

## *p*-Formamidobenzoic acid

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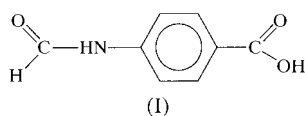
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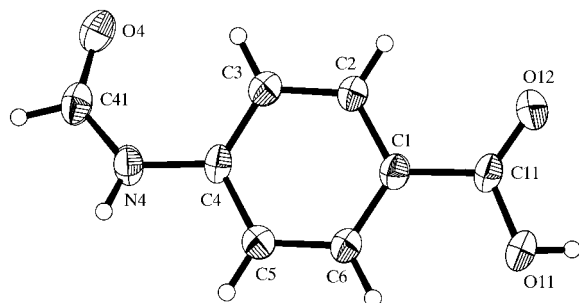
The title compound, C<sub>8</sub>H<sub>7</sub>NO<sub>3</sub>, is an aromatic amide that forms an extensive hydrogen-bond network within the crystal. The crystals were obtained while preparing derivatives of benzoic acid as intermediaries in the synthesis of acridones.

### Comment

The title compound, (I), is an aromatic amide obtained during the final stages of the synthesis of 2-[(4-carboxyphenyl)-amino]benzoic acid (Novoa de Armas *et al.*, 2000), which is used as an intermediate in the synthesis of acridones. Crystals of (I) were isolated as pale-yellow plates, possibly formed as a colateral by-product in the reaction of 2-chlorobenzoic acid with 4-aminobenzoic acid in the presence of K<sub>2</sub>CO<sub>3</sub>, dimethylformamide and copper powder. In previous work on *N*-aromatic amides, the crystal structures of the amides have



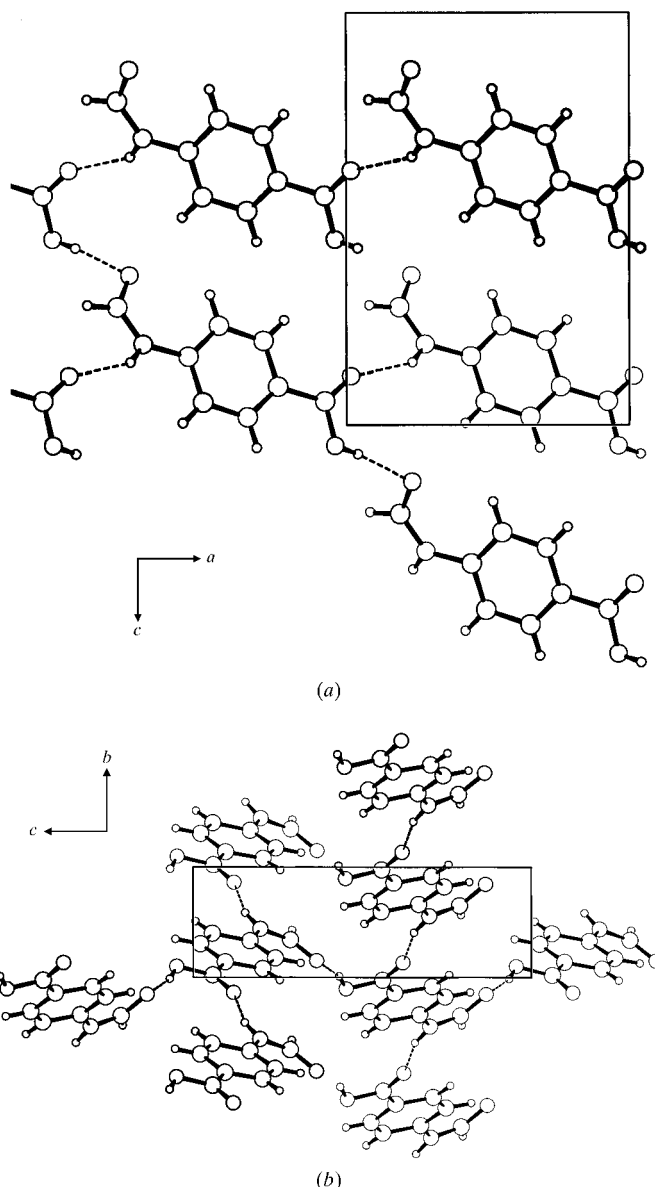
been classified into three types according to their scheme of N—H···O=C hydrogen bonds, *i.e.* 2<sub>1</sub>, glide and translation (Haisa *et al.*, 1980). The hydrogen-bond pattern in the present structure may be used to corroborate previous works in the systematization of crystal structures of aromatic compounds.



