

Synthesis and reactions of *N*-aryl-*N'*-(3-furoyl)thioureas

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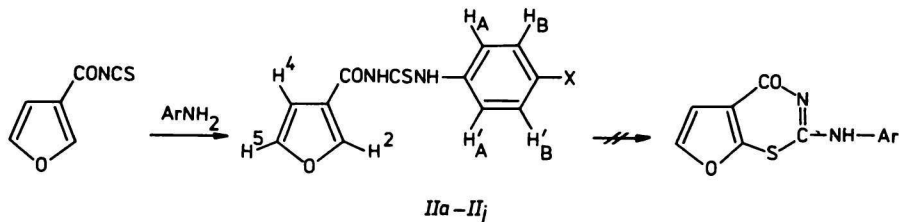
Dedicated to Professor Ing. J. Kováč, DrSc., in honour of his 60th birthday

3-Furoyl isothiocyanate reacts with 4-*X*-anilines (*X* = H, CH₃, OH, OCH₃, N(C₂H₅)₂, NHCOCH₃, Cl, Br, NO₂, COOH) under formation of the respective *N*-(4-*X*-phenyl)-*N'*-(3-furoyl)thioureas. By treatment with bromine in acetic acid, thionyl chloride in chloroform, and methyl iodide in methanol, respectively, these thioureas do not cyclize to furo-fused derivatives. In the reaction with bromoacetone and ω -bromoacetophenone they afford the corresponding 2-(4-*X*-phenylimino)-3-(3-furoyl)-4-*R*-4-thiazolines (*R* = CH₃, C₆H₅). The structures of the newly synthesized compounds were proved by IR, UV, ¹H NMR, and mass spectra.

3-Фууроилизотиоцианат взаимодействует с 4-*X*-анилинами (*X* = H, CH₃, OH, OCH₃, N(C₂H₅)₂, NHCOCH₃, Cl, Br, NO₂, COOH) с образованием соответствующих *N*-(4-*X*-фенил)-*N'*-(3-фууроил)тиомочевин. При действии на эти тиомочевины бромом в уксусной кислоте, хлористым тионилем в хлороформе и иодистым метилом в метаноле они не циклизуются в фууро-конденсированные производные. В реакциях с бромацетоном и ω -бромацетофеноном они дают соответствующие 2-(4-*X*-фенилимино)-3-(3-фууроил)-4-*R*-4-тиазолины (*R* = CH₃, C₆H₅). Строение новосинтезированных соединений было подтверждено с помощью ИК-, УФ-, ¹H ЯМР и масс-спектрометрии.

It is known that ureas and thioureas possess high biological activity [1]. Of acyl derivatives of furan only α -furoylthioureas have been studied so far [2—4]. Many of them showed strong antibacterial activity.

3-Furoylthioureas mentioned in this work were synthesized according to Scheme 1. 3-Furoyl isothiocyanate *I* was synthesized from the appropriate



Scheme 1

3-furoyl chloride [5] and potassium thiocyanate in 78 % yield. By the reaction of *I* with aromatic amines the corresponding *N,N'*-disubstituted thioureas *IIa–IIj* (Table 1) were obtained. The original aim of this work was to perform cyclization of 3-furoylthioureas to the vicinal α -position (or β -position) and prepare the respective furo-fused heterocycles.

In treatment of *IIb* with the most frequently used cyclization reagents, *i.e.* bromine in acetic acid and methyl iodide in methanol, we have not obtained the cyclization product.

Unwillingness of 3-furoylthioureas to cyclize to the vicinal α -position, known to be very reactive for electrophilic substitution, is surprising. On treatment with bromine in acetic acid or chloroform the formation of sulfenyl bromide (from the intermediate disulfide by action of bromine [6]) [7–15] is assumed. This can enter electrophilic substitution reactions for example with benzene ring [11, 15] or electrophilic addition reactions on double bond [8, 9, 11]. It follows from many works on cyclization of thioureas of furan [13], thiophene [14], and benzene series [11] that successful cyclization strongly depends on reactivity of that position in the substrate where cyclization is to occur. In the case of the studied 3-furoylthioureas the vicinal positions are deactivated by the electron-accepting carbonyl group. This is probably the reason why cyclization does not proceed. As the mentioned thiourea *IIb* did not cyclize, we attempted to modify the carbonyl to thiocarbonyl group in order to reduce the deactivation effect upon furan ring. Under conditions of the Lawesson agent [16] the starting compound was obtained quantitatively, while under conditions of P_2S_5 [17, 18] the reaction mixture became resinous. Cyclization was unsuccessful also under conditions of photocyclization at 6 h irradiation of thiourea *IIb* in methanol by a 150 W high-pressure mercury discharge lamp, *i.e.* under conditions when 2-furoylchloroanilines cyclize [19].

