

## Crystal Structure of 4,8,9,10-Tetraphenyl-1,3-diazaadamantane

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In the crystal structure of the title compound, C<sub>32</sub>H<sub>30</sub>N<sub>2</sub>, all of the four six-membered rings that constitute the diazaadamantanone cage adopt chair conformations. Two of the four phenyl substituents occupy axial, and the other two occupy equatorial positions relative to their respective C<sub>5</sub>N rings of the adamantane framework. There are no hydrogen-bonded interactions between the two molecules in the unit cell. The crystal packing is characterized by weak C-H... $\pi$  interactions.

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1,3-Diazaadamantane systems are of pharmacological significance and are potentially interesting as anticholinergic compounds.<sup>1</sup> To prepare the title compound, 4,8,9,10-tetraphenyl-1,3-diazatri cyclo[3.3.1]decan-6-one (2 g), diethylene glycol (7.5 mL) and hydrazene hydrate (10 mL), were taken into a 100 mL RB flask,

and the contents were heated at 100°C for an hour. Then, the mixture was allowed to cool and KOH (1.2 g) was added to it. Excess hydrazine hydrate was distilled off. The resulting solution was heated at 200°C for an hour, and then the mixture

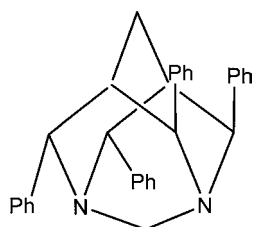


Fig. 1 Schematic diagram of the title compound.

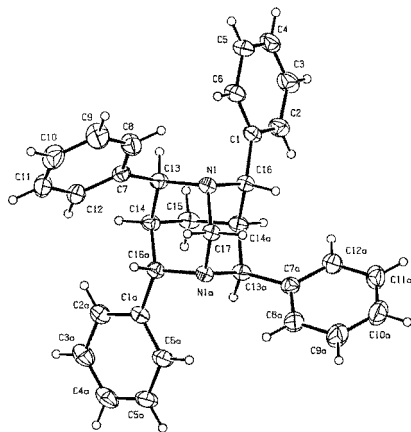


Fig. 2 ORTEP diagram of the molecule at 30% probability.

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Table 1 Crystal and experimental data

Empirical formula	C <sub>32</sub> H <sub>30</sub> N <sub>2</sub>
Formula weight	442.58
Temperature	293(2)K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	$a = 16.7700(10)$ Å $b = 11.1431(9)$ Å $c = 12.7593(8)$ Å $\alpha = 90^\circ$ $\beta = 90.331(2)^\circ$ $\gamma = 90^\circ$
Volume	$2384.3(3)$ Å <sup>3</sup>
Z, Calculated density	4, 1.233 Mg/m <sup>3</sup>
Absorption coefficient	$0.071 \text{ mm}^{-1}$
$F(0\ 0\ 0)$	944
Crystal size	$0.18 \times 0.15 \times 0.11 \text{ mm}$
Theta range for data collection	$2.19$ to $24.97^\circ$
Limiting indices	$-19 \leq h \leq 12$ , $-11 \leq k \leq 3$ , $-15 \leq l \leq 15$
Reflections collected/unique	4809/2090 [ $R(\text{int}) = 0.0257$ ]
Completeness to theta = 25	100.00%
Absorption correction	$\psi$ -scan
Max. and min. transmission	0.9922 and 0.9873
Refinement method	full-matrix least-squares on $F^2$
Data/restraints/parameters	2090/0/155
Goodness-of-fit on $F^2$	1.033
Final $R$ indices [ $I > 2\sigma(I)$ ]	$R1 = 0.0398$ , $wR2 = 0.0888$
$R$ indices (all data)	$R1 = 0.0769$ , $wR2 = 0.1035$
Largest diff. peak and hole	$0.135$ and $-0.153 \text{ e.Å}^{-3}$
Measurement	Enraf Nonius CAD-4
Structure determination and refinement	SHELXS97/SHELXL97
Structure drawing	ORTEP

