Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-*p*-Tolyl-*N*-[*N*-(*p*-tolyl)carbamoyl]-acetamide

Ming Zhong[a] and Sihui Long[b]*

[a]Department of Chemistry, Xinyang Vocational and Technical College, Xinyang, Henan 464000, People's Republic of China, and [b]Department of Pharmaceutical Sciences, University of Kentucky, Lexington, KY 40506-0082, USA
Correspondence e-mail: slong0@uky.edu

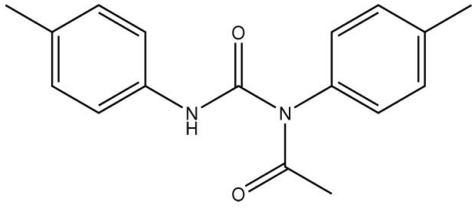
Received 1 July 2007; accepted 3 July 2007

Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.140; data-to-parameter ratio = 17.3.

The title compound, $C_{17}H_{18}N_2O_2$, was obtained by reacting *N*,*N*′-diphenyl-1*H*-imidazole-1-carboximidamide with *tert*-butyl acetate. There is an intramolecular *S*(6) hydrogen-bonded loop formed between the urea NH group and the acetyl O atom. While the other potential hydrogen-bond acceptor, the carbonyl O atom of urea, does not participate in any hydrogen bonds, there are short contacts between each molecule and four adjacent molecules, indicating that they contribute to the stabilization of the crystal structure.

Related literature

Synthesis: Mu *et al.* (2006); Smith *et al.* (1958); Zetzsche *et al.* (1938). Geometry: Allen *et al.* (1987); Etter (1990).



Experimental

Crystal data

$C_{17}H_{18}N_2O_2$
M_r = 282.33
Triclinic, $P\overline{1}$
a = 8.2562 (3) Å
b = 9.5498 (3) Å
c = 10.4583 (4) Å
α = 65.0882 (16)°
β = 83.8718 (16)°
γ = 76.5805 (16)°
V = 727.41 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 90 (2) K
0.30 × 0.30 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.975, T_{max} = 0.992
6620 measured reflections
3345 independent reflections
1857 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 0.97
3345 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.88	1.90	2.6167 (19)	137

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

SL is grateful to Dr Sean Parkin for providing support and laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2145).

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.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Mu, X., Zou, J., Qian, Q. & Zhang, W. (2006). *Tetrahedron Lett.* **47**, 2323–2325.
Nonius (2002). *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. (1995). *XP* in *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Smith, M., Moffatt, J. G. & Khorana, H. G. (1958). *J. Am. Chem. Soc.* **80**, 6204–12.
Zetzsche, F., Luscher, E. & Meyer, H. E. (1938). *Ber. Dtsch. Chem. Ges. Abt. B Abh.* **71**, 1088–1093.

 doi:10.1107/S1600536807032394

supplementary materials

Acta Cryst. (2007). E**63**, o3405 [**doi:10.1107/S1600536807032394**]

N-*p*-Tolyl-*N*-[*N*-(*p*-tolyl)carbamoyl]acetamide

M. Zhong and S. Long

Comment

N-acetylureas, including *N*-*p*-tolyl-*N*-(*p*-tolylcarbamoyl)acetamide (I), were first synthesized by reacting AcOH with carbodiimides (Zetzsche *et al.*, 1938; Smith *et al.*, 1958). In 2006, a new method to prepare *N*-acetylureas was reported by Mu *et al.* (2006)·The structures of two *N*-acetylureas including the title compound were confirmed by X-ray crystallography analysis, but no details of the structures were given. Here, we report the crystal structure of I obtained by a different synthesis method.

The asymmetric unit of (I), (Fig. 1), contains one molecule and the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the crystal structure, an S(6) hydrogen bond forms between the urea NH and the O of the acetyl group (Etter, 1990). The other hydrogen bond acceptor, the carbonyl O of the urea, does not participate in hydrogen bonds. Although no intermolecular hydrogen bonds are discovered in the crystal structure, short contacts exist between molecules. Each molecule is in short contact with four adjacent molecules, suggesting that weak interactions stablize the crystal structure.

Experimental

A saturated solution was prepared by dissolving 20 mg of *N*,*N*'-diphenyl-1*H*-imidazole-1-carboximidamide in 5 ml of *tert*-butyl acetate at room temperature. The resulted solution was set for crystal growth by slow evaporation. Single crystals of the title compound were obtained in a week.

