Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[(Diphenylamino)methyl]acetamide

Ganesan Venkatesa Prabhu,[a] Nagarajan Vembu,[b]* Loganathan Muruganandam[a] and Anthony Linden[c]

[a]Department of Chemistry, National Institute of Technology, Tiruchirappalli 620 015, India, [b]Department of Chemistry, Urumu Dhanalakshmi College, Tiruchirappalli 620 019, India, and [c]Institute of Organic Chemistry, University of Zurich, Winterthurerstrasse 190, CH-8057 Zurich, Switzerland
Correspondence e-mail: vembu57@yahoo.com

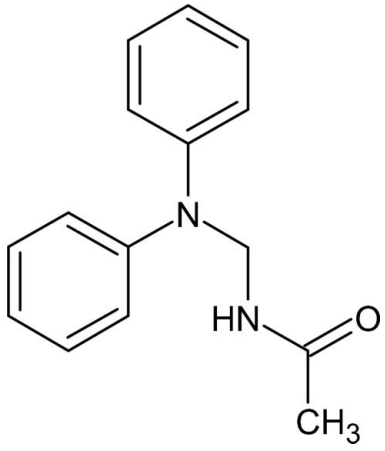
Received 18 April 2007; accepted 30 April 2007

Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.002 Å; R factor = 0.056; wR factor = 0.156; data-to-parameter ratio = 16.4.

The dihedral angles between the mean planes of the two phenyl rings for the two molecules in the asymmetric unit of the title compound, $C_{15}H_{16}N_2O$, are 55.34 (5) and 71.24 (5)°. In one of the molecules, the entire aminomethylacetamide chain is largely planar, while in the second the chain is twisted significantly at the methylene C atom. In the crystal structure, extended chains form along c through N—H···O hydrogen bonds between the amide groups. Further aggregation is completed by the presence of C—H···O and C—H···π interactions.

Related literature

For applications of diphenylamine derivatives, see Tomlin (1997), Bettaieb & Aaron (2001) and Shirota (2005). A structural isomer [$(C_6H_5)_2CHNHCH_2CONH_2$] of the title compound [$(C_6H_5)_2NCH_2NHCOCH_3$] has been reported (Mancilla *et al.*, 2003). For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$C_{15}H_{16}N_2O$
M_r = 240.30
Monoclinic, $P2_1/c$
a = 15.5339 (2) Å
b = 8.7382 (2) Å
c = 18.7931 (4) Å
β = 91.3265 (10)°
V = 2550.26 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 160 (2) K
0.33 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
76517 measured reflections
7428 independent reflections
5265 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.156
S = 1.06
7428 reflections
453 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N9—H9···O11A^{i}	0.923 (19)	2.097 (18)	2.9924 (15)	163.2 (16)
C18—H18A···O11A^{i}	0.98 (2)	2.48 (2)	3.329 (2)	145.1 (17)
N9A—H9′···O11	0.85 (2)	2.03 (2)	2.8671 (16)	169.7 (18)
C17A—H17′···O11A	1.016 (18)	2.440 (18)	3.4288 (18)	164.5 (14)
C18A—H18E···O11	0.99 (2)	2.42 (2)	3.254 (2)	141.2 (14)
C3—H3···$Cg4^{ii}$	0.95 (2)	2.89	3.68	141
C8—H8A···$Cg3^{ii}$	0.98 (2)	3.14	3.92	139
C8A—H8C···$Cg4^{iii}$	1.03 (1)	3.23	3.96	129
C14—H14···$Cg1^{iv}$	0.94 (2)	2.91	3.74	148
C16A—H16′···$Cg3^{i}$	0.97 (2)	3.19	3.97	139
C17—H17···$Cg1^{v}$	0.93 (2)	2.95	3.49	118
C18—H18C···$Cg2$	0.97 (2)	2.80	3.54	134

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x, -y+1, -z$; (v) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$. $Cg1$, $Cg2$, $Cg3$ are $Cg4$ are the centroids of rings C1–C6, C12–C17, C1A–C6A and C12A–C17A, respectively.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2298).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bettaieb, L. & Aaron, J. J. (2001). *Turk. J. Chem.* **25**, 165–171.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Mancilla, T., Zamudio-Rivera, L. S., Carrillo, L., Beltran, H. I. & Farfan, N. (2003). *ARKIVOC*, **xi**, 37–47.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shirota, Y. (2005). *J. Mater. Chem.* **15**, 75–93.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tomlin, C. D. S. (1997). *The Pesticide Manual*, 11th ed. British Crop Protection Council, Farnham, Surrey, England.

supplementary materials

Acta Cryst. (2007). E**63**, o2841-o2842 [**doi:10.1107/S1600536807021459**]

N-[(Diphenylamino)methyl]acetamide

G. V. Prabhu, N. Vembu, L. Muruganandam and A. Linden

Comment

Diphenylamine derivatives find applications in DNA detection, plant growth regulation, pesticides (Tomlin, 1997), pharmaceuticals (Bettaieb & Aaron, 2001) and materials science (Shirota, 2005). The present investigation is aimed at the study of the molecular and supramolecular architecture of the title compound, (I). This study may serve as a forerunner to a study of the correlation between the molecular and supramolecular features of this compound and its biological activity.

