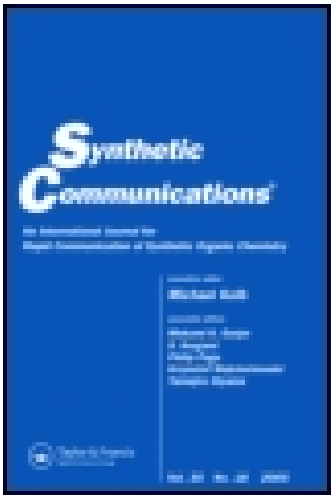

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A Novel Route to Acyl Ureas: Syntheses of N-[5-(2-Chlorophenyl)-2-Furoyl]-N'-Arylthioureas and Ureas

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A NOVEL ROUTE TO ACYL UREAS: SYNTHESES OF N-[5-(2-CHLOROPHENYL)-2-FUROYL]-N'-ARYLTHIOUREAS AND UREAS

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ABSTRACT: A novel route to acyl ureas is described. Reaction of 5-(2-chlorophenyl)-2-furoyl chloride with ammonium thiocyanate first, then arylamine under phase transfer catalysis gives N-[5-(2-chlorophenyl)-2-furoyl]-N'-aryl-thioureas (**1a-o**). Compounds **1a-o** on oxidation with potassium iodate respectively under reflux result in the formation of N-[5-(2-chlorophenyl)-2-furoyl]-N'-arylureas (**2a-o**).

The chemistry of acyl thioureas and acyl ureas has drawn considerable attention of a number of investigations due to their varied biological activities, e.g. insecticidal[1,2], herbicidal[3] and plant-growth regulator[4] activities. These prompt us to synthesize new acyl thioureas and acyl ureas bearing a 5-aryl-2-furoyl moiety as the objective of obtaining new biologically active compounds.

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The phase transfer catalysis technique has been reported as a convenient technique for the synthesis of acyl thioureas using acyl chloride, ammonium thiocyanate and arylamine at mild conditions[5]. However, acyl ureas have to be prepared via the catalysis of metal halides, such as $SnCl_4$ and $ZnCl_2$, using acyl chloride, sodium cyanate and arylamine under argon[6,7]. Ramadas et al. have reported an effective and safe procedure for the preparation of trisubstituted ureas derived from their thio analogues using sodium metaperiodate, sodium chlorite or ammonium persulfate as oxidants[8].

Keeping in view the above facts and varied biological activities associated with acyl thioureas and acyl ureas, in the present paper, attempts have been made to synthesize thioureas under the condition of solid-liquid phase transfer catalysis and then to synthesize corresponding ureas in the presence of oxidant, potassium iodate, from resulting thioureas.

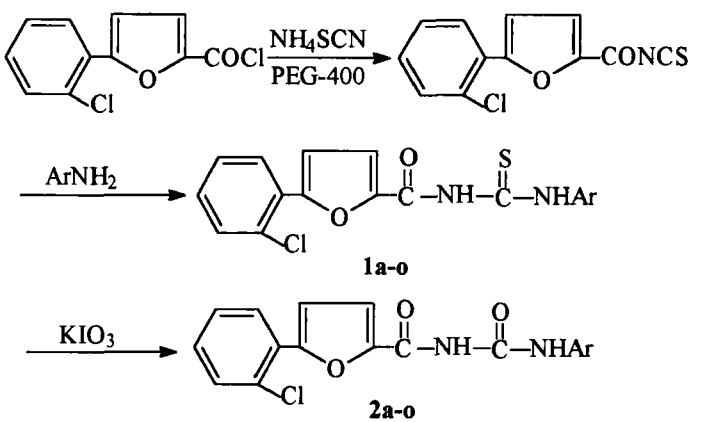
N-[5-(2-chlorophenyl)-2-furoyl]-N'-arylthioureas(**1a-o**) have been obtained by the reactions of 5-(2-chlorophenyl)-2-furoyl chloride with ammonium thiocyanate catalyzed by polyethylene glycol-400 (PEG-400)and then addition of arylamine to the mixture at room temperature in excellent yields. Further, compounds **1a-o** on treatment with potassium iodate in the water under reflux afford N-[5-(2-chlorophenyl)-2-furoyl]-N'-arylureas(**2a-o**) in excellent yields (Scheme).

It is observed that oxidant, potassium iodate, reacts spontaneously with acyl thioureas (**1a-o**) leading to the acyl ureas (**2a-o**) fairly rapidly in respectable yields in the all of cases studied. Moreover, this oxidation reaction can easily take place in the aqueous slurry under gentle reflux.

