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N-(*p*-Chlorophenyl)maleamic acid

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Key indicators

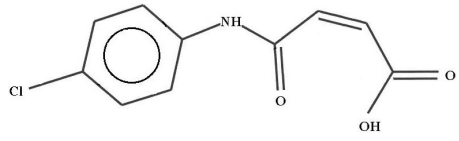
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.140
Data-to-parameter ratio = 12.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title molecule, $C_{10}H_8ClNO_3$, is nearly planar, with the mean planes through the *p*-chlorophenyl and maleamic acid groups inclined at an angle of 4.45 (1)° to each other. Symmetry-related molecules are linked by N—H···O, C—H···O and C—H···Cl intermolecular hydrogen bonds, to form molecular layers parallel to the *bc* plane.

Comment

The structure of *N*-(*p*-chlorophenyl)-maleamic acid, (I), was briefly reported by Prasad & Mandal (1978), with an *R* value of 0.16, using photographic X-ray diffraction data. The structure has now been refined using diffractometer X-ray data and the results are presented here. We have previously reported the structure of a related compound, *N*-(*p*-tolyl)maleamic acid (Prasad *et al.*, 2002). The two structures are similar but not exactly isostructural. The substitution of CH_3 by a Cl atom has reduced the unit-cell volume by 37.8 $Å^3$.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1. The bond lengths and angles of the maleamic acid group agree with those in *N*-(*p*-tolyl)maleamic acid and also with those in maleic acid (James & Williams, 1974). The molecule is nearly planar, with atom O3 deviating by a maximum by 0.116 (2) Å. The dihedral angle between the mean planes through the *p*-chlorophenyl and maleamic acid groups is 4.45 (1)°. The carboxyl H atom is involved in an intramolecular O—H···O hydrogen bond with carbonyl atom O3. In the crystal, symmetry-related molecules are linked by N—H···O, C—H···O and C—H···Cl intermolecular hydrogen bonds (Table 2 and Fig. 2), to form molecular layers parallel to the *bc* plane, approximately at $x = \frac{1}{4}$ and $\frac{3}{4}$. These layers are stacked alternately at distances of 3.405 and 3.341 Å, indicating significant interactions between the phenyl and maleamic acid groups, as observed in *N*-(*p*-tolyl)maleamic acid (Prasad *et al.*, 2002). A list of some short intermolecular contacts is given in Table 3.

Experimental

The title compound was prepared by a solid-state reaction between *p*-chloroaniline and maleic anhydride, by Professor R. P. Rastogi (Ghorakhpur University) and his co-workers (private communication). It was recrystallized from methanol at room temperature.

Crystal data

$C_{10}H_8ClNO_3$	D_x = 1.513 Mg m^{-3}
M_r = 225.62	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 7.306 (3) Å	θ = 8.2–18.9°
b = 11.765 (4) Å	μ = 0.37 mm^{-1}
c = 12.828 (4) Å	T = 293 (2) K
β = 116.09 (4)°	Needle, light yellow
V = 990.3 (6) Å^3	0.25 × 0.23 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 25.0°
ω–2θ scans	$h = 0 \rightarrow 8$
Absorption correction: none	$k = 0 \rightarrow 13$
1707 measured reflections	$l = -15 \rightarrow 13$
1707 independent reflections	3 standard reflections
1224 reflections with $I > 2\sigma(I)$	every 50 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.102P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.140	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
S = 0.92	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
1707 reflections	Extinction correction: *SHELXL*97
137 parameters	Extinction coefficient: 0.006 (2)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—Cl	1.729 (3)	C7—N1	1.340 (3)
C4—N1	1.407 (4)	C10—O2	1.201 (3)
C7—O3	1.239 (3)	C10—O1	1.302 (3)
C2—C1—Cl	120.7 (2)	O3—C7—N1	122.2 (3)
C6—C1—Cl	119.2 (2)	O3—C7—C8	123.3 (2)
C3—C4—N1	124.1 (2)	N1—C7—C8	114.4 (2)
C5—C4—N1	116.8 (2)	C7—N1—C4	128.9 (2)
O3—C7—C8—C9	3.5 (5)	C3—C4—N1—C7	−8.4 (4)
O3—C7—N1—C4	0.7 (4)	C5—C4—N1—C7	172.0 (3)
C8—C7—N1—C4	−179.1 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.86	2.02	2.870 (3)	169
O1—H1O···O3	0.82	1.68	2.496 (3)	174
C3—H3···O3	0.93	2.26	2.847 (4)	121
C5—H5···O2^{i}	0.93	2.58	3.315 (3)	137
C8—H8···O1^{i}	0.93	2.65	3.567 (4)	168
C9—H9···Cl^{ii}	0.93	2.86	3.720 (3)	154

Symmetry codes: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (ii) $x, 1 + y, z$.

Table 3
Short contact distances (Å).

Cl···O1^{iii}	3.121 (3)	C4···O2^{i}	3.576 (3)
Cl···O3^{iii}	3.458 (3)	C6···C8^{v}	3.588 (4)
C2···C8^{iv}	3.494 (5)	C7···O3^{vi}	3.538 (4)
C3···C8^{iv}	3.454 (5)	O3···O3^{vi}	3.440 (3)

Symmetry codes: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (iii) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $1 - x, 1 - y, 1 - z$; (v) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (vi) $-x, 1 - y, 1 - z$.

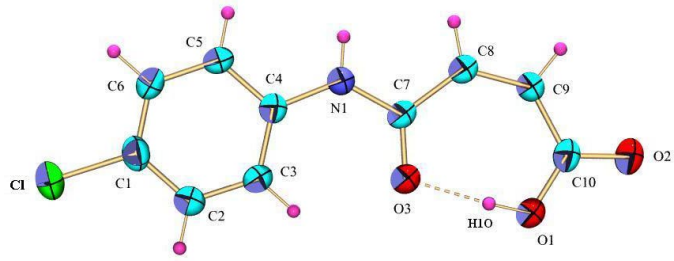


Figure 1
An *ORTEP*-3 plot (Farrugia, 1997) of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

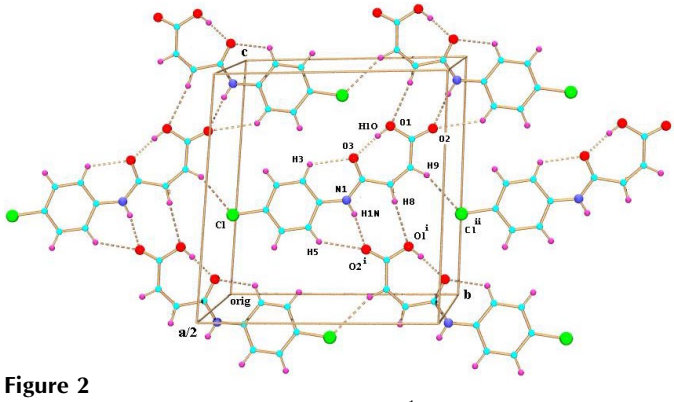


Figure 2
A view of the molecular layer, close to $x = \frac{1}{4}$.

After location in a difference map, all the H atoms were fixed geometrically and were treated as riding on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å and O—H = 0.82 Å. The initial coordinates were taken from the related previous work (Prasad & Mandal, 1978).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: see above; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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