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Crystal structure of 3-(4-fluorophenyl)-1-(2-methylimidazo-[1,2-*a*]-pyridin-3-yl)prop-2-en-1-one, $C_{17}H_{13}FN_2O$

Yvon Bibila Mayaya Bisseyou*,I, Adjo Adohi-Krou[I], Mahamat Ouattara[II], Drissa Sissouma[III], Emma G. Zoro-Diama[I] and N'dédé Ebby[I]

[I] UFR SSMT Université de Cocody, Laboratoire de Cristallographie et Physique Moléculaire, 22 BP 582 Abidjan 22, Côte d'Ivoire
[II] Université de Cocody, Laboratoire de Chimie Thérapeutique et Synthèse de Médicaments, UFR Sciences Pharmaceutiques, 01 BP V 34 Abidjan 01, Côte d'Ivoire
[III] UFR SSMT Université de Cocody, Laboratoire de Chimie Organique Structurale, 22 BP 582 Abidjan 22, Côte d'Ivoire

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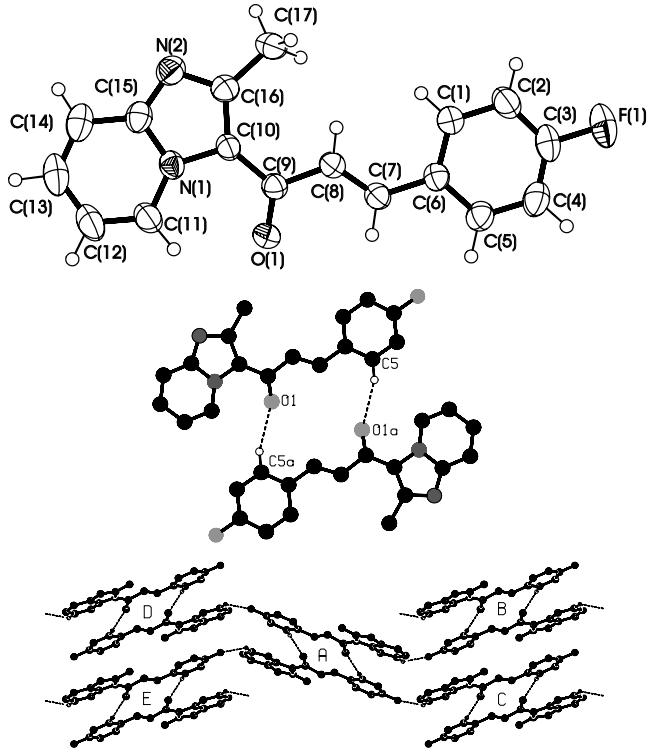

Abstract

$C_{17}H_{13}FN_2O$, monoclinic, $P12_1/n1$ (no. 14), a = 6.7597(2) Å, b = 28.4154(8) Å, c = 7.3112(2) Å, β = 106.472(1)°, V = 1346.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.048, $wR_{ref}(F^2)$ = 0.104, T = 295 K.

Source of material

10 ml of NaOH (20 %) were added to a cold solution of ethanol (20 ml) and 1-(2-methylimidazo[1,2-*a*]pyridin-3-yl)ethanone (1 g, 5.8 mmol). After 5 minutes of cold agitation, 4-fluorobenzaldehyde (7 mmol) was added in small quantities. The mixture was stirred at ambient temperature for 3 h, then 100 ml of water was added to this suspension. The resulting mixture was neutralized with acetic acid (20 %). The precipitate was then filtered, washed many times with water, dried and recrystallized from a mixture of hexane/dichlormethane (3:1). Yellow single crystals of parallel-epipedic shape were obtained for X-ray diffraction analysis (yield, 74 %; m.p. 459 K).

* Correspondence author (e-mail: bibilamayayabisseyou@yahoo.fr)

Experimental details

The H atoms were all located in a difference Fourier map. They were all initially refined with soft restraints on the bond lengths and angles to regularize their geometry [d(C—H) in the range 0.92 - 1.00 Å and U_{iso}(H) in the range 1.2 - 1.5 U_{eq}(parent atom)]. Finally their positions were refined with riding constraints.