EXPERIMENTAL SECTION

IR spectra were recorded using KBr pellets on an Alpha Centauri FTIR spectrophotometer and ^{1}H NMR spectra on a FT-80A instrument using $(CD_3)_2SO$

Scheme



1, 2	Ar	1, 2	Ar
a	C_6H_5	**i**	4-ClC_6H_4
b	2-$O_2NC_6H_4$	**j**	4-BrC_6H_4
c	3-$O_2NC_6H_4$	**k**	2-$CH_3OC_6H_4$
d	4-$O_2NC_6H_4$	**l**	4-$CH_3COC_6H_4$
e	2-$CH_3C_6H_4$	**m**	4-$CH_3CONHC_6H_4$
f	3-$CH_3C_6H_4$	**n**	1-Naphthyl
g	4-$CH_3C_6H_4$	**o**	2-Naphthyl
h	2-ClC_6H_4		

as solvent and Me_4Si as internal standard. Elemental analyses were performed on a Carlo-Erba 1106 Elemental Analysis instrument. Melting points were observed in an open capillary tube and uncorrected. 5-(2-chlorophenyl)-2-furoyl chloride was prepared according to literature procedures[9]. Ammonium thiocyanate and PEG-400 were commercially available and used as received.

General procedure for the preparation of compounds 1a-o

To a solution of 3 mmol of 5-(2-chlorophenyl)-2-furoyl chloride in 15 mL of methylene dichloride, 4.5 mmol of ammonium thiocyanate and 0.1 mmol of polyethylene glycol-400 were added. The mixture was stirred for 1 h at room temperature. Then 2.95 mmol of an arylamine was added and the reaction mixture was stirred for another 0.5 h at room temperature. To the resulting mixture, 10 mL of water was added so that the inorganic salts were dissolved, then the slurry was filtered, the solid was washed with 3×5 mL water and recrystallized from DMF-EtOH-H_2O, and the product was given. The physical and spectral data of compounds **1a-o** are reported below.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-phenylthiourea (**1a**): Yield: 93%. m.p.: 114-115 ℃. ^1H NMR (DMSO-d_6) δ 12.16(s,1H,NH), 11.71(s,1H,NH), 7.28-8.23(m,11H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3390(N-H), 1668(C=O), 1151(C=S). Anal. Calcd. for $C_{18}H_{13}N_2O_2SCl$: C,60.59; H,3.67; N,7.85. Found: C,60.38; H,3.51; N,7.79.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-nitrophenyl)thiourea (**1b**): Yield: 96%. m.p.: 137-138

℃. ^{1}H NMR (DMSO-d_6) δ 12.38(s,1H,NH), 12.03(s,1H,NH), 7.37-8.19(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3406(N-H), 1691(C=O), 1154(C=S). Anal. Calcd. for $C_{18}H_{12}N_3O_4SCl$: C,53.80; H,3.01; N,10.46. Found: C,53.71; H,2.96; N,10.57.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(3-nitrophenyl)thiourea (**1c**): Yield: 89%. m.p.: 164-165℃. ^{1}H NMR (DMSO-d_6) δ 12.35(s,1H,NH), 12.02(s,1H,NH), 7.36-8.17(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3408(N-H), 1689(C=O), 1150(C=S). Anal. Calcd. for $C_{18}H_{12}N_3O_4SCl$: C,53.80; H,3.01; N,10.46. Found: C,53.86; H,3.02; N,10.30.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-nitrophenyl)thiourea (**1d**): Yield: 93%. m.p.: 199-200 ℃. ^{1}H NMR (DMSO-d_6) δ 12.34(s,1H,NH), 12.01(s,1H,NH), 7.34-8.17(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3410(N-H), 1687(C=O), 1161(C=S). Anal. Calcd. for $C_{18}H_{12}N_3O_4SCl$: C,53.80; H,3.01; N,10.46. Found: C,53.62; H,2.93; N,10.51.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-methylphenyl)thiourea (**1e**): Yield: 85%. m.p.: 157-158 ℃. ^{1}H NMR (DMSO-d_6) δ 12.13(s,1H,NH), 11.69(s,1H,NH), 7.24-8.16(m,10H,Ar-H&Fu-H), 2.28(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3387(N-H), 1670(C=O), 1168(C=S). Anal. Calcd. for $C_{19}H_{15}N_2O_2SCl$: C,61.54; H,4.08; N,7.55. Found: C,61.39; H,4.14; N,7.67.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(3-methylphenyl)thiourea (**1f**): Yield: 83%. m.p.: 148-149 ℃. ^{1}H NMR (DMSO-d_6) δ 12.14(s,1H,NH), 11.69(s,1H,NH), 7.25-8.16(m,10H,Ar-H&Fu-H), 2.29(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3388(N-H), 1669(C=O), 1169(C=S). Anal. Calcd. for $C_{19}H_{15}N_2O_2SCl$: C,61.54; H,4.08; N,7.55. Found: C,61.43; H,4.10; N,7.60.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-methylphenyl)thiourea (**1g**): Yield: 84%. m.p.: 156-157 ℃. ^{1}H NMR (DMSO-d_6) δ 12.11(s,1H,NH), 11.69(s,1H,NH), 7.22-8.15(m,10H,Ar-H&Fu-

