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Crystal structure analysis and synthesis of N-(Phenylcarbamothioyl) Furan -2- Carboxamide

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Abstract: Single crystals of N-(phenylcarbamothioyl) furan -2- carboxamide were grown by slow evaporation method and X-ray diffraction analysis reveals monoclinic *P21/n* space group with unit cell dimensions of a = 4.7662 (4) Å, b= 20.983(4) Å, c= 11.781 (3) Å and β= 92.80(4)°. The furan group (O1/C1-C4) makes a dihedral angle of 3.7(2)° & 2.57(19)° with the phenyl ring (C7-C12) and carbamothioyl (N1/C6/S1/N2). Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct method and refined on F^2 by full-matrix least-squares procedure to the final R_1 of 0.059 using SHELXL programs.
Key Words: Furan, Carbamothioyl, Crystal packing and Crystal structure.

Introduction

Furans are well known heterocyclic compounds which are common and have important feature of a variety of medicinal agents. Furan is a 5-membered planer ring, which is soluble in most organic solvents. It is the most reactive compound of the 5-membered heterocyclic compounds. It is a nonpolar compound. The oxygen, nitrogen and sulfur donor atoms of thiourea derivatives provide a multitude of bonding possibilities. Both the ligands and their metal complexes display a wide range of biological activity including antibacterial, antifungal,antitubercular, antithroid, antihelmintic, rodenticidal, insecticidal, herbicidal, and plant-growth regulator properties[1-5].

Experimental

X-ray Structure Determination

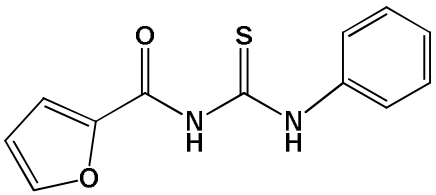
Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a Bruker[6] SMART APEX CCD Diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at Department of chemistry, IIT, Chennai, India. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[7]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[8].The crystallographic data for the compound are listed in Table 1.

Table 1: Crystal data and structure refinement of the titled compound

Compound	**Parameters**
Empirical formula	$C_{12}H_{10}N_2O_2S$
Formula weight	246.28
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *P21/n*
Unit cell dimensions	a = 4.7662(10) Å alpha = 90º b = 20.983(4) Å beta = 92.807º c = 11.781(3) Å gamma = 90º
Volume	1176.8(4) $Å^3$
Z, Calculated density	4, 1.390 Mg/m^3
Absorption coefficient	0.265 mm^{-1}
F(000)	512
Crystal size	0.30 x 0.25 x 0.20 mm
Theta range for data collection	1.94 to 23.33 deg.
Limiting indices	-5<=h<=5, -23<=k<=23, -13<=l<=11
Reflections collected / unique	3706 / 1638 [R(int) = 0.0263]
Completeness to theta = 23.33	95.7%
Max. and min. transmission	0.948 and 0.924
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1638 / 0 / 154
Goodness-of-fit on F^2	1.088
Final R indices [I>2sigma(I)]	**R1 = 0.0596**, wR2 = 0.1389
R indices (all data)	R1 = 0.0913, wR2 = 0.1561
Extinction coefficient	0.0023(4)
Largest diff. peak and hole	0.199 and -0.264 e. $Å^{-3}$

Synthesis of the compound

A solution of furan-2-carbonyl isothiocyanate(1.53 g, 10 mmol) in acetone (20 mL) was added drop wise to aniline (0.931 g, 10 mmol) in anhydrous acetone (20 mL). The reaction mixture was stirred for 2 h at room temperature. Hydrochloric acid (0.1 N, 500 mL) was added and the resulting white solid was filtered off, washed with water and dried *in vacuo*. Single crystals for X-ray diffraction method were grown at room temperature from DMF/Chloroform solutions of the furan based thioura compound.



Results and Discussion

The molecular structure of (I) in shown in fig 1.The furan and phenyl ring are essentially planar with making dihedral angle 3.7(2)º. The carbamothioyl group assumes an extended conformation as can be send from the C5/N1/C6/S1 torsion angle is 180.0(3)º. Atoms O2 and N1 deviate from the furan ring by 0.004 Å and 0.051 Å with respectively.

The formation of relatively strong intramolecular bonds between the central fragment and the furan ring, in some similar systems can presence the planarity of the 2-Furan Carboxamide moiety[9]. The O1-C1 and O1-O4 within the furan ring are within the expected range [1.36 Å]

and the other bond distance are also with expected values[10].

