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N-(4-Chlorophenyl)-N'-(4-methylphenyl)succinamide

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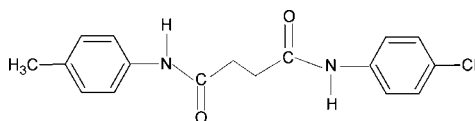
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.048; wR factor = 0.131; data-to-parameter ratio = 13.0.

The asymmetric unit of the title compound, $\text{C}_{17}\text{H}_{17}\text{ClN}_2\text{O}_2$, contains one half-molecule with a center of symmetry at the mid-point of the central C—C bond. The dihedral angle between the benzene ring and the adjacent NH—C(O)—CH₂ group is 39.9 (1)°. The methyl and Cl groups are disordered with respect to the *para*-positions of the benzene ring, with site-occupation factors of 0.5 each. In the crystal, intermolecular N—H···O hydrogen bonds link the molecules into chains parallel to the *b* axis.

Related literature

For our studies on the effects of substituents on the structures of *N*-(aryl)-amides, see: Arjunan *et al.* (2004); Bhat & Gowda (2000); Saraswathi *et al.* (2011), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and on arylsulfonamides, see: Gowda *et al.* (2003). For a similar structure, see Pierrot *et al.* (1984). For restrained geometry, see: Nardelli (1999).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{17}\text{ClN}_2\text{O}_2$
 $M_r = 316.78$
Monoclinic, $P2_1/c$

$a = 17.305$ (3) Å
 $b = 4.8446$ (6) Å
 $c = 9.726$ (1) Å

$\beta = 101.58$ (2)°
 $V = 798.79$ (19) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.25$ mm⁻¹
 $T = 293$ K
 $0.46 \times 0.36 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)
 $T_{\min} = 0.895$, $T_{\max} = 0.952$
2538 measured reflections
1452 independent reflections
1103 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.009$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.131$
 $S = 1.03$
1452 reflections
112 parameters
16 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.85 (2)	2.11 (2)	2.918 (2)	160 (2)

 Symmetry code: (i) $x, y + 1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2134).

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supporting information

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N*-(4-Chlorophenyl)-*N'*-(4-methylphenyl)succinamide*B. S. Saraswathi, Sabine Foro and B. Thimme Gowda****S1. Comment**

The amide and sulfonamide moieties are important constituents of many biologically significant compounds. As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Arjunan *et al.*, 2004; Bhat & Gowda, 2000; Saraswathi *et al.*, 2011), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and arylsulfonamides (Gowda *et al.*, 2003), in the present work, the structure of *N*-(4-Chlorophenyl),*N'*-(4-methylphenyl)-succinamide (I) has been determined (Fig.1). The asymmetric unit of (I) contains half a molecule with a center of symmetry at the mid-point of the central C—C bond, similar to that observed in bis(2-chlorophenylaminocarbonylmethyl)disulfide (II) (Pierrot *et al.*, 1984), *N*-(3-Chlorophenyl),*N'*-(3-methylphenyl)-succinamide (III) (Saraswathi *et al.*, 2011)

The conformations of the amide O atoms are *anti* to the H atoms attached to the adjacent C atoms.

The dihedral angle between the benzene ring and the NH—C(O)—CH₂ segment in the two halves of the molecule is 39.9 (1)°, compared to the value of 43.5 (1)° in (III).

The packing of molecules in the crystal linked by of N—H···O hydrogen bonds (Table 1) is shown in Fig. 2.

S2. Experimental

Succinic anhydride (0.01 mol) in toluene (25 ml) was treated drop wise with 4-chloroaniline (0.01 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for one hour and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove unreacted 4-chloroaniline. The resultant solid *N*-(4-chlorophenyl)-succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. The compound was recrystallized to constant melting point from ethanol. The purity of the compound was checked by elemental analysis and characterized by its infrared and NMR spectra.

The *N*-(4-chlorophenyl)succinamic acid obtained was then treated with phosphorous oxychloride and excess of 4-methylaniline at room temperature with constant stirring. The resultant mixture was stirred for 4 h, kept aside for additional 6 h for completion of the reaction and poured slowly into crushed ice with constant stirring. It was kept aside for a day. The resultant solid, *N*-(4-chlorophenyl), *N'*-(4-methylphenyl)-succinamide was filtered under suction, washed thoroughly with water, dilute sodium hydroxide solution and finally with water. It was recrystallized to constant melting point from a mixture of acetone and toluene (3:1 *v/v*). The compound was characterized by its infrared and NMR spectra.