H), 2.26(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3401(N-H), 1672(C=O), 1174(C=S). Anal. Calcd. for $C_{19}H_{15}N_2O_2SCl$: C,61.54; H,4.08; N,7.55. Found: C,61.61; H,4.01; N,7.49.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-chlorophenyl)thiourea (**1h**): Yield: 86%. m.p.: 170-171 ℃. ^{1}H NMR (DMSO-d_6) δ 12.14(s,1H,NH), 11.80(s,1H,NH), 7.35-8.21(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3392(N-H), 1682(C=O), 1153(C=S). Anal. Calcd. for $C_{18}H_{12}N_2O_2SCl_2$: C,55.25; H,3.09; N,7.16. Found: C,55.21; H,3.01; N,7.11.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-chlorophenyl)thiourea (**1i**): Yield: 83%. m.p.: 183-184 ℃. ^{1}H NMR (DMSO-d_6) δ 12.11(s,1H,NH), 11.76(s,1H,NH), 7.36-8.19(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3341(N-H), 1667(C=O), 1150(C=S). Anal. Calcd. for $C_{18}H_{12}N_2O_2SCl_2$: C,55.25; H,3.09; N,7.16. Found: C,55.15; H,2.89; N,7.01.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-bromophenyl)thiourea (**1j**): Yield:95%. m.p.:181-182 ℃. ^{1}H NMR (DMSO-d_6) δ 12.12(s,1H,NH), 11.71(s,1H,NH), 7.32-8.19(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3396(N-H), 1668(C=O), 1148(C=S). Anal. Calcd. for $C_{18}H_{12}N_2O_2SClBr$: C,49.62; H,2.78; N,6.43. Found: C,49.81; H,2.81; N,6.29.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-methoxyphenyl)thiourea (**1k**): Yield: 86%. m.p.: 134-135℃. ^{1}H NMR (DMSO-d_6) δ 12.53(s,1H,NH), 11.79(s,1H,NH), 7.33-8.21(m,10H,Ar-H&Fu-H), 2.38(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3406(N-H), 1680(C=O), 1178(C=S). Anal. Calcd. for $C_{19}H_{15}N_2O_3SCl$: C,58.99; H,3.91; N,7.24. Found: C,59.24; H,4.01; N,7.36.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-acetylphenyl)thiourea (**1l**): Yield: 86%. m.p.: 203-204 ℃. ^{1}H NMR (DMSO-d_6) δ 12.06(s,1H,NH), 11.81(s,1H,NH), 7.42-8.39(m,10H,Ar-H&Fu-H), 2.44(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3405(N-H), 1665(C=O), 1153(C=S). Anal. Calcd. for

$C_{20}H_{15}N_2O_3SCl$: C,60.23; H,3.79; N,7.02. Found: C,60.48; H,3.82; N,7.16.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-acetamidophenyl)thiourea (**1m**): Yield: 85%. m.p.: 185-186℃. ^{1}H NMR (DMSO-d_6) δ 12.01(s,1H,NH), 11.66(s,1H,NH), 10.02(s,1H,NH), 7.52-8.39(m,10H,Ar-H&Fu-H), 2.48(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3401(N-H), 1671(C=O), 1156(C=S). Anal. Calcd. for $C_{20}H_{16}N_3O_3SCl$: C,58.04; H,3.90; N,10.15. Found: C,58.18; H,3.91; N,10.28.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(1-naphthyl)thiourea (**1n**): Yield:81%. m.p.:184-185℃. ^{1}H NMR (DMSO-d_6) δ 12.12(s,1H,NH), 11.73(s,1H,NH), 7.34-8.33(m,13H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3402(N-H), 1671(C=O), 1156(C=S). Anal. Calcd. for $C_{22}H_{15}N_2O_2SCl$: C,64.94; H,3.72; N,6.88. Found: C,65.13; H,3.78; N,6.73.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-naphthyl)thiourea (**1o**): Yield:83%. m.p.:165-166℃. ^{1}H NMR (DMSO-d_6) δ 12.10(s,1H,NH), 11.74(s,1H,NH), 7.35-8.32(m,13H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3393(N-H), 1668(C=O), 1150(C=S). Anal. Calcd. for $C_{22}H_{15}N_2O_2SCl$: C,64.94; H,3.72; N,6.88. Found: C,65.09; H,3.79; N,6.78.

