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3-[(4-Bromoanilino)carbonyl]prop-2-enoic acid

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

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The crystal structure of the title compound, $C_{10}H_8BrNO_3$, establishes that condensation of 4-bromoaniline with maleic anhydride gives the title compound at room temperature. There are strong intermolecular N—H···O hydrogen bonds [N···O = 2.814 (3) Å and 2.817 (3) Å] linking the molecules into sheets lying parallel to the *bc* plane. The carboxyl groups adopt an *anti*-planar conformation and participate in intramolecular O—H···O hydrogen bonds [O···O = 2.484 (3) and 2.487 (3) Å]. There are two molecules in the asymmetric unit.

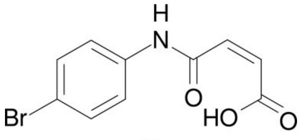
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.083
Data-to-parameter ratio = 16.3

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

Comment

Maleic anhydride, also known as 2,5-furandione, *cis*-butenedioic anhydride, dihydro-2,5-dioxofuran and toxilic anhydride, is used to prepare certain agricultural chemicals, dye intermediates and pharmaceuticals (Budavari, 1989). Inhalation of sub-acute levels of maleic anhydride can cause severe headache, nosebleeds, nausea and temporary impairment of vision. Besides these toxic effects, maleic anhydride has gained importance in the synthesis of carboxylic acids because of its high reactivity towards nucleophiles, *e.g.* aniline, alcohol and phenol. We therefore used maleic anhydride to synthesize 2-[(4-bromoanilino)carbonyl]propinoic acid, (I), and to unequivocally establish that the condensation of 4-bromoaniline with maleic anhydride gives (I) at room temperature. In this article, the structure of (I) is reported.



(I)

The structure of (I) has two molecules in the asymmetric unit (Fig. 1). In each molecule, the bromoaniline and carbonylpropionic acid moieties are almost coplanar. The mean planes formed by atoms C1–C6 and by O1–O3/C1–C4 are inclined at 6.30 (15)° in one molecule, while the corresponding angle in the second molecule has a value of 7.67 (15)°. However, a close examination of the deviations of atoms from these mean planes shows that atoms N1 and Br1 lie 0.027 (3) and 0.019 (3) Å, respectively, out of the plane of the benzene ring on the same side. In the second molecule, atoms N2 and Br2 are also displaced on the same side of the benzene ring, by 0.011 (4) and 0.020 (3) Å, respectively. The carbonylpropionic acid moiety in one of the molecules is more twisted than that in the other molecule, the maximum deviations from the O1–O3/C1–C4 and O4–O6/C11–C14 mean planes being 0.043 (2) (for O2) and 0.024 (2) Å (for O5); atoms N1 and N2 lie

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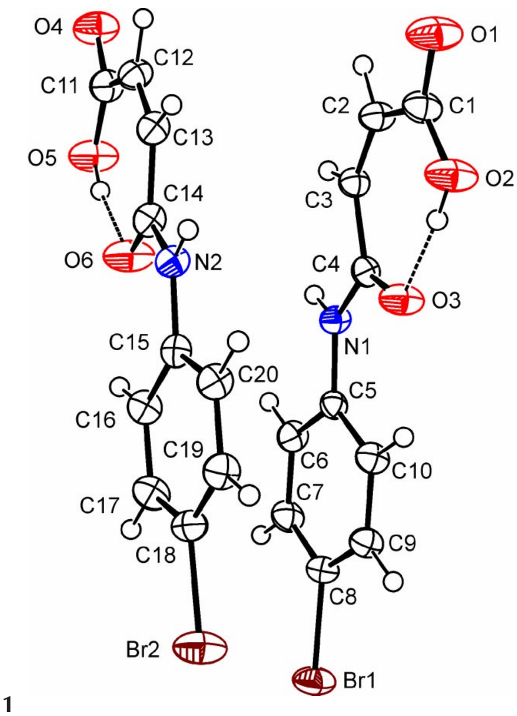

Figure 1
*ORTEP*II (Johnson, 1976) drawing of the asymmetric unit of (I), with displacement ellipsoids plotted at the 50% probability level.

