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Condensation of 3-methyl/ethyl-5-mercapto-*s*-triazole with 3-acetylcoumarin and its derivatives

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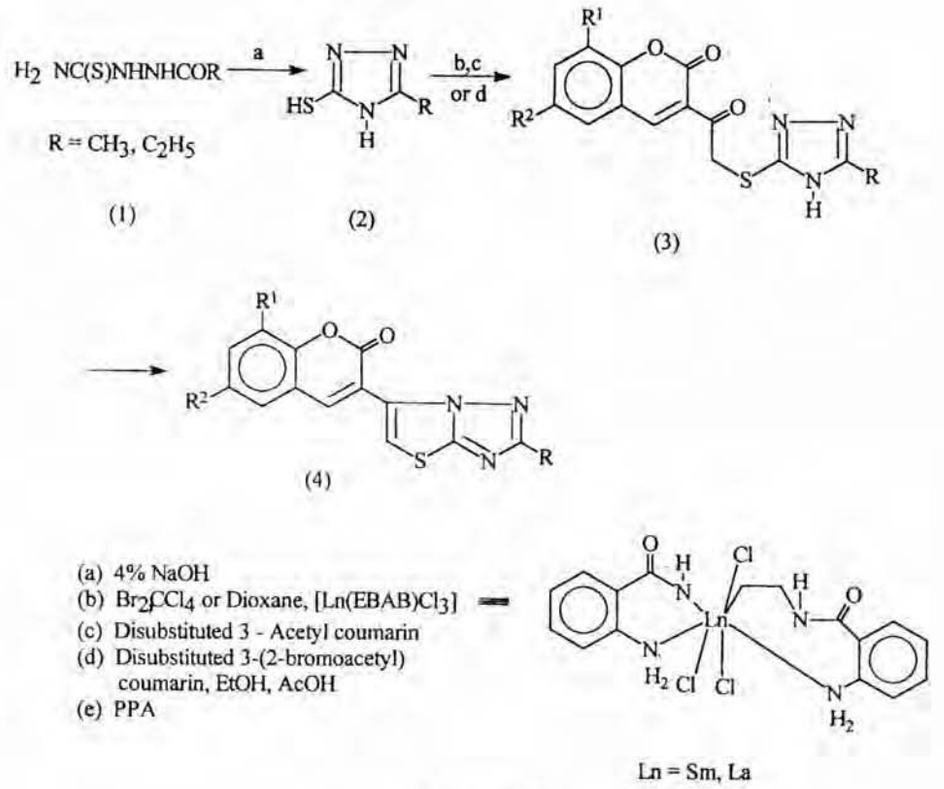
3-Methyl/ethyl-5-mercapto-*s*-triazoles **2** on condensation with either 3-(2-bromoacetyl)coumarins or with substituted 3-acetyl comarins using bromine in the presence of trichloro-(*N,N'*-ethylene-bis-aminobenzamide) lanthanum and samarium as a catalyst, followed by cyclization of the intermediate 3-alkyl-5-coumarinacyl-thio-*s*-triazole **3** using PPA furnish 3-alkyl-5-coumarinylthiazolo[3,2-*b*]-*s*-triazoles **4** and not the isomeric 3-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazoles **6**. 2-Acetyl- or 2-propanoylhydrazino-4-coumarinyl-thiazolo hydrobromides **5**, obtained from the reaction of acetyl/propanoyl thiosemicarbazides **1** with either substituted 3-(2-bromoacetyl) coumarins or with bromine and substituted 3-acetylcoumarin using trichloro-(*N,N'*-ethylene-bis-aminobenzamide)lanthanum (III) and samarium (III) as a catalyst, on treatment with phosphoryl chloride undergo facile cyclization yielding 5-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole **6**.