General procedure for the preparation of compounds 2a-o

A suspension of 0.5 mmol of compound **1** and 0.75 mmol of potassium iodate in 30 mL of water was refluxed for 1 h. The resulting mixture was filtered, the solid was washed with 3×5 mL water and recrystallized from DMF-EtOH-H_2O, then the product was given. The physical and spectral data of compounds **2a-o** are reported below.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-phenylurea (**2a**): Yield: 96%. m.p.: 188-189

℃. ^{1}H NMR (DMSO-d_6) δ 10.61(s,1H,NH), 10.20(s,1H,NH), 7.11-8.30 (m,11H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3220, 3134(N-H), 1698(C=O). Anal. Calcd. for $C_{18}H_{13}N_2O_3Cl$: C,63.45; H,3.85; N,8.22. Found: C,63.48; H,3.74; N,8.10.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-nitrophenyl)urea (**2b**): Yield: 90%. m.p.: 225-226℃. ^{1}H NMR (DMSO-d_6) δ 12.11(s,1H,NH), 11.40(s,1H,NH), 7.26-8.52(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3265, 3166(N-H), 1637(C=O). Anal. Calcd. for $C_{18}H_{12}N_3O_5Cl$: C,56.02; H,3.14; N,10.89. Found: C,56.11; H,3.20; N,10.82.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(3-nitrophenyl)urea (**2c**): Yield: 88%. m.p.: 235-236℃. ^{1}H NMR (DMSO-d_6) δ 12.10(s,1H,NH), 11.38(s,1H,NH), 7.20-8.53(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3261, 3160(N-H), 1640(C=O). Anal. Calcd. for $C_{18}H_{12}N_3O_5Cl$: C,56.02; H,3.14; N,10.89. Found: C,56.11; H,3.20; N,10.82.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-nitrophenyl)urea (**2d**): Yield: 91%. m.p.: 215-216℃. ^{1}H NMR (DMSO-d_6) δ 12.13(s,1H,NH), 11.42(s,1H,NH), 7.24-8.51(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3264, 3158(N-H), 1635(C=O). Anal. Calcd. for $C_{18}H_{12}N_3O_5Cl$: C,56.02; H,3.14; N,10.89. Found: C,56.08; H,3.10; N,10.91.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-methylphenyl)urea (**2e**): Yield: 95%. m.p.: 215-216℃. ^{1}H NMR (DMSO-d_6) δ 11.28(s,1H,NH), 10.66(s,1H,NH), 7.03-8.33(m,10H,Ar-H&Fu-H), 2.31(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3233, 3137(N-H), 1691(C=O). Anal. Calcd. for $C_{19}H_{15}N_2O_3Cl$: C,64.32; H,4.26; N,7.90. Found: C,64.23; H,4.21; N,7.87.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(3-methylphenyl)urea (**2f**): Yield: 86%. m.p.: 202-203℃.

