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N-(2,4,6-Trichlorophenyl)maleamic acid

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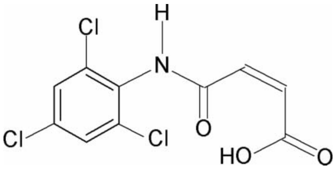
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.094; data-to-parameter ratio = 15.1.

In the crystal structure of the title compound, $C_{10}H_6Cl_3NO_3$, the conformation of the amide bond is *trans*. The C═O and O—H bonds of the acid group are in the relatively rare *anti* position to each other. This is a consequence of the intramolecular O—H···O hydrogen bond donated to the amide carbonyl group stabilizing the molecular structure. In the crystal, intermolecular N—H···O hydrogen bonds link the molecules into zigzag chains along the c axis.

Related literature

For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Arjunan *et al.* (2004); Bhat & Gowda (2000); Gowda *et al.* (2000, 2009); Lo & Ng (2009); Prasad *et al.* (2002), and on *N*-(aryl)-methanesulfonamides, see: Jayalakshmi & Gowda (2004). For modes of interlinking carboxylic acids by hydrogen bonds, see: Leiserowitz (1976).



Experimental

Crystal data

$C_{10}H_6Cl_3NO_3$
M_r = 294.51
Monoclinic, $C2/c$
a = 21.928 (3) Å
b = 8.2678 (8) Å
c = 13.248 (2) Å
β = 99.08 (1)°
V = 2371.7 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 293 K
0.44 × 0.44 × 0.40 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.729, T_{max} = 0.749
4862 measured reflections
2436 independent reflections
2000 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.094
S = 1.08
2436 reflections
161 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.84 (2)	2.04 (2)	2.884 (2)	175 (2)
O3—H3O···O1	0.82 (2)	1.69 (2)	2.498 (2)	168 (3)

Symmetry code: (i) $x, -y, z + \frac{1}{2}$.

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: BT5581).

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supplementary materials

Acta Cryst. (2011). E**67**, o2149 [**doi:10.1107/S1600536811029436**]

N-(2,4,6-Trichlorophenyl)maleamic acid

K. Shakuntala, S. Foro and B. T. Gowda

Comment

The amide moiety is an important constituent of many biologically significant compounds. As part of our studies on the effects of ring and side chain substitutions on the structures and other aspects of *N*-(aryl)-amides (Arjunan *et al.*, 2004; Bhat & Gowda, 2000; Gowda *et al.*, 2000, 2009; Prasad *et al.*, 2002) and *N*-(aryl)-methanesulfonamides (Jayalakshmi & Gowda, 2004), the crystal structure of *N*-(2,4,6-trimethylphenyl)-maleamic acid (I) has been determined. The conformation of the amide O atom is *anti* to the H atom attached to the adjacent C atom, while the carboxyl O atom is *syn* to the H atom attached to its adjacent C atom (Fig.1). The rare anti conformation of the C=O and O–H bonds of the acid group has been observed, similar to that obsrved in *N*-(2,4,6-trimethylphenyl)-maleamic acid (Gowda *et al.*, 2009) and *N*-phenylmaleamic acid (Lo & Ng, 2009), but contrary to the more general *syn* conformation observed for C=O and O–H bonds. The various modes of interlinking carboxylic acids by hydrogen bonds is described elsewhere (Leiserowitz, 1976).

The maleamic moiety includes a short intramolecular hydrogen O–H···O bond (Table 1). The C8–C9 bond length of 1.331 (3)Å clearly indicates the double bond character. The dihedral angle between the phenyl ring and the amido group –NHCO– is 83.2 (2)°. In the crystal structure, the intermolecular N–H···O hydrogen bonds link the molecules into column like chains along *b*-axis (Fig. 2).