Rod like colorless single crystals used in X-ray diffraction studies were grown in a mixture of acetone and toluene (3:1 *v/v*) at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (2) Å.

The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å, methyl C—H = 0.97 Å, and the methylene C—H = 0.97 Å.

All H atoms were refined with isotropic displacement parameters. The $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{C-aromatic, N})$ and $1.5U_{\text{eq}}(\text{C-methyl})$.

C9 and CL1 are disordered and were refined using a split model. The corresponding site-occupation factors were fixed to 0.50:0.50. The bond lengths C4—C9 were restrained to 1.54 (1) Å and C4—CL1 to 1.74 (1) Å, respectively. The U^{ij} components of these atoms were restrained to approximate isotropic behavior (Nardelli, 1999).

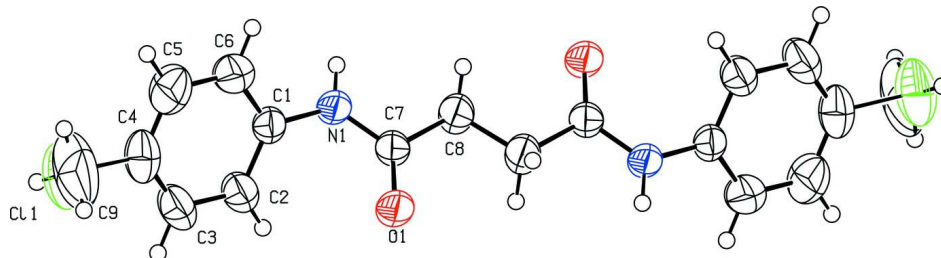


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level. symmetry code: (i) $-x + 1, -y, -z + 1$.

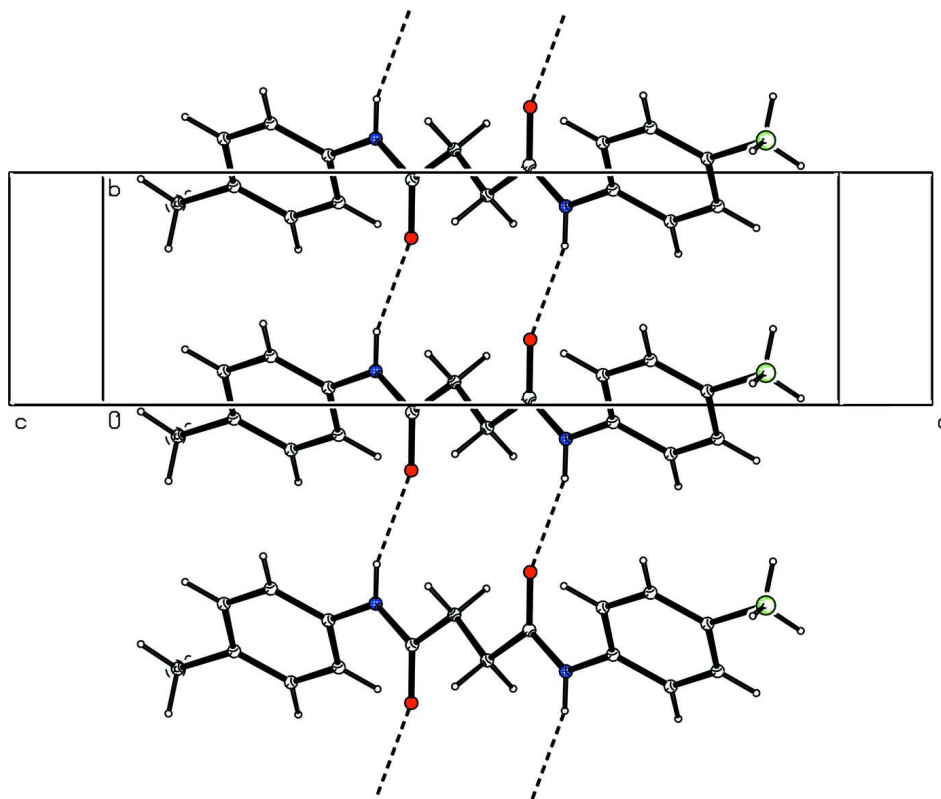


Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N*-(4-Chlorophenyl)-*N'*-(4-methylphenyl)succinamideCrystal data*C₁₇H₁₇ClN₂O₂ $M_r = 316.78$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 17.305 (3) \text{ \AA}$ $b = 4.8446 (6) \text{ \AA}$ $c = 9.726 (1) \text{ \AA}$ $\beta = 101.58 (2)^\circ$ $V = 798.79 (19) \text{ \AA}^3$ $Z = 2$ $F(000) = 332$ $D_x = 1.317 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1260 reflections

