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Synthesis and Crystal Structure of 1-(2-Fluorophenyl)-3-(1*H*-1,2,4-triazol-1-yl)propan-1-one

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The compound 1-(2-fluorophenyl)-3-(1*H*-1,2,4-triazol-1-yl)propan-1-one, was synthesized and characterized by means of elemental analysis, IR spectrum and X-ray diffraction. It crystallizes in monoclinic, space group P2(1)/c with a = 11.757(15), b = 4.904(6), c = 21.50(3) Å, β = 118.11(6)º, $C_{11}H_{10}N_3OF$, M_r = 219.21, V = 1093(2)Å^3, Z = 4, Dc = 1.326 g/cm^3, F(000) = 452, μ = 0.101 mm^{-1}, final R_1 = 0.1101. The triazole ring and 2-fluorophenyl ring makes dihedral angles of 85.97º. There is obvious potentially weak intra- and intermolecular C-H···O hydrogen bonds in the crystal, which stabilizes the crystal structure.

Key Words: Synthesis, Crystal structure, 1-(2-Fluorophenyl)-3-(1*H*-1,2,4-triazol-1-yl)propan-1-one.

INTRODUCTION

Many N-heterocyclic compounds have attracted considerable attention in industry and agriculture because of their significant biological activities including efficient antifungal, antibacterial antitumor and pesticides activities[1-3]. The compounds containing a triazole ring system are well known as efficient fungi in pesticides and medicine by inhibiting the biosynthesis of ergosterol. Meanwhile they have also a good plant growth regulatory activity for a wide variety of crops. Lots of compounds containing a triazole group were synthesized in recent years[4-7]. However the compounds that contain triazole group and 2-fluorophenyl group in a single molecule have rarely been found. Herein, the synthesis, IR and crystal structure of the compound, 1-(2-fluorophenyl)-3-(1*H*-1,2,4-triazol-1-yl) propan-1-one have been reported (**Scheme-I**).

EXPERIMENTAL

All the reagents and solvents from commercial sources were used without further purification. Elemental analyses were obtained using an American Perkin-Elmer 2400 analyzer. IR spectra (4000-400 cm^{-1}), were recorded on a Nicolet FT-IR 510P spectrometer. Melting points were measure by using a melting point apparatus made in Shanghai Instrument Limited Company.

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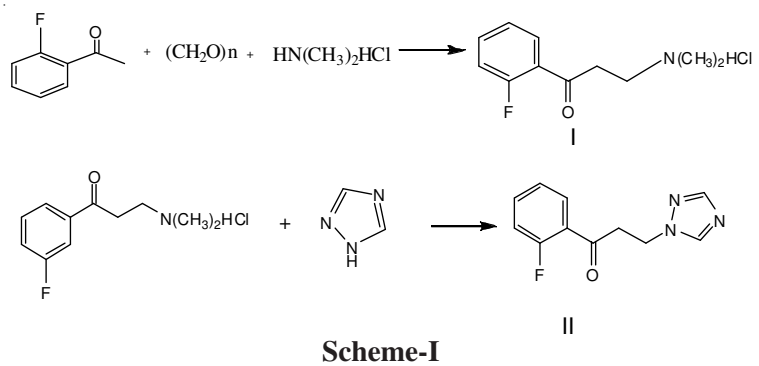

Scheme-I

The intermediates I was prepared according to the literatural report[8]. The synthesis of the compound is described below. To a solution of 3-(dimethylamino)-1-(2-fluorophenyl) propan-1-one hydrochloride (4.62 g, 0.02 mol) in water (25 mL) was added triazole (1.38 g, 0.02 mol). The mixture was heated under reflux for 5 h, yielding a copious precipitate. The colourless single crystal suitable for X-ray diffraction analysis was obtained by evaporation for petroleum ether and ethyl acetate (1:1 v/v) after a few days. Yields 45 %. m.p. 68.6-69.4 ºC. Anal. calcd. (%) for $C_{11}H_{10}N_3OF$: C, 60.27; H, 4.60, N; 19.17, found: C, 60.89; H, 4.54, N; 19.87. Selected IR (KBr pellet, ν_{max}, cm^{-1}): 1683 (C=O), 1508 (C=N).

