Crystal structure of *N*-benzyl-4-fluorobenzamide, $C_{14}H_{12}FNO$, *at 173 K*

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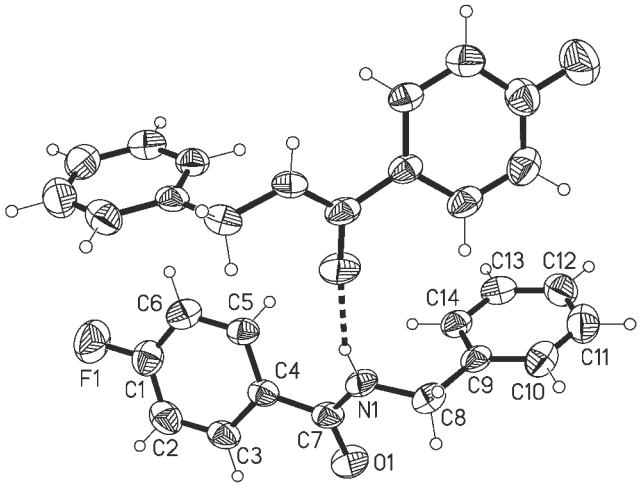

Abstract

$C_{14}H_{12}FNO$, monoclinic, $P2_1/n$ (no. 14), a = 5.6653(2) Å, b = 25.305(1) Å, c = 8.2832(3) Å, β = 92.237(3)°, V = 1186.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.123, T = 173 K.

Source of material

Benzylazide [1] (60 mg, 0.45 mmol) and 2-(diphenylphosphano)-phenyl 4-fluorobenzoate [2] (150 mg, 0.37 mmol) were dissolved in a DMF/water mixture (0.5 mL: 0.05 mL) and heated under microwave conditions (50 Watt) for 20 min. After removal of the solvent under reduced pressure the crude product was purified via column chromatography (PE/EtOAc, 20:1); yield: 71 mg (83 %) of colourless crystals; mp. 143 - 144 °C; elemental analysis — found: C, 73.61 %; H, 5.45 %; N, 6.14 %; calculatd for $C_{14}H_{12}FNO$: C, 73.35 %; H, 5.28 %; N, 6.11 %. After the purification via column chromatography colourless crystals were obtained by slow evaporation of the solvent of a saturated solution of this compound in a mixture of petroleum ether/ethyl acetate. NMR and MS data as well as the melting point are in accordance with the previously published [1].

Experimental details

X-ray intensity data were collected at –100 °C. All atoms (except hydrogen) were treated anisotropically. The coordinates and isotropic displacement parameter of H1 atom were refined isotropically, whereas all the other hydrogen atoms were placed on calculated positions and refined using riding models with isotropic thermal parameters based on the bonded atom and a restrain allowing for a group-wise optimisation of the C—H distances (AFIX 44 and AFIX 24 options [2]).

Discussion

The title compound was obtained as main product from the traceless Staudinger ligation of 2-(diphenylphosphano)phenyl-4-fluorobenzoate with benzylazide in high yield [3]. A similar method using a flow reactor is described in [4]. Other preparation methods, where the respective acid chloride is treated with benzyl amine, are published in [5]. Compounds with amide functions are found in a large number of biomolecules and radiopharmaceuticals. The introduction of fluorine-18 or carbon-11 into biocompatible compounds allows for selective radio-labelling. The title compound has been used already in such investigations [6].

The structure is build up of dimers of neutral molecules, interconnected through a N1–H1···O1' hydrogen bond (symmetry operation for O1': x–½,½–y,z–½) with a N1···O1' distance of 2.780(2) Å. All bond lengths within the $C_{14}H_{12}FNO$ molecule are within the expected ranges. The mean planes through the two phenyl rings are tilded with respect to each other by 65°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.19 × 0.21 × 0.65 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.92 cm^{-1}
Diffractometer, scan mode:	Bruker-Nonius Apex X8CCD, φ/ω
$2\theta_{max}$:	52.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16757, 2399
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1610
$N(param)_{refined}$:	169
Programs:	SHELXL-97, SHELXTL [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.656(3)	0.2466(8)	0.142(2)	0.054(6)
H(2)	4*e*	0.762(2)	0.4454(7)	0.472(1)	0.067
H(3)	4*e*	0.938(3)	0.3611(2)	0.473(1)	0.056
H(5)	4*e*	0.402(2)	0.3065(6)	0.184(1)	0.051
H(6)	4*e*	0.220(3)	0.3896(2)	0.182(1)	0.062
H(8A)	4*e*	1.001(2)	0.18527(7)	0.2734(8)	0.056
H(8B)	4*e*	0.8838(6)	0.1764(1)	0.103(2)	0.056
H(10)	4*e*	0.889(3)	0.0810(2)	0.192(1)	0.063
H(11)	4*e*	0.642(1)	0.0139(9)	0.2886(6)	0.074
H(12)	4*e*	0.308(2)	0.0352(6)	0.4334(9)	0.068
H(13)	4*e*	0.224(3)	0.1239(2)	0.485(1)	0.061
H(14)	4*e*	0.4613(8)	0.1937(8)	0.3929(5)	0.052

