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## Structure Reports

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## *N*-*p*-Tolyladamantane-1-carboxamide

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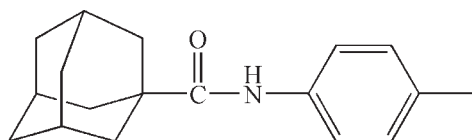
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.073;  $wR$  factor = 0.174; data-to-parameter ratio = 18.9.

In the crystal of the title compound,  $\text{C}_{18}\text{H}_{23}\text{NO}$ , the molecules are linked into chains along the  $c$  axis by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

### Related literature

For bond-length data, see: Allen *et al.* (1987). For the synthesis of the title compound, see: Karle *et al.* (1997); Tadashi *et al.* (1969)



### Experimental

#### Crystal data

$\text{C}_{18}\text{H}_{23}\text{NO}$   
 $M_r = 269.37$   
Orthorhombic,  $Pccn$   
 $a = 30.708$  (7) Å  
 $b = 9.7927$  (2) Å  
 $c = 10.0203$  (6) Å

$V = 3013.2$  (7) Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation  
 $\mu = 0.07$  mm<sup>-1</sup>  
 $T = 298$  K  
0.50 × 0.30 × 0.30 mm

#### Data collection

Rigaku SCXmini diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.964$ ,  $T_{\max} = 0.978$

27405 measured reflections  
3443 independent reflections  
2652 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.080$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.073$   
 $wR(F^2) = 0.174$   
 $S = 1.16$   
3443 reflections

182 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.15$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.86	2.12	2.962 (2)	166

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

The author is grateful to the starter fund of Southeast University for financial support to buy the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2072).

### References

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Karle, I. S., Ranganathan, D. & Haridas, D. (1997). *J. Am. Chem. Soc.* **119**, 2777–2783.  
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**supplementary materials**

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## *N-p*-Tolyladamantane-1-carboxamide

### W. SiMa

#### Comment

The unique structure of adamantane and the pharmacutical effects of adamantane-containing agents on virus (Davis *et al.*, 1964) have attracted many chemists and pharmacologists to do considerable work on the syntheses of adamantane derivatives (Fort *et al.*, 1964). The crystal structure of the title compound (I) is reported herein.

The molecular structure of compound (I), C<sub>18</sub>H<sub>23</sub>ON, is shown in Figure 1. All bond lengths and bond correspond to the geometry parameters expected for atom types and the type of hybridization (Allen *et al.*, 1987). The bonds to nitrogen of the title amide, Fig. 1, the torsion angles of O1—C8—N1—C1 and C9—C8—N1—C1 are 1.70 (3)° and 178.59 (18)°, respectively. The C8—N1 bond has considerable double-bond characer, at 1.349 (2) Å, is substantially shorter than the normal C—N single-bond distance observed in amines. In the crystal of (I), the intermolecular N<sub>1</sub>—H···O<sub>1</sub> H-bonds linked molecules to chains along the *c* axis (Fig.2). And the N<sub>1</sub>—H···O<sub>1</sub> bond length is 2.962 (2) Å.

#### Experimental

A solution of freshly prepared 1-adamantane carbonyl chloride (1 mmol, prepared by refluxing 1-adamantane carboxylic acid with 3*M* excess of SOCl<sub>2</sub>) in dry CH<sub>2</sub>Cl<sub>2</sub> was added dropwise to a well stirred and ice-cooled solution of *p*-toluidine (1 mmol) and triethylamine (2 mmol) in the same solvent. After 24 h of stirring at room temperature, the solvents were removed *in vacuo* and the residue was recrystallized from methanol. Colorless single crystals of the title compound suitable for X-ray diffraction analysis were obtained then and the yield was 80% (Isabella *et al.* 1997; Tadashi *et al.*, 1969).

#### Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Figures

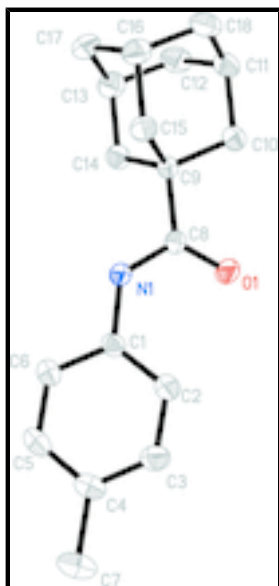


Fig. 1. The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and all H atoms have been omitted for clarity.

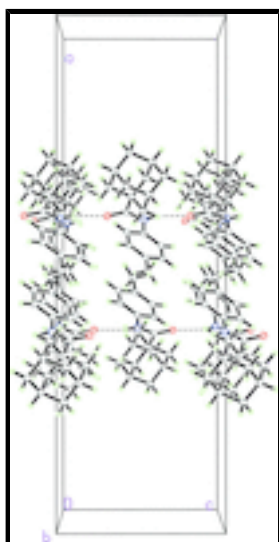


Fig. 2. A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

***N-p-Tolyladamantane-1-carboxamide***

*Crystal data*

$C_{18}H_{23}NO$

$M_r = 269.37$

Orthorhombic, *Pccn*

Hall symbol: -P 2ab 2ac

$a = 30.708 (7) \text{ \AA}$

$b = 9.7927 (2) \text{ \AA}$

$c = 10.0203 (6) \text{ \AA}$

$V = 3013.2 (7) \text{ \AA}^3$

$F_{000} = 1168$

$D_x = 1.188 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4945 reflections

$\theta = 2.5\text{--}27.5^\circ$

$\mu = 0.07 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colourless

$Z = 8$   $0.50 \times 0.30 \times 0.30$  mm

*Data collection*

Rigaku SCXmini diffractometer	3443 independent reflections
Radiation source: fine-focus sealed tube	2652 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.080$
Detector resolution: 13.6612 pixels $\text{mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 298$ K	$\theta_{\text{min}} = 2.7^\circ$
CCD Profile fitting scans	$h = -39 \rightarrow 39$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$k = -12 \rightarrow 12$
$T_{\text{min}} = 0.964$ , $T_{\text{max}} = 0.978$	$l = -12 \rightarrow 12$
27405 measured reflections	

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.073$	H-atom parameters constrained
$wR(F^2) = 0.174$	$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.7031P]$
$S = 1.16$	where $P = (F_o^2 + 2F_c^2)/3$
3443 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
182 parameters	$\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.41659 (6)	0.06223 (19)	-0.01785 (19)	0.0377 (4)
C2	0.41516 (7)	-0.0339 (2)	0.0824 (2)	0.0487 (5)
H2	0.3978	-0.0195	0.1570	0.058*