Experimental

The solution of maleic anhydride (0.025 mol) in toluene (25 ml) was treated dropwise with the solution of 2,4,6-trichloroaniline (0.025 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about 30 min and set aside for an additional 30 min at room temperature for the completion of reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 2,4,6-trichloroaniline. The resultant solid *N*-(2,4,6-trichlorophenyl)maleamic acid was filtered under suction and washed thoroughly with water to remove the unreacted maleic anhydride and maleic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like colorless single crystals used in X-ray diffraction studies were grown in an ethanol solution by slow evaporation at room temperature.

Refinement

The H atoms of the NH group and the OH group were located in a difference map and later restrained to the distance N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, respectively. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

Figures

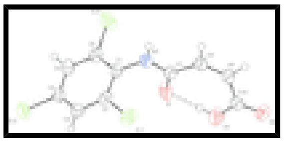

Fig. 1. Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.

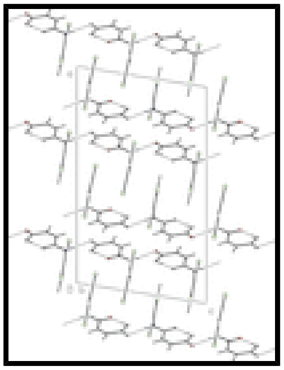

Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

N-(2,4,6-Trichlorophenyl)maleamic acid

Crystal data

$C_{10}H_6Cl_3NO_3$

$M_r = 294.51$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 21.928$ (3) Å

$b = 8.2678$ (8) Å

$c = 13.248$ (2) Å

$\beta = 99.08$ (1)°

$V = 2371.7$ (5) Å^3

$Z = 8$

$F(000) = 1184$

$D_x = 1.650$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2201 reflections

$\theta = 2.6$–27.9°

$\mu = 0.77$ mm^{-1}

$T = 293$ K

Prism, colourless

0.44 × 0.44 × 0.40 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

graphite

Rotation method data acquisition using ω and φ scans

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.729$, $T_{\max} = 0.749$

4862 measured reflections

2436 independent reflections

2000 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 26.3°$, $\theta_{\min} = 2.6°$

$h = -21 \rightarrow 27$

$k = -9 \rightarrow 10$

$l = -14 \rightarrow 16$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.094$

$S = 1.08$

2436 reflections

161 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 2.1238P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.008$

$\Delta\rho_{max} = 0.55$ e Å^{-3}

$\Delta\rho_{min} = -0.44$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0069 (5)

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 (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
Cl1	0.01367 (3)	0.14374 (7)	0.38180 (5)	0.0547 (2)
Cl2	−0.05298 (3)	0.76688 (8)	0.35643 (5)	0.0546 (2)
Cl3	0.18696 (3)	0.61105 (7)	0.44095 (6)	0.0594 (2)
O1	0.13622 (8)	0.26049 (19)	0.24188 (11)	0.0489 (4)
O2	0.20438 (7)	−0.1698 (2)	0.11143 (12)	0.0491 (4)
O3	0.15810 (9)	0.0634 (2)	0.10918 (12)	0.0554 (5)
H3O	0.1526 (15)	0.138 (3)	0.147 (2)	0.083*
N1	0.14268 (8)	0.2673 (2)	0.41225 (12)	0.0356 (4)
H1N	0.1593 (10)	0.234 (3)	0.4704 (14)	0.043*
C1	0.09629 (9)	0.3880 (2)	0.40607 (13)	0.0325 (4)
C2	0.03409 (10)	0.3452 (2)	0.38660 (14)	0.0355 (4)
C3	−0.01220 (9)	0.4607 (3)	0.37139 (15)	0.0389 (5)
H3	−0.0535	0.4305	0.3569	0.047*
C4	0.00448 (9)	0.6214 (3)	0.37825 (14)	0.0362 (5)
C5	0.06509 (10)	0.6697 (3)	0.40216 (16)	0.0394 (5)
H5	0.0753	0.7787	0.4096	0.047*
C6	0.11040 (9)	0.5516 (2)	0.41482 (15)	0.0358 (4)
C7	0.15744 (9)	0.2035 (2)	0.32652 (15)	0.0344 (4)
C8	0.19895 (10)	0.0623 (3)	0.34006 (15)	0.0391 (5)