There are two independent molecules in the asymmetric unit (Fig. 1). The dihedral angles between the mean planes of the two phenyl rings in each independent molecule are 55.34 (5)° and 71.24 (5)°, respectively. The bond lengths are comparable to the values reported for similar compounds (Allen *et al.*, 1987). The plane defined by atoms N7, C8 and N9 is almost coplanar with the mean plane through atoms C8, N9, C10, O11 and C18 (dihedral angle is 16.27 (8)°) in molecule 1, whereas the corresponding planes in molecule 2 intersect at the much larger angle of 61.51 (8)°.

The crystal structure of (I) is stabilized by the interplay of N—H···O, C—H···O and C—H···π interactions (Table 1). The N9—H9···O11A^{i} (see Table 1 for symmetry codes) and C18—-H18A···O11A^{i} interactions constitute a pair of bifurcated acceptor bonds generating a motif of graph set (Bernstein *et al.*, 1995; Etter, 1990) $R^{1}_{2}(6)$. Similarly, the N9A—H9'···O11 and C18A—H18E···O11 interactions link the two molecules of the asymmetric unit to give a $R^{1}_{2}(6)$ motif. The N—H···O interactions generate an infinite chain of alternating symmetry-independent molecules along [001] (Fig. 2), which can be designated with the binary motif of $C^{2}_{2}(8)$. An S(8) motif is formed by the C17A—H17'···O11A interaction.

The C18—H18C···Cg2 interaction (Table 1) generates an S(7) motif. This is a special type of motif whose atom count in the pattern is assigned by taking the entire aromatic ring (C12—C17) as a single acceptor atom. The C3—H3···Cg4^{ii} and C8—H8A···Cg3^{ii} interactions together generate an $R^{2}_{2}(10)$ motif. Cg1, Cg2, Cg3 & Cg4 (Table 1) refer to the centroids of the C1—C6, C12—C17, C1A—C6A & C12A—C17A rings, respectively.

Experimental

The title compound was synthesized by a Mannich condensation reaction involving acetamide, formaldehyde and diphenylamine in a 1:1:0.5 molar ratio. Acetamide was dissolved in ethanol and, to this solution, formaldehyde was added with stirring. Diphenylamine dissolved in acetone was added in small quantities to the above mixture and stirred. After 10 days, the solid product formed was filtered and washed with distilled water, followed by small quantities of acetone. The compound was dried at 333 K and diffraction quality crystals were obtained by recrystallization from ethanol. Microelemental analysis (Calcd & exptl. values) C 75.0 & 73.01, H 6.6 & 6.19 and N 11.66 & 11.58. Mass spectrum (m/e): 240, 197, 182, 168 Molecular Weight Determination by Rast method : exptl. 246 expected 240.

Refinement

All H atoms were located in difference maps and their positions and isotropic displacement parameters were refined freely.

Figures

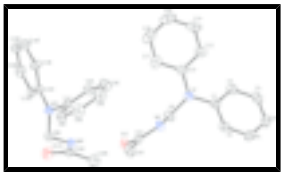


Fig. 1. The asymmetric unit of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. H-atoms are omitted for clarity.

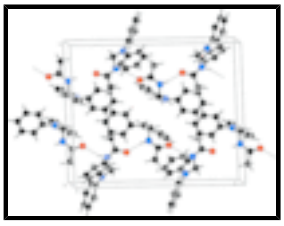


Fig. 2. View of the molecule along y-axis showing the 1D chain of N—H···O interactions along [001].

N-[(Diphenylamino)methyl]acetamide

Crystal data

$C_{15}H_{16}N_2O$	F_{000} = 1024
M_r = 240.30	D_x = 1.252 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 391-392 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 15.5339 (2) Å	Cell parameters from 7775 reflections
b = 8.7382 (2) Å	θ = 2.0–30.0º
c = 18.7931 (4) Å	μ = 0.08 mm^{-1}
β = 91.3265 (10)º	T = 160 (2) K
V = 2550.26 (9) Å^3	Prism, colourless
Z = 8	0.33 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer	7428 independent reflections
Radiation source: Nonius FR590 sealed tube generator	5265 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	R_{int} = 0.086
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 30.1º
T = 160(1) K	θ_{min} = 2.2º
φ and ω scans with κ offsets	h = −21→21
Absorption correction: none	k = −12→12