## supplementary materials

C3	0.43961 (8)	-0.1518 (2)	0.0718 (2)	0.0531 (6)
H3	0.4382	-0.2159	0.1401	0.064*
C4	0.46586 (7)	-0.1775 (2)	-0.0360 (2)	0.0497 (5)
C5	0.46711 (7)	-0.0805 (2)	-0.1354 (2)	0.0531 (6)
H5	0.4846	-0.0952	-0.2097	0.064*
C6	0.44298 (7)	0.0383 (2)	-0.1273 (2)	0.0485 (5)
H6	0.4445	0.1024	-0.1956	0.058*
C7	0.49221 (9)	-0.3075 (3)	-0.0462 (3)	0.0744 (8)
H7A	0.5182	-0.2903	-0.0967	0.112*
H7B	0.4752	-0.3765	-0.0901	0.112*
H7C	0.4999	-0.3382	0.0417	0.112*
C8	0.37650 (6)	0.2542 (2)	0.08852 (18)	0.0364 (4)
C9	0.34923 (6)	0.38031 (19)	0.05641 (18)	0.0351 (4)
C10	0.33115 (7)	0.4418 (2)	0.1861 (2)	0.0472 (5)
H10A	0.3550	0.4667	0.2446	0.057*
H10B	0.3133	0.3746	0.2317	0.057*
C11	0.30364 (8)	0.5690 (2)	0.1545 (2)	0.0534 (6)
H11	0.2923	0.6076	0.2378	0.064*
C12	0.33198 (9)	0.6739 (2)	0.0850 (3)	0.0618 (7)
H12A	0.3561	0.6990	0.1424	0.074*
H12B	0.3151	0.7555	0.0667	0.074*
C13	0.34927 (8)	0.6146 (2)	-0.0453 (2)	0.0549 (6)
H13	0.3671	0.6831	-0.0910	0.066*
C14	0.37712 (7)	0.4883 (2)	-0.0142 (2)	0.0446 (5)
H14A	0.3888	0.4509	-0.0964	0.053*
H14B	0.4014	0.5140	0.0425	0.053*
C15	0.31066 (7)	0.3420 (2)	-0.0341 (2)	0.0437 (5)
H15A	0.3214	0.3033	-0.1169	0.052*
H15B	0.2928	0.2737	0.0098	0.052*
C16	0.28321 (7)	0.4687 (2)	-0.0641 (2)	0.0535 (6)
H16	0.2587	0.4435	-0.1217	0.064*
C17	0.31163 (8)	0.5726 (3)	-0.1349 (2)	0.0591 (6)
H17A	0.2944	0.6523	-0.1578	0.071*
H17B	0.3229	0.5335	-0.2169	0.071*
C18	0.26607 (8)	0.5283 (3)	0.0655 (3)	0.0599 (6)
H18A	0.2482	0.6076	0.0466	0.072*
H18B	0.2481	0.4612	0.1108	0.072*
N1	0.39146 (6)	0.18381 (17)	-0.01773 (15)	0.0414 (4)
H1	0.3850	0.2166	-0.0948	0.050*
O1	0.38450 (5)	0.21905 (16)	0.20311 (13)	0.0529 (4)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0387 (10)	0.0392 (10)	0.0352 (10)	0.0041 (8)	-0.0014 (8)	-0.0063 (8)
C2	0.0507 (12)	0.0489 (12)	0.0466 (12)	0.0075 (10)	0.0105 (10)	0.0025 (10)
C3	0.0563 (13)	0.0424 (12)	0.0605 (14)	0.0078 (10)	0.0049 (11)	0.0054 (10)
C4	0.0426 (11)	0.0435 (12)	0.0630 (14)	0.0054 (10)	-0.0001 (11)	-0.0114 (10)

C5	0.0505 (12)	0.0568 (14)	0.0521 (13)	0.0072 (11)	0.0102 (10)	-0.0147 (11)
C6	0.0549 (13)	0.0503 (12)	0.0401 (11)	0.0064 (10)	0.0073 (9)	-0.0032 (9)
C7	0.0662 (16)	0.0551 (15)	0.102 (2)	0.0201 (13)	0.0048 (16)	-0.0105 (15)
C8	0.0393 (10)	0.0396 (10)	0.0301 (9)	0.0009 (8)	-0.0011 (8)	-0.0010 (8)
C9	0.0373 (10)	0.0367 (10)	0.0314 (9)	0.0023 (8)	0.0015 (8)	-0.0010 (8)
C10	0.0554 (13)	0.0460 (12)	0.0401 (11)	0.0084 (10)	0.0054 (9)	-0.0034 (9)
C11	0.0580 (13)	0.0476 (13)	0.0546 (13)	0.0119 (11)	0.0107 (11)	-0.0057 (10)
C12	0.0654 (15)	0.0386 (12)	0.0813 (18)	0.0055 (11)	0.0027 (13)	-0.0045 (12)
C13	0.0574 (13)	0.0424 (12)	0.0650 (15)	-0.0040 (10)	0.0069 (12)	0.0133 (11)
C14	0.0436 (11)	0.0420 (11)	0.0480 (11)	-0.0012 (9)	0.0047 (9)	0.0015 (9)
C15	0.0420 (11)	0.0435 (11)	0.0455 (11)	-0.0007 (9)	-0.0027 (9)	-0.0001 (9)
C16	0.0459 (12)	0.0566 (14)	0.0580 (13)	0.0049 (11)	-0.0076 (10)	0.0047 (11)
C17	0.0641 (15)	0.0552 (14)	0.0580 (14)	0.0144 (12)	-0.0006 (12)	0.0184 (12)
C18	0.0455 (12)	0.0543 (14)	0.0799 (17)	0.0116 (11)	0.0081 (12)	0.0055 (13)
N1	0.0521 (10)	0.0437 (9)	0.0284 (8)	0.0141 (8)	0.0000 (7)	0.0007 (7)
O1	0.0778 (11)	0.0511 (9)	0.0297 (7)	0.0213 (8)	-0.0035 (7)	-0.0015 (6)