Discussion

Chalcones are one of very used compounds classes because of their biopharmacological profiles including several interesting properties [1-5]. We are interested in this class of compounds and have attempted the synthesis of new halogeno substituted chalcones containing a imidazo[1,2-*a*]pyridine ring system in order to design organic molecules having new potential therapeutic uses.

The values of bond lengths and angles in the molecule of the title compound (figure, top) are within range obtained for his chlorinated analogue [6]. The imidazopyridine ring, essentially planar, makes a dihedral angle of 5.46° with benzene ring. Enone group (C7/C8/C9/O1) is planar, with a maximun deviation of 0.0124(1) Å for C9 from the mean plane. In the crystal structure, molecules are linked into a three-dimensional framework by means of a pair of C–H···O and C–H···F hydrogen bonds. The formation of this framework may be analysed in terms of two distinct low-dimensional substructures, each involving a single hydrogen bond type. The first of these substructures which acts as the basic building motif in the fomation of the supramolecular structure is a centrosymmetric dimeric unit. Here, carbon atom C5 in the molecule at (x,y,z) acts as a hydrogen bond donor, *via* H5, to carbonyl atom O1 in the molecule at (–x,–y,–z), so forming a centrosymmetric dimeric unit characterized by an $R_2^2(14)$ motif [7] (figure, middle). In the second substructure, carbon atoms C12 in molecules at (x,y,z) and(–x,–y,–z), which are components of $R_2^2(14)$ dimeric motif so formed and centred at (0,0,0) act as hydrogen bond donors *via* H12 to fluorine atoms F1 in molecules at (½–x,½+y,½–z) and (–½+x,–½–y,–½+z) respectively, which are themselves components of the dimeric motifs centred at (½,½,½) and (–½,–½,–½) respectively. In the same way, fluorine atoms F1 in molecules at (x,y,z) and (–x,–y,–z) accept hydrogen bonds from atoms C12 in the molecules at (½–x,–½+y,½–z) and (–½+x,½–y,–½+z) respectively, which are components of the dimeric motifs centred at (½,–½,½) and (–½,½,–½). Thus, each basic building motif is linked *via* C–H···F hydrogen bonds to four adjacent $R_2^2(14)$ dimeric motifs (figure, bottom). The propagation of this C–H···F hydrogen bond then leads to the formation of a

sheet in zigzag fashion along the [010] direction built from alternating centrosymmetric $R_2^2(14)$ and $R_6^6(50)$ motifs.

Table 1. Data collection and handling.

Crystal:	yellow parallelepiped, size 0.20 × 0.20 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.97 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	56.45°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10734, 3226
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2177
$N(param)_{refined}$:	190
Programs:	SIR92 [8], CRYSTALS [9], ORTEP-III [10], PLATON [11]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	4*e*	0.1901	0.1399	0.0114	0.063
H(7)	4*e*	0.1862	–0.0241	0.1248	0.050
H(4)	4*e*	0.1578	–0.1737	0.3590	0.071
H(173)	4*e*	1.0434	0.0583	0.3072	0.082
H(8)	4*e*	0.5817	0.0064	0.3109	0.054
H(14)	4*e*	0.8090	0.2221	0.0799	0.074
H(172)	4*e*	0.9029	0.0428	0.4349	0.082
H(2)	4*e*	0.7627	–0.1416	0.5165	0.070
H(5)	4*e*	0.0766	–0.0982	0.2236	0.060
H(13)	4*e*	0.4896	0.2614	–0.0536	0.080
H(12)	4*e*	0.1834	0.2210	–0.0791	0.078
H(1)	4*e*	0.6880	–0.0673	0.3758	0.063
H(171)	4*e*	0.8680	0.0198	0.2301	0.082

