

## 2-Furancarboxanilide

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## Key indicators

Single-crystal X-ray study

$T = 296\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

$R$  factor = 0.050

$wR$  factor = 0.130

Data-to-parameter ratio = 14.5

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

In the title compound, *N*-phenylfuran-2-carboxamide,  $\text{C}_{11}\text{H}_9\text{NO}_2$ , the  $\text{C}=\text{O}$  group is *anti* in relation to the amide  $\text{NH}$  group. The phenyl ring is twisted by  $147.67(14)^\circ$  about the  $\text{C}(\text{phenyl})-\text{N}$  bond with respect to the planar 2-furamide fragment. The amide  $\text{H}$  atom is the donor in a bifurcated hydrogen bond involving an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  [ $2.682(2)\text{ \AA}$ ] interaction with the furan  $\text{O}$  atom and an intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond [ $3.084(2)\text{ \AA}$ ] with the amide  $\text{O}$  atom. According to the graph-set assignment, the intramolecular hydrogen-bond pattern is of the  $S(5)$  type. The intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond links the molecules into infinite  $C(4)$  chains along the  $a$  axis.

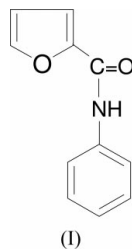
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## Comment

The title compound, (I), first prepared by Baum (1904), has now been investigated by X-ray single-crystal diffraction.



Compound (I) was prepared as part of our study of aryl-substituted benzothiazole derivatives. These compounds exhibit complex biological activity, such as anti-infective, antifungal, anthelmintic or antitumor activities (Tralić-Kulenović *et al.*, 1993; Racané *et al.*, 2001). The thionation of the corresponding *N*-arylfuramides leads to *N*-arylfuramides, which are precursors in the synthesis of 2-(2- or 3-furyl)benzothiazoles by oxidative cyclization (Fišer-Jakić *et al.*, 1980). 2-Furancarboxanilide is known for its antifungal activity. A survey of the Cambridge Structural Database (Version 5.25, November 2003; Allen, 2002) reveals only two structures of 2-furancarboxamides, *N*-(4,6-dimethylpyridine-2-yl)(furan-2-yl)carboxamide, (II) (Rodier *et al.*, 1991), and *N*-(2-chloroethyl)-2-furamide, (III) (Galešić *et al.*, 1987).

The molecule of (I) is not planar: the dihedral angle between the phenyl and furan rings is  $32.30(9)^\circ$ . The planarity of the 2-furancarboxamide moiety is preserved by the presence of a strong intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond between the amide  $\text{N}$  atom and the furan  $\text{O}$  atom, thus forming a five-membered chelate ring (Fig. 1 and Table 2). A corresponding intramolecular hydrogen bond is formed in (II) [ $\text{N}\cdots\text{O}$   $2.668(2)\text{ \AA}$  and  $108(2)^\circ$ ] and in (III) [ $\text{N}\cdots\text{O}$   $2.727(4)\text{ \AA}$  and  $110(4)^\circ$ ].

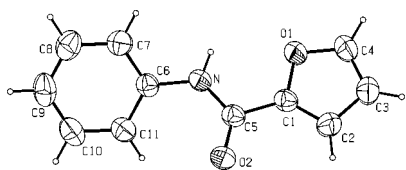


Figure 1

The molecular structure of (I), with the atom-labelling scheme, showing displacement ellipsoids at the 50% probability level. Atoms N and O1 are involved in the formation of an intramolecular hydrogen bond.

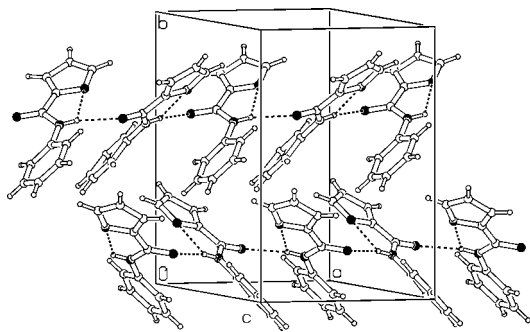


Figure 2

A view of the crystal structure of (I), showing infinite  $C(4)$  chains extending along the  $a$  axis. Hydrogen bonds are indicated by dashed lines. O atoms are shown as solid circles, C atoms as open circles and N atoms as shaded circles.

The C5—N bond length of 1.350 (2) Å shows remarkable  $\pi$ -electron delocalization within the planar 2-furamide moiety. On the other hand, the C6—N bond, with a length of 1.416 (2) Å, can be regarded as a single bond. The analogous C—N bond lengths in (II) are 1.360 (2) and 1.403 (2) Å, respectively, and 1.337 (6) and 1.437 (6) Å, respectively, in (III). The O1—C1 and O1—C4 bonds are 1.368 (2) and 1.361 (2) Å, respectively, which corresponds to an average O—C $sp^2$  distance of 1.368 Å (Allen *et al.*, 1987). The C1=C2 bond [1.347 (2) Å] within the furan ring is within the expected range, while the C3=C4 bond [1.328 (2) Å] is slightly shorter using a  $3\sigma$  criterion (1.341 Å; Allen *et al.*, 1987). Other bond distances in both the furan and the phenyl rings are consistent with expected values (Allen *et al.*, 1987).