*Geometric parameters (Å, °)*

C1—C2	1.377 (3)	C10—H10B	0.9700
C1—C6	1.383 (3)	C11—C18	1.512 (3)
C1—N1	1.419 (2)	C11—C12	1.516 (3)
C2—C3	1.381 (3)	C11—H11	0.9800
C2—H2	0.9300	C12—C13	1.525 (3)
C3—C4	1.371 (3)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C4—C5	1.377 (3)	C13—C17	1.520 (3)
C4—C7	1.512 (3)	C13—C14	1.536 (3)
C5—C6	1.382 (3)	C13—H13	0.9800
C5—H5	0.9300	C14—H14A	0.9700
C6—H6	0.9300	C14—H14B	0.9700
C7—H7A	0.9600	C15—C16	1.530 (3)
C7—H7B	0.9600	C15—H15A	0.9700
C7—H7C	0.9600	C15—H15B	0.9700
C8—O1	1.224 (2)	C16—C17	1.517 (3)
C8—N1	1.349 (2)	C16—C18	1.518 (3)
C8—C9	1.526 (3)	C16—H16	0.9800
C9—C14	1.534 (3)	C17—H17A	0.9700
C9—C10	1.536 (3)	C17—H17B	0.9700
C9—C15	1.538 (3)	C18—H18A	0.9700
C10—C11	1.537 (3)	C18—H18B	0.9700
C10—H10A	0.9700	N1—H1	0.8600
C2—C1—C6	118.76 (19)	C11—C12—C13	109.58 (19)
C2—C1—N1	123.74 (17)	C11—C12—H12A	109.8
C6—C1—N1	117.48 (18)	C13—C12—H12A	109.8
C1—C2—C3	119.8 (2)	C11—C12—H12B	109.8
C1—C2—H2	120.1	C13—C12—H12B	109.8
C3—C2—H2	120.1	H12A—C12—H12B	108.2
C4—C3—C2	122.3 (2)	C17—C13—C12	110.1 (2)