76517 measured reflections

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.156$

$S = 1.06$

7428 reflections

453 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.3971P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

Extinction correction: none

Special details

Experimental. IR spectrum in KBr : 3257 (NH stretch), 3060 (Ar CH stretch), 2366 (ring C=C), 1637 (CO stretch), 1588 (NH in plane bend), 1493 (CN stretch), 751 & 693 (mono substituted Ar ring) ^{1}H NMR in dmso-d_6 :7.3038-6.9624 (Ar), 8.4598 (NH), 1.8418 s (CH_3), 5.0 d (CH_2) & 3.3552 (water in dmso-d6) ppm ^{13}C NMR in dmso-d_6 :169.598 (CO), 146.606, 129.228, 121.715 & 120.912 (Ar), 146.606 (substituted C in ring), 56.116 (CH_2 bonded to N) & 22.572 (CH_3) ppm.

Solvent used: EtOH Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.438 (1) Frames collected: 415 Seconds exposure per frame: 36 Degrees rotation per frame: 1.9 Crystal-Detector distance (mm): 30.0

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.01832 (8)	0.29539 (15)	0.14883 (7)	0.0211 (3)
C2	−0.09719 (9)	0.36679 (17)	0.13395 (8)	0.0252 (3)
C3	−0.16398 (9)	0.28519 (18)	0.10190 (8)	0.0302 (3)
C4	−0.15435 (10)	0.13199 (19)	0.08551 (8)	0.0309 (3)
C5	−0.07661 (9)	0.06029 (17)	0.10076 (8)	0.0284 (3)
C6	−0.00904 (9)	0.14121 (16)	0.13188 (8)	0.0250 (3)
N7	0.05056 (7)	0.37343 (13)	0.18421 (6)	0.0245 (3)

C8	0.09226 (9)	0.29551 (17)	0.24309 (8)	0.0253 (3)
N9	0.17586 (7)	0.22970 (13)	0.22465 (6)	0.0218 (2)
C10	0.23004 (8)	0.18362 (15)	0.27643 (7)	0.0216 (3)
O11	0.21192 (7)	0.19959 (13)	0.33981 (5)	0.0313 (3)
C12	0.07677 (8)	0.52154 (15)	0.16468 (7)	0.0220 (3)
C13	0.05035 (9)	0.58546 (17)	0.09968 (8)	0.0269 (3)
C14	0.07814 (10)	0.72975 (18)	0.07981 (10)	0.0360 (4)
C15	0.13260 (11)	0.81400 (19)	0.12401 (11)	0.0417 (5)
C16	0.15846 (11)	0.7520 (2)	0.18810 (11)	0.0405 (4)
C17	0.13206 (10)	0.60693 (19)	0.20888 (9)	0.0314 (3)
C18	0.31169 (10)	0.10702 (19)	0.25530 (9)	0.0316 (3)
C1A	0.41376 (8)	0.47118 (14)	0.36084 (7)	0.0203 (3)
C2A	0.35280 (9)	0.52647 (16)	0.31177 (8)	0.0250 (3)
C3A	0.36483 (10)	0.50847 (18)	0.23963 (8)	0.0316 (3)
C4A	0.43723 (10)	0.43434 (19)	0.21546 (8)	0.0335 (4)
C5A	0.49697 (10)	0.37669 (19)	0.26369 (9)	0.0343 (4)
C6A	0.48578 (9)	0.39454 (17)	0.33625 (8)	0.0285 (3)
N7A	0.40232 (7)	0.49085 (12)	0.43557 (6)	0.0229 (3)
C8A	0.38415 (9)	0.35492 (15)	0.47658 (8)	0.0225 (3)
N9A	0.29531 (7)	0.30152 (13)	0.46900 (6)	0.0211 (2)
C10A	0.24228 (9)	0.28198 (15)	0.52379 (7)	0.0235 (3)
O11A	0.26156 (8)	0.31970 (13)	0.58554 (5)	0.0367 (3)
C12A	0.38663 (8)	0.63759 (15)	0.46278 (7)	0.0200 (3)
C13A	0.41939 (9)	0.76591 (16)	0.42782 (8)	0.0260 (3)
C14A	0.40855 (10)	0.91092 (17)	0.45551 (9)	0.0311 (3)
C15A	0.36551 (10)	0.93288 (17)	0.51808 (9)	0.0308 (3)
C16A	0.33210 (9)	0.80765 (17)	0.55239 (8)	0.0285 (3)
C17A	0.34189 (9)	0.66043 (16)	0.52556 (7)	0.0237 (3)
C18A	0.15771 (11)	0.2075 (2)	0.50598 (9)	0.0335 (4)
H2	−0.1035 (10)	0.473 (2)	0.1444 (9)	0.029 (4)*
H3	−0.2181 (12)	0.3335 (19)	0.0918 (9)	0.034 (4)*
H4	−0.2006 (12)	0.074 (2)	0.0649 (10)	0.041 (5)*
H5	−0.0677 (11)	−0.049 (2)	0.0879 (10)	0.040 (5)*
H6	0.0476 (10)	0.0891 (18)	0.1419 (8)	0.025 (4)*
H8A	0.0546 (11)	0.2130 (19)	0.2584 (9)	0.030 (4)*
H8B	0.1035 (10)	0.3654 (18)	0.2828 (9)	0.026 (4)*
H9	0.1913 (11)	0.2184 (19)	0.1778 (10)	0.034 (5)*
H13	0.0134 (11)	0.528 (2)	0.0675 (10)	0.036 (5)*
H14	0.0616 (14)	0.772 (2)	0.0355 (13)	0.057 (6)*
H15	0.1532 (13)	0.913 (3)	0.1108 (11)	0.058 (6)*
H16	0.1992 (14)	0.811 (2)	0.2187 (12)	0.054 (6)*
H17	0.1503 (12)	0.568 (2)	0.2528 (11)	0.045 (5)*
H18A	0.3210 (14)	0.105 (2)	0.2040 (13)	0.060 (6)*
H18B	0.3085 (15)	−0.006 (3)	0.2706 (13)	0.071 (7)*
H18C	0.3611 (13)	0.153 (2)	0.2790 (11)	0.052 (6)*
H2'	0.2999 (11)	0.577 (2)	0.3275 (9)	0.036 (4)*
H3'	0.3208 (12)	0.548 (2)	0.2048 (10)	0.045 (5)*
H4'	0.4457 (12)	0.427 (2)	0.1645 (11)	0.046 (5)*
H5'	0.5507 (12)	0.3211 (19)	0.2476 (10)	0.036 (4)*

