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N,*N*′-Bis(4-chlorophenyl)urea

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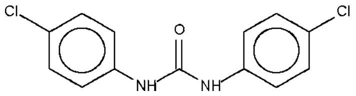
Key indicators: single-crystal X-ray study; *T* = 100 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.032; *wR* factor = 0.097; data-to-parameter ratio = 15.9.

The carbonyl unit of the title compound, $C_{13}H_{10}Cl_2N_2O$, lies on a twofold rotation axis. The ring is aligned at 51.6 (1)° with respect to the N—C(=O)—N fragment. The two –NH– fragments of one molecule form hydrogen bonds [2.845 (2) Å] to the C=O fragment of an adjacent molecule, giving rise to the formation of a linear hydrogen-bonded chain.

Related literature

For isostructural *N*,*N*′-bis(4-bromophenyl)urea, see: Lin *et al.* (2004). *N*,*N*′-Bis-(4-chlorophenyl)urea has been isolated as a co-crystal with a phthalazinium chloride; see: Wamhoff *et al.* (1994). For the self-condensation of 4-chlorophenyl isocyanate to yield the title symmetrical urea, see: Fu *et al.* (2007); Jimenez Blanco *et al.* (1999).



Experimental

Crystal data

$C_{13}H_{10}Cl_2N_2O$
$M_r = 281.13$
Monoclinic, *C*2/*c*
$a = 27.093$ (3) Å
$b = 4.5768$ (5) Å
$c = 9.901$ (1) Å
$\beta = 96.389$ (2)°
$V = 1220.1$ (2) Å^3
$Z = 4$
Mo *Kα* radiation
$\mu = 0.52$ mm^{-1}
$T = 100$ (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.862$, $T_{\max} = 0.950$
3703 measured reflections
1386 independent reflections
1210 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.096$
$S = 1.11$
1386 reflections
87 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.87 (1)	2.05 (1)	2.845 (2)	152 (2)

Symmetry code: (i) *x*, *y* − 1, *z*.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2256).

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supplementary materials

Acta Cryst. (2008). E**64**, o922 [**doi:10.1107/S1600536808011069**]

N,*N*'-Bis(4-chlorophenyl)urea

K. M. Lo and **S. W. Ng**

Comment

The title compound, a symmetrical urea derivative, was the unexpected product from the reaction of 4-chlorophenyl isocyanate with *p*-tolylsulfonic acid in ethanol. The carbonyl unit of (Cl-4-C_6H_4)NH–C(=O)–NH(C_6H_4-4-Cl) lies on a twofold rotation axis, Fig. 1, that relates one aromatic ring to the other. The ring is aligned at 51.6 (1) ° with respect to the N–C(=O)–N fragment. The two –NH– fragments of one molecule forms hydrogen bonds to the C=O fragment of an adjacent molecule, giving rise to the formation of a linear hydrogen-bonded chain (Table 1). The compound has previously been synthesized from the self-condensation of 4-chlorophenyl isocyanate in acetone (Fu *et al.*, 2007) and in water catalyzed by pyridine (Jimenez Blanco *et al.*, 1999).

Experimental

4-Chlorophenyl isocyanate (1.0 g, 6.5 mmol) and *p*-toluenesulfonic acid (1.2 g, 6.5 mmol) were heated in ethanol (100 ml) for 1 h. The solution was filtered; evaporation of the solvent gave plates of the symmetrical urea.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with U(H) set to $1.2U_{eq}$(C).

The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N–H 0.88±0.01 Å; its temperature factor was freely refined.

Figures

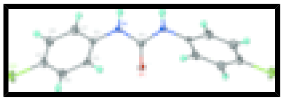

Fig. 1. The molecular structure of (I) showing the atom-numbering scheme and 70% probability displacement ellipsoids. Hydrogen atoms are drawn as spheres of arbitrary radius. The unlablled atoms related by a 2-fold axis of symmetry.

N,*N*'-Bis(4-chlorophenyl)urea

Crystal data

$C_{13}H_{10}Cl_2N_2O$

$M_r = 281.13$

Monoclinic, *C*2/*c*

Hall symbol: -C 2yc

$a = 27.093$ (3) Å

$F_{000} = 576$

$D_x = 1.530$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 1510 reflections

$\theta = 3.0–28.2°$

b = 4.5768 (5) Å
c = 9.901 (1) Å
β = 96.389 (2)°
V = 1220.1 (2) Å^3
Z = 4

μ = 0.52 mm^{-1}
T = 100 (2) K
Block, colorless
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
T = 100(2) K
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.862, T_{max} = 0.950
3703 measured reflections