Thiazole derivatives exhibit a wide spectrum of biological activities[1-4]. Thiazolo-*s*-triazoles have been shown to exhibit remarkable anthelmentic activity[5] and also coumarin derivatives with a heterocyclic system at position-3 exhibit a promising biological activity[6]. In view of this and in continuation of earlier work on the synthesis of heterocyclic systems derived from coumarins[7-9], we report herein the preparation of triazolo thiazoles. Two routes for the synthesis of thiazolo[3,2-*b*]-*s*-triazole have been documented in the literature.[10-18] The first route in which a triazole ring is built onto a thiazolo ring involves the reaction of 3-amino-2-iminothiazoline with acid[10], anhydride[11] or phosgene imonium chloride[12] or via cyclization of 2-acylimino-3-aminothiazoline-*o*-mesitylene sulphonate salt with polyphosphoric acid.[13] The second route in which a thiazole ring is built onto a triazole ring involves the reaction of 5-mercapto-*s*-triazole with α-halogenoketones[14-18] or propargyl bromide[16,17] followed by cyclization of the intermediates. While the former route provides unequivocal synthesis of alkyl-5-coumarinyl-thiazolo[3,2-*b*]-*s*-triazole **4**, the formation of 3-alkyl-5-coumarinyl-thiazolo[3,2-*b*]-*s*-triazole **4** or/and 3-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole **6** by the latter is a plausible alternative.

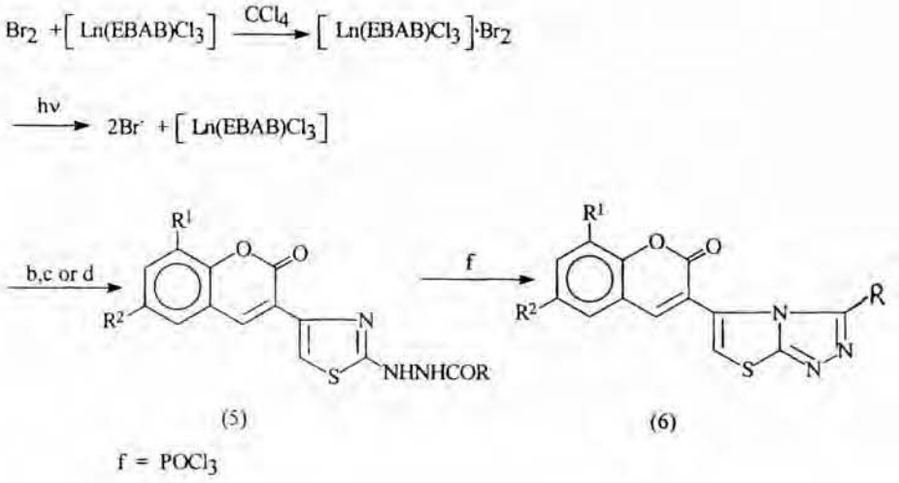
Condensation of 3-methyl or ethyl-5-mercapto-*s*-triazole **2** with either 3-(2-bromoacetyl) coumarins in acetic acid or anhyd. ethanol or via direct reaction of **2** with substituted 3-acetylcoumarins in carbon tetrachloride or dioxane under photohalogenation and using bromine in the presence of chloro-(*N, N'*-ethylene-bis-aminobenzamide) lanthanum (III) or samarium (III) catalyst[19], yielded the uncyclized ketone **3**. It subsequently underwent PPA cyclization giving thiazolo-[3,2-*b*]-*s*-triazole **4** and not **6** (**Scheme I**). Refluxing **2** and 3-(2-bromoacetyl)-coumarin for a prolonged time in anhyd. ethanol or acetic acid gave only the uncyclized ketone **3**. Unlike in the previous experiments reported from our laboratories[7], it has now been reported, that bromination, catalysed by the lanthanide complexes goes to completion within a few minutes.

The structures of **3** and **4** have been established by IR spectral data - **3a** exhibited a band at 1684 cm^{-1} which was absent in **4** thereby suggesting the cyclic structure for **4**. The ^1H NMR spectra of **4h** (R^1, R^2 = H) exhibit singlets at δ 9.6 for H(6) thereby corroborating the 3-ethyl-5-coumarinyl-thiazolo[3,2-*b*]-*s*-triazole **4** structure.