H6'	0.5273 (11)	0.3603 (19)	0.3721 (10)	0.031 (4)*
H8C	0.4265 (9)	0.2723 (17)	0.4602 (8)	0.018 (4)*
H8D	0.3939 (9)	0.3741 (17)	0.5278 (9)	0.022 (4)*
H9'	0.2768 (12)	0.272 (2)	0.4283 (11)	0.043 (5)*
H13'	0.4509 (10)	0.7530 (18)	0.3859 (9)	0.025 (4)*
H14'	0.4322 (13)	1.002 (2)	0.4266 (11)	0.054 (6)*
H15'	0.3571 (11)	1.030 (2)	0.5386 (10)	0.038 (5)*
H16'	0.3000 (11)	0.8205 (19)	0.5954 (10)	0.033 (4)*
H17'	0.3179 (10)	0.570 (2)	0.5525 (9)	0.034 (4)*
H18D	0.1136 (15)	0.255 (3)	0.5299 (13)	0.068 (7)*
H18E	0.1435 (11)	0.206 (2)	0.4542 (11)	0.040 (5)*
H18F	0.1647 (14)	0.103 (3)	0.5248 (13)	0.071 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0203 (6)	0.0233 (7)	0.0198 (6)	−0.0002 (5)	0.0026 (5)	0.0043 (5)
C2	0.0233 (7)	0.0262 (7)	0.0261 (7)	0.0041 (5)	0.0029 (6)	0.0021 (6)
C3	0.0207 (7)	0.0413 (9)	0.0286 (8)	0.0027 (6)	0.0000 (6)	0.0071 (7)
C4	0.0279 (7)	0.0393 (8)	0.0255 (8)	−0.0107 (6)	−0.0005 (6)	0.0021 (6)
C5	0.0339 (8)	0.0255 (7)	0.0260 (7)	−0.0052 (6)	0.0047 (6)	0.0014 (6)
C6	0.0241 (7)	0.0238 (7)	0.0272 (7)	0.0022 (5)	0.0040 (6)	0.0043 (6)
N7	0.0239 (6)	0.0237 (6)	0.0258 (6)	−0.0014 (4)	−0.0042 (5)	0.0056 (5)
C8	0.0246 (7)	0.0316 (8)	0.0199 (7)	0.0026 (6)	0.0022 (5)	0.0040 (6)
N9	0.0240 (6)	0.0267 (6)	0.0145 (6)	0.0011 (5)	−0.0010 (4)	0.0003 (5)
C10	0.0240 (6)	0.0214 (6)	0.0192 (6)	−0.0057 (5)	−0.0038 (5)	0.0015 (5)
O11	0.0319 (5)	0.0452 (6)	0.0168 (5)	−0.0048 (5)	−0.0036 (4)	0.0028 (4)
C12	0.0211 (6)	0.0213 (6)	0.0239 (7)	0.0032 (5)	0.0064 (5)	−0.0013 (5)
C13	0.0283 (7)	0.0250 (7)	0.0276 (8)	0.0038 (6)	0.0063 (6)	0.0020 (6)
C14	0.0334 (8)	0.0280 (8)	0.0473 (10)	0.0088 (6)	0.0152 (8)	0.0124 (7)
C15	0.0350 (9)	0.0220 (8)	0.0693 (13)	0.0015 (7)	0.0237 (9)	0.0005 (8)
C16	0.0318 (8)	0.0341 (9)	0.0563 (12)	−0.0088 (7)	0.0144 (8)	−0.0161 (8)
C17	0.0273 (7)	0.0372 (8)	0.0299 (8)	−0.0038 (6)	0.0057 (6)	−0.0069 (7)
C18	0.0254 (7)	0.0361 (9)	0.0331 (9)	0.0034 (6)	−0.0051 (6)	0.0023 (7)
C1A	0.0219 (6)	0.0187 (6)	0.0204 (6)	−0.0026 (5)	0.0034 (5)	−0.0004 (5)
C2A	0.0246 (7)	0.0267 (7)	0.0238 (7)	0.0054 (6)	0.0047 (5)	0.0016 (6)
C3A	0.0350 (8)	0.0367 (8)	0.0233 (7)	0.0047 (6)	0.0022 (6)	0.0056 (6)
C4A	0.0392 (8)	0.0389 (9)	0.0230 (7)	0.0013 (7)	0.0103 (6)	−0.0019 (7)
C5A	0.0274 (7)	0.0407 (9)	0.0353 (9)	0.0040 (7)	0.0091 (7)	−0.0077 (7)
C6A	0.0224 (7)	0.0319 (8)	0.0312 (8)	0.0031 (6)	−0.0013 (6)	−0.0015 (6)
N7A	0.0310 (6)	0.0179 (5)	0.0199 (6)	−0.0013 (4)	0.0027 (5)	0.0012 (4)
C8A	0.0266 (7)	0.0203 (6)	0.0204 (7)	−0.0006 (5)	−0.0035 (5)	0.0039 (5)
N9A	0.0264 (6)	0.0221 (6)	0.0150 (6)	−0.0025 (4)	−0.0006 (5)	0.0003 (4)
C10A	0.0327 (7)	0.0204 (6)	0.0174 (7)	−0.0010 (5)	0.0024 (5)	0.0026 (5)
O11A	0.0500 (7)	0.0442 (7)	0.0160 (5)	−0.0127 (5)	0.0043 (5)	−0.0013 (5)
C12A	0.0187 (6)	0.0212 (6)	0.0199 (6)	−0.0018 (5)	−0.0028 (5)	−0.0001 (5)
C13A	0.0283 (7)	0.0237 (7)	0.0263 (7)	−0.0037 (6)	0.0046 (6)	0.0005 (6)
C14A	0.0357 (8)	0.0216 (7)	0.0360 (8)	−0.0048 (6)	0.0014 (7)	0.0016 (6)