The dihedral angle between the furan and pyridine rings in (II) is 6.6 (3)°, in contrast with the analogous dihedral angle in (I), between the furan and phenyl rings, of 32.30 (9)°. The additional C—H $\cdots$ O intramolecular hydrogen bond [C $\cdots$ O 2.927 (2) Å and 120 (2)°] between the pyridine C atom and the keto O atom contributes to the planarity of (II). Similar intramolecular hydrogen bonding is not found in the structure of (I). Although the C11 $\cdots$ O2 distance is 2.953 (2) Å, the H11 $\cdots$ O2 distance is 2.504 (1) Å and the C11—H11 $\cdots$ O2 angle is only 109.9 (1)°. Therefore, it is obvious that atom H11 is not directed towards the O2 electron lone pair, explaining the twisted molecular conformation of (I).

Molecules of (I) are joined by N—H1N $\cdots$ O2<sup>i</sup> [symmetry code: (i)  $x - \frac{1}{2}, y, \frac{1}{2} - z$ ] intermolecular hydrogen bonds [N $\cdots$ O 3.084 (2) Å and 161 (2)°] into infinite chains running parallel to the  $a$  axis (Table 2). The pattern is specified as  $C(4)$  chains. A similar packing mode is found in the structure of

(III) [N—H $\cdots$ O 2.872 (5) Å and 150 (4)°], where intermolecular hydrogen bonding connects the molecules into infinite  $C(4)$  chains along the  $b$  axis, while in the structure of (II), the molecules form pairs through weak C—H $\cdots$ O hydrogen bonds [C $\cdots$ O 3.316 (2) Å and 149 (2)°].

## Experimental

Compound (I) was prepared according to the procedure of Baum (1904). Single crystals were obtained by evaporation of an ethanol solution.

### Crystal data

$C_{11}H_9NO_2$   
 $M_r = 187.19$   
 Orthorhombic,  $Pbca$   
 $a = 8.699$  (2) Å  
 $b = 12.983$  (2) Å  
 $c = 16.712$  (2) Å  
 $V = 1887.4$  (5) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.318$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 5046 reflections  
 $\theta = 15$ –27°  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
 Prism, colourless  
 0.64 × 0.51 × 0.42 mm

### Data collection

Oxford Diffraction Xcalibur2 diffractometer with Sapphire 3 CCD detector  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 32 112 measured reflections  
 1903 independent reflections

1758 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.049$   
 $\theta_{max} = 26.5^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -16 \rightarrow 16$   
 $l = -20 \rightarrow 20$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.130$   
 $S = 1.19$   
 1903 reflections  
 131 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 0.0157P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.15$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.14$  e Å<sup>-3</sup>

H atoms treated by a mixture of independent and constrained refinement

Table 1

Selected geometric parameters (Å, °).

O1—C4	1.361 (2)	C1—C2	1.347 (2)
O1—C1	1.368 (2)	C1—C5	1.474 (2)
O2—C5	1.232 (2)	C2—C3	1.416 (3)
N—C5	1.349 (2)	C3—C4	1.328 (2)
N—C6	1.416 (2)		
C5—N—C6	126.6 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N—H1N $\cdots$ O1	0.85 (2)	2.28 (2)	2.682 (2)	109 (2)
N—H1N $\cdots$ O2 <sup>i</sup>	0.85 (2)	2.27 (2)	3.084 (2)	161 (2)

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

H atoms bonded to the phenyl and furan C atoms were introduced at calculated positions and refined using a riding model [ $U_{iso}(H) = 1.2U_{eq}(C)$  and C—H = 0.93 Å]. The H atom on the amide N atom was found in a difference Fourier electron-density map and refined freely.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2003); data reduction:

*CrysAlisRED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON98* (Spek, 1998); software used to prepare material for publication: *SHELXL97*.

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## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Allen, F. H., Kennard, O., Watson, D. G., Brammer L. & Orpen, A. G. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Baum, E. (1904). *Chem. Ber.* **37**, 2949.
- Fišer-Jakić, L., Karaman, B. & Jakopčić, K. (1980). *Croat. Chem. Acta*, **53**, 69–79.
- Galešić, N., Kovačević, K., Vlahov A. & Galešić, M. (1987). *Acta Cryst.* **C43**, 2350–2353.
- Oxford Diffraction (2003). *CrysAlisCCD* and *CrysAlisRED*. Versions 1.170. Oxford Diffraction Ltd, Abingdon, Oxford, England.
- Racané, L., Tralić-Kulenović, V., Fišer-Jakić, L., Boykin, D. W. & Karminski-Zamola, G. (2001). *Heterocycles*, **55**, 2085–2098.
- Rodier, P. N., Cense, J. M., Robert, J.-M. & Le Baut G. (1991). *Acta Cryst.* **C47**, 2688–2690.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (1998). *PLATON98 for Windows*. University of Utrecht, The Netherlands.
- Tralić-Kulenović, V., Fišer-Jakić, L. & Lazarević, Z. (1993). *Spectrosc. Lett.* **26**, 1771–1784.