 $\theta = 2.7\text{--}27.6^\circ$ $\mu = 0.25 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Rod, colourless

 $0.46 \times 0.36 \times 0.20 \text{ mm}$ *Data collection*

Oxford Diffraction Xcalibur

diffractometer with Sapphire CCD detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2009)

 $T_{\min} = 0.895$, $T_{\max} = 0.952$

2538 measured reflections

1452 independent reflections

1103 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.009$ $\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 3.6^\circ$ $h = -15 \rightarrow 20$ $k = -4 \rightarrow 5$ $l = -11 \rightarrow 11$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.131$ $S = 1.03$

1452 reflections

112 parameters

16 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.3157P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.21 \text{ e \AA}^{-3}$ *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.29114 (11)	0.0736 (4)	0.17026 (19)	0.0472 (5)	
C2	0.29237 (13)	-0.1283 (5)	0.0720 (2)	0.0635 (6)	
H2	0.3390	-0.2218	0.0696	0.076*	

C3	0.22412 (15)	-0.1929 (6)	-0.0237 (2)	0.0764 (7)	
H3	0.2255	-0.3312	-0.0895	0.092*	
C4	0.15544 (13)	-0.0589 (6)	-0.0236 (2)	0.0711 (6)	
C5	0.15403 (14)	0.1444 (6)	0.0728 (3)	0.0846 (8)	
H5	0.1074	0.2391	0.0735	0.102*	
C6	0.22151 (15)	0.2106 (5)	0.1694 (3)	0.0750 (7)	
H6	0.2198	0.3496	0.2347	0.090*	
C7	0.41174 (11)	-0.0321 (4)	0.34385 (18)	0.0468 (5)	
C8	0.47503 (13)	0.0983 (4)	0.4538 (2)	0.0634 (6)	
H8A	0.5084	0.2107	0.4071	0.076*	
H8B	0.4501	0.2202	0.5110	0.076*	
C9	0.0768 (8)	-0.134 (5)	-0.1191 (19)	0.169 (10)	0.50
H9A	0.0666	-0.3271	-0.1101	0.203*	0.50
H9B	0.0792	-0.0930	-0.2148	0.203*	0.50
H9C	0.0352	-0.0283	-0.0924	0.203*	0.50
C11	0.07114 (18)	-0.1382 (10)	-0.1455 (5)	0.1135 (11)	0.50
N1	0.35940 (10)	0.1458 (3)	0.27126 (17)	0.0535 (5)	
H1N	0.3632 (13)	0.315 (3)	0.294 (2)	0.064*	
O1	0.40838 (9)	-0.2812 (3)	0.32397 (15)	0.0642 (5)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0506 (11)	0.0398 (10)	0.0471 (10)	-0.0047 (9)	0.0000 (8)	0.0038 (8)
C2	0.0569 (12)	0.0745 (15)	0.0547 (12)	0.0060 (11)	0.0009 (10)	-0.0138 (11)
C3	0.0749 (17)	0.0884 (18)	0.0576 (13)	-0.0052 (14)	-0.0061 (11)	-0.0202 (12)
C4	0.0550 (13)	0.0906 (16)	0.0607 (13)	-0.0145 (13)	-0.0048 (10)	0.0112 (10)
C5	0.0548 (14)	0.104 (2)	0.0894 (17)	0.0146 (14)	0.0000 (13)	-0.0008 (13)
C6	0.0700 (15)	0.0706 (15)	0.0775 (16)	0.0144 (13)	-0.0018 (12)	-0.0158 (12)
C7	0.0537 (11)	0.0347 (10)	0.0483 (10)	-0.0079 (8)	0.0011 (8)	0.0012 (8)
C8	0.0693 (14)	0.0389 (11)	0.0692 (13)	-0.0098 (10)	-0.0173 (11)	0.0008 (9)
C9	0.119 (11)	0.237 (18)	0.120 (12)	-0.019 (11)	-0.051 (7)	-0.022 (10)
C11	0.0731 (14)	0.155 (3)	0.0934 (14)	-0.0297 (16)	-0.0288 (11)	-0.0018 (14)
N1	0.0601 (10)	0.0317 (8)	0.0599 (10)	-0.0019 (7)	-0.0090 (8)	-0.0033 (7)
O1	0.0761 (11)	0.0309 (7)	0.0738 (10)	-0.0050 (6)	-0.0134 (8)	-0.0028 (6)