The molecular structure is stabilized by an intramolecular N---H…O hydrogen bond generating an S(6) and S(5) ring motif. In the crystal molecules are linked by pairs of C---H…O hydrogen bonds forming inversion dimmer with on $R_2^2(10)$ ring motif. Within the chains there is C---H…π interactions. The chains are linked via slipped parallel π--π interactions forming a three dimensional structure.

Table 2: Hydrogen-bond geometry [Å]

Distance (Å)				**Angle (°)**
D—H…A	D—H	H…A	D…A	D—H…A
C3—H3…O2^{i}	0.93	2.41	3.268(6)	152
N1—H1…O1	0.86	2.31	2.726(4)	110
N2—H2…O2	0.86	1.88	2.619	144

Symmetry code: i) -1-x,1-y,1-z

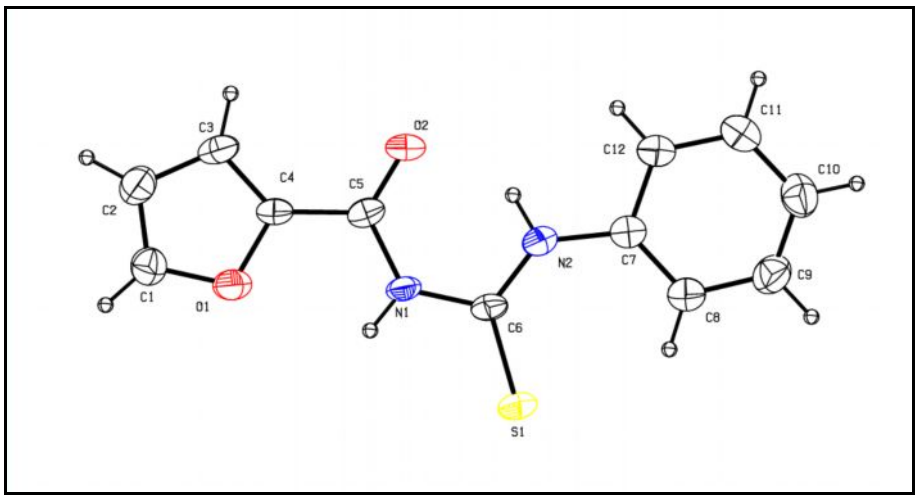


Fig 1. The molecular structure of the title compound, with atom labeling. Displacement ellipsoids are drawn at the 30% probability level.

Table 3: Selected Bond lengths (Å)

Atom	**Length**	**Atom**	**Length**
O(1)-C(1)	1.360(5)	C(5)-C(4)	1.451(6)
O(1)-C(4)	1.367(5)	C(6)-S(1)	1.663(4)
O(2)-C(5)	1.229(5)	C(9)-C(10)	1.350(7)
N(2)-C(6)	1.339(5)	C(9)-C(8)	1.394(6)
N(2)-C(7)	1.409(5)	C(3)-C(2)	1.406(7)
N(1)-C(5)	1.369(5)	C(8)-H(8)	0.9300
N(1)-H(1)	0.8600	C(12)-C(11)	1.369(7)
C(7)-C(8)	1.373(6)	C(10)-C(11)	1.369(7)
N(2)-H(2)	0.8600	C(10)-H(10)	0.9300
N(1)-C(6)	1.391(5)	C(2)-H(2A)	0.9300
C(1)-C(2)	1.322(7)	C(7)-C(12)	1.385(6)
C(3)-H(3)	0.9300	C(11)-H(11)	0.9300

Table 4: Selected Bond angles (°)

Atom	Angle	Atom	Angle
C(1)-O(1)-C(4)	107.0(4)	C(8)-C(7)-C(12)	118.7(4)
C(6)-N(2)-H(2)	113.5	C(12)-C(7)-N(2)	114.9(4)
C(5)-N(1)-C(6)	128.3(4)	C(10)-C(9)-H(9)	118.9
O(2)-C(5)-N(1)	123.5(4)	C(4)-C(3)-C(2)	107.2(5)
N(1)-C(5)-C(4)	116.6(4)	C(2)-C(3)-H(3)	126.4
C(3)-C(4)-O(1)	108.7(4)	C(2)-C(1)-O(1)	110.0(4)
O(1)-C(4)-C(5)	119.0(4)	O(1)-C(1)-H(1A)	125.0
N(2)-C(6)-N(1)	114.5(4)	C(12)-C(11)-H(11)	119.6
C(7)-C(8)-C(9)	119.0(5)	C(10)-C(11)-C(12)	120.8(5)
C(6)-N(1)-H(1)	115.9	C(9)-C(10)-C(11)	118.4(5)
C(8)-C(7)-N(2)	126.4(4)	O(2)-C(5)-C(4)	119.9(4)
N(2)-C(6)-S(1)	127.5(3)	C(6)-N(2)-C(7)	132.9(4)

