) and protect these from solvent interactions (Petoud *et al.*, 2003). As a result, these compounds often offer many advantages in the extraction and analysis of rare earth ions (Zhang *et al.*, 2002). We are interested in luminescent lanthanide complexes because of their potential applications, for example as diagnostic tools in biomedical analysis, as responsive analytical systems, and as luminescent labels for fluoroimmunoassays (Zucchi *et al.*, 2002). Nevertheless, to achieve good fluorescence properties, it is important to achieve the correct balance between flexibility and rigidity (Adam *et al.*, 2001).



(I)

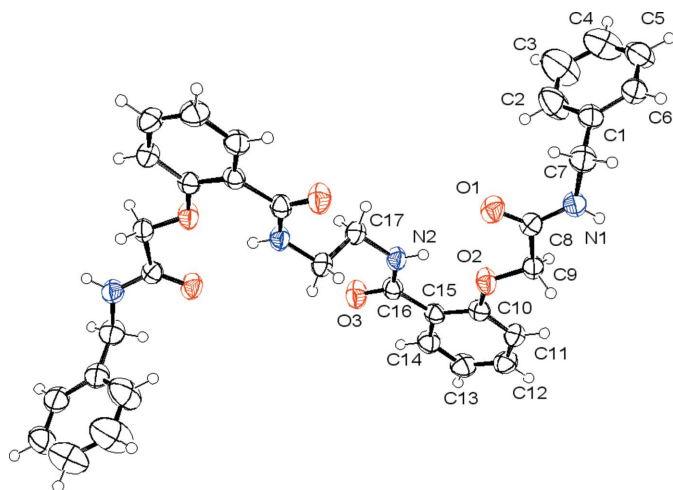


Figure 1
The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by $1-x$, $-y$, $3-z$.

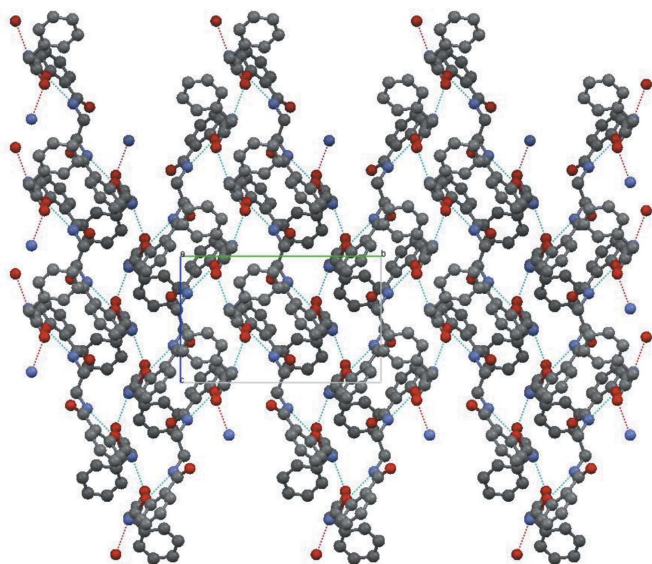


Figure 2
The molecular packing of (I), viewed along the a axis. H atoms have been omitted for clarity. Atoms are coloured as follows: O, red; N, blue; C, black. Dashed lines indicate the hydrogen-bonding interactions.

earth ions and enhance the fluorescence of rare earth complexes, by incorporating amide groups which coordinate more strongly to lanthanide ions than ether groups, conferring strong luminescence on the resulting complexes (Tang *et al.*, 2005). Here we report the synthesis and structure of a new doubly functionalized and amide-based open-chain crown ether ligand, namely the title compound, (I).

As seen from Fig. 1, (I) has crystallographically imposed inversion symmetry. The amide groups of the molecule appear to form hydrogen bonds with both the carbonyl O atom and phenoxy O atom. The principal interaction is an intramolecular hydrogen bond between the phenoxy O atom and the benzamide N atom (Table 1), which forms a six-membered ring; however, the $N2 \cdots O1$ contact of $3.524(3) \text{ \AA}$ is so much

longer that it is difficult to regard this as representing a significantly bifurcated interaction.

Intermolecular hydrogen bonds between the amide NH and carbonyl O atoms in neighbouring molecules (Fig. 2) results in the arrangement of molecules into a two-dimensional network which runs parallel to (011).

Experimental

To *N,N'*-1,2-ethanediyl-bis(2-hydroxybenzamide) (1.8 g, 6 mmol) (Gandhi & Kulkarni, 1994) in DMF (100 ml) was added sodium hydroxide (0.48 g, 12 mmol). The mixture was heated to 353 K and stirred for about 0.5 h. A solution of *N*-benzyl-2-chloroacetamide (2.424 g, 13.2 mmol) and potassium iodide (1.1 g, 6.6 mmol) in DMF (30 ml) was then added dropwise at a constant rate over a period of 1 h. The reaction mixture was stirred at *ca* 353 K for an additional 10 h. The solvent was removed under vacuum, and then the residue was treated with water (50 ml). The precipitate was collected by filtration and washed with water, then twice recrystallized from DMF–water (5:1 *v/v*) to give colourless crystals. Single crystals suitable for X-ray analysis were obtained by slow evaporation of the DMF solvent at room temperature. Yield 2.85 g (80%); m.p. 484–486 K; elemental analysis: calculated for $C_{34}H_{34}N_4O_6$: C 68.67, H 5.76, N 9.42%; found: C 68.72, H 5.72, N 9.47%.

Crystal data

$C_{34}H_{34}N_4O_6$	$Z = 2$
$M_r = 594.65$	$D_x = 1.330 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.7401(3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 13.6100(4) \text{ \AA}$	$T = 294(2) \text{ K}$
$c = 8.8838(2) \text{ \AA}$	Block, colourless
$\beta = 105.457(1)^\circ$	$0.42 \times 0.28 \times 0.10 \text{ mm}$
$V = 1484.67(7) \text{ \AA}^3$	

Data collection

Bruker APEXII CCD area-detector diffractometer	2913 independent reflections
φ and ω scans	2210 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.022$
8214 measured reflections	$\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.258P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta\rho)_{\text{max}} = 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
2913 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
200 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0079 (18)

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2' \cdots O2$	0.86	1.96	2.642 (3)	135
$N1-H1' \cdots O1^i$	0.86	2.20	3.028 (3)	160

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

H atoms were positioned geometrically and treated as riding, with $C-H = 0.93$ and 0.97 \AA , $N-H = 0.86 \text{ \AA}$, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXTL*.

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