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.7568(3)	0.23843(6)	0.2157(2)	0.0427(9)	0.049(1)	0.0350(8)	0.0044(7)	–0.0155(7)	0.0044(7)
O(1)	4*e*	0.9540(3)	0.26235(6)	0.4434(2)	0.075(1)	0.070(1)	0.0513(8)	–0.0002(8)	–0.0424(7)	0.0075(7)
F(1)	4*e*	0.3759(3)	0.47174(5)	0.3263(2)	0.100(1)	0.0591(9)	0.099(1)	0.0206(8)	–0.0077(8)	–0.0131(7)
C(1)	4*e*	0.4790(4)	0.42319(8)	0.3260(2)	0.063(1)	0.049(1)	0.054(1)	0.006(1)	0.003(1)	–0.0025(9)
C(2)	4*e*	0.6888(4)	0.41655(9)	0.4128(2)	0.065(1)	0.052(1)	0.052(1)	–0.012(1)	–0.003(1)	–0.0065(9)
C(3)	4*e*	0.7916(3)	0.36687(8)	0.4123(2)	0.048(1)	0.055(1)	0.037(1)	–0.0118(9)	–0.0097(8)	0.0028(8)
C(4)	4*e*	0.6876(3)	0.32524(7)	0.3264(2)	0.0395(9)	0.049(1)	0.0271(8)	–0.0073(8)	–0.0052(7)	0.0054(7)
C(5)	4*e*	0.4733(3)	0.33393(8)	0.2410(2)	0.040(1)	0.051(1)	0.0360(9)	–0.0030(8)	–0.0062(7)	–0.0009(8)
C(6)	4*e*	0.3676(4)	0.38337(8)	0.2406(2)	0.046(1)	0.062(1)	0.046(1)	0.006(1)	–0.0044(9)	–0.0021(9)
C(7)	4*e*	0.8083(3)	0.27278(7)	0.3329(2)	0.041(1)	0.052(1)	0.0331(9)	–0.0074(8)	–0.0122(7)	0.0093(8)
C(8)	4*e*	0.8527(3)	0.18547(7)	0.2131(2)	0.041(1)	0.052(1)	0.046(1)	0.0079(9)	–0.0038(8)	0.0054(9)
C(9)	4*e*	0.6946(3)	0.14329(7)	0.2818(2)	0.0382(9)	0.046(1)	0.0318(8)	0.0067(8)	–0.0109(7)	0.0010(7)
C(10)	4*e*	0.7454(4)	0.09056(8)	0.2539(2)	0.057(1)	0.053(1)	0.046(1)	0.013(1)	–0.0006(9)	0.0012(9)
C(11)	4*e*	0.6033(4)	0.05120(9)	0.3099(3)	0.074(2)	0.049(1)	0.062(1)	0.006(1)	–0.002(1)	–0.003(1)
C(12)	4*e*	0.4090(4)	0.06335(9)	0.3949(2)	0.062(1)	0.054(1)	0.055(1)	–0.009(1)	–0.008(1)	0.007(1)
C(13)	4*e*	0.3558(3)	0.11559(8)	0.4263(2)	0.041(1)	0.068(1)	0.044(1)	0.002(1)	–0.0016(8)	0.0012(9)
C(14)	4*e*	0.4994(3)	0.15587(8)	0.3698(2)	0.039(1)	0.049(1)	0.041(1)	0.0044(8)	–0.0063(8)	–0.0005(8)

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