When treating *IIb* with thionyl chloride as an effective cyclization agent [20] we have not obtained the cyclization product either, but sulfur in thiourea was replaced for oxygen, *i.e.* *N*-(3-furoyl)-*N'*-(4-methylphenyl)urea *IIk* was formed by the reaction of the intermediate carbodiimide with water (Scheme 2). This fact was confirmed by the reaction of thiourea *IIb* with mercury(II) oxide where the respective carbodiimide is generated in the reaction medium and, after addition of water, provides thiourea *IIk*.

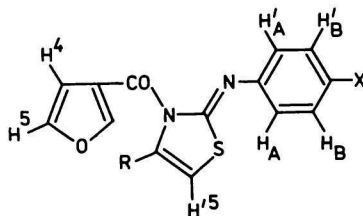
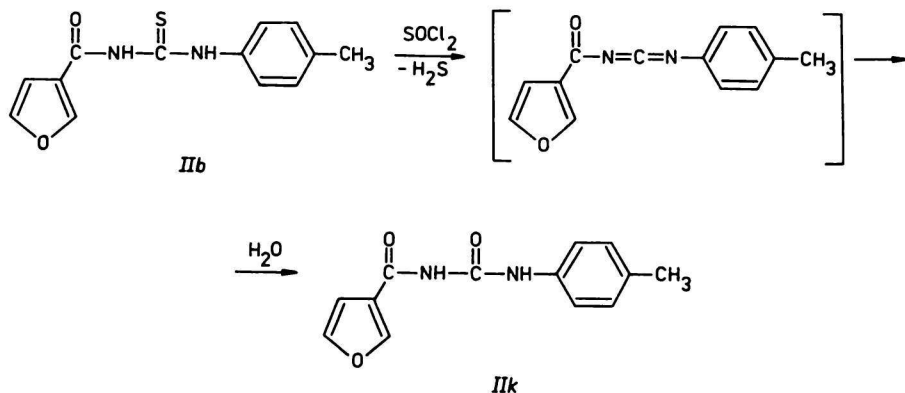


Table 1

N-Aryl-*N'*-(3-furoyl)thioureas (*Ila*—*Ilj*)

Compound	X	Formula	M_r	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$		Yield %	M.p. °C	$M_r(M^{+\bullet})$	$\nu(\text{C=O})$ $\tilde{\nu}/\text{cm}^{-1}$	$\lambda_{\text{max}}/\text{nm}$	log ($\epsilon/(\text{m}^2 \text{mol}^{-1})$)
				N	S						
<i>Ila</i>	H	$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_2\text{S}$	246.3	11.37	13.01	89	129	246	1637	219	3.20
				11.52	12.83					269	3.28
<i>Ilb</i>	CH_3	$\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$	260.3	10.76	12.31	94	127	260	1670	214	3.30
				10.88	12.38					270	3.26
<i>Ilc</i>	OH	$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3\text{S}$	262.3	10.68	12.22	100	170	262	1677	216	3.47
				10.82	11.98					274	3.43
<i>Ild</i>	OCH_3	$\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3\text{S}$	276.3	10.13	11.60	86	141	276	1671	222	3.37
				10.33	11.42					275	3.38
<i>Ile</i>	$\text{N}(\text{Et})_2$	$\text{C}_{16}\text{H}_{19}\text{N}_3\text{O}_2\text{S}$	317.4	13.23	10.10	91	122	317	1672	214	3.37
				13.42	10.02					268	3.45
										365	3.23
<i>Ilf</i>	NHCOCH_3	$\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3\text{S}$	303.3	13.85	10.57	100	206	303	1675	217	3.55
				14.02	10.41					267	3.65
<i>Ilg^a</i>	Cl	$\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_2\text{S}$	280.7	9.97	11.42	89	145	280	1679	219	3.30
				10.12	11.40					269	3.37
<i>Ilh^b</i>	Br	$\text{C}_{12}\text{H}_9\text{BrN}_2\text{O}_2\text{S}$	325.2	8.61	9.86	81	153	325	1676	219	3.32
				8.73	10.03					268	3.38
<i>Ili</i>	NO_2	$\text{C}_{12}\text{H}_9\text{N}_3\text{O}_4\text{S}$	291.3	14.42	11.00	93	185	291	1678	215	3.38
				14.31	10.92					286	3.51
										345	3.30
<i>Ilj</i>	COOH	$\text{C}_{13}\text{H}_{10}\text{N}_2\text{O}_4\text{S}$	290.3	9.64	11.04	94	184	290	1672	218	3.42
				9.81	11.23					274	3.28

