4,8,9,10-Tetrakis(4-methoxyphenyl)-1,3-diaza-adamantan-6-one benzene solvate

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Mirror symmetry is retained in the crystals of the title compound, $C_{36}H_{36}N_2O_5 \cdot C_6H_6$. The trapping of the solvent (benzene) molecule in the crystal lattice as space filler with no strong interactions with the solute molecule presumably plays an important role in the retention of molecular site symmetry. The crystal structure is stabilized by van der Waals interactions.

Comment

Crystallographic studies on symmetrically shaped molecules, with a view to understanding the nature and strength of intermolecular interactions, are expected to provide information regarding their role in effecting symmetry carry-over from the free state to the solid and obtain information about specific interactions. The symmetry of a molecule in the solid state is often found to be less than that observed in the free state as the molecules tend to pack as closely as possible upon crystallization (Kitaigorodskii, 1973), giving rise to a variety of intermolecular interactions. While an inversion centre is often carried over into the crystal, the retention of mirror or twofold symmetry by molecules seems to depend on the presence, nature and strength of the intermolecular interactions. The present crystal structure, (I), is a good example of the retention of mirror symmetry by a molecule in the crystal state.
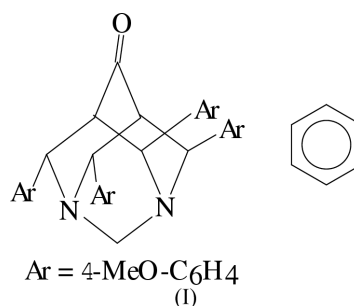

Ar = 4-MeO-C6H4

(I)

Fig. 1 shows the crystallographic numbering scheme adopted. All four six-membered rings which constitute the diazaadamantanone cage adopt the chair conformation which is the most preferred conformation for adamantanes, irrespective of substitution. The molecule is bisected by a mirror plane passing through atoms C2, N1, N3, C10 and O11 of the diazaadamantanone cage. The solvent benzene molecule is trapped in the crystal lattice and held inside the channel running along the c axis by van der Waals interactions (Fig. 2). In the absence of C—H···X-type hydrogen bonds, the solvent molecule presumably plays a role in optimizing packing efficiency within the lattice. The absence of C—H···X-type interactions may be attributed to the deficiency of acceptors

DOI: 101107/S1600536801003956

compared to donors (phenyl C—H) whose presence otherwise might have had a considerable influence on the molecular conformation.

Experimental

Colourless single crystals were obtained as transparent needles by slow evaporation of a benzene solution at room temperature.

Crystal data

$C_{36}H_{36}N_2O_5 \cdot C_6H_6$
$M_r = 654.78$
Orthorhombic, $Pnma$
$a = 16.6240 (5)$ Å
$b = 26.0007 (16)$ Å
$c = 8.2207 (7)$ Å
$V = 3553.3 (4)$ Å^3
$Z = 4$
$D_x = 1.224$ Mg m^{-3}

Cu $K\alpha$ radiation
Cell parameters from 25
  reflections
$\theta = 16–26°$
$\mu = 0.64$ mm^{-1}
$T = 293 (2)$ K
Needles, colourless
$0.33 \times 0.22 \times 0.18$ mm

Data collection

Enraf–Nonius CAD-4 diffract-
  ometer
ω–θ scans
Absorption correction: ψ scan
  (North *et al.*, 1968)
  $T_{min} = 0.864$, $T_{max} = 0.892$
3401 measured reflections
3401 independent reflections
2226 reflections with $I > 2\sigma(I)$

$R_{int} = 0.010$
$\theta_{max} = 70.0°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 31$
$l = 0 \rightarrow 10$
2 standard reflections
  every 200 reflections
  intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.191$
$S = 1.13$
3401 reflections
230 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 6.1627P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: $SHELXL$97
Extinction coefficient: 0.00090 (11)

All the H atoms were located from a difference Fourier map and were allowed to ride on the heavier atoms to which they are attached using default values for bond lengths and displacement parameters.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: CAD-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.
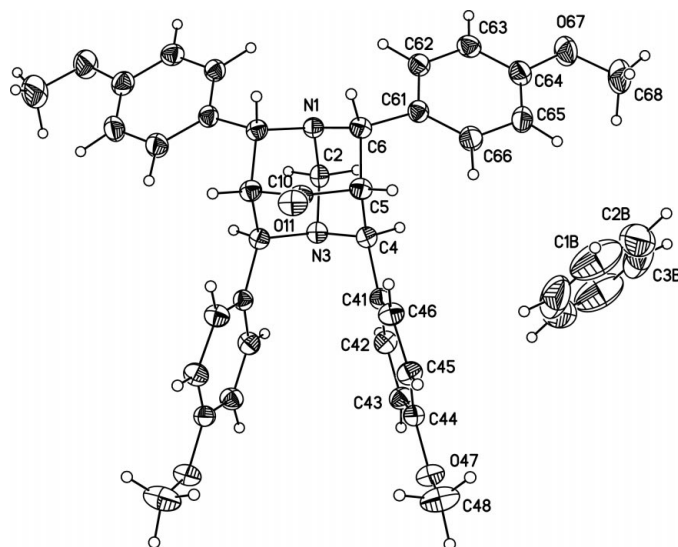

Figure 1
ORTEP diagram (*PLATON*; Spek, 1999) with displacement ellipsoids drawn at the 40% probability level showing the numbering scheme adopted.

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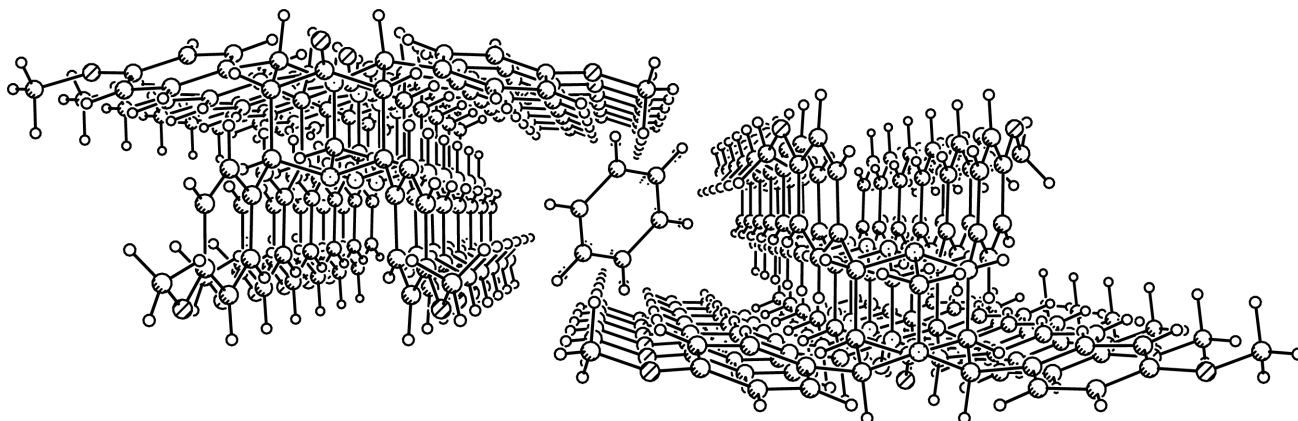Spek, A. L. (1999). *PLATON for Windows*. Utrecht University, The Netherlands.

Figure 2
Figure showing the solvent benzene molecule trapped inside channels running along the *c* axis.