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12-Benzoyl-2,3,5,6,9,10,11,12-octahydro-8*H*-1,4,7,12-benzotrioxaazacyclotetradecine

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.114
Data-to-parameter ratio = 22.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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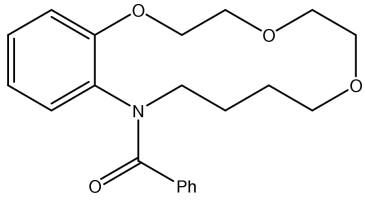
The title compound, $C_{21}H_{25}NO_4$, is the benzoyl derivative of a benzo-aza-crown ether.

Comment

Incorporation of N-donor atoms into the macrocyclic framework of crown ethers gives aza-crown analogues which can display increased binding strength and selectivity towards transition and post-transition metal ions (Bradshaw *et al.*, 1996). We have reported the preparation of a range of benzo-aza-crown ethers by a protocol involving reductive cyclization of nitroarenes bearing aldehyde-containing side chains (Ventrice *et al.*, 1999, 2001). An X-ray structure determination of the benzoyl derivative, (I), was undertaken to establish the conformation of this compound (Fig. 1).



(I)

Experimental

Sodium hydroxide (10% aq.) (10 ml) was added to 2,3,5,6,9,10,11,12-octahydro-8*H*-1,4,7,12-benzotrioxaazacyclotetradecine (50 mg, 0.20 mmol) dissolved in THF (10 ml). This mixture was allowed to stir for 10 min, benzoyl chloride (56 mg, 0.40 mmol) was added dropwise and the resulting mixture was allowed to stir for 24 h. The reaction was poured into water (50 ml) and extracted into CH_2Cl_2 (3 × 50 ml), dried (Na_2SO_4), filtered and the solvent was removed under reduced pressure to give a yellow oil. Excess benzoyl chloride was removed on the a high vacuum pump. Column chromatography of the residue (SiO_2, 20% EtOAc/hexane) gave amide (I) as a yellow oil which solidified on standing (70 mg, 99%). Dichloromethane (10 ml) was added to the amide and single crystals suitable for X-ray structure determination were obtained by slow evaporation of the solvent; m.p: 415.4–417.6 K.

Crystal data

$C_{21}H_{25}NO_4$
M_r = 355.42
Monoclinic, $P2_1/c$
a = 16.825 (1) Å
b = 13.550 (1) Å
c = 8.173 (1) Å
β = 95.02 (1)°
V = 1856.1 (3) Å^3
Z = 4
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 477 reflections
θ = 2.4–30.0°
μ = 0.09 mm^{-1}
T = 123 (2) K
Tabular, colourless
0.22 × 0.16 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.056$
CCD rotation images, thick-slice scans	$\theta_{max} = 30.1^\circ$
	$h = -23 \rightarrow 23$
25 477 measured reflections	$k = -18 \rightarrow 18$
5351 independent reflections	$l = -11 \rightarrow 11$
3438 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.2125P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	
$wR(F^2) = 0.114$	
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
5351 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
H-atom parameters constrained	

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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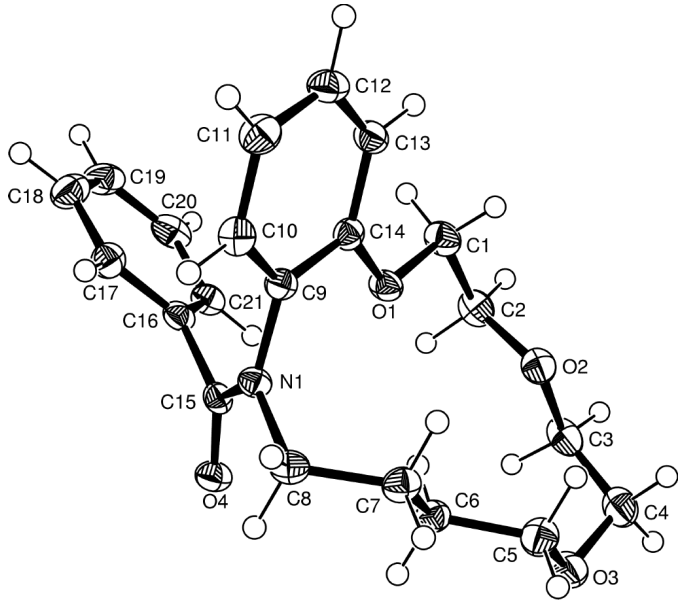

Figure 1
View of (I) (50% probability displacement ellipsoids).

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