a) $w(\text{Cl, calc.}) = 12.62\%$; $w(\text{Cl, found}) = 12.48\%$; *b*) $w(\text{Br, calc.}) = 24.57\%$; $w(\text{Br, found}) = 24.31\%$.



Scheme 2

The synthesized thioureas *IIa*–*IIj* were utilized for preparation of 2-(4-X-phenylimino)-3-(3-furoyl)-4-R-4-thiazolines [21, 22] in reaction with bromoacetone (*IIIa*–*IIIj*; R = CH₃) and ω-bromoacetophenone (*IVa*–*IVj*; R = C₆H₅) (see Formula; p. 694).

The reactions were performed in boiling ethyl acetate and gave high yields. The structures of the synthesized compounds were proved by IR, ¹H NMR, and mass spectra (Tables 2–5). Besides bands characteristic of furan, in the IR spectra of derivatives *IIa*–*IIj* also the bands ν(NH) appeared in the region of $\tilde{\nu} = 3145\text{--}3405\text{ cm}^{-1}$ which in some cases were overlapped by the bands ν(CH). The bands ν(CO) had relatively stable position at $\tilde{\nu} = 1670\text{--}1679\text{ cm}^{-1}$. The characteristic bands belonging to NH–C=S grouping appeared in a relatively broad range at $\tilde{\nu} = 1110\text{--}1173\text{ cm}^{-1}$. The UV spectra of derivatives *IIa*–*IIj* (Table 1) revealed two absorption bands. The first one in the region of $\lambda = 214\text{--}222\text{ nm}$ ($\log \{\epsilon\} = 3.20\text{--}3.70$) and the second one in the region of $\lambda = 267\text{--}286\text{ nm}$ ($\log \{\epsilon\} = 3.28\text{--}3.65$) which correspond to electron transitions localized in the furan and benzene rings, respectively. The ¹H NMR and mass spectra of the synthesized compounds *II*, *III*, and *IV* proved the suggested structures unambiguously.

Experimental

Melting points were determined on a Kofler block. IR spectra were measured with a Specord IR-71 (Zeiss, Jena) spectrophotometer in the region of $\tilde{\nu} = 700\text{--}4000\text{ cm}^{-1}$ applying KBr technique. UV spectra were recorded with a Specord UV VIS (Zeiss, Jena)

Table 2

2-(4-X-Phenylimino)-3-(3-furoyl)-4-methyl-4-thiazolines (IIIa—IIIj)

Compound	X	Formula	M_r	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$		Yield %	M.p. °C	$M_r(M^{+})$	$\nu(\text{C=O})$ $\tilde{\nu}/\text{cm}^{-1}$	$\lambda_{\text{max}}/\text{nm}$	\log $(\epsilon/(\text{m}^2 \text{mol}^{-1}))$
				N	S						
IIIa	H	$\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$	284.3	9.85 9.75	11.27 11.38	90	92	284	1601	328	3.27
IIIb	CH_3	$\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$	298.3	9.38 9.37	10.75 10.88	82	150	298	1603	331	3.18
IIIc	OH	$\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}_3\text{S}$	300.3	9.32 9.42	10.67 10.78	94	156	300	1602	330	3.28
III d	OCH_3	$\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$	314.3	8.99 9.18	10.18 10.08	88	155	314	1601	331	3.26
IIIe	$\text{N}(\text{Et})_2$	$\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_2\text{S}$	335.4	11.92 12.05	9.02 9.21	79	160	355	1602	268 327	3.59 3.44
III f	NHCOCH_3	$\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_3\text{S}$	341.4	12.31 12.01	9.39 9.03	84	223	341	1602	253 330	3.21 3.18
III g ^a	Cl	$\text{C}_{15}\text{H}_{11}\text{ClN}_2\text{O}_2\text{S}$	318.8	8.78 8.95	10.06 10.17	88	142	318	1602	329	3.24
III h ^b	Br	$\text{C}_{15}\text{H}_{11}\text{BrN}_2\text{O}_2\text{S}$	362.2	7.71 7.73	9.18 9.01	91	161	363	1600	329	3.32
III i	NO_2	$\text{C}_{15}\text{H}_{11}\text{N}_3\text{O}_4\text{S}$	329.3	12.76 12.91	9.78 9.93	91	219	329	1601	259 329	3.10 3.19
III j	COOH	$\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_4\text{S}$	328.3	8.54 8.66	9.78 9.96	78	282	328	1601	333	3.27

