



NOTE

Solvent Evaporation Method and Crystal Structure of [5-Benzoyl-2-methyl-4-(1-phenyl[1,2,3]triazolidin-4-ylmethoxy)benzofuran-7-yl]phenyl-methanone

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One new compound *i.e.*, [5-benzoyl-2-methyl-4-(1-phenyl[1,2,3]triazolidin-4-ylmethoxy)benzofuran-7-yl]phenyl-methanone with m.f. C₃₂H₂₃N₃O₄ has been successfully synthesized by 1-azidobenzene, the appropriate alkyne, copper sulphate and sodium ascorbate. Compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Sodium ascorbate, Crystal structure, Methanone.

Metal organic frameworks (MOFs) have received much attention in the field of crystal engineering and supramolecular chemistry because of their diverse structures and promising applications in functional materials such as luminescent materials, gas adsorption and magnetism¹⁻⁴. Hydrogen bonds are well suited for the design of polymeric arrangement and crystal engineering because of their important directional interactions and because they can interlink 1-D, or 3-D structures into higher-dimensionality systems^{5,6}.

All reagent and solvents employed were commercially available and used as received without further purification.

A mixture of the 1-azidobenzene (2 mmol), the appropriate alkyne (2 mmol), copper sulphate (0.025 mmol) and sodium ascorbate (0.5 mmol) in 6 mL of *t*-BuOH/H₂O (1:1) was stirred at 65 °C overnight. The crude products precipitate upon adding water and were purified by chromatography recrystallized from EtOH/H₂O. Finally colourless crystals were formed.

Detection method: Diffraction intensity data of the single crystal of the compound was collected on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite monochromated MoK_α radiation ($\lambda = 0.71073 \text{ \AA}$) by using a ω -scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F² using the program SHELXL 97⁷. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses

for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2.

The crystal structure determined by X-ray diffraction reveals is [5-benzoyl-2-methyl-4-(1-phenyl[1,2,3]triazolidin-4-ylmethoxy)benzofuran-7-yl]phenyl-methanone. The bond connecting the benzene ring and the triazole ring is a single bond, which can rotate and the bond length of N(1)-C1 is 1.420(3) Å, in addition. The dihedral angle between the benzene ring C19 to C24 and the benzene ring C26 to C31 is

TABLE-1
CRYSTALLOGRAPHIC DATA AND
STRUCTURE REFINEMENT SUMMARY

Empirical formula	C ₃₂ H ₂₃ N ₃ O ₄
Formula weight	513.53
Crystal system space group	Triclinic, P-1
Unit cell dimensions	a = 10.019(10) Å b = 10.819(11) Å c = 11.554(12) Å
Volume (Å ³)	1246(2)
θ range for data collection	2.48-25.50
Final R indices [I > 2σ(I)]	R ₁ = 0.0485, wR ₂ = 0.1155
Z, Calculated density (mg/m ³)	2, 1.368
Absorption coefficient (mm ⁻¹)	0.092
F(000)	536
Limiting indices	-12 ≤ h ≤ 11; -12 ≤ k ≤ 13; -13 ≤ l ≤ 13
Largest diff. peak and hole (e/Å ³)	0.282 and -0.464
Goodness-of-fit on F ²	0.991
R indices (all data)	R ₁ = 0.0763; wR ₂ = 0.1337

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

N(1)-N(2)	1.366(3)	N(1)-C(7)	1.355(3)
N(2)-N(3)	1.305(3)	N(1)-C(1)	1.420(3)
O(1)-C(10)	1.365(3)	N(3)-C(8)	1.371(3)
C(7)-N(1)-N(2)	109.96(19)	N(3)-N(2)-N(1)	107.04(19)
C(7)-N(1)-C(1)	130.44(19)	N(2)-N(3)-C(8)	109.24(19)
N(2)-N(1)-C(1)	119.53(19)	C(10)-O(1)-C(9)	118.06(18)
C(12)-O(4)-C(17)	106.03(17)	C(6)-C(1)-N(1)	121.2(2)

60.75°. The dihedral angle between the benzene ring C19 to C24 and the benzene ring C1 to C6 is 8.61°. The dihedral angle between the benzene ring C19 to C24 and the triazole ring C7 N1 N2 N3C8 is 12.73°. The dihedral angle between the benzene ring C19 to C24 and the benzene ring C10 to C15 is 66.48° (Fig. 1). The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

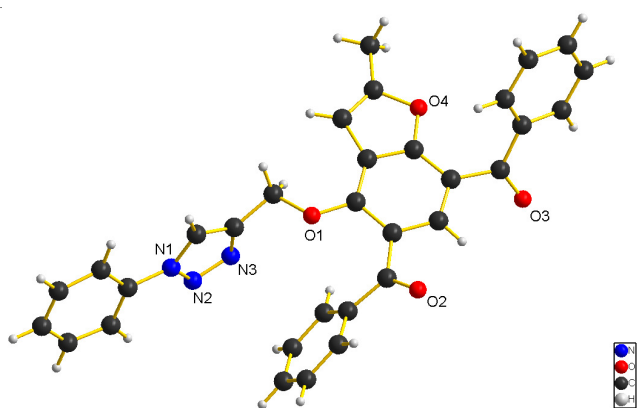


Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

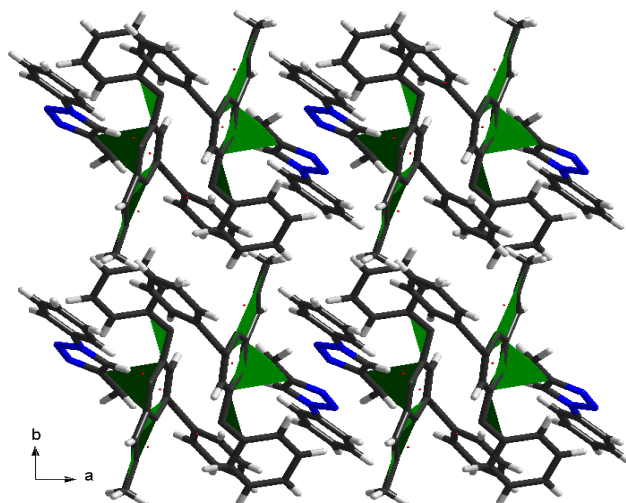


Fig. 2. 3D structure formed *via* hydrogen bonding interactions

REFERENCES

1. B.O. Patrick, C.L. Stevens, A. Storr and R.C. Thompson, *Polyhedron*, **24**, 2242 (2005).
2. Y.H. Wen, J.K. Cheng, Y.L. Feng, J. Zhang, Z.L. Li and Y.G. Yao, *Inorg. Chim. Acta*, **358**, 3347 (2005).
3. X.L. Wang, C. Qin, E.B. Wang and L. Xu, *J. Mol. Struct.*, **749**, 45 (2005).
4. S.R. Batten and K.S. Murray, *Coord. Chem. Rev.*, **246**, 103 (2003).
5. T.L. Hu, R.Q. Zou, J.R. Li and X.H. Bu, *J. Chem. Soc., Dalton Trans.*, 1302 (2008).
6. J.F. Song, Y. Chen, Z.G. Li, R.S. Zhou, X.Y. Xu and J.Q. Xu, *J. Mol. Struct.*, **842**, 125 (2007).
7. G.M. Sheldrick, SHELXTL97, Program for the Refinement of Crystal Structure, University of Gottingen, Germany (1997).