1041 reflections
133 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.115P)^2 + 0.82P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Cl(1) | 0.2617 (1) | 0.0006 (1) | 0.6465 (1) | 0.053 (1) |
| N(1) | 0.3631 (3) | 0.2008 (3) | 0.4717 (2) | 0.043 (1) |
| C(2) | 0.5161 (4) | 0.2907 (3) | 0.4568 (2) | 0.038 (1) |
| C(21) | 0.6955 (4) | 0.2773 (3) | 0.5328 (2) | 0.046 (1) |
| O(22) | 0.8347 (3) | 0.3757 (2) | 0.5201 (2) | 0.056 (1) |
| O(23) | 0.6987 (3) | 0.1826 (3) | 0.5958 (2) | 0.074 (1) |
| C(3) | 0.5008 (5) | 0.3839 (3) | 0.3769 (2) | 0.046 (1) |
| C(4) | 0.3265 (5) | 0.3803 (4) | 0.3121 (2) | 0.056 (1) |
| C(5) | 0.1732 (5) | 0.2860 (4) | 0.3294 (2) | 0.054 (1) |
| C(6) | 0.1938 (4) | 0.1955 (3) | 0.4112 (2) | 0.050 (1) |
| O(100) | 0.1349 (3) | 0.3455 (3) | 0.6517 (2) | 0.058 (1) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| N(1)—C(6) | 1.330 (3) | C(21)—O(22) | 1.300 (3) |
| N(1)—C(2) | 1.335 (3) | C(3)—C(4) | 1.388 (4) |
| C(2)—C(3) | 1.366 (4) | C(4)—C(5) | 1.369 (4) |
| C(2)—C(21) | 1.505 (4) | C(5)—C(6) | 1.373 (4) |
| C(21)—O(23) | 1.197 (3) | | |
| C(6)—N(1)—C(2) | 123.0 (2) | O(22)—C(21)—C(2) | 112.8 (2) |
| N(1)—C(2)—C(3) | 119.7 (2) | C(2)—C(3)—C(4) | 118.7 (3) |
| N(1)—C(2)—C(21) | 114.7 (2) | C(5)—C(4)—C(3) | 120.0 (3) |
| C(3)—C(2)—C(21) | 125.6 (2) | C(4)—C(5)—C(6) | 119.5 (3) |
| O(23)—C(21)—O(22) | 127.2 (3) | N(1)—C(6)—C(5) | 119.2 (3) |
| O(23)—C(21)—C(2) | 120.0 (3) | | |

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refined by full-matrix least squares (*SHELXL*93; Sheldrick, 1993) with anisotropic displacement parameters for all non-H atoms. H atoms were located by difference methods and included with both positional and displacement parameters refined. Other programs used were *Xtal*3.2 (Hall, Flack & Stewart, 1992) and *PLATON* (Spek, 1990).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1154). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). C**51**, 1427–1429

# 2,3,4,5-Tetrafluoro-*N*-(4-fluorophenyl)-6-hydroxybenzamide: an Example of a Combined Inter- and Intramolecular O···H···O Bifurcated Hydrogen Bond

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## Abstract

Although the amide dimensions in the title molecule, $C_{13}H_6F_5NO_2$, show little difference from those of its unfluorinated analogue [Kashino, Matsushita, Iwamoto, Yamaguchi & Haisa (1986). *Acta Cryst.* C**42**, 457–462], the hydroxyl positions are reversed. Whereas in the former, the OH takes part in intra- [O···O 2.508 (3), H···O 1.69 (4) Å; O—H···O 153 (4)°] and intermolecular [O···O 3.029 (3), H···O 2.57 (4) Å; O—H···O 113 (3)°] bifurcated hydrogen bonding, in the latter structure the hydroxyl associates intramolecularly with the amide H atom as well as the amide O atom of a neighbouring molecule.

## Comment

The structure determination reported here was carried out as part of a general investigation into the preparation of fluorine-containing arylnitrones and their use as 1,3-dipolar reagents (DuBoisson, 1986). From the reaction of *C*-(pentafluorophenyl)-*N*-(4-fluorophenyl)nitrone