a) $w(\text{Cl, calc.}) = 11.12\%$; $w(\text{Cl, found}) = 11.28\%$; b) $w(\text{Br, calc.}) = 22.0\%$; $w(\text{Br, found}) = 22.3\%$.

Table 3

2-(4-X-Phenylimino)-3-(3-furoyl)-4-phenyl-4-thiazolines (IVa—IVj)

Compound	X	Formula	M_r	$w_i(\text{calc.})/\%$		Yield	M.p.	$M_r(M^{+})$	$\nu(\text{C}=\text{O})$ $\tilde{\nu}/\text{cm}^{-1}$	$\lambda_{\text{max}}/\text{nm}$	\log $(\epsilon/(\text{m}^2 \text{mol}^{-1}))$
				N	S						
IVa	H	$\text{C}_{20}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$	346.4	8.11 8.31	9.26 9.42	98	210	346	1600	333	3.30
IVb	CH_3	$\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2\text{S}$	360.4	7.80 8.06	8.90 9.08	92	247	360	1604	332	3.15
IVc	OH	$\text{C}_{20}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$	362.4	7.74 7.79	8.84 8.92	80	241	362	1603	333	3.33
IVd	OCH_3	$\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_3\text{S}$	376.4	7.75 7.65	8.51 8.52	97	236	376	1602	334	3.07
IVe	$\text{N}(\text{Et})_2$	$\text{C}_{24}\text{H}_{23}\text{N}_3\text{O}_2\text{S}$	417.5	10.07 10.11	7.69 7.79	86	170	417	1604	273 330	3.46 3.33
IVf	NHCOCH_3	$\text{C}_{22}\text{H}_{17}\text{N}_3\text{O}_3\text{S}$	403.4	10.42 10.66	7.95 8.09	86	290	403	1601	258 336	3.26 3.22
IVg ^a	Cl	$\text{C}_{20}\text{H}_{13}\text{ClN}_2\text{O}_2\text{S}$	380.8	7.36 7.40	8.45 8.65	93	240	380	1603	334	3.21
IVh ^b	Br	$\text{C}_{20}\text{H}_{13}\text{BrN}_2\text{O}_2\text{S}$	425.3	6.58 6.45	7.54 7.69	98	249	425	1608	334	3.36
IVi	NO_2	$\text{C}_{20}\text{H}_{13}\text{N}_3\text{O}_4\text{S}$	391.4	10.74 10.86	8.21 8.33	83	262	391	1609	267 329	3.05 3.10
IVj	COOH	$\text{C}_{21}\text{H}_{14}\text{N}_2\text{O}_4\text{S}$	390.4	7.18 7.34	8.21 8.21	82	301	390	1605	334	3.30

a) $w(\text{Cl}, \text{calc.}) = 9.30\%$; $w(\text{Cl}, \text{found}) = 9.08\%$; b) $w(\text{Br}, \text{calc.}) = 18.78\%$; $w(\text{Br}, \text{found}) = 18.96\%$.