Mercapto-*s*-triazole **2**, being unsymmetrical, on condensation with either 6,8-disubstituted-3-acetylcoumarin or 3-(2-bromoacetyl)coumarins, and subsequent cyclization, is expected to give the triazole **4** or its isomer **6**, or both depending on the mode of cyclization. The ketone **3**, however, on cyclization with PPA gave only one product (via TLC). PMR data of the cyclized product was not of use in judging in favour of either structure **4** or **6**. Hence, the



Scheme I



Scheme II

unequivocal synthesis of **6** has been carried out as follows. Acetyl thiosemicarbazides **1**, when condensed with 6,8-disubstituted-3-acetyl-coumarins in the presence of bromine in dry carbon tetrachloride or dioxane using lanthanum catalyst or when reacted with 6,8-disubstituted 3-(2-bromo-acetyl)coumarins in anhyd. ethanol yielded 2-acetyl-hydryazino-4-coumarinyl-thiazole **5**. Subsequent treatment of **5** with $POCl_3$ induced facile cyclization to give the triazole **6** (**Scheme II**). The compounds **4,5** and **6** were characterized on the basis of spectra. **5a** exhibited a band at 1668 cm^{-1} (C=O), which was absent in **6b** thereby establishing its cyclic structure. The signal at δ 7.86 (s) for H(6) shown by **6h** (R^1, R^2=H; R=CH_2CH_3) in its PMR spectrum confirmed the cyclic structure for it. Compound **6** was not identical

with the cyclized product **4** obtained from ketone **3**. A modified procedure has been used in the preparation of 2-mercapto-5-ethyl-1,3,4-triazole. It has been found that aq. sodium hydroxide is better and more efficient cyclization agent than aq. sodium carbonate.[14]

Experimental Section

All melting points were determined in open capillary tubes using sulphuric acid bath and are uncorrected. IR spectra (ν_{max} in cm^{-1}) were recorded on Perkin-Elmer-282 instrument. The ^1H NMR spectra were recorded on Varian 90 MHz spectrometer using TMS as internal standard and chemical shifts are expressed in δ, ppm. Mass spectra were scanned on a JEOL-JMS-300 spectrometer using 70 eV. The purity of the compounds were monitored by TLC, performed on silica gel plates (Merck) and using chloroform-methanol (3:1) as the eluent.

2-Mercapto-5-ethyl-1,3,4-triazole 2b. Propanoyl thio-semicarbazide (1.47 g; 0.01 mole) was dissolved in 4% aq. NaOH and heated on water bath for 1 hr. The reaction mixture was acidified with acetic acid to yield 2-mercapto-5-ethyl-1,3,4-triazole (1.24 g; 96%), m.p. 249-50°C (Lit.[20] 248-51°C).

3-Alkyl-5-coumarinacylthio-*s*-triazole 3: Method I. A mixture of 3-alkyl-5-mercapto-*s*-triazole (0.01 mole) and 6, 8-disubstituted-3-(2-bromoacetyl)-coumarin (0.01 mole) in abs. ethanol or in gl. acetic acid (50 mL) was heated under reflux for 1 hr. The reaction mixture was cooled to room temperature and the hydrobromide thus separated was removed and washed with water, filtered and recrystallized from suitable solvent (**Table 1**).

3h : IR : 1605 (C=N), 1680 (C=O) and 1720 cm^{-1} (lactone C=O); ^1H-NMR (DMSO-d_6) : 1.25 (t, 3H, CH_3), 2.70 (2H, q, CH_2 of ethyl group), 4.7 (s, 2H, $-S-CH_2$), 7.0 - 7.9 (m, 5H, Ar-H and NH) and 8.60 (s, 1H, C_4); MS: m/z 315, M^+ (16.5).

Method II. Lanthanide complex (5 mg) was suspended in dry carbon tetrachloride (100 mL) containing corres-ponding 6, 8-disubstituted-3-

Table 1—Physical data of ketones **3**, thiazolo[3,2-*b*]-*s*-triazoles **4**, 2-acylhydrazino-4-coumarinylthiazole hydrobromides **5** and 2-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole **6**