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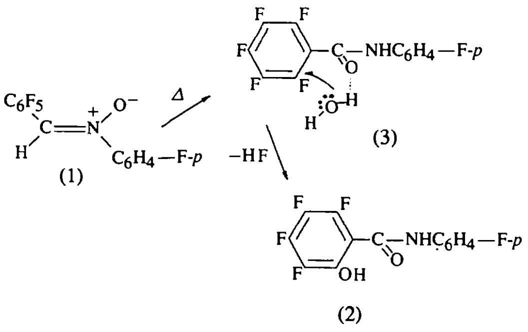
(1) with styrene in toluene under reflux, a white solid (m.p. 303–304 K) was isolated by dry column flash chromatography (DCFC) on silica, the NMR spectra of which indicated that rearrangement of the nitrone (1) had taken place. To confirm this, (1) was heated alone in toluene under reflux and the major component of the product mixture was purified by DCFC and found to be identical to that obtained from the reaction of (1) with styrene. It was found to have the molecular formula $C_{13}H_6F_5NO_2$, compared to that of nitrone (1), $C_{13}H_5F_6NO$, indicating that an F atom had been replaced by a hydroxyl group and an X-ray determination was required to determine the structure as the hydroxybenzamide (2). It is thought that adventitious moisture present was responsible for the replacement of F by OH with hydrogen bonding of the water to the carbonyl O atom of the initially formed amide (3), causing replacement of an *ortho*-fluorine to give the title product (2).



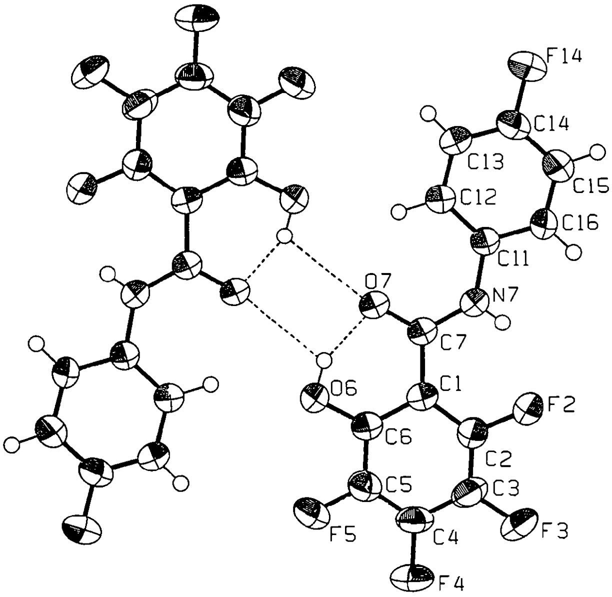


Fig. 1. The title molecule and hydrogen-bonded neighbour, with atomic numbering scheme, drawn using *ORTEPII* (Johnson, 1976). Ellipsoids have been drawn at the 50% probability level.

## Experimental

*C*-Pentafluorophenyl-*N*-(4-fluorophenyl)nitrone (1) (1.00 g, 3.28 mmol) in anhydrous toluene (30 ml) was heated under reflux for 30 h in a flask which had been purged with nitrogen. The crude product mixture was separated by dry column flash chromatography [Merck Kieselgel $60_{GF284}$; eluant light petroleum (b.p. 313–333 K) and $CH_2Cl_2$ 9:1 to 6:4 *v*/*v*] to give a white solid identified as the title compound (2) (0.45 g, 1.49 mmol, 45%; found C 51.8, H 1.8, F 31.5, N 4.7%, $M^+$ 303; $C_{13}H_6F_5NO_2$ requires C 51.5, H 2.0, F 31.4, N 4.6%, *M* 303; m.p. 403–404 K). The product was recrystallized from toluene.

*Crystal data*

$C_{13}H_6F_5NO_2$
$M_r = 303.19$
Triclinic
$P\bar{1}$
$a = 5.842\,(1)$ Å
$b = 10.309\,(1)$ Å
$c = 11.062\,(1)$ Å
$\alpha = 66.53\,(1)°$
$\beta = 76.15\,(1)°$
$\gamma = 73.03\,(1)°$
$V = 578.7\,(3)$ Å$^3$
$Z = 2$
$D_x = 1.740$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 7.50–12.50°$
$\mu = 0.1630$ mm$^{-1}$
$T = 296$ K
Plate
0.50 × 0.50 × 0.05 mm
Colourless

*Data collection*

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2071 measured reflections
2019 independent reflections
1303 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.029$

$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 11$
$l = -11 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity decay: insignificant