Table 4

¹H NMR data of *N*-(4-*X*-phenyl)-*N'*-(3-furoyl)thioureas (*IIa*—*IIj*)
 δ_i /ppm

Compound	H ²	H ⁴	H ⁵	H _A \equiv H' _A	H _B \equiv H' _B	Others
<i>IIa</i>	8.75 s	7.84 s	7.10 s			H _{ar} : 7.25—7.75 m (5H)
<i>IIb</i>	8.76 s	7.83 s	7.10 s	7.51 d	7.17 d	CH ₃ : 2.53 s
<i>IIc</i>	8.75 s	7.84 s	7.10 s	7.44 d	6.81 d	OH: 9.6
<i>IId</i>	8.79 s	7.66 s	7.06 s	7.53 d	6.88 d	OCH ₃ : 3.78 s
<i>IIe</i>	8.75 s	7.83 s	7.10 s	7.44 d	6.66 d	NCH ₂ : 3.35 q, CH ₃ : 1.1 t
<i>IIf</i>	8.75 s	7.85 s	7.10 s	7.44 s	7.44 s	CH ₃ : 3.38 s, NH: 10.45 s
<i>IIg</i>	8.76 s	7.85 s	7.10 s	8.73 d	7.44 d	
<i>IIh</i>	8.75 s	7.84 s	7.09 s	8.75 d	7.64 d	
<i>IIi</i>	8.78 s	7.80 s	7.09 s	8.09 d	8.31 d	NH: 11.65
<i>IIj</i>	8.75 s	7.84 s	7.10 s	8.34 d	7.52 d	

Table 5

¹H NMR data of 2-(4-X-phenylimino)-3-(3-furoyl)-4-methyl-4-thiazolines (*IIIa—IIIj*) and 2-(4-X-phenylimino)-3-(3-furoyl)-4-phenyl-4-thiazolines (*IVa—IVj*)
 δ_i /ppm

Compound	H ²	H ⁴	H ⁵	H _A ≡ H' _A	H _B ≡ H' _B	H ^{5'}	R ^{a,b}	Others
<i>IIIa</i>	7.78 s	7.30 s	6.69 s			6.34 s	2.05 s ^a	H _{ar} : 7.23—7.65 m (5H)
<i>IIIb</i>	7.80 s	7.27 s	6.69 s	7.32 d	7.12 d	6.31 s	2.01 s ^a	CH ₃ : 2.45 s
<i>IIIc</i>	7.82 s	7.30 s	6.71 s	7.21 d	7.01 d	6.31 s	2.04 s ^a	OH: 3.90 s
<i>III d</i>	7.81 s	7.30 s	6.70 s	7.21 d	7.01 d	6.31 s	2.05 s ^a	OCH ₃ : 3.90 s
<i>IIIe</i>	7.86 s	7.30 s	6.75 s	7.07 d	6.79 d	6.30 s	2.07 s ^a	NCH ₂ : 3.44 q, CH ₃ : 1.25 t
<i>III f</i>	7.81 s	7.30 s	6.71 s	7.76 d	7.23 d	6.33 s	2.04 s ^a	CH ₃ : 3.90 s
<i>III g</i>	7.83 s	7.31 s	6.71 s	7.55 d	7.26 d	6.35 s	2.05 s ^a	
<i>III h</i>	7.80 s	7.30 s	6.70 s	7.69 d	7.18 d	6.34 s	2.05 s ^a	
<i>III i</i>	7.80 s	7.32 s	6.67 s	7.56 d	8.46 d	6.41 s	2.10 s ^a	
<i>III j</i>	7.83 s	7.58 s	6.53 s	7.56 d	8.16 d	6.80 s	2.01 s ^a	
<i>IV a</i>	7.83 s	7.55 s	6.55 s			7.08 s	7.14—7.42 m (11H) ^b	
<i>IV b</i>	7.86 s	7.59 s	6.57 s				7.05—7.35 m (10H) ^b	
<i>IV c</i>	7.88 s	7.58 s	6.60 s		6.91 d	7.06 s	7.10—7.32 m (7H) ^b	
<i>IV d</i>	7.86 s	7.56 s	6.56 s		6.90 d	7.05 s	7.10—7.30 m (7H) ^b	OCH ₃ : 3.72 s
<i>IV e</i>	7.88 s	7.55 s	6.60 s	7.01 d	6.58 d	7.03 s	7.21 s (5H) ^b	NCH ₂ : 3.30 q, CH ₃ : 1.04 t
<i>IV f</i>	7.89 s	7.60 s	6.58 s	7.64 d		7.09 s	7.15—7.32 m (7H) ^b	CH ₃ : 2.03 s
<i>IV g</i>	7.91 s	7.60 s	6.56 s			7.10 s	7.15—7.42 m (9H) ^b	
<i>IV h</i>	7.90 s	7.60 s	6.58 s	7.57 d		7.08 s	7.20—7.32 m (7H) ^b	
<i>IV i</i>	7.95 s	7.60 s	6.60 s	7.62 d	8.24 d	7.15 s	7.15—7.70 m (5H) ^b	
<i>IV j</i>	7.94 s	7.61 s	6.60 s	7.45 d	7.99 d	7.14 s	7.20—7.36 m (5H) ^b	