Geometric parameters (Å, °)

C1—C2	1.371 (3)	C6—H6	0.9300
C1—C6	1.374 (3)	C7—O1	1.222 (2)
C1—N1	1.420 (2)	C7—N1	1.344 (2)
C2—C3	1.385 (3)	C7—C8	1.507 (3)
C2—H2	0.9300	C8—C8 ⁱ	1.466 (4)
C3—C4	1.354 (3)	C8—H8A	0.9700
C3—H3	0.9300	C8—H8B	0.9700
C4—C5	1.363 (4)	C9—H9A	0.9600
C4—C9	1.529 (9)	C9—H9B	0.9600
C4—C11	1.728 (4)	C9—H9C	0.9600

C5—C6	1.382 (3)	N1—H1N	0.847 (15)
C5—H5	0.9300		
C2—C1—C6	118.5 (2)	C5—C6—H6	119.6
C2—C1—N1	122.07 (18)	O1—C7—N1	123.02 (17)
C6—C1—N1	119.45 (18)	O1—C7—C8	122.08 (17)
C1—C2—C3	120.0 (2)	N1—C7—C8	114.89 (16)
C1—C2—H2	120.0	C8 ⁱ —C8—C7	114.7 (2)
C3—C2—H2	120.0	C8 ⁱ —C8—H8A	108.6
C4—C3—C2	121.4 (2)	C7—C8—H8A	108.6
C4—C3—H3	119.3	C8 ⁱ —C8—H8B	108.6
C2—C3—H3	119.3	C7—C8—H8B	108.6
C3—C4—C5	118.9 (2)	H8A—C8—H8B	107.6
C3—C4—C9	124.0 (9)	C4—C9—H9A	109.5
C5—C4—C9	116.9 (9)	C4—C9—H9B	109.5
C3—C4—C11	120.9 (3)	H9A—C9—H9B	109.5
C5—C4—C11	120.2 (3)	C4—C9—H9C	109.5
C9—C4—C11	5.7 (9)	H9A—C9—H9C	109.5
C4—C5—C6	120.4 (2)	H9B—C9—H9C	109.5
C4—C5—H5	119.8	C7—N1—C1	125.78 (16)
C6—C5—H5	119.8	C7—N1—H1N	118.4 (15)
C1—C6—C5	120.8 (2)	C1—N1—H1N	115.3 (15)
C1—C6—H6	119.6		
C6—C1—C2—C3	-1.0 (3)	C2—C1—C6—C5	0.7 (4)
N1—C1—C2—C3	179.5 (2)	N1—C1—C6—C5	-179.8 (2)
C1—C2—C3—C4	0.5 (4)	C4—C5—C6—C1	0.1 (4)
C2—C3—C4—C5	0.3 (4)	O1—C7—C8—C8 ⁱ	-7.8 (4)
C2—C3—C4—C9	-175.4 (10)	N1—C7—C8—C8 ⁱ	171.6 (3)
C2—C3—C4—C11	179.0 (3)	O1—C7—N1—C1	4.5 (3)
C3—C4—C5—C6	-0.5 (4)	C8—C7—N1—C1	-174.84 (18)
C9—C4—C5—C6	175.4 (9)	C2—C1—N1—C7	-42.7 (3)
C11—C4—C5—C6	-179.3 (3)	C6—C1—N1—C7	137.8 (2)

Symmetry code: (i) $-x+1, -y, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<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—H1N \cdots O1 ⁱⁱ	0.85 (2)	2.11 (2)	2.918 (2)	160 (2)

Symmetry code: (ii) $x, y+1, z$.