1386 independent reflections
1210 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°
θ_{min} = 1.5°
$h = -34 \rightarrow 27$
$k = -5 \rightarrow 5$
$l = -10 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.096
S = 1.11
1386 reflections
87 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 1.607P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}}$ = 0.31 e Å^{-3}
$\Delta\rho_{\text{min}}$ = −0.29 e Å^{-3}
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.293344 (15)	0.99597 (10)	0.33207 (4)	0.02417 (17)
O1	0.5000	0.9101 (4)	0.7500	0.0163 (4)
N1	0.46380 (5)	0.4789 (3)	0.67795 (15)	0.0149 (3)
H1	0.4640 (8)	0.292 (2)	0.691 (2)	0.024 (5)*
C1	0.5000	0.6399 (5)	0.7500	0.0130 (4)
C2	0.42311 (6)	0.6073 (3)	0.59591 (15)	0.0131 (3)
C3	0.43093 (6)	0.8150 (4)	0.49760 (16)	0.0152 (3)
H3	0.4638	0.8730	0.4854	0.018*
C4	0.39102 (6)	0.9373 (4)	0.41754 (17)	0.0175 (4)
H4	0.3963	1.0823	0.3520	0.021*

C5	0.34334 (6)	0.8455 (4)	0.43438 (16)	0.0162 (3)
C6	0.33491 (6)	0.6357 (4)	0.52957 (17)	0.0183 (4)
H6	0.3021	0.5729	0.5391	0.022*
C7	0.37498 (6)	0.5180 (4)	0.61096 (17)	0.0176 (4)
H7	0.3695	0.3754	0.6774	0.021*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0159 (2)	0.0299 (3)	0.0249 (3)	0.00200 (17)	−0.00584 (17)	0.00510 (17)
O1	0.0173 (8)	0.0094 (8)	0.0207 (8)	0.000	−0.0037 (6)	0.000
N1	0.0146 (7)	0.0082 (6)	0.0208 (7)	−0.0002 (5)	−0.0028 (6)	0.0007 (5)
C1	0.0128 (10)	0.0123 (11)	0.0141 (10)	0.000	0.0025 (8)	0.000
C2	0.0141 (7)	0.0104 (7)	0.0142 (7)	0.0001 (6)	−0.0006 (6)	−0.0027 (6)
C3	0.0124 (7)	0.0162 (8)	0.0167 (8)	−0.0028 (6)	0.0004 (6)	−0.0013 (6)
C4	0.0184 (8)	0.0178 (8)	0.0159 (8)	−0.0013 (6)	−0.0001 (6)	0.0016 (6)
C5	0.0138 (8)	0.0189 (8)	0.0152 (8)	0.0018 (6)	−0.0023 (6)	−0.0017 (6)
C6	0.0118 (8)	0.0233 (9)	0.0199 (8)	−0.0016 (6)	0.0024 (6)	−0.0009 (7)
C7	0.0176 (8)	0.0174 (8)	0.0178 (8)	−0.0019 (6)	0.0026 (6)	0.0026 (6)

Geometric parameters (Å, °)

Cl1—C5	1.741 (2)	C3—C4	1.386 (2)
O1—C1	1.237 (3)	C3—H3	0.9500
N1—C1	1.363 (2)	C4—C5	1.386 (2)
N1—C2	1.422 (2)	C4—H4	0.9500
N1—H1	0.87 (1)	C5—C6	1.382 (2)
C1—N1^{i}	1.363 (2)	C6—C7	1.387 (2)
C2—C7	1.390 (2)	C6—H6	0.9500
C2—C3	1.393 (2)	C7—H7	0.9500
C1—N1—C2	122.9 (1)	C5—C4—C3	119.2 (2)
C1—N1—H1	118 (1)	C5—C4—H4	120.4
C2—N1—H1	119 (1)	C3—C4—H4	120.4
O1—C1—N1	122.7 (1)	C4—C5—C6	121.3 (2)
O1—C1—N1^{i}	122.7 (1)	C4—C5—Cl1	119.0 (1)
N1—C1—N1^{i}	114.6 (2)	C6—C5—Cl1	119.65 (13)
C7—C2—C3	119.5 (2)	C7—C6—C5	119.21 (15)
C7—C2—N1	119.6 (1)	C7—C6—H6	120.4
C3—C2—N1	120.8 (1)	C5—C6—H6	120.4
C4—C3—C2	120.4 (2)	C6—C7—C2	120.42 (15)
C4—C3—H3	119.8	C6—C7—H7	119.8
C2—C3—H3	119.8	C2—C7—H7	119.8
C2—N1—C1—O1	0.4 (2)	C3—C4—C5—C6	0.3 (3)
C2—N1—C1—N1^{i}	−179.6 (2)	C3—C4—C5—Cl1	−179.1 (1)
C1—N1—C2—C7	−129.4 (2)	C4—C5—C6—C7	0.9 (3)
C1—N1—C2—C3	52.6 (2)	Cl1—C5—C6—C7	−179.8 (1)
C7—C2—C3—C4	1.6 (2)	C5—C6—C7—C2	−0.8 (3)
N1—C2—C3—C4	179.6 (2)	C3—C2—C7—C6	−0.5 (2)

C2—C3—C4—C5	−1.5 (2)	N1—C2—C7—C6	−178.5 (2)

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{ii}	0.87 (1)	2.05 (1)	2.845 (2)	152 (2)

Symmetry codes: (ii) *x*, *y*−1, *z*.

Fig. 1