Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# 3-[(2,4,6-Trichloroanilino)carbonyl]prop-2-enoic acid

**Masood Parvez,[a]* Saira Shahzadi,[b] Khadija Shahid[b] and Saqib Ali[b]**

[a]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and [b]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

**Key indicators**

Single-crystal X-ray study
$T$ = 173 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.038
$wR$ factor = 0.104
Data-to-parameter ratio = 17.0

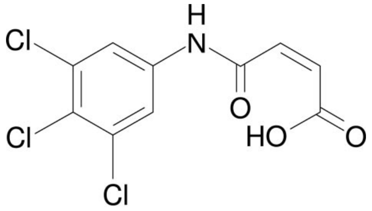
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 September 2004
Accepted 14 October 2004
Online 22 October 2004

The crystal structure of the title compound, $C_{10}H_6Cl_3NO_3$, establishes that condensation of 2,4,6-trichloroaniline with maleic anhydride gives the title compound at room temperature. There are strong intermolecular N—H···O hydrogen bonds [N···O = 2.832 (2) and 2.867 (2) Å], resulting in a hydrophobic region in the middle of the unit cell. The carboxyl groups adopt an *anti*-planar conformation and participate in intramolecular O—H···O hydrogen bonds [both O···O = 2.486 (2) Å]. There are two molecules in the asymmetric unit.

## Comment

Maleic anhydride is a multifunctional chemical intermediate with applications in nearly every field of industrial chemistry (Lutz, 1940; Westrenen *et al.*, 1990). It is an important raw material used in the manufacture of unsaturated polyester resins, surface coatings, lubricant additives and agricultural chemicals (Budavari, 1989). Extensive descriptions of the chemistry of maleic anhydride and its derivatives have been published (Luskin, 1974; Flett & Gardver, 1952). The high reactivity of maleic anhydride is due to the presence of a double bond in conjugation with two carbonyl O atoms. We have synthesized the title compound, (I), by amidation of 2,3,4-trichloroaniline with maleic anhydride, and report its structure here.



(I)

The structure of (I) unequivocally establishes that condensation of 2,4,6-trichloroaniline with maleic anhydride gives the title compound at room temperature. It contains two molecules in the asymmetric unit (Fig. 1). In each molecule, the trichloroaniline and carbonylpropionic acid moieties are individually planar. The mean planes formed by the ring C5–C10 and by atoms O1/C1–C4/O3/O2 are inclined at 60.38 (6)° in one molecule, while the corresponding angle in the second molecule is 58.30 (6)°. This is in contrast with the observation of coplanar moieties in the structure of 2-[(4-bromoanilino)-carbonyl]propenoic acid (Parvez *et al.*, 2004). However, a close examination of the deviations of the atoms from these mean planes shows that atoms N1 and N2 lie 0.047 (3) and 0.025 (3) Å, respectively, out of the plane of the benzene rings.