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}
F(1)	4*e*	0.5110(2)	–0.20650(4)	0.5244(2)	0.116(1)	0.0399(6)	0.089(1)	0.0081(7)	0.0267(8)	0.0162(6)
O(1)	4*e*	0.2149(2)	0.05954(5)	0.0220(2)	0.0400(7)	0.0478(7)	0.0702(9)	0.0015(6)	0.0003(6)	0.0107(6)
N(1)	4*e*	0.4981(2)	0.13603(5)	0.0901(2)	0.0496(8)	0.0342(7)	0.0401(7)	0.0018(6)	0.0067(6)	–0.0020(6)
N(2)	4*e*	0.8440(2)	0.13161(6)	0.1975(2)	0.0512(9)	0.0518(9)	0.0505(9)	–0.0076(7)	0.0108(7)	0.0001(7)
C(1)	4*e*	0.5787(3)	–0.08826(7)	0.3780(3)	0.047(1)	0.043(1)	0.061(1)	0.0003(8)	0.0108(8)	0.0057(9)
C(2)	4*e*	0.6252(3)	–0.13250(7)	0.4577(3)	0.061(1)	0.048(1)	0.063(1)	0.0118(9)	0.013(1)	0.0093(9)
C(3)	4*e*	0.4651(4)	–0.16285(6)	0.4466(3)	0.083(2)	0.0332(9)	0.052(1)	0.0031(9)	0.020(1)	0.0029(8)
C(4)	4*e*	0.2652(4)	–0.15135(7)	0.3631(3)	0.072(1)	0.043(1)	0.060(1)	–0.015(1)	0.015(1)	–0.0009(9)
C(5)	4*e*	0.2202(3)	–0.10695(7)	0.2841(3)	0.053(1)	0.046(1)	0.051(1)	–0.0077(8)	0.0073(8)	–0.0010(8)
C(6)	4*e*	0.3765(3)	–0.07470(6)	0.2913(2)	0.0475(9)	0.0369(8)	0.0369(8)	–0.0010(7)	0.0091(7)	–0.0020(7)
C(7)	4*e*	0.3248(3)	–0.02805(6)	0.2048(2)	0.0425(9)	0.0398(9)	0.0406(8)	0.0029(7)	0.0080(7)	0.0009(7)
C(8)	4*e*	0.4504(3)	0.00853(6)	0.2281(2)	0.0412(9)	0.0397(9)	0.0485(9)	0.0012(7)	0.0047(7)	0.0031(7)
C(9)	4*e*	0.3914(3)	0.05359(6)	0.1279(2)	0.0401(9)	0.0369(8)	0.0441(9)	0.0021(7)	0.0089(7)	–0.0024(7)
C(10)	4*e*	0.5468(3)	0.08997(6)	0.1557(2)	0.0429(9)	0.0333(8)	0.0393(8)	0.0028(7)	0.0075(7)	0.0006(6)
C(11)	4*e*	0.3119(3)	0.15829(7)	0.0199(3)	0.056(1)	0.042(1)	0.059(1)	0.0086(8)	0.0043(9)	–0.0008(8)
C(12)	4*e*	0.3112(4)	0.20439(7)	–0.0305(3)	0.083(2)	0.041(1)	0.068(1)	0.016(1)	0.007(1)	0.0011(9)
C(13)	4*e*	0.4962(4)	0.22884(7)	–0.0108(3)	0.108(2)	0.0355(9)	0.060(1)	0.001(1)	0.019(1)	0.0010(9)
C(14)	4*e*	0.6797(4)	0.20711(7)	0.0639(3)	0.085(2)	0.044(1)	0.056(1)	–0.013(1)	0.019(1)	–0.0045(9)
C(15)	4*e*	0.6826(3)	0.15967(6)	0.1187(3)	0.058(1)	0.0414(9)	0.0426(9)	–0.0063(8)	0.0128(8)	–0.0037(7)
C(16)	4*e*	0.7620(3)	0.08940(6)	0.2212(2)	0.0453(9)	0.0460(9)	0.0382(8)	–0.0002(7)	0.0083(7)	–0.0021(7)
C(17)	4*e*	0.9053(3)	0.04977(7)	0.3055(3)	0.042(1)	0.060(1)	0.061(1)	0.0077(9)	0.0087(8)	0.009(1)

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