C15A	0.0353 (8)	0.0232 (7)	0.0336 (8)	0.0020 (6)	−0.0037 (6)	−0.0056 (6)
C16A	0.0292 (7)	0.0337 (8)	0.0227 (7)	−0.0012 (6)	−0.0005 (6)	−0.0072 (6)
C17A	0.0250 (7)	0.0270 (7)	0.0192 (7)	−0.0050 (6)	−0.0015 (5)	−0.0012 (6)
C18A	0.0327 (8)	0.0416 (9)	0.0263 (8)	−0.0077 (7)	0.0060 (7)	0.0018 (7)

Geometric parameters (Å, °)

C1—C6	1.3926 (19)	C1A—C6A	1.3919 (19)
C1—C2	1.3974 (19)	C1A—C2A	1.393 (2)
C1—N7	1.4208 (18)	C1A—N7A	1.4301 (17)
C2—C3	1.385 (2)	C2A—C3A	1.382 (2)
C2—H2	0.956 (17)	C2A—H2'	0.984 (18)
C3—C4	1.383 (2)	C3A—C4A	1.384 (2)
C3—H3	0.955 (18)	C3A—H3'	1.00 (2)
C4—C5	1.385 (2)	C4A—C5A	1.377 (2)
C4—H4	0.952 (19)	C4A—H4'	0.97 (2)
C5—C6	1.384 (2)	C5A—C6A	1.388 (2)
C5—H5	0.996 (18)	C5A—H5'	1.017 (18)
C6—H6	1.005 (16)	C6A—H6'	0.969 (18)
N7—C12	1.4080 (17)	N7A—C12A	1.4039 (17)
N7—C8	1.4403 (18)	N7A—C8A	1.4473 (17)
C8—N9	1.4690 (18)	C8A—N9A	1.4606 (18)
C8—H8A	0.976 (17)	C8A—H8C	1.028 (15)
C8—H8B	0.977 (17)	C8A—H8D	0.986 (16)
N9—C10	1.3339 (18)	N9A—C10A	1.3443 (17)
N9—H9	0.923 (19)	N9A—H9'	0.85 (2)
C10—O11	1.2381 (16)	C10A—O11A	1.2365 (17)
C10—C18	1.496 (2)	C10A—C18A	1.497 (2)
C12—C13	1.396 (2)	C12A—C17A	1.3974 (19)
C12—C17	1.397 (2)	C12A—C13A	1.4013 (19)
C13—C14	1.387 (2)	C13A—C14A	1.382 (2)
C13—H13	0.967 (19)	C13A—H13'	0.944 (16)
C14—C15	1.384 (3)	C14A—C15A	1.379 (2)
C14—H14	0.94 (2)	C14A—H14'	1.04 (2)
C15—C16	1.372 (3)	C15A—C16A	1.378 (2)
C15—H15	0.96 (2)	C15A—H15'	0.942 (18)
C16—C17	1.391 (2)	C16A—C17A	1.391 (2)
C16—H16	0.99 (2)	C16A—H16'	0.966 (18)
C17—H17	0.93 (2)	C17A—H17'	1.016 (18)
C18—H18A	0.98 (2)	C18A—H18D	0.93 (2)
C18—H18B	1.03 (2)	C18A—H18E	0.99 (2)
C18—H18C	0.97 (2)	C18A—H18F	0.99 (3)
C6—C1—C2	118.76 (13)	C6A—C1A—C2A	119.11 (13)
C6—C1—N7	119.38 (12)	C6A—C1A—N7A	120.19 (13)
C2—C1—N7	121.77 (12)	C2A—C1A—N7A	120.70 (12)
C3—C2—C1	120.11 (13)	C3A—C2A—C1A	120.32 (13)
C3—C2—H2	120.7 (10)	C3A—C2A—H2'	118.6 (10)
C1—C2—H2	119.2 (10)	C1A—C2A—H2'	121.1 (10)
C4—C3—C2	120.77 (14)	C2A—C3A—C4A	120.32 (15)