Compd	R	R^1	R^2	m.p (°C)[a]	Recryst Solvent	Mol. formula (Mol.wt)	Calc. (Found) % N	S
3a	CH_3	H	H	227-29	MeOH	$C_{13}H_{11}N_3O_3S$ (301)	13.92 (13.92	10.63 10.60)
3b	CH_3	OCH_3	H	177-79	MeOH	$C_{15}H_{13}N_3O_4S$ (331)	12.68 (12.65	9.66 9.62)
3c	CH_3	H	Br	220-22	$CHCl_3/CH_3OH$	$C_{14}H_{10}N_3BrO_3S$ (300)	11.05 (11.00	8.42 8.40)
3d	CH_3	Br	Br	222-24	$CHCl_3$/MeOH	$C_{14}H_9N_3Br_2O_2S$ (459)	9.15 (9.00	6.97 6.94)
3e	CH_3	H	Cl	226-28	$CHCl_3/CH_3OH$	$C_{14}H_{10}N_3ClO_3S$ (335.5)	12.51 (12.50	9.53 9.50)
3f	CH_3	5,6-benzo-		218-20	$CHCl_3/CH_3OH$	$C_{18}N_{13}N_3O_3S$ (351)	11.96 (11.93	9.11 9.00)
3g	CH_3	H	CH_3	201-03	DMF/CH_3OH	$C_{15}H_{13}N_3O_3S$ (315)	13.33 (13.30	10.15 10.00)
3h	Et	H	H	220-22	Pet.ether/$CHCl_3$	$C_{15}H_{13}N_3O_3S$ (315)	13.33 (13.30	10.15 10.00)
3i	Et	OCH_3	H	215-17	Aq./ACOH	$C_{16}H_{15}N_3O_3S$ (345)	12.17 (12.14	9.27 9.25)
3j	Et	H	Br	201-03	n-butanol	$C_{15}H_{12}N_3BrO_3S$ (394)	10.65 (10.60	12.15 12.16)
3k	Et	Br	Br	210-12	Aq./DMF	$C_5H_{11}N_3Br_2O_3S$ (473)	8.87 (8.82	6.76 6.75)
3l	Et	H	Cl	140-42	Aq./DMF	$C_{15}H_{12}N_3ClO_3S$ (349.5)	12.01 (12.00	9.15 9.00)
3m	Et	5,6-benzo-		235-37	Pet.ether/$CHCl_3$	$C_{19}H_{15}N_3O_2S$ (365)	11.50 (11.48	8.76 8.75)
3n	Et	H	CH_3	230-32	MeOH	$C_{16}H_{15}N_3O_2S$ (329)	12.76 (12.72	9.76 9.70)

Contd.

Table I—Physical data of ketones **3**, thiazolo[3,2-*b*]-*s*-triazoles **4**, 2-acylhydrazino-4-coumarinylthiazole hydrobromides **5** and 2-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole **6** (Contd.)