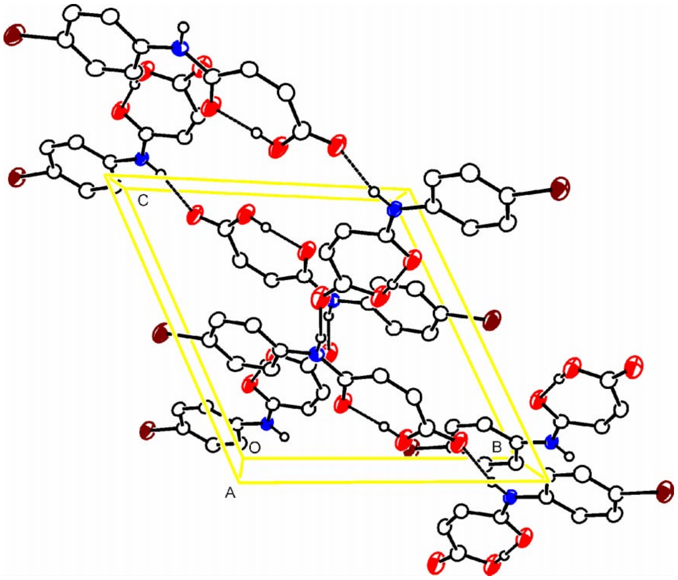

Figure 2
*ORTEP*II (Johnson, 1976) drawing of the crystal packing, showing the hydrogen bonding (dashed lines).

0.034 (3) and 0.018 (3) Å, respectively, out of these planes. In each case, the HC═CH H atoms are *cis* to each other while the –NH and –C═O group are *trans* with respect to each other. All bond lengths and bond angles are within expected values. There is a network of strong intermolecular hydrogen bonds of the type N—H···O involving the aniline N and carbonyl O atoms of the acid groups, thus linking the molecules into sheets lying parallel to the *bc* plane, with N···O separations of 2.814 (3) and 2.817 (3) Å. The carboxyl groups adopt an *anti*-planar conformation wherein the hydroxyl H atoms participate in intramolecular O—H···O hydrogen bonds, with O···O separations of 2.484 (3) and 2.487 (3) Å, forming seven-membered rings. Details of hydrogen bonds are given in Table 1 and Fig. 2.

Experimental

The title compound was prepared by the method reported by Shahid *et al.* (2003). Crystals suitable for crystallographic study were grown from a concentrated solution of (I) in ethanol. Slow evaporation of the solvent at room temperature after several days yielded fine crystals.

Crystal data

$C_{10}H_8BrNO_3$
$M_r = 270.08$
Triclinic, $P\overline{1}$
$a = 10.481$ (2) Å
$b = 10.844$ (2) Å
$c = 11.151$ (3) Å
$\alpha = 106.576$ (9)°
$\beta = 106.920$ (10)°
$\gamma = 111.192$ (10)°
$V = 1016.4$ (4) Å^3
$Z = 4$
$D_x = 1.765$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8520 reflections
$\theta = 2.2$–27.5°
$\mu = 4.03$ mm^{-1}
$T = 173$ (2) K
Block, colorless
0.12 × 0.12 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.631$, $T_{\max} = 0.724$
8520 measured reflections
4621 independent reflections
3578 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.5°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.083$
$S = 1.04$
4621 reflections
283 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.49$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O3	0.94 (4)	1.56 (4)	2.487 (3)	171 (3)
O5—H5O···O6	0.85 (4)	1.65 (4)	2.484 (3)	166 (4)
N1—H1N···O4$^{\rm i}$	0.88 (3)	1.95 (3)	2.817 (3)	173 (2)
N2—H2N···O1$^{\rm ii}$	0.80 (3)	2.02 (3)	2.813 (3)	173 (3)

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $2 - x, 1 - y, 1 - z$.

H atoms were located in a difference map and those bonded to O and N atoms were allowed to refine freely, whereas all other H atoms were included in the refinements as riding, with C—H = 0.95 Å; $U_{\rm iso}$(H) values were set at 1.5 (hydroxyl H atoms) and 1.2 (other H atoms) times $U_{\rm eq}$ of the atoms to which they are bonded. 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.

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