C4—C3—H3	118.8 (10)	C2A—C3A—H3'	119.9 (11)
C2—C3—H3	120.4 (10)	C4A—C3A—H3'	119.7 (11)
C3—C4—C5	119.36 (14)	C5A—C4A—C3A	119.70 (14)
C3—C4—H4	121.2 (11)	C5A—C4A—H4'	121.2 (11)
C5—C4—H4	119.4 (11)	C3A—C4A—H4'	119.1 (11)
C6—C5—C4	120.37 (14)	C4A—C5A—C6A	120.52 (14)
C6—C5—H5	119.0 (10)	C4A—C5A—H5'	121.6 (10)
C4—C5—H5	120.6 (10)	C6A—C5A—H5'	117.9 (11)
C5—C6—C1	120.63 (13)	C5A—C6A—C1A	120.01 (14)
C5—C6—H6	120.0 (9)	C5A—C6A—H6'	123.6 (10)
C1—C6—H6	119.3 (9)	C1A—C6A—H6'	116.4 (10)
C12—N7—C1	122.57 (11)	C12A—N7A—C1A	119.61 (11)
C12—N7—C8	120.47 (12)	C12A—N7A—C8A	121.19 (11)
C1—N7—C8	116.94 (11)	C1A—N7A—C8A	117.04 (11)
N7—C8—N9	112.84 (11)	N7A—C8A—N9A	113.93 (11)
N7—C8—H8A	108.2 (10)	N7A—C8A—H8C	106.4 (8)
N9—C8—H8A	108.7 (9)	N9A—C8A—H8C	110.8 (8)
N7—C8—H8B	111.1 (9)	N7A—C8A—H8D	110.7 (9)
N9—C8—H8B	106.3 (9)	N9A—C8A—H8D	105.8 (9)
H8A—C8—H8B	109.6 (13)	H8C—C8A—H8D	109.1 (12)
C10—N9—C8	119.49 (12)	C10A—N9A—C8A	124.07 (12)
C10—N9—H9	119.4 (11)	C10A—N9A—H9'	116.7 (13)
C8—N9—H9	121.1 (11)	C8A—N9A—H9'	118.9 (13)
O11—C10—N9	120.94 (13)	O11A—C10A—N9A	123.07 (13)
O11—C10—C18	121.25 (13)	O11A—C10A—C18A	121.19 (13)
N9—C10—C18	117.76 (13)	N9A—C10A—C18A	115.73 (13)
C13—C12—C17	118.09 (13)	C17A—C12A—C13A	118.34 (12)
C13—C12—N7	120.99 (13)	C17A—C12A—N7A	122.14 (12)
C17—C12—N7	120.90 (13)	C13A—C12A—N7A	119.47 (12)
C14—C13—C12	120.82 (15)	C14A—C13A—C12A	120.56 (13)
C14—C13—H13	119.4 (10)	C14A—C13A—H13'	119.6 (10)
C12—C13—H13	119.8 (10)	C12A—C13A—H13'	119.8 (10)
C15—C14—C13	120.79 (17)	C15A—C14A—C13A	120.96 (14)
C15—C14—H14	118.2 (13)	C15A—C14A—H14'	121.5 (11)
C13—C14—H14	121.0 (13)	C13A—C14A—H14'	117.5 (11)
C16—C15—C14	118.65 (15)	C16A—C15A—C14A	118.93 (14)
C16—C15—H15	119.4 (13)	C16A—C15A—H15'	117.8 (11)
C14—C15—H15	121.9 (13)	C14A—C15A—H15'	123.3 (11)
C15—C16—C17	121.59 (17)	C15A—C16A—C17A	121.29 (14)
C15—C16—H16	118.3 (12)	C15A—C16A—H16'	120.4 (10)
C17—C16—H16	120.1 (12)	C17A—C16A—H16'	118.3 (10)
C16—C17—C12	120.06 (16)	C16A—C17A—C12A	119.91 (13)
C16—C17—H17	119.9 (12)	C16A—C17A—H17'	119.7 (10)
C12—C17—H17	120.0 (12)	C12A—C17A—H17'	120.3 (10)
C10—C18—H18A	114.5 (13)	C10A—C18A—H18D	110.5 (15)
C10—C18—H18B	108.0 (13)	C10A—C18A—H18E	113.5 (10)
H18A—C18—H18B	105.4 (18)	H18D—C18A—H18E	109.5 (18)
C10—C18—H18C	111.0 (12)	C10A—C18A—H18F	103.6 (13)