Figures

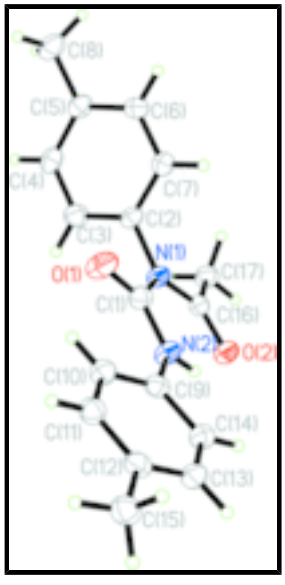


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

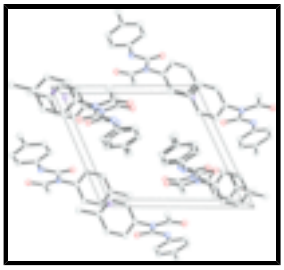


Fig. 2. A packing diagram of (I) viewed along the *a* axis.

N-*p*-Tolyl-N-[N-(*p*-tolyl)carbamoyl]acetamide

Crystal data

$C_{17}H_{18}N_2O_2$	$Z = 2$
$M_r = 282.33$	$F_{000} = 300$
Triclinic, $P\overline{1}$	$D_x = 1.289$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.2562$ (3) Å	Cell parameters from 3311 reflections
$b = 9.5498$ (3) Å	$\theta = 1$–27.5º
$c = 10.4583$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 65.0882$ (16)º	$T = 90$ (2) K
$\beta = 83.8718$ (16)º	Colourless, block
$\gamma = 76.5805$ (16)º	0.30 × 0.30 × 0.10 mm
$V = 727.41$ (4) Å^3	

Data collection

Nonius KappaCCD diffractometer	3345 independent reflections
Radiation source: fine-focus sealed tube	1857 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.056$
Detector resolution: 18 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 90(2)$ K	$\theta_{min} = 2.2$º
ω scans at fixed $\chi = 55°$	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$k = -12 \rightarrow 12$
$T_{min} = 0.975$, $T_{max} = 0.992$	$l = -13 \rightarrow 13$
6620 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
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.140$	$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
3345 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
193 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.46825 (18)	0.80888 (17)	0.72922 (15)	0.0237 (4)
N2	0.73670 (18)	0.70652 (17)	0.66213 (15)	0.0261 (4)
H2	0.6883	0.7319	0.5821	0.031*
O1	0.68725 (16)	0.70943 (16)	0.87994 (14)	0.0378 (4)
O2	0.47319 (15)	0.83026 (14)	0.50216 (12)	0.0289 (3)
C1	0.6403 (2)	0.7371 (2)	0.76391 (19)	0.0259 (4)
C2	0.3756 (2)	0.8486 (2)	0.84037 (18)	0.0241 (4)
C3	0.2991 (2)	0.7396 (2)	0.94701 (18)	0.0291 (5)
H3	0.3013	0.6400	0.9463	0.035*
C4	0.2186 (2)	0.7773 (2)	1.05552 (18)	0.0306 (5)
H4	0.1646	0.7030	1.1284	0.037*
C5	0.2156 (2)	0.9213 (2)	1.05963 (18)	0.0277 (5)
C6	0.2924 (2)	1.0291 (2)	0.95016 (19)	0.0302 (5)
H6	0.2905	1.1288	0.9504	0.036*
C7	0.3719 (2)	0.9935 (2)	0.84048 (18)	0.0286 (5)
H7	0.4234	1.0687	0.7660	0.034*
C8	0.1338 (2)	0.9583 (2)	1.18071 (18)	0.0360 (5)
H8A	0.2054	0.9007	1.2636	0.054*
H8B	0.0260	0.9264	1.2026	0.054*
H8C	0.1168	1.0720	1.1547	0.054*
C9	0.9093 (2)	0.6371 (2)	0.67291 (18)	0.0244 (4)
C10	0.9928 (2)	0.5569 (2)	0.79996 (19)	0.0296 (5)
H10	0.9353	0.5492	0.8856	0.035*
C11	1.1606 (2)	0.4881 (2)	0.80180 (19)	0.0280 (5)
H11	1.2159	0.4325	0.8898	0.034*
C12	1.2499 (2)	0.4976 (2)	0.68017 (19)	0.0269 (4)
C13	1.1649 (2)	0.5796 (2)	0.55315 (19)	0.0287 (5)