H8	0.2183	0.0426	0.4066	0.047*
C9	0.21238 (10)	−0.0406 (3)	0.26948 (16)	0.0407 (5)
H9	0.2405	−0.1206	0.2950	0.049*
C10	0.19092 (9)	−0.0518 (3)	0.15725 (15)	0.0371 (5)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0623 (4)	0.0373 (3)	0.0631 (4)	−0.0113 (3)	0.0060 (3)	−0.0095 (3)
Cl2	0.0499 (3)	0.0574 (4)	0.0557 (4)	0.0228 (3)	0.0064 (3)	−0.0018 (3)
Cl3	0.0376 (3)	0.0433 (3)	0.0960 (5)	−0.0025 (2)	0.0072 (3)	0.0033 (3)
O1	0.0625 (10)	0.0509 (10)	0.0327 (8)	0.0257 (8)	0.0056 (7)	0.0070 (7)
O2	0.0507 (9)	0.0483 (9)	0.0485 (9)	0.0001 (7)	0.0086 (7)	−0.0150 (7)
O3	0.0717 (12)	0.0589 (11)	0.0340 (8)	0.0200 (9)	0.0034 (7)	−0.0018 (7)
N1	0.0442 (10)	0.0324 (9)	0.0292 (8)	0.0089 (7)	0.0026 (7)	0.0037 (7)
C1	0.0398 (10)	0.0322 (10)	0.0263 (9)	0.0039 (8)	0.0073 (7)	0.0011 (8)
C2	0.0437 (11)	0.0346 (10)	0.0288 (9)	−0.0037 (8)	0.0075 (8)	−0.0044 (8)
C3	0.0345 (10)	0.0498 (13)	0.0330 (10)	−0.0009 (9)	0.0073 (8)	−0.0067 (9)
C4	0.0391 (11)	0.0403 (11)	0.0302 (10)	0.0108 (9)	0.0085 (8)	−0.0009 (8)
C5	0.0453 (12)	0.0307 (10)	0.0433 (11)	0.0040 (9)	0.0108 (9)	0.0018 (9)
C6	0.0351 (10)	0.0341 (10)	0.0390 (11)	0.0007 (8)	0.0082 (8)	0.0021 (8)
C7	0.0379 (10)	0.0326 (10)	0.0322 (10)	0.0033 (8)	0.0039 (8)	0.0019 (8)
C8	0.0456 (12)	0.0380 (11)	0.0315 (10)	0.0102 (9)	−0.0006 (8)	0.0018 (8)
C9	0.0425 (12)	0.0352 (11)	0.0429 (11)	0.0107 (9)	0.0018 (9)	0.0007 (9)
C10	0.0320 (10)	0.0415 (11)	0.0387 (11)	−0.0035 (9)	0.0087 (8)	−0.0040 (9)

Geometric parameters (Å, °)

Cl1—C2	1.723 (2)	C2—C3	1.385 (3)
Cl2—C4	1.733 (2)	C3—C4	1.377 (3)
Cl3—C6	1.731 (2)	C3—H3	0.9300
O1—C7	1.237 (2)	C4—C5	1.376 (3)
O2—C10	1.210 (3)	C5—C6	1.384 (3)
O3—C10	1.299 (3)	C5—H5	0.9300
O3—H3O	0.819 (18)	C7—C8	1.473 (3)
N1—C7	1.338 (3)	C8—C9	1.331 (3)
N1—C1	1.418 (2)	C8—H8	0.9300
N1—H1N	0.843 (16)	C9—C10	1.490 (3)
C1—C6	1.388 (3)	C9—H9	0.9300
C1—C2	1.393 (3)		

C10—O3—H3O	112 (2)	C4—C5—H5	120.9
C7—N1—C1	119.74 (16)	C6—C5—H5	120.9
C7—N1—H1N	121.4 (16)	C5—C6—C1	122.08 (19)
C1—N1—H1N	118.8 (16)	C5—C6—Cl3	118.60 (16)
C6—C1—C2	117.51 (18)	C1—C6—Cl3	119.32 (15)
C6—C1—N1	122.16 (18)	O1—C7—N1	120.82 (18)
C2—C1—N1	120.28 (18)	O1—C7—C8	123.28 (18)
C3—C2—C1	121.69 (19)	N1—C7—C8	115.89 (17)