doi: 10.1107/S1600536804026030

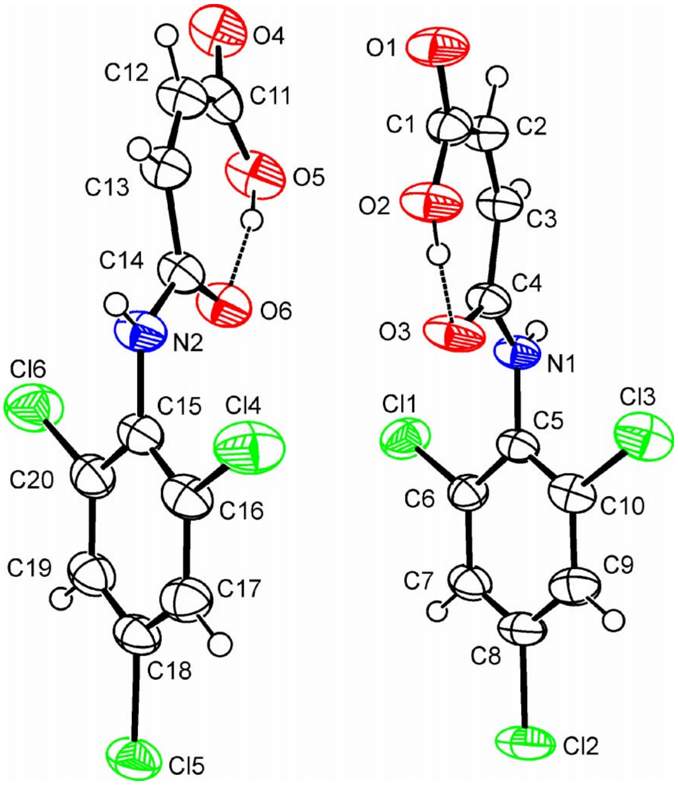


**Figure 1**
A view of the two independent molecules of (I), with the atom-labeling scheme. Displacement ellipsoids are plotted at the 50% probability level. Dashed lines indicate hydrogen bonds.

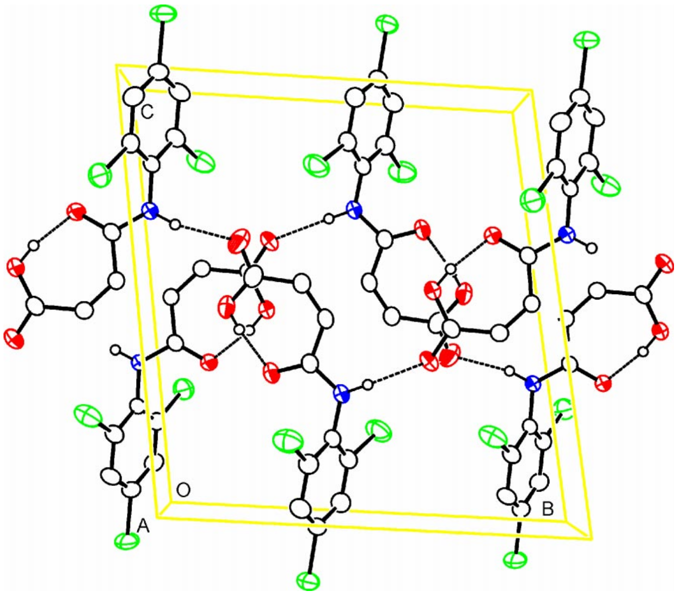


**Figure 2**
The crystal packing of (I), showing the hydrogen bonding (dashed lines).

It is interesting to note that atom Cl1 lies in the plane of the benzene ring to which it is attached, while the remaining Cl atoms deviate substantially from the planes of the benzene rings to which they are bonded [Cl2 0.013 (3), Cl3 0.049 (3), Cl4 0.058 (3), Cl5 0.024 (3) and Cl6 0.062 (3) Å].

The carbonylpropenoic acid in one of the molecules is more twisted than in the other, with the maximum deviations from the O1/C1–C4/O3/O2 and O4/C11–C14/O6/O5 mean planes being 0.0661 (12) Å for atom O3 and 0.1055 (13) Å for atom O4. Atoms N1 and N2 lie 0.163 (3) and 0.333 (3) Å, respectively, out of these planes. In each case, HC=CH H atoms are *cis* to each other, while —NH and —C=O groups are *trans* with respect to each other. All bond lengths and angles are within expected ranges.

There is a network of strong intermolecular N—H···O hydrogen bonds, involving aniline N and carbonyl O atoms of the acid groups. This results in a hydrophobic region in the middle of the unit cell, with N···O separations of 2.832 (2) and 2.867 (2) Å. The carboxyl groups adopt an *anti*-planar conformation, wherein the hydroxyl H atoms participate in intramolecular O—H···O hydrogen bonds, with an O···O separation of 2.486 (2) Å in both molecules, resulting in seven-membered rings. Details of the hydrogen bonds are listed in Table 2 and depicted in Fig. 2.