Table 2 Fractional atomic coordinates ( $\times 10^{-4}$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

atom	x	y	z	U(eq)
C(1)	6748(1)	5755(2)	7075(1)	42(1)
C(2)	7163(1)	4851(2)	7584(2)	55(1)
C(3)	7838(1)	4340(2)	7152(2)	64(1)
C(4)	8101(1)	4707(2)	6191(2)	65(1)
C(5)	7702(1)	5604(2)	5672(2)	65(1)
C(6)	7033(1)	6133(2)	6115(2)	56(1)
C(7)	4716(1)	7077(2)	5261(1)	44(1)
C(8)	5150(1)	8004(2)	4823(2)	65(1)
C(9)	4846(2)	8688(2)	4017(2)	82(1)
C(10)	4093(2)	8478(2)	3638(2)	74(1)
C(11)	3653(1)	7572(2)	4055(2)	65(1)
C(12)	3959(1)	6874(2)	4857(1)	53(1)
C(13)	5085(1)	6320(1)	6134(1)	41(1)
C(14)	4506(1)	5535(1)	6764(1)	41(1)
C(15)	5000	4737(2)	7500	47(1)
C(16)	6044(1)	6364(1)	7599(1)	40(1)
C(17)	5000	7814(2)	7500	39(1)
N(1)	5556(1)	7097(1)	6864(1)	39(1)

$$*U_{\text{eq}} = (1/3)\sum_j U_{ij} a_i^* a_j^* a_i a_j$$

was poured into crushed ice and the compounds were extracted using  $\text{CHCl}_3$ . Recrystallization was performed in a  $\text{CHCl}_3$  and  $\text{C}_6\text{H}_6$  solvent mixture. Yield 67% (1.3 g); melting point  $242^\circ\text{C}$ . A schematic diagram of the molecule is shown in Fig. 1.

An ORTEP plot of the molecule is shown in Fig. 2. No significant differences in the geometry of the diazaadamantanone cage are seen, since it is known to be inherently rigid and symmetrical. All of the four six-membered rings which constitute the diaza-adamantanone cage, adopt chair conformations; this is the most preferred conformation for adamantanes, irrespective of substitutions, as in related structures previously studied.<sup>2,3</sup>

The crystal and experimental details are given in Table 1. The structure was solved by direct methods and refined by full-matrix least-squares procedures to a final reliability value of 0.0420. H atoms were placed at calculated positions and allowed to ride on their carrier atoms with  $\text{C-H} = 0.93 - 0.98 \text{\AA}$ , and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for  $\text{CH}_2$  and  $\text{CH}$  groups. The atomic

Table 3 Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C6-H6...N1	0.93	2.52	2.869(2)	103
C4-H4...Cg1 <sup>i</sup>	0.93	2.93	3.795(2)	156

Symmetry codes: (i) =  $1/2+x, -1/2+y, z$ . Cg1 is C7-C12 centroid.

coordinates and temperature factors for the non-hydrogen atoms are presented in Table 2.

In this structure, two of the four phenyl substituents occupy axial and the other two occupy equatorial positions relative to their respective  $\text{C}_5\text{N}$  rings of the adamantane framework as shown by the torsion angles ( $\text{C15-C14a-C13-C7} = 173.0(2)^\circ$ ,  $\text{C15-C14a-C16a-C1a} = -72.0(2)^\circ$ ,  $\text{C15-C14-C16-C1} = -72.0(2)^\circ$ ,  $\text{C15-C14-C13a-C7a} = 173.0(2)^\circ$ ). The intramolecular  $\text{C-H}\cdots\text{N}$  bonds determine (Table 3) which of the two phenyl rings prefer a axial/equatorial configuration. There are no noticeable intermolecular  $\text{C-H}\cdots\text{O}$  hydrogen bonds; however, the packing is effected by weak  $\text{C-H}\cdots\pi$  interactions (Table 3) and van der Waals interactions.

## Acknowledgements

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## References

1. M. J. Fernández, E. Gálvez, A. Lorente, and J. A. Camuñas, *J. Heterocycl. Chem.*, **1990**, *27*, 1355.
2. R. V. Krishnakumar, M. Subha Nandhini, V. Vijayakumar, S. Natarajan, M. Sundaravadivelu, S. Perumal, and A. Mostad, *Acta Cryst.*, **2001**, *E57*, o860.
3. M. Subha Nandhini, R. V. Krishnakumar, T. Narasimhamurthy, V. Vijayakumar, M. Sundaravadivelu, and S. Natarajan, *Acta Cryst.*, **2002**, *E58*, o675.