Data collection and structure determination: A selected crystal of the reported compound was mounted on a SMART CCD diffractometer. The reflection data were measured at 293 K, using a graphite monochromator MoK_α (λ = 0.071073 nm) radiation with an ω scan mode. A total of 4475 reflections were collected and 1816 were independent (R_{int} = 0.0592) in the range of $1.96 < \theta < 24.99°$, of which 1063 reflections were observed with $I > 2\sigma(I)$.

The structure of the present compound was solved by SHELXS-97 program and was refined by SHELXL-97 program[9]. All the non-hydrogen atoms were refined by full-matrix least squares method. All hydrogen atoms were placed in the geometrically calculated positions. The contributions of these hydrogen atoms were included in the structure-factor calculations. The atomic scattering factors and anomalous dispersion corrections were taken from International Table for X-ray crystallography[10]. The final least-square cycle gave R = 0.1101 and wR = 0.2720 ($w = /[\sigma^2(F_o^2) + (0.2000P)^2 + 0.0000P]$, where $P = (F_o^2 + 2F_C^2)/3$). S = 1.216. $(\Delta/\sigma)_{max}$ = 0.000, $(\Delta\rho)_{min}$ = -0.638 and $(\Delta\rho)_{max}$ = 0.600e/Å^3.

RESULTS AND DISCUSSION

The atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms in the present compound are given in Table-1 and the selected bond distances and bond angles in Table-2. A displacement ellipsoid plot with atomic numbering scheme is shown in Fig. 1 and a perspective view of the crystal packing in the unit cell in Fig. 2.

TABLE-1
ATOMIC COORDINATES (× 10^4) AND THERMAL PARAMETERS (Å^2 × 10^3)

Atom	x	y	Z	U (eq)
F(1)	809(3)	2998(7)	690(2)	82(1)
O(2)	3996(3)	1814(8)	344(2)	73(1)
O(2)	3996(3)	1814(8)	344(2)	73(1)
N(2)	2080(3)	497(7)	-1190(2)	51(1)
N(3)	1052(4)	2065(9)	-1648(2)	65(1)
N(5)	2759(4)	2728(10)	-1841(2)	78(2)
C(7)	2949(4)	4054(8)	904(2)	45(1)
C(8)	2986(4)	2090(9)	373(2)	48(1)
C(9)	4056(4)	5624(9)	1298(2)	51(1)
C(14)	3068(4)	946(12)	-1315(3)	72(2)
C(15)	1796(4)	460(9)	-116(2)	53(1)
C(16)	1919(4)	4476(9)	1045(2)	52(1)
C(19)	1956(5)	6303(11)	1543(3)	64(1)
C(27)	1989(5)	-1196(10)	-659(3)	60(1)
C(28)	1529(5)	3319(12)	-2013(3)	70(2)
C(29)	3051(5)	7826(11)	1913(3)	66(1)

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (º)

C(7)-C(16)	1.394(6)	C(7)-C(8)	1.510(6)
C(7)-C(9)	1.402(6)	C(8)-C(15)	1.522(6)
C(15)-C(27)	1.524(6)	N(2)-C(27)	1.454(6)
N(2)-C(14)	1.329(6)	N(2)-C(27)	1.454(6)
N(2)-N(3)	1.379(5)	N(3)-C(28)	1.313(7)
O(2)-C(8)	1.227(5)	N(5)-C(14)	1.336(7)
Angles	(º)	Angles	(º)
C(14)-N(2)-N(3)	108.5(4)	O(2)-C(8)-C(7)	118.9(4)
C(14)-N(2)-C(27)	130.4(4)	O(2)-C(8)-C(15)	120.2(4)
N(3)-N(2)-C(27)	121.0(3)	C(7)-C(8)-C(15)	120.9(3)
N(2)-C(27)-C(15)	112.9(4)	C(16)-C(7)-C(8)	126.6(4)
C(8)-C(15)-C(27)	113.0(4)	C(9)-C(7)-C(8)	117.3(4)

In the compound, the bond lengths and angles in the 1,2,4-triazole ring and phenyl ring are generally normal[11,12]. The bond lengths of C(7)-C(8) (1.510(6), Å), C(8)-C(15) (1.522(6), Å), C(27)-C(15) (1.524(6), Å) are shorter than that of standard of C-C single bond length of 1.54 Å; while the bond lengths of C(27)-N(2) (1.454(6), Å) is similar to that of the standard C-N of 1.47 Å. The phenyl ring [C7, C9, C30, C29, C19, C16] with conjunction carbon atom C8 and oxygen O2 are quite planar (plane equation: -0.0171x + 0.7181y - 0.6958z = 0.1880) and the largest deviation is 0.089 Å. The five atoms in the 1,2,4-triazole ring with the conjunction carbon atom