*Refinement*

Refinement on *F*
$R = 0.0528$
$wR = 0.0530$
$S = 1.390$
1303 reflections
214 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{\rm max} = 0.0818$
$\Delta\rho_{\rm max} = 0.252$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.192$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| C1 | 0.4903 (5) | 0.3711 (3) | 0.6878 (3) | 0.050 (2) |
| C2 | 0.2789 (6) | 0.3306 (3) | 0.7636 (3) | 0.058 (3) |
| C3 | 0.1906 (6) | 0.2262 (3) | 0.7560 (3) | 0.063 (3) |

| | | | | |
|---|---|---|---|---|
| C4 | 0.3123 (7) | 0.1556 (3) | 0.6684 (3) | 0.063 (3) |
| C5 | 0.5211 (7) | 0.1921 (3) | 0.5922 (3) | 0.060 (3) |
| C6 | 0.6123 (6) | 0.2969 (3) | 0.5993 (3) | 0.052 (3) |
| C7 | 0.5979 (6) | 0.4830 (3) | 0.6917 (3) | 0.049 (2) |
| C11 | 0.5603 (6) | 0.6597 (3) | 0.8006 (3) | 0.052 (2) |
| C12 | 0.7621 (6) | 0.7168 (3) | 0.7310 (3) | 0.061 (3) |
| C13 | 0.8193 (7) | 0.8174 (4) | 0.7645 (4) | 0.065 (3) |
| C14 | 0.6774 (7) | 0.8613 (3) | 0.8636 (3) | 0.067 (3) |
| C15 | 0.4746 (7) | 0.8090 (4) | 0.9323 (3) | 0.068 (3) |
| C16 | 0.4168 (7) | 0.7074 (3) | 0.9004 (3) | 0.061 (3) |
| O6 | 0.8170 (4) | 0.3240 (2) | 0.5177 (2) | 0.065 (2) |
| O7 | 0.7885 (4) | 0.5078 (2) | 0.6190 (2) | 0.065 (2) |
| N7 | 0.4866 (6) | 0.5548 (3) | 0.7752 (3) | 0.061 (3) |
| F2 | 0.1467 (3) | 0.3965 (2) | 0.8510 (2) | 0.072 (2) |
| F3 | −0.0141 (4) | 0.1927 (2) | 0.8318 (2) | 0.089 (2) |
| F4 | 0.2253 (4) | 0.0552 (2) | 0.6602 (3) | 0.089 (2) |
| F5 | 0.6380 (4) | 0.1236 (2) | 0.5064 (2) | 0.082 (2) |
| F14 | 0.7374 (5) | 0.9589 (2) | 0.8962 (2) | 0.097 (2) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| C1—C2 | 1.389 (4) | C7—N7 | 1.341 (4) |
| C1—C6 | 1.413 (4) | C11—C12 | 1.392 (4) |
| C1—C7 | 1.486 (4) | C11—C16 | 1.383 (4) |
| C6—O6 | 1.342 (4) | C11—N7 | 1.416 (4) |
| C7—O7 | 1.236 (4) | | |
| C2—C1—C6 | 115.8 (3) | O7—C7—N7 | 121.2 (3) |
| C2—C1—C7 | 126.3 (3) | C12—C11—C16 | 119.6 (3) |
| C6—C1—C7 | 117.9 (3) | C12—C11—N7 | 124.2 (3) |
| C1—C7—O7 | 119.6 (3) | C16—C11—N7 | 116.3 (3) |
| C1—C7—N7 | 119.2 (3) | C7—N7—C11 | 129.2 (3) |

Data collection: *CAD*-4 *Diffractometer Control Software* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Diffractometer Control Software*. Data reduction: *PROCESS TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *LS TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *FINISH TEXSAN*. Literature survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1141). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). C**51**, 1429–1431

# 1-(2-Benzothiazolyl)-3-methoxycarbonylmethyl-2-thioxo-4-imidazolidinone, $C_{13}H_{11}N_3O_3S_2$

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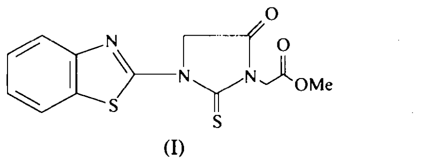
## Abstract

The reaction of methyl isothiocyanatoacetate with 2-aminothiophenol gave as the unexpected major product the title compound, methyl 1-(2-benzothiazolyl)-4-oxo-2-thioxo-3-imidazolidinylacetate, which was identified by X-ray crystal structure analysis.

## Comment

In a continuation of our studies on the synthesis of heterocyclic compounds obtained from isothiocyanato carboxylates, we have reported the synthesis of 2-amino substituted derivatives of benzimidazoles (Floch, Uher & Leško, 1989). It was expected that the synthetic procedures developed there should be similarly applicable to the synthesis of 2-amino-substituted derivatives of benzothiazoles. Nevertheless, the reaction of methyl isothiocyanatoacetate with 2-aminothiophenol furnished, in high yield, an unexpected compound (I) having the



(I)

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