

N*-tert-Butyl-4-chloro-5-methyl-2-nitroaniline*Ying Zhang, Tian-Yang Feng* and
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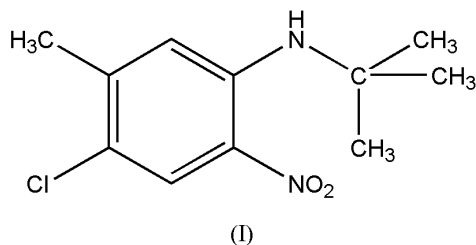
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Key indicatorsSingle-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.036
 wR factor = 0.098
Data-to-parameter ratio = 13.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{11}\text{H}_{15}\text{ClN}_2\text{O}_2$, lies on a crystallographic mirror plane of symmetry; thus all of its atoms, except for two of the *tert*-butyl methyl C atoms and the H atoms, are exactly coplanar. There is an intramolecular hydrogen bond between the amide N atom and a nitro O atom. There are no exceptionally close intermolecular contacts, but the planar portions of the molecules stack with their planes only 3.39 Å apart.

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The title compound, (I) (Fig. 1), is one of the key intermediates in the preparation of quinoxalinediones, which are potential *N*-methyl-D-aspartate (NMDA) antagonists and can be used to reduce damage to the nervous system during cerebral stroke and brain injury (Ilyin *et al.*, 1996; Cai *et al.*, 1997). The molecules are exactly planar, excluding C9, C9A and the H atoms, and lie on crystallographic mirror planes of symmetry that occur at $y = \frac{1}{4}$ and $y = \frac{3}{4}$ in the unit cell. The bond lengths and angles are unexceptional.



The structure is stabilized by one intramolecular hydrogen bond, between the amide N atom and a nitro O atom (Table 1), with $\text{N}\cdots\text{O} = 2.628$ (3) Å and $\text{N}-\text{H}\cdots\text{O} = 133^\circ$. It is similar to a corresponding hydrogen bond in 2-chloro-*N*-(5-chloro-4-fluoro-2-nitrophenyl)acetamide [$\text{N}\cdots\text{O} = 2.611$ (9) Å and $\text{N}-\text{H}\cdots\text{O} = 135$ (2) $^\circ$; Zhang & Feng, 2004]. No intermolecular hydrogen bonds are found in the crystal structure of (I), but there are several $\text{C}\cdots\text{C}$ and $\text{C}\cdots\text{N}$ contacts in the 3.4–3.6 Å range, all of which are between stacked parallel planar parts of the molecules (see Fig. 2).

Experimental

The title compound was prepared from 2-chloro-4-fluoro-1-methylbenzene, through nitration and substitution, according to the method described by Kher *et al.* (1995). Single crystals suitable for X-ray analysis were grown by slow evaporation of an ethanol solution at 298 K.

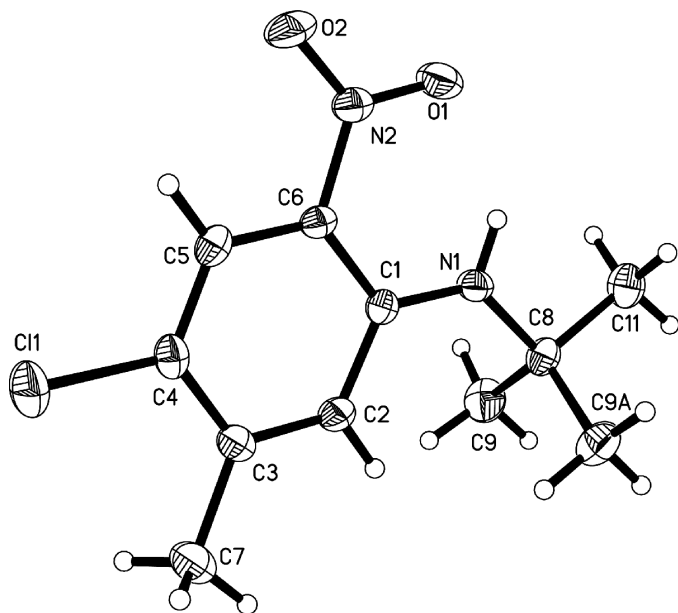


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

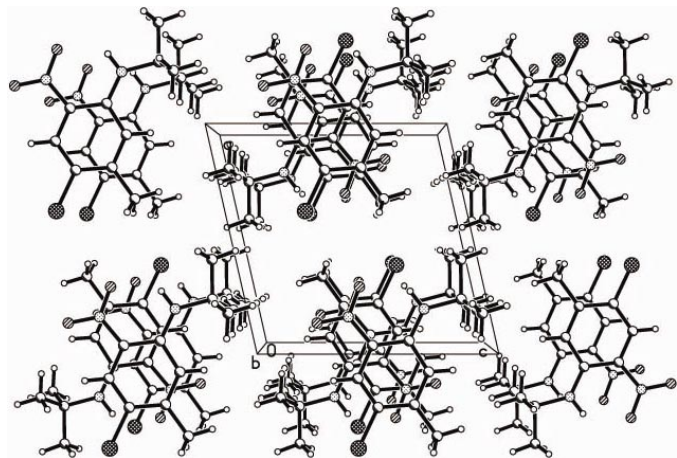


Figure 2
Stacking of the molecules in the crystal structure of (I), viewed down the *b* axis of the unit cell.

Crystal data

$C_{11}H_{15}ClN_2O_2$
 $M_r = 242.70$
 Monoclinic, $P2_1/m$
 $a = 9.354$ (3) Å
 $b = 6.7787$ (12) Å
 $c = 9.566$ (3) Å
 $\beta = 102.720$ (5)°
 $V = 591.7$ (3) Å³
 $Z = 2$

$D_x = 1.362$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 579
 reflections
 $\theta = 3.7$ – 25.0°
 $\mu = 0.31$ mm⁻¹
 $T = 293$ (2) K
 Prism, yellow
 $0.22 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART 1000 CCD area-
 detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 1997)
 $T_{\min} = 0.925$, $T_{\max} = 0.963$
 3456 measured reflections

1314 independent reflections
 921 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -11 \rightarrow 11$
 $k = -8 \rightarrow 8$
 $l = -9 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.099$
 $S = 1.05$
 1314 reflections
 95 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.1063P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···O1	0.87	1.96	2.628 (3)	133

Atom H1, attached to N1, was located initially in a difference Fourier map, and was then constrained to the N atom with an N—H distance of 0.87 Å. All other H atoms were positioned geometrically and refined in the riding-model approximation. C—H distances were set at 0.93 Å for C2 and C5, and at 0.96 Å for the methyl groups (C7, C9, C9A and C11); $U_{\text{iso}}(\text{H})$ values were constrained to be $1.5U_{\text{eq}}(\text{carrier})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{carrier})$ for other H atoms.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

X-ray data were collected at the X-ray Crystallographic Service, University of Nankai, China. The authors thank the staff for all their help and advice.

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