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Crystal structure of 2,2-dibromo-1-(4-fluoro-2-(4-fluorophenoxy)phenyl) ethanone, $C_{14}H_8Br_2F_2O_2$

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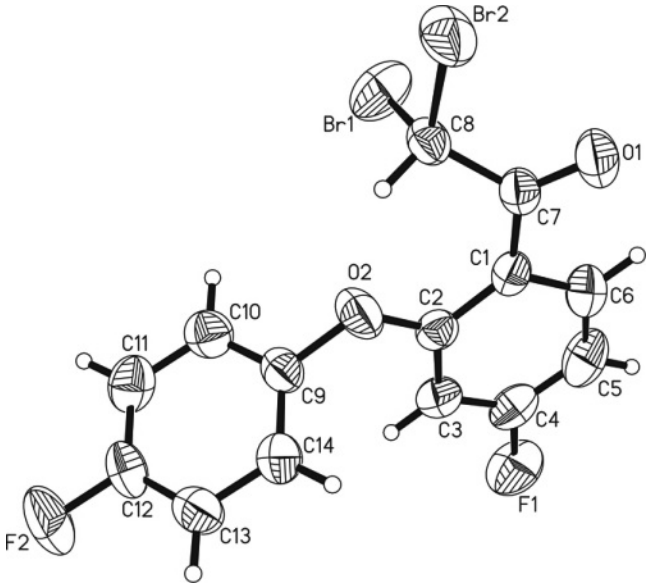

Abstract

$C_{14}H_8Br_2F_2O_2$, monoclinic, $P2_1/c$ (no. 14), a = 11.009(1) Å, b = 15.787(2) Å, c = 8.687(1) Å, β = 112.912(1)°, V = 1390.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0449, $wR_{ref}(F^2)$ = 0.1309, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.13×0.15×0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	58.49 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9717, 2598
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1945
$N(param)_{refined}$:	181
Programs:	SHELX [1]

Source of material

The mixture of sodium *p*-fluorophenol (1.34 g, 10 mmol), 2,4-difluoroacetophenone (1.56 g, 10 mmol), copper(II)chloride (0.02 g) and toluene (10 mL) was stirred at reflux 2 h, and then concentrated under vacuum. After cooling to room temperature, hexamethylene (20 mL) was added to the residue, whereafter the organic layer was washed with brine (5 mL) three times. Subsequently, bromine (3.20 g, 20 mmol) in hexamethylene (5 mL) was added dropwise to the above mentioned solution and stirred for 2 h at room temperature, and washed with saturated sodium bisulfite solution (5 mL). The title compound was obtained in 75% yield when the organic layer was washed with water to neutral, dried over $MgSO_4$, stripped under the condition of reduced pressure. Single crystals suitable for X-ray measurements were obtained by slow evaporation of its ethanol solution at room temperature.

Experimental details

All hydrogen atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 or 0.98 Å and with U_{iso}(H) = 1.2 times U_{eq}(C).

Discussion

Recently, interest in fluorine-containing compounds is continuously growing since fluorinated organic products exhibit unique properties that are of great interest for a variety of applications [2, 3]. The title compound has been obtained during a search for new fuorine-containing compounds with better biological activity. In the title structure, all bond lengths are normal [4] and in a good agreement with those reported previously [5-6]. The atom F1, O2, C7, C9 and the phenyl ring (C1-C6) are coplanar. The dihedral angle between the phenyl rings is 73.22°. The bond angle of C2–O2–C9 is 121.21°.