## supplementary materials

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C4—C3—H3	118.9	C17—C13—C14	109.01 (19)
C2—C3—H3	118.9	C12—C13—C14	109.09 (19)
C3—C4—C5	117.4 (2)	C17—C13—H13	109.5
C3—C4—C7	121.5 (2)	C12—C13—H13	109.5
C5—C4—C7	121.1 (2)	C14—C13—H13	109.5
C4—C5—C6	121.6 (2)	C9—C14—C13	109.75 (17)
C4—C5—H5	119.2	C9—C14—H14A	109.7
C6—C5—H5	119.2	C13—C14—H14A	109.7
C5—C6—C1	120.2 (2)	C9—C14—H14B	109.7
C5—C6—H6	119.9	C13—C14—H14B	109.7
C1—C6—H6	119.9	H14A—C14—H14B	108.2
C4—C7—H7A	109.5	C16—C15—C9	109.99 (17)
C4—C7—H7B	109.5	C16—C15—H15A	109.7
H7A—C7—H7B	109.5	C9—C15—H15A	109.7
C4—C7—H7C	109.5	C16—C15—H15B	109.7
H7A—C7—H7C	109.5	C9—C15—H15B	109.7
H7B—C7—H7C	109.5	H15A—C15—H15B	108.2
O1—C8—N1	121.90 (18)	C17—C16—C18	110.0 (2)
O1—C8—C9	122.38 (17)	C17—C16—C15	108.61 (18)
N1—C8—C9	115.72 (15)	C18—C16—C15	109.57 (19)
C8—C9—C14	110.41 (15)	C17—C16—H16	109.5
C8—C9—C10	109.71 (15)	C18—C16—H16	109.5
C14—C9—C10	108.77 (16)	C15—C16—H16	109.5
C8—C9—C15	110.43 (16)	C16—C17—C13	110.00 (19)
C14—C9—C15	109.04 (16)	C16—C17—H17A	109.7
C10—C9—C15	108.43 (16)	C13—C17—H17A	109.7
C9—C10—C11	110.01 (17)	C16—C17—H17B	109.7
C9—C10—H10A	109.7	C13—C17—H17B	109.7
C11—C10—H10A	109.7	H17A—C17—H17B	108.2
C9—C10—H10B	109.7	C11—C18—C16	109.94 (19)
C11—C10—H10B	109.7	C11—C18—H18A	109.7
H10A—C10—H10B	108.2	C16—C18—H18A	109.7
C18—C11—C12	110.2 (2)	C11—C18—H18B	109.7
C18—C11—C10	109.11 (18)	C16—C18—H18B	109.7
C12—C11—C10	109.15 (18)	H18A—C18—H18B	108.2
C18—C11—H11	109.4	C8—N1—C1	127.93 (16)
C12—C11—H11	109.4	C8—N1—H1	116.0
C10—C11—H11	109.4	C1—N1—H1	116.0
C6—C1—C2—C3	-0.4 (3)	C11—C12—C13—C14	-61.2 (2)
N1—C1—C2—C3	177.7 (2)	C8—C9—C14—C13	-179.79 (16)
C1—C2—C3—C4	0.3 (4)	C10—C9—C14—C13	-59.4 (2)
C2—C3—C4—C5	-0.1 (4)	C15—C9—C14—C13	58.7 (2)
C2—C3—C4—C7	-179.7 (2)	C17—C13—C14—C9	-59.7 (2)
C3—C4—C5—C6	0.1 (4)	C12—C13—C14—C9	60.5 (2)
C7—C4—C5—C6	179.6 (2)	C8—C9—C15—C16	179.22 (16)
C4—C5—C6—C1	-0.2 (3)	C14—C9—C15—C16	-59.3 (2)
C2—C1—C6—C5	0.4 (3)	C10—C9—C15—C16	59.0 (2)
N1—C1—C6—C5	-177.82 (19)	C9—C15—C16—C17	60.4 (2)
O1—C8—C9—C14	114.5 (2)	C9—C15—C16—C18	-59.8 (2)



N1—C8—C9—C14	-65.3 (2)	C18—C16—C17—C13	58.4 (2)
O1—C8—C9—C10	-5.4 (3)	C15—C16—C17—C13	-61.5 (3)
N1—C8—C9—C10	174.87 (17)	C12—C13—C17—C16	-58.3 (3)
O1—C8—C9—C15	-124.8 (2)	C14—C13—C17—C16	61.3 (2)
N1—C8—C9—C15	55.4 (2)	C12—C11—C18—C16	59.4 (2)
C8—C9—C10—C11	180.00 (17)	C10—C11—C18—C16	-60.5 (2)
C14—C9—C10—C11	59.1 (2)	C17—C16—C18—C11	-58.9 (2)
C15—C9—C10—C11	-59.3 (2)	C15—C16—C18—C11	60.4 (2)
C9—C10—C11—C18	60.4 (2)	O1—C8—N1—C1	1.7 (3)
C9—C10—C11—C12	-60.1 (2)	C9—C8—N1—C1	-178.59 (18)
C18—C11—C12—C13	-59.0 (2)	C2—C1—N1—C8	28.1 (3)
C10—C11—C12—C13	60.8 (3)	C6—C1—N1—C8	-153.8 (2)
C11—C12—C13—C17	58.4 (3)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O1 <sup>i</sup>	0.86	2.12	2.962 (2)	166

Symmetry codes: (i)  $x, -y+1/2, z-1/2$ .