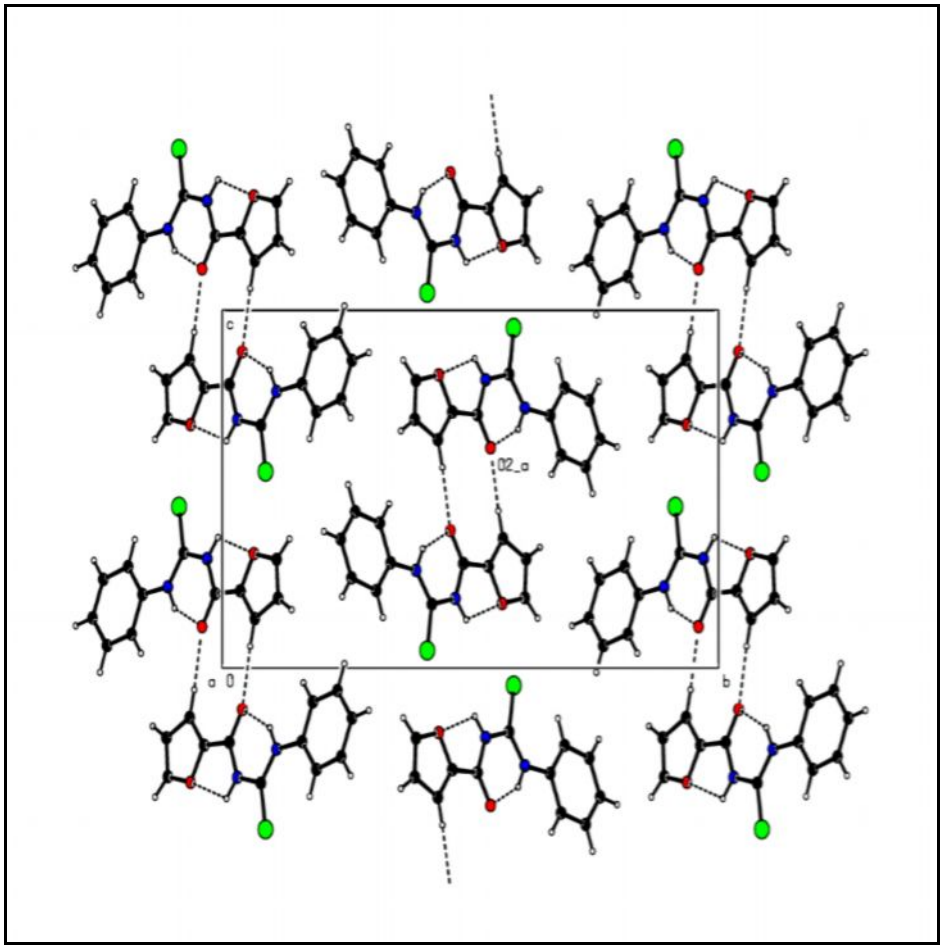


Fig 2. The crystal packing of the titled compound forming centrosymmetric dimmer described by graph-set ring motif $R^2_2(10)$ viewed along c axis. The hydrogen bonds are shown as dashed lines (see Table 2 for details).

Conclusion

The crystal structure analysis of a novel furan and carbamothioyl compound was studied using x-ray diffraction method. In the crystal, molecules linked via C---H…O hydrogen bond forming $R_2^2(10)$ rings motif

and it forms chains. The crystal packing is further stabilized by intramolecular interactions hydrogen bonds, forming $R_2^2(5)$ and $R_2^2(6)$ rings motif.

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Crystallographic data for the structure reported here have been deposited with CCDC (Deposition No's.CCDC: 1435195). These data can be obtained free of charge via http:// www .ccdc. cam.ac. uk/ conts/retrieving.html or from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK,
E-mail: deposit@ccdc.cam.ac.uk.

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