C3—C2—Cl1	118.74 (16)	C9—C8—C7	128.44 (19)
C1—C2—Cl1	119.58 (16)	C9—C8—H8	115.8
C4—C3—C2	118.33 (19)	C7—C8—H8	115.8
C4—C3—H3	120.8	C8—C9—C10	132.11 (19)
C2—C3—H3	120.8	C8—C9—H9	113.9
C5—C4—C3	122.17 (19)	C10—C9—H9	113.9
C5—C4—Cl2	119.15 (16)	O2—C10—O3	120.37 (19)
C3—C4—Cl2	118.69 (16)	O2—C10—C9	119.2 (2)
C4—C5—C6	118.12 (19)	O3—C10—C9	120.47 (18)

C7—N1—C1—C6	−98.7 (2)	C4—C5—C6—Cl3	178.00 (15)
C7—N1—C1—C2	78.7 (2)	C2—C1—C6—C5	−1.7 (3)
C6—C1—C2—C3	3.1 (3)	N1—C1—C6—C5	175.71 (18)
N1—C1—C2—C3	−174.36 (17)	C2—C1—C6—Cl3	179.01 (14)
C6—C1—C2—Cl1	−176.52 (14)	N1—C1—C6—Cl3	−3.5 (3)
N1—C1—C2—Cl1	6.0 (2)	C1—N1—C7—O1	8.1 (3)
C1—C2—C3—C4	−1.5 (3)	C1—N1—C7—C8	−170.97 (18)
Cl1—C2—C3—C4	178.18 (15)	O1—C7—C8—C9	−11.8 (4)
C2—C3—C4—C5	−1.7 (3)	N1—C7—C8—C9	167.2 (2)
C2—C3—C4—Cl2	178.53 (14)	C7—C8—C9—C10	−1.0 (4)
C3—C4—C5—C6	3.0 (3)	C8—C9—C10—O2	−170.9 (2)
Cl2—C4—C5—C6	−177.20 (15)	C8—C9—C10—O3	9.0 (4)
C4—C5—C6—C1	−1.2 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2^{i}	0.84 (2)	2.04 (2)	2.884 (2)	175 (2)
O3—H3O···O1	0.82 (2)	1.69 (2)	2.498 (2)	168 (3)

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

Fig. 1

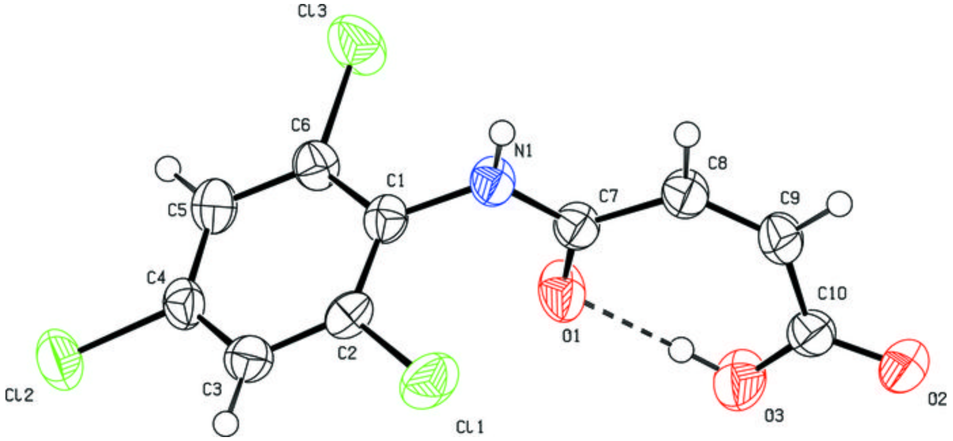

Fig. 2