Fig. 1

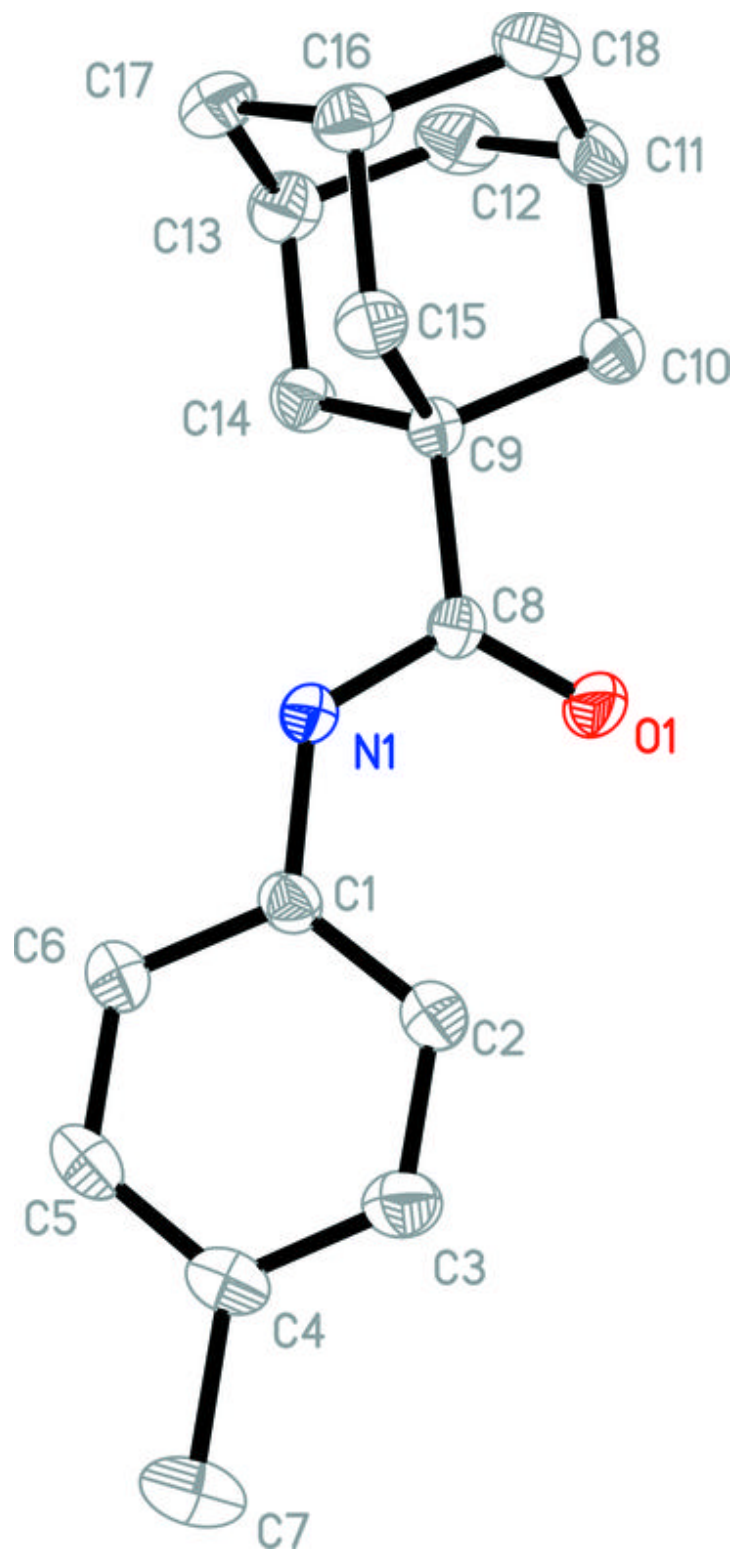


Fig. 2