H13	1.2233	0.5888	0.4676	0.034*
C14	0.9967 (2)	0.6482 (2)	0.54892 (19)	0.0279 (4)
H14	0.9411	0.7029	0.4611	0.033*
C15	1.4328 (2)	0.4226 (2)	0.6862 (2)	0.0332 (5)
H15A	1.4985	0.5049	0.6534	0.050*
H15B	1.4559	0.3650	0.6257	0.050*
H15C	1.4627	0.3493	0.7836	0.050*
C16	0.3945 (2)	0.85026 (19)	0.60228 (18)	0.0243 (4)
C17	0.2113 (2)	0.9195 (2)	0.59209 (19)	0.0298 (5)
H17A	0.1730	0.9457	0.4976	0.045*
H17B	0.1897	1.0153	0.6095	0.045*
H17C	0.1515	0.8426	0.6625	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0216 (9)	0.0285 (9)	0.0222 (8)	−0.0046 (7)	0.0026 (7)	−0.0125 (7)
N2	0.0225 (9)	0.0343 (9)	0.0241 (8)	−0.0055 (7)	0.0021 (7)	−0.0152 (7)
O1	0.0325 (8)	0.0508 (9)	0.0318 (8)	0.0018 (7)	−0.0031 (6)	−0.0233 (7)
O2	0.0289 (8)	0.0339 (8)	0.0248 (7)	−0.0057 (6)	0.0046 (6)	−0.0146 (6)
C1	0.0251 (11)	0.0275 (10)	0.0274 (10)	−0.0086 (8)	0.0040 (8)	−0.0130 (9)
C2	0.0217 (10)	0.0298 (10)	0.0221 (10)	−0.0040 (8)	0.0035 (8)	−0.0133 (8)
C3	0.0295 (11)	0.0296 (11)	0.0313 (11)	−0.0098 (9)	0.0029 (9)	−0.0145 (9)
C4	0.0296 (11)	0.0362 (12)	0.0236 (10)	−0.0114 (9)	0.0056 (9)	−0.0090 (9)
C5	0.0219 (10)	0.0343 (11)	0.0267 (10)	−0.0036 (8)	0.0025 (8)	−0.0142 (9)
C6	0.0354 (12)	0.0277 (11)	0.0306 (11)	−0.0057 (9)	0.0042 (9)	−0.0162 (9)
C7	0.0320 (11)	0.0274 (11)	0.0252 (10)	−0.0080 (9)	0.0048 (8)	−0.0099 (9)
C8	0.0330 (12)	0.0450 (13)	0.0291 (11)	−0.0044 (10)	0.0056 (9)	−0.0175 (10)
C9	0.0220 (10)	0.0224 (10)	0.0299 (11)	−0.0043 (8)	0.0019 (8)	−0.0126 (8)
C10	0.0306 (12)	0.0288 (11)	0.0291 (11)	−0.0065 (9)	0.0045 (9)	−0.0125 (9)
C11	0.0284 (11)	0.0264 (10)	0.0283 (10)	−0.0038 (9)	−0.0025 (9)	−0.0108 (9)
C12	0.0258 (11)	0.0219 (10)	0.0345 (11)	−0.0057 (8)	0.0031 (9)	−0.0133 (9)
C13	0.0277 (11)	0.0292 (11)	0.0290 (11)	−0.0064 (9)	0.0071 (9)	−0.0132 (9)
C14	0.0281 (11)	0.0281 (10)	0.0268 (10)	−0.0045 (9)	0.0016 (8)	−0.0118 (9)
C15	0.0296 (12)	0.0304 (11)	0.0374 (12)	−0.0046 (9)	0.0032 (9)	−0.0135 (9)
C16	0.0289 (11)	0.0204 (10)	0.0242 (10)	−0.0085 (8)	0.0026 (8)	−0.0088 (8)
C17	0.0285 (11)	0.0316 (11)	0.0282 (10)	−0.0023 (9)	−0.0001 (8)	−0.0135 (9)

Geometric parameters (Å, °)

N1—C16	1.381 (2)	C8—H8B	0.9800
N1—C1	1.438 (2)	C8—H8C	0.9800
N1—C2	1.458 (2)	C9—C10	1.386 (2)
N2—C1	1.351 (2)	C9—C14	1.390 (2)
N2—C9	1.420 (2)	C10—C11	1.387 (3)
N2—H2	0.8800	C10—H10	0.9500
O1—C1	1.215 (2)	C11—C12	1.380 (2)
O2—C16	1.233 (2)	C11—H11	0.9500
C2—C7	1.378 (2)	C12—C13	1.392 (2)