^{1}H NMR (DMSO-d_6) δ 11.27(s,1H,NH), 10.63(s,1H,NH), 7.05-8.30(m,10H,Ar-H&Fu-H), 2.29(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3236, 3134(N-H), 1695(C=O). Anal. Calcd. for $C_{19}H_{15}N_2O_3Cl$: C,64.32; H,4.26; N,7.90. Found: C,64.38; H,4.19; N,7.83.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-methylphenyl)urea (**2g**): Yield: 93%. m.p.: 211-212℃. ^{1}H NMR (DMSO-d_6) δ 11.25(s,1H,NH), 10.62(s,1H,NH), 7.01-8.34(m,10H,Ar-H&Fu-H), 2.31(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3234, 3131(N-H), 1688(C=O). Anal. Calcd. for $C_{19}H_{15}N_2O_3Cl$: C,64.32; H,4.26; N,7.90. Found: C,64.29; H,4.22; N,7.95.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-chlorophenyl)urea (**2h**): Yield: 94%. m.p.: 226-227℃. ^{1}H NMR (DMSO-d_6) δ 12.03(s,1H,NH), 11.22(s,1H,NH), 7.19-8.51(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3391, 3233(N-H), 1700, 1673(C=O). Anal. Calcd. for $C_{18}H_{12}N_2O_3Cl_2$: C,57.62; H,3.22; N,7.47. Found: C,57.54; H,3.15; N,7.39.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-chlorophenyl)urea (**2i**): Yield: 92%. m.p.: 206-207℃. ^{1}H NMR (DMSO-d_6) δ 12.00(s,1H,NH), 11.19(s,1H,NH), 7.18-8.52(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3393, 3230(N-H), 1702, 1670(C=O). Anal. Calcd. for $C_{18}H_{12}N_2O_3Cl_2$: C,57.62; H,3.22; N,7.47. Found: C,57.58; H,3.18; N,7.40.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-bromophenyl)urea (**2j**): Yield:89%. m.p.:191-192℃. ^{1}H NMR (DMSO-d_6) δ 12.02(s,1H,NH), 11.22(s,1H,NH), 7.21-8.57(m,10H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3386, 3236(N-H), 1705, 1668(C=O). Anal. Calcd. for $C_{18}H_{12}N_2O_3ClBr$: C,51.52; H,2.88; N,6.68. Found: C,51.48; H,2.84; N,6.66.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-methoxyphenyl)urea (**2k**): Yield: 81%. m.p.: 158-159 ℃. ^{1}H NMR (DMSO-d_6) δ 11.88(s,1H,NH), 11.31(s,1H,NH), 7.30-8.41(m,10H,Ar-H&Fu-

H), 2.40(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3241, 3143(N-H), 1681(C=O). Anal. Calcd. for $C_{19}H_{15}N_2O_4Cl$: C,61.55; H,4.08; N,7.56. Found: C,61.50; H,4.01; N,7.49.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-acetylphenyl)urea (**2l**): Yield: 84%. m.p.: 210-211℃. ^1H NMR (DMSO-d_6) δ 11.35(s,1H,NH), 10.89(s,1H,NH), 7.31-8.39(m,10H,Ar-H&Fu-H), 2.45(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3235, 3147(N-H), 1707, 1684, 1673(C=O). Anal. Calcd. for $C_{20}H_{15}N_2O_4Cl$: C,62.75; H,3.95; N,7.32. Found: C,62.79; H,3.89; N,7.25.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(4-acetamidophenyl)urea (**2m**): Yield: 90%. m.p.: 283-284℃. ^1H NMR (DMSO-d_6) δ 11.15(s,1H,NH), 10.55(s,1H,NH), 9.92(s,1H,NH), 7.34-8.31(m,10H,Ar-H&Fu-H), 2.03(s,3H,CH_3). IR(KBr, ν ,cm^{-1}): 3239, 3141(N-H), 1687, 1657(C=O). Anal. Calcd. for $C_{20}H_{16}N_3O_4Cl$: C,60.36; H,4.05; N,10.60. Found: C,60.28; H,3.99; N,10.57.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(1-naphthyl)urea (**2n**): Yield:87%. m.p.:197-198℃. ^1H NMR (DMSO-d_6) δ 11.59(s,1H,NH), 11.34(s,1H,NH), 7.33-8.35(m,13H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3222, 3124(N-H), 1696, 1683(C=O). Anal. Calcd. for $C_{22}H_{15}N_2O_3Cl$: C,67.61; H,3.87; N,7.17. Found: C,67.56; H,3.83; N,7.09.

N-[5-(2-chlorophenyl)-2-furoyl]-N'-(2-naphthyl)urea (**2o**): Yield:91%. m.p.:215-216℃. ^1H NMR (DMSO-d_6) δ 11.61(s,1H,NH), 11.32(s,1H,NH), 7.36-8.37(m,13H,Ar-H&Fu-H). IR(KBr, ν ,cm^{-1}): 3227, 3120(N-H), 1698, 1685(C=O). Anal. Calcd. for $C_{22}H_{15}N_2O_3Cl$: C,67.61; H,3.87; N,7.17. Found: C,67.59; H,3.80; N,7.11.

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