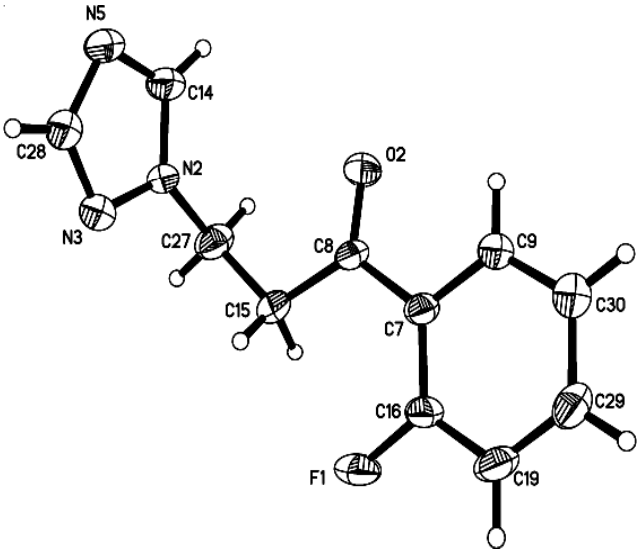

Fig. 1. Molecular structure with atomic numbering Scheme

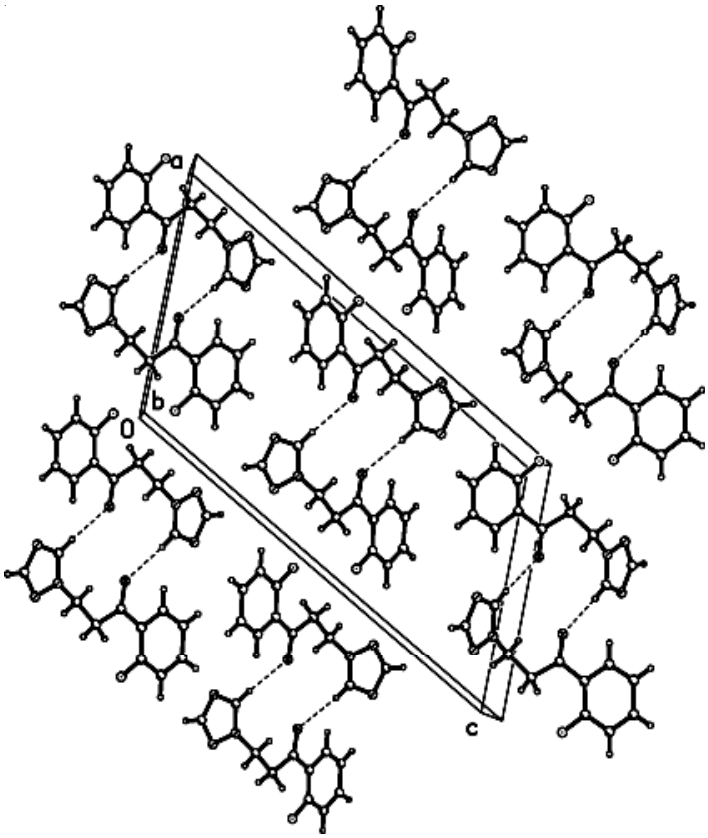

Fig. 2. View of crystal packing down the b axis

C27 are also planar, the plane equation: 0.0019x + 0.7559y + 0.6547z = 1.2694 and the largest deviation is 0.016 Å. The triazole ring and 2-fluorophenyl ring makes dihedral angles of 85.97º.

There exist some potential weak intra- and intermolecular interactions C-H···O in the lattice (Table-3). The O(2) atom with C(14) atoms form weak C-H···O intermolecular interactions and the donor and acceptor distance are 3.3638 Å. While the O(2) atom with C(9) atoms form weak C-H···O intramolecular interactions and the donor and acceptor distance are 2.7519 Å. In solid state, all above extensive hydrogen bond net works which stabilize the crystal structure.

TABLE-3
INTERMOLECULAR INTERACTION DISTANCES (Å)

D-H-A	Symmetry	D-H	H···A	D···A	D-H···A
C(14)-H(14A) ···O(2)	1-x, -y, -z	0.9300	2.4358	3.3638	175.54
C(9)-H(9A) ···O(2)	–	0.9300	2.4255	2.7519	100.54

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