C2—C3	1.380 (2)	C12—C15	1.508 (2)
C3—C4	1.390 (2)	C13—C14	1.388 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.388 (2)	C14—H14	0.9500
C4—H4	0.9500	C15—H15A	0.9800
C5—C6	1.389 (2)	C15—H15B	0.9800
C5—C8	1.506 (2)	C15—H15C	0.9800
C6—C7	1.387 (2)	C16—C17	1.499 (2)
C6—H6	0.9500	C17—H17A	0.9800
C7—H7	0.9500	C17—H17B	0.9800
C8—H8A	0.9800	C17—H17C	0.9800
C16—N1—C1	126.12 (14)	C10—C9—C14	118.99 (16)
C16—N1—C2	120.99 (14)	C10—C9—N2	123.41 (16)
C1—N1—C2	112.70 (13)	C14—C9—N2	117.59 (16)
C1—N2—C9	125.73 (16)	C9—C10—C11	119.83 (17)
C1—N2—H2	117.1	C9—C10—H10	120.1
C9—N2—H2	117.1	C11—C10—H10	120.1
O1—C1—N2	125.47 (17)	C12—C11—C10	122.23 (17)
O1—C1—N1	118.43 (15)	C12—C11—H11	118.9
N2—C1—N1	116.09 (16)	C10—C11—H11	118.9
C7—C2—C3	120.65 (16)	C11—C12—C13	117.36 (16)
C7—C2—N1	119.03 (15)	C11—C12—C15	120.70 (17)
C3—C2—N1	120.27 (15)	C13—C12—C15	121.94 (17)
C2—C3—C4	119.17 (17)	C14—C13—C12	121.40 (17)
C2—C3—H3	120.4	C14—C13—H13	119.3
C4—C3—H3	120.4	C12—C13—H13	119.3
C5—C4—C3	121.39 (17)	C13—C14—C9	120.19 (18)
C5—C4—H4	119.3	C13—C14—H14	119.9
C3—C4—H4	119.3	C9—C14—H14	119.9
C4—C5—C6	118.11 (16)	C12—C15—H15A	109.5
C4—C5—C8	120.90 (17)	C12—C15—H15B	109.5
C6—C5—C8	120.97 (17)	H15A—C15—H15B	109.5
C7—C6—C5	121.11 (17)	C12—C15—H15C	109.5
C7—C6—H6	119.4	H15A—C15—H15C	109.5
C5—C6—H6	119.4	H15B—C15—H15C	109.5
C2—C7—C6	119.56 (17)	O2—C16—N1	122.47 (16)
C2—C7—H7	120.2	O2—C16—C17	120.98 (16)
C6—C7—H7	120.2	N1—C16—C17	116.54 (15)
C5—C8—H8A	109.5	C16—C17—H17A	109.5
C5—C8—H8B	109.5	C16—C17—H17B	109.5
H8A—C8—H8B	109.5	H17A—C17—H17B	109.5
C5—C8—H8C	109.5	C16—C17—H17C	109.5
H8A—C8—H8C	109.5	H17A—C17—H17C	109.5
H8B—C8—H8C	109.5	H17B—C17—H17C	109.5
C9—N2—C1—O1	−0.1 (3)	N1—C2—C7—C6	−176.25 (16)
C9—N2—C1—N1	179.67 (15)	C5—C6—C7—C2	−0.4 (3)
C16—N1—C1—O1	179.03 (16)	C1—N2—C9—C10	16.3 (3)
C2—N1—C1—O1	4.0 (2)	C1—N2—C9—C14	−165.16 (16)

C16—N1—C1—N2	−0.7 (2)	C14—C9—C10—C11	−0.6 (3)
C2—N1—C1—N2	−175.77 (14)	N2—C9—C10—C11	177.90 (15)
C16—N1—C2—C7	−87.1 (2)	C9—C10—C11—C12	0.7 (3)
C1—N1—C2—C7	88.2 (2)	C10—C11—C12—C13	−0.1 (3)
C16—N1—C2—C3	95.6 (2)	C10—C11—C12—C15	179.34 (16)
C1—N1—C2—C3	−89.01 (19)	C11—C12—C13—C14	−0.4 (3)
C7—C2—C3—C4	−0.4 (3)	C15—C12—C13—C14	−179.92 (16)
N1—C2—C3—C4	176.79 (15)	C12—C13—C14—C9	0.5 (3)
C2—C3—C4—C5	−0.8 (3)	C10—C9—C14—C13	0.0 (3)
C3—C4—C5—C6	1.4 (3)	N2—C9—C14—C13	−178.57 (16)
C3—C4—C5—C8	−177.59 (17)	C1—N1—C16—O2	−0.9 (3)
C4—C5—C6—C7	−0.8 (3)	C2—N1—C16—O2	173.77 (15)
C8—C5—C6—C7	178.18 (17)	C1—N1—C16—C17	178.22 (15)
C3—C2—C7—C6	1.0 (3)	C2—N1—C16—C17	−7.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.88	1.90	2.6167 (19)	137