**Figure 1**

Plot showing the atomic numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

The bond lengths and angles in (I) are similar to those reported for *p*-amidoperbenzoic acids (Feeder & Jones, 1996) and *N*-aromatic amides (Kashino *et al.*, 1986). In the crystal, nearly flat molecules lie almost parallel to (010) and extend approximately along **a**. The dihedral angle between the least-square planes involving the benzene ring (*A*) and the amide group (*B*) is 3.3 (1)°. The dihedral angle between the least-square planes involving *A* and the formyl group (*C*) is 15.0 (1)°, and between planes *B* and *C* is 12.9 (1)°. The formyl group is out of the plane formed by *A* and *B*. An extensive hydrogen-bond network is formed within the crystal. Hydrogen bonds from the acid proton to the amido carbonyl O atom (O11···O4) are formed by a combination of a *c* glide and a translation in *a*. The imido H atom is hydrogen bonded to the acid carbonyl O12 atom of molecules related by *a* and *b* translations (Fig. 2*a*). The hydrogen-bond motif forming this



**Figure 2**

Projections of the crystal structure viewed (a) along **b** and (b) along **a** showing the crosslinked network.

two-dimensional network is similar to those observed in *p*-amidoperbenzoic acids (Feeder & Jones, 1996). An N4—H4···O12 intermolecular hydrogen-bond connects translated molecules into a one-dimensional infinite chain running parallel to the  $[\bar{1}10]$  and  $[110]$  directions, respectively. The chains are crosslinked by O11—H12···O4 hydrogen bonds, resulting in a three-dimensional network (Fig. 2*b*). The hydrogen-bond motifs in the crystal structure can be described using Etter's graph-set notation (Etter *et al.*, 1990) as a first-order  $C(10)$  network and a second-order network of the type  $C_2^2(8)C(10)$ , obtained using the *RPLUTO* program implemented in a recent version of the Cambridge Structural Database (April 2000 release; Allen & Kennard, 1993).

## Experimental

During the recrystallization of 2-[(4-carboxyphenyl)amino]benzoic acid (Novoa de Armas *et al.*, 2000) from ethanol–water (1:1), pale-yellow plates of the title compound were obtained.

### Crystal data

$C_8H_7NO_3$	$D_x = 1.491 \text{ Mg m}^{-3}$
$M_r = 165.15$	Cu $K\alpha$ radiation
Monoclinic, $Pc$	Cell parameters from 58 reflections
$a = 8.0595 (7) \text{ \AA}$	$\theta = 7.50\text{--}27.64^\circ$
$b = 3.8631 (2) \text{ \AA}$	$\mu = 0.980 \text{ mm}^{-1}$
$c = 11.812 (1) \text{ \AA}$	$T = 293 \text{ K}$
$\beta = 90.160 (8)^\circ$	Plate, pale yellow
$V = 367.77 (5) \text{ \AA}^3$	$0.48 \times 0.36 \times 0.06 \text{ mm}$
$Z = 2$	

### Data collection

Siemens <i>P4</i> four-circle diffractometer	$R_{\text{int}} = 0.080$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 69.07^\circ$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$h = -1 \rightarrow 9$
$T_{\text{min}} = 0.609$ , $T_{\text{max}} = 0.943$	$k = -1 \rightarrow 4$
1166 measured reflections	$l = -14 \rightarrow 14$
789 independent reflections	3 standard reflections every 100 reflections
769 reflections with $F^2 > 2\sigma(F^2)$	intensity decay: none

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0921P)^2 + 0.0348P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.129$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.134$	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
789 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
111 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.066 (8)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O11—C11	1.331 (4)	N4—C4	1.412 (4)
C11—O12	1.209 (4)	N4—C41	1.337 (4)
O4—C41	1.227 (4)		
C1—C11—O11	113.6 (3)	C3—C4—N4	123.0 (3)
C1—C11—O12	123.6 (3)	N4—C4—C5	116.7 (3)
O11—C11—O12	122.8 (3)	O4—C41—N4	126.7 (3)
C4—N4—C41	128.5 (3)		

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{---}H\text{---}A$	$D\text{---}H$	$H\text{---}A$	$D\text{---}A$	$D\text{---}H\text{---}A$
N4—H4···O12 <sup>i</sup>	0.86	2.09	2.832 (3)	145
O11—H11···O4 <sup>ii</sup>	0.82	1.82	2.618 (3)	164

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

The H atoms were calculated geometrically and included in the refinement, but were constrained to ride on their parent atoms (O—H = 0.82, N—H = 0.86 and C—H = 0.93  $\text{\AA}$ ). The isotropic displacement parameters of the H atoms were fixed to  $1.5U_{\text{eq}}$  of their parent atoms. The absolute structure was not determined as the Flack (1983) parameter was indeterminate [given as 0.0 (4)] due to the lack of sufficient anomalous scatterers.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 1990), *PARST* (Nardelli, 1983, 1995) and *PARSTCIF* (Nardelli, 1991).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1154). Services for accessing these data are described at the back of the journal.

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