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.6362	0.9284	0.2903	0.058
H(5)	4*e*	0.7974	1.0977	0.0825	0.081
H(6)	4*e*	0.9325	1.1460	0.3450	0.066
H(8)	4*e*	0.9033	1.0377	0.8056	0.053
H(10)	4*e*	0.5668	0.9652	0.6643	0.064
H(11)	4*e*	0.4429	0.8498	0.6816	0.071
H(13)	4*e*	0.6735	0.6958	0.5520	0.070
H(14)	4*e*	0.8008	0.8107	0.5375	0.067

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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}
Br(1)	4*e*	0.76868(7)	1.16552(5)	0.75140(8)	0.0677(4)	0.1039(6)	0.0682(4)	0.0236(4)	0.0307(3)	–0.0044(3)
Br(2)	4*e*	1.06522(6)	1.13287(4)	0.99619(7)	0.0621(4)	0.0856(5)	0.0488(3)	–0.0275(3)	0.0100(3)	–0.0032(3)
C(1)	4*e*	0.8662(5)	1.0678(3)	0.4794(6)	0.042(2)	0.042(2)	0.043(2)	0.006(2)	0.022(2)	0.005(2)
C(2)	4*e*	0.7772(5)	1.0021(3)	0.4591(6)	0.044(3)	0.039(2)	0.041(3)	0.005(2)	0.019(2)	0.000(2)
C(3)	4*e*	0.6958(5)	0.9721(3)	0.3025(6)	0.053(3)	0.050(3)	0.042(3)	0.001(2)	0.019(2)	–0.005(2)
C(4)	4*e*	0.7073(5)	1.0099(4)	0.1663(6)	0.055(3)	0.068(3)	0.039(3)	0.012(3)	0.018(2)	–0.002(2)
C(5)	4*e*	0.7928(6)	1.0738(4)	0.1779(7)	0.079(4)	0.088(5)	0.045(3)	0.010(4)	0.035(3)	0.017(3)
C(6)	4*e*	0.8729(6)	1.1026(4)	0.3351(7)	0.058(3)	0.060(3)	0.055(3)	–0.003(3)	0.030(3)	0.008(2)
C(7)	4*e*	0.9577(5)	1.1054(3)	0.6397(6)	0.039(2)	0.040(2)	0.051(3)	0.001(2)	0.021(2)	0.004(2)
C(8)	4*e*	0.9245(5)	1.0970(3)	0.7937(6)	0.041(2)	0.047(3)	0.043(3)	–0.010(2)	0.013(2)	–0.001(2)
C(9)	4*e*	0.6937(5)	0.8981(3)	0.5975(6)	0.055(3)	0.044(3)	0.033(2)	–0.012(2)	0.013(2)	0.001(2)
C(10)	4*e*	0.5882(5)	0.9109(3)	0.6416(6)	0.060(3)	0.047(3)	0.052(3)	0.005(2)	0.020(3)	0.006(2)
C(11)	4*e*	0.5148(6)	0.8426(4)	0.6518(7)	0.046(3)	0.070(4)	0.067(4)	0.011(3)	0.028(3)	0.012(3)
C(12)	4*e*	0.5489(5)	0.7645(3)	0.6176(7)	0.039(3)	0.055(3)	0.061(3)	–0.008(2)	0.017(2)	0.013(2)
C(13)	4*e*	0.6531(6)	0.7503(3)	0.5746(7)	0.063(3)	0.043(3)	0.071(4)	–0.004(2)	0.029(3)	–0.003(2)
C(14)	4*e*	0.7281(5)	0.8185(3)	0.5653(7)	0.053(3)	0.056(3)	0.067(3)	–0.008(3)	0.032(3)	–0.005(3)
F(1)	4*e*	0.6317(4)	0.9796(3)	0.0149(4)	0.083(2)	0.110(3)	0.037(2)	0.001(2)	0.016(2)	–0.007(2)
F(2)	4*e*	0.4764(4)	0.6965(2)	0.6275(5)	0.073(2)	0.071(2)	0.125(3)	–0.024(2)	0.046(2)	0.015(2)
O(1)	4*e*	1.0534(4)	1.1454(2)	0.6483(5)	0.059(2)	0.075(3)	0.069(3)	–0.021(2)	0.034(2)	–0.003(2)
O(2)	4*e*	0.7741(4)	0.9675(2)	0.6032(4)	0.094(3)	0.061(2)	0.039(2)	–0.037(2)	0.022(2)	–0.004(2)

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