Fig. 1

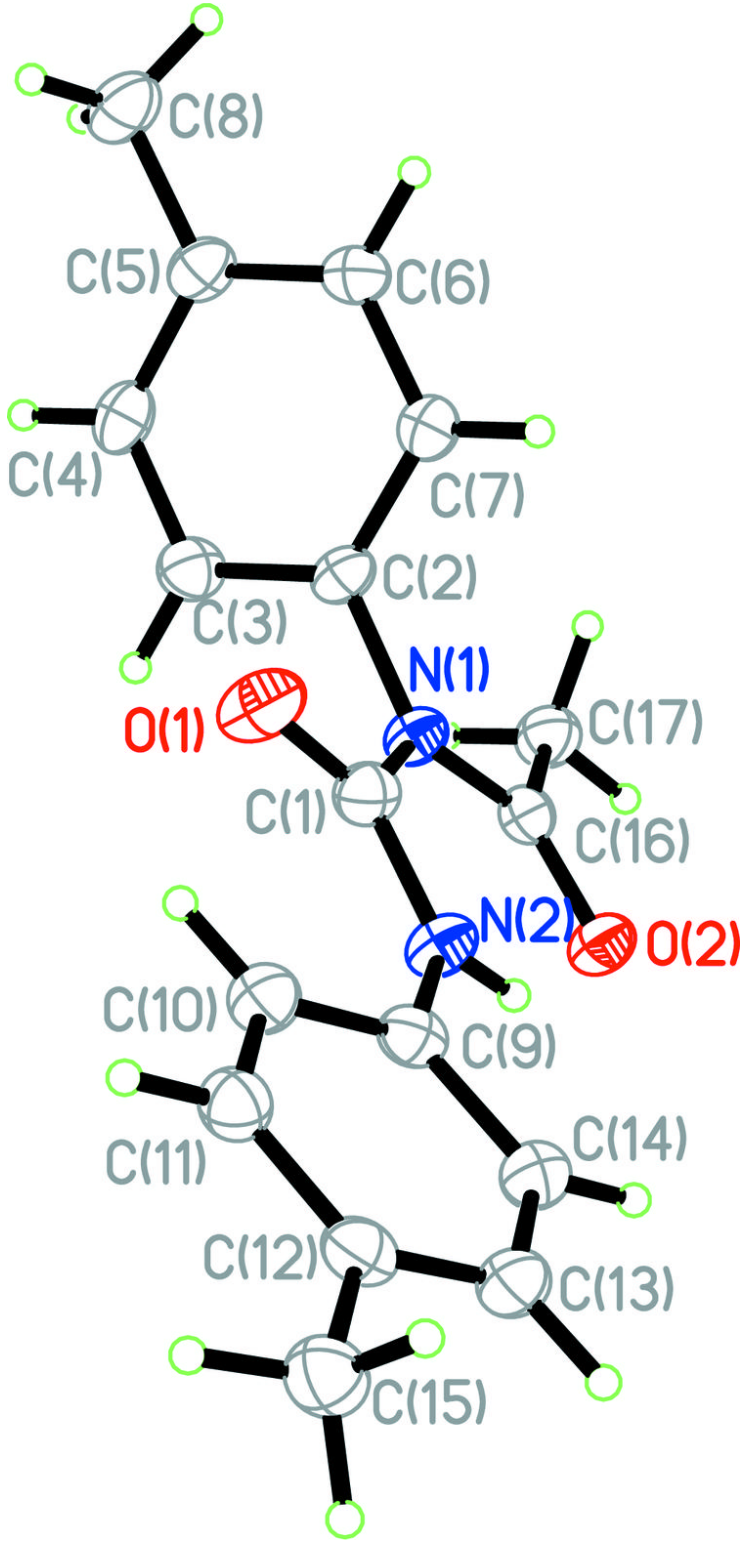

C(8)
C(5)
C(6)
C(4)
C(7)
C(2)
C(3)
N(1)
O(1)
C(17)
C(1)
C(16)
N(2)
O(2)
C(10)
C(9)
C(14)
C(11)
C(12)
C(13)
C(15)

Fig. 2