## Experimental

The title compound, (I), was synthesized by the condensation of 2,4,6-trichloroaniline with maleic anhydride. To a solution of maleic anhydride (1 mmol) in HOAc (300 ml) was added a solution of 2,4,6-trichloroaniline (1 mmol) in HOAc (150 ml) and the mixture was stirred at room temperature overnight. The light-gray precipitate was filtered, washed with cold distilled $H_2O$ (200 ml) and air dried. Crystals suitable for crystallographic study were grown from a solution of (I) in ethanol by slow evaporation at room temperature.

### *Crystal data*

$C_{10}H_6Cl_3NO_3$
$M_r = 294.51$
Triclinic, $P\bar{1}$
$a = 7.0620$ (10) Å
$b = 12.665$ (2) Å
$c = 13.875$ (3) Å
$\alpha = 98.372$ (7)°
$\beta = 102.792$ (8)°
$\gamma = 92.732$ (9)°
$V = 1193.2$ (4) Å$^3$

$Z = 4$
$D_x = 1.639$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 10 295 reflections
$\theta = 3.3$–27.5°
$\mu = 0.76$ mm$^{-1}$
$T = 173$ (2) K
Prism, colorless
0.10 × 0.08 × 0.08 mm

### *Data collection*

Nonius KappaCCD area-detector diffractometer
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.928$, $T_{\max} = 0.942$
10 295 measured reflections

5428 independent reflections
3762 reflections with $>2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 15$
$l = -17 \rightarrow 17$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.104$
$S = 1.02$
5428 reflections
319 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.16P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.42$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C6 | 1.725 (2) | Cl4—C16 | 1.725 (2) |
| Cl2—C8 | 1.731 (2) | Cl5—C18 | 1.737 (2) |
| Cl3—C10 | 1.730 (2) | Cl6—C20 | 1.728 (2) |
| O1—C1 | 1.211 (2) | O4—C11 | 1.215 (3) |
| O2—C1 | 1.307 (2) | O5—C11 | 1.310 (3) |
| O3—C4 | 1.238 (2) | O6—C14 | 1.238 (2) |
| N1—C4 | 1.340 (2) | N2—C14 | 1.344 (3) |
| N1—C5 | 1.420 (2) | N2—C15 | 1.416 (2) |
| C4—N1—C5 | 122.71 (16) | C14—N2—C15 | 122.62 (16) |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O2—H1O⋯O3 | 0.86 (2) | 1.64 (3) | 2.486 (2) | 167 (2) |
| N1—H1N⋯O4$^{i}$ | 0.86 (2) | 1.99 (2) | 2.832 (2) | 166 (2) |
| O5—H5O⋯O6 | 0.83 (3) | 1.67 (3) | 2.486 (2) | 169 (3) |
| N2—H2N⋯O1$^{ii}$ | 0.85 (2) | 2.02 (2) | 2.867 (2) | 173 (2) |

Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $1-x, 1-y, 1-z$.

H atoms were located in a difference map and the positions of those bonded to O and N atoms were allowed to refine, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O) and 1.2$U_{\rm eq}$(N). The remaining H atoms were included in the refinement in geometrically idealized positions, with C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

## References

Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.

Budavari, S. (1989). *Merck Index*, 11th ed., pp. 895–896. Rahway, New Jersey: Merck & Co.

Fan, H.-F. (1991). *SAPI*91. Rigaku Corporation, Tokyo, Japan.

Flett, L. H. & Gardver, W. H. (1952). *Maleic Anhydride Derivatives*. New York: John Wiley Inc.

Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Luskin, L. S. (1974). *Functional Monomers*, Vol. 2, edited by R. H. Yokum & E. B. Nyquist, pp. 357–554. New York: Marcel Dekker Inc.

Lutz, R. E. (1940). *Org. Synth.* **20**, 29–32.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Parvez, M., Shahzadi, S., Shahid, S. & Ali, S. (2004). *Acta Cryst.* E**60**, o2079–o2081.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Westrenen, J. V., Roggen, R. M., Hoefinagal, M. A., Peters, J. A., Kieboom, A. P. G. & Bekkam, H. V. (1990). *Tetrahedron*, **46**, 5741–5758.