a) R = CH₃; b) R = Ph; c) compounds *IVa* and *IIIj* measured in DMSO.

spectrometer in methanol at $1-5 \times 10^{-5} \text{ mol dm}^{-3}$ concentration. Mass spectra were measured with an AEI (Manchester) MS 902 S apparatus (direct inlet) at ionizing voltage 70 eV, ionizing current 100 μA , and ion source temperature 70–170 °C.

3-Furoyl isothiocyanate (I)

To the solution of potassium thiocyanate (0.16 mol) 3-furoyl chloride (0.16 mol) was added at room temperature during 10 min. The mixture was boiled for 10 min. The precipitate of potassium chloride was filtered off and the mixture was evaporated under reduced pressure. The crude product was distilled at 66–67 °C (65 Pa).

For $\text{C}_6\text{H}_3\text{NO}_2\text{S}$ ($M_r = 153.16$) $w_i(\text{calc.})$: 9.14 % N, 20.9 % S; $w_i(\text{found})$: 9.29 % N, 21.05 % S; b.p. = 66–67 °C (65 Pa), yield = 70–73 %. ^1H NMR spectrum (C^2HCl_3), δ/ppm : 8.17 (s, 1H, H^2 furan), 7.50 (d, 1H, H^4), 6.83 (d, 1H, H^5). IR spectrum (chloroform), $\tilde{\nu}/\text{cm}^{-1}$: $\nu(\text{CO})$ 1692, $\nu(\text{NCS})$ 1955.

N-(4-X-Phenyl)-N'-(3-furoyl)thioureas (IIa–IIj)

To the solution of 4-X-aniline (10 mmol) in acetone (50 cm^3) 3-furoyl isothiocyanate I (10 mmol) in acetone (10 cm^3) was added slowly. The mixture was stirred at 5–10 °C for 1–3 h. The precipitate was sucked off and washed with cyclohexane. By this reaction analytically pure compounds were obtained.

2-(4-X-Phenylimino)-3-(3-furoyl)-4-methyl-4-thiazolines (IIIa–IIIj) and 2-(4-X-phenylimino)-3-(3-furoyl)-4-phenyl-4-thiazolines (IVa–IVj)

To the solution of thioureas IIa–IIj in ethyl acetate (60 cm^3) bromoacetone and ω -bromoacetophenone (4.3 mmol), respectively, in ethyl acetate (10 cm^3) were added in one portion. After 3 h the precipitate of bromide was sucked off, dissolved in hot water, and neutralized with NaOH to pH = 8. The product was sucked off and crystallized.

N-(4-Tolyl)-N'-(3-furoyl)urea (IIk)

To the solution of thiourea IIb (40 mmol) in acetone (50 cm^3) mercury(II) oxide (0.02 mol) was added in one portion. The mixture was boiled for 3–4 h. The precipitate of mercury sulfide was sucked off, the mixture was concentrated, and the product was crystallized from aqueous acetone.

For $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$ ($M_r = 244.25$) $w_i(\text{calc.})$: 11.47 % N; $w_i(\text{found})$: 11.58 % N; m.p. = 206–208 °C, yield = 98 %. ^1H NMR spectrum (DMSO), δ/ppm : 10.84 (s, 1H, NH), 10.71 (s, 1H, NH), 8.61 (s, 1H, H^2 furan), 7.83 (d, 1H, H^4 furan), 7.43 (d, 2H, Ph),

7.13 (d, 2H, Ph), 7.08 (s, 1H, H⁵ furan), 2.28 (s, 3H, CH₃). IR spectrum (KBr), $\tilde{\nu}/\text{cm}^{-1}$: $\nu(\text{C}=\text{O})$ 1670, 1692.

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