H18A—C18—H18C	109.1 (17)	H18D—C18A—H18F	109 (2)

H18B—C18—H18C	108.4 (18)	H18E—C18A—H18F	110.8 (17)
C6—C1—C2—C3	−1.0 (2)	C6A—C1A—C2A—C3A	−1.5 (2)
N7—C1—C2—C3	−177.43 (13)	N7A—C1A—C2A—C3A	179.28 (13)
C1—C2—C3—C4	1.3 (2)	C1A—C2A—C3A—C4A	0.5 (2)
C2—C3—C4—C5	−0.7 (2)	C2A—C3A—C4A—C5A	0.8 (2)
C3—C4—C5—C6	−0.2 (2)	C3A—C4A—C5A—C6A	−1.0 (2)
C4—C5—C6—C1	0.5 (2)	C4A—C5A—C6A—C1A	0.0 (2)
C2—C1—C6—C5	0.1 (2)	C2A—C1A—C6A—C5A	1.3 (2)
N7—C1—C6—C5	176.64 (12)	N7A—C1A—C6A—C5A	−179.52 (13)
C6—C1—N7—C12	136.58 (13)	C6A—C1A—N7A—C12A	127.70 (14)
C2—C1—N7—C12	−47.00 (18)	C2A—C1A—N7A—C12A	−53.09 (17)
C6—C1—N7—C8	−44.83 (17)	C6A—C1A—N7A—C8A	−68.79 (16)
C2—C1—N7—C8	131.58 (13)	C2A—C1A—N7A—C8A	110.42 (14)
C12—N7—C8—N9	−78.30 (16)	C12A—N7A—C8A—N9A	86.00 (15)
C1—N7—C8—N9	103.09 (14)	C1A—N7A—C8A—N9A	−77.22 (15)
N7—C8—N9—C10	165.61 (12)	N7A—C8A—N9A—C10A	−123.28 (14)
C8—N9—C10—O11	−2.1 (2)	C8A—N9A—C10A—O11A	6.2 (2)
C8—N9—C10—C18	175.69 (13)	C8A—N9A—C10A—C18A	−172.44 (13)
C1—N7—C12—C13	−15.10 (19)	C1A—N7A—C12A—C17A	153.53 (13)
C8—N7—C12—C13	166.36 (12)	C8A—N7A—C12A—C17A	−9.28 (19)
C1—N7—C12—C17	166.83 (12)	C1A—N7A—C12A—C13A	−28.97 (18)
C8—N7—C12—C17	−11.70 (19)	C8A—N7A—C12A—C13A	168.23 (13)
C17—C12—C13—C14	−0.2 (2)	C17A—C12A—C13A—C14A	0.9 (2)
N7—C12—C13—C14	−178.29 (12)	N7A—C12A—C13A—C14A	−176.72 (13)
C12—C13—C14—C15	−0.1 (2)	C12A—C13A—C14A—C15A	0.1 (2)
C13—C14—C15—C16	−0.2 (2)	C13A—C14A—C15A—C16A	−0.9 (2)
C14—C15—C16—C17	0.8 (2)	C14A—C15A—C16A—C17A	0.8 (2)
C15—C16—C17—C12	−1.1 (2)	C15A—C16A—C17A—C12A	0.3 (2)
C13—C12—C17—C16	0.7 (2)	C13A—C12A—C17A—C16A	−1.1 (2)
N7—C12—C17—C16	178.87 (13)	N7A—C12A—C17A—C16A	176.48 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O11A^{i}	0.923 (19)	2.097 (18)	2.9924 (15)	163.2 (16)
C18—H18A···O11A^{i}	0.98 (2)	2.48 (2)	3.329 (2)	145.1 (17)
N9A—H9'···O11	0.85 (2)	2.03 (2)	2.8671 (16)	169.7 (18)
C17A—H17'···O11A	1.016 (18)	2.440 (18)	3.4288 (18)	164.5 (14)
C18A—H18E···O11	0.99 (2)	2.42 (2)	3.254 (2)	141.2 (14)
C3—H3···Cg4^{ii}	0.95 (2)	2.89	3.68	141
C8—H8A···Cg3^{ii}	0.98 (2)	3.14	3.92	139
C8A—H8C···Cg4^{iii}	1.03 (1)	3.23	3.96	129
C14—H14···Cg1^{iv}	0.94 (2)	2.91	3.74	148
C16A—H16'···Cg3^{i}	0.97 (2)	3.19	3.97	139
C17—H17···Cg1^{v}	0.93 (2)	2.95	3.49	118
C18—H18C···Cg2	0.97 (2)	2.80	3.54	134

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*, −*y*+1, −*z*; (v) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

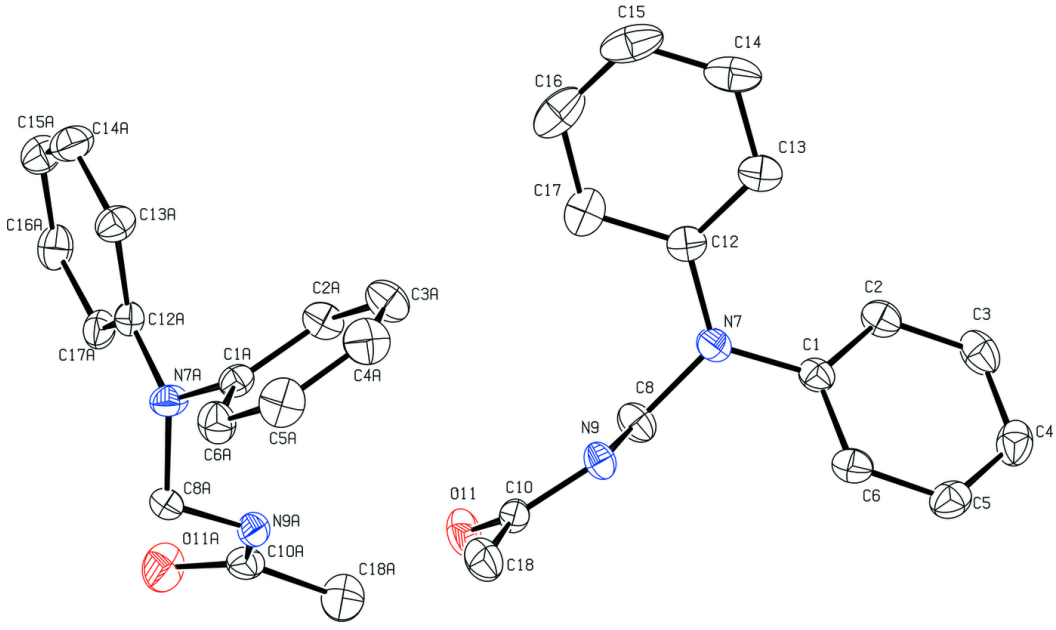

C15A
C14A
C16A
C13A
C12A
C17A
N7A
C1A
C2A
C3A
C4A
C5A
C6A
C8A
N9A
O11A
C10A
C18A
C15
C14
C16
C13
C17
C12
N7
C1
C2
C3
C8
N9
C4
O11
C10
C6
C5
C18

Fig. 2