Compd	R	R^1	R^2	m.p. (°C)[a]	Recryst Solvent	Mol. formula (Mol.wt)	Calc. (Found) % N	S
4a	CH_3	H	H	274-76	Pet. ether/ chloroform	$C_{12}H_9N_3O_2S$ (283)	14.84 (14.83	11.30 11.27)
4b	CH_3	OCH_3	H	239-41	Aq/DMF	$C_{15}H_{11}N_3O_3S$ (313)	13.41 (13.39	15.33 15.30)
4c	CH_3	H	Br	283-85	DMF/CH_3OH	$C_{14}H_8N_3BrO_2S$ (362)	11.60 (11.56	8.83 8.81)
4d	CH_3	Br	Br	224-26	DMF/CH_3OH	$C_{14}H_7N_3Br_2O_2S$ (441)	9.52 (9.50	7.25 7.20)
4e	CH_3	H	Cl	272-74	Aq./DMF	$C_{14}H_8N_3ClO_2S$ (317.5)	13.22 (13.20	10.07 10.00)
4f	CH_3	5,6-benzo-		267-69	DMF/CH_3OH	$C_{14}H_7N_3Cl_2O_2S$ (352)	12.61 (12.60	9.60 9.56)
4g	CH_3	H	CH_3	233-35	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (297)	14.14 (14.11	10.77 10.73)
4h	Et	H	H	180-82	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (297)	14.14 (14.10	10.77 10.74)
4i	Et	OCH_3	H	275-77	Aq./DMF	$C_{16}H_{13}N_3O_3S$ (311)	12.84 (12.81	10.28 10.24)
4j	Et	H	Br	> 360	Aq./DMF	$C_{15}H_{10}N_3BrO_2S$ (376)	11.17 (11.13	8.51 8.50)
4k	Et	Br	Br	283-85	DMF/ethanol	$C_{15}H_9N_3Br_2O_2S$ (455)	9.23 (9.20	7.03 7.00)
4l	Et	H	Cl	230-32	Aq./DMF	$C_{15}H_{10}N_3ClO_2S$ (331.5)	12.66 (12.63	9.66 9.61)
4m	Et	5,6-benzo		285-87	DMF/ethanol	$C_{19}H_{13}N_3O_2S$ (347)	12.10 (12.00	9.22 9.19)
4n	Et	H	CH_3	201-03	CH_3OH	$C_{16}H_{13}N_3O_2S$ (295)	13.50 (13.46	10.20 10.24)
5a	CH_3	H	H	250-52	Aq./AcOH	$C_{14}H_{11}N_3O_3S$ (301)	13.95 (13.92	10.63 10.60)
5b	CH_3	OCH_3	H	257-59	n-butanol	$C_{15}H_{13}N_3O_4S$ (331)	12.68 (12.61	9.66 9.63)
5c	CH_3	H	Br	268-70	Aq./AcOH	$C_{14}H_{10}N_3BrO_3S$ (380)	11.05 (11.00	8.42 8.40)
5d	CH_3	Br	Br	259-61	C_6H_6/MeOH	$C_{14}H_9N_3Br_2O_3S$ (459)	9.15 (9.12	6.79 6.94)
5e	CH_3	H	Cl	250-52	DMF/CH_3OH	$C_{14}H_{10}N_3ClO_3S$ (335.5)	12.50 (12.50	9.53 9.50)
5f	CH_3	5,6-benzo-		241-43	Aq./DMF	$C_{18}H_{13}N_3O_3S$ (351)	11.96 (11.94	13.67 13.64)
5g	CH_3	H	CH_3	243-45	Aq /DMF	$C_{15}H_{13}N_3O_3S$ (315)	13.33 (13.30	10.15 10.10)
5h	Et	H	H	216-18	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (297)	13.33 13.30	10.15 10.12)
5i	Et	H	OCH_3	202-04	Aq./DMF	$C_{16}H_{13}N_3O_3S$ (345)	12.17 (12.14	9.27 9.24)
5j	Et	H	Br	221-23	Aq./DMF	$C_{15}H_{12}N_3BrO_3S$ (394)	10.65 (10.62	8.12 8.10)
5k	Et	Br	Br	175-77	Benzene	$C_{15}H_{11}N_3Br_2O_3S$ (473)	8.87 (8.85	6.76 6.73)
5l	Et	H	Cl	216-18	Benzene/MeOH	$C_{13}H_{12}N_3ClO_3S$ (349.5)	12.01 (12.00	13.73 13.72)
5m	Et	5,6-benzo-		240-42	Aq/DMF	$C_{19}H_{15}N_3O_2S$ (365)	11.50 (11.49	8.76 8.72)

Contd.

Table I—Physical data of ketones **3**, thiazolo[3,2-*b*]-*s*-triazoles **4**, 2-acylhydrazino-4-coumarinylthiazole hydrobromides **5** and 2-alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole **6** (Contd.)

Compd	R	R^1	R^2	m.p. (°C)[a]	Recryst Solvent	Mol. formula (Mol.wt)	Calc. (Found) % N	S
5n	Et	H	CH_3	190-92	AcOH	$C_{16}H_{15}N_3O_2S$ (329)	12.76 (12.73	9.72 9.71)
6a	CH_3	H	H	> 360	Aq./DMF	$C_{14}H_9N_3O_2S$ (283)	14.84 (14.81	11.30 11.28)
6b	CH_3	OCH_3	H	285-87	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (313)	13.41 (13.40	15.33 15.30)
6c	CH_3	H	Br	217-19	Aq./AcOH	$C_{14}H_8N_3BrO_2S$ (362)	11.60 (11.58	8.83 8.80)
6d	CH_3	Br	Br	218-20	Aq./AcOH	$C_{14}H_7N_3Br_2O_2S$ (441)	9.52 (9.51	7.25 7.21)
6e	CH_3	H	Cl	> 360	Aq./DMF	$C_{14}H_8N_3ClO_2S$ (317.5)	13.22 (13.20	10.07 10.00)
6f	CH_3	5,6-benzo-		255-57	Aq./DMF	$C_{18}H_{11}N_3O_2S$ (333)	12.68 (12.69	9.66 9.64)
6g	CH_3	H	CH_3	> 360	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (297)	14.14 (14.10	10.77 10.74)
6h	Et	H	H	283-85	Aq./DMF	$C_{15}H_{11}N_3O_2S$ (297)	14.14 (14.12	10.73 10.70)
6i	Et	OCH_3	H	> 360	n-butanol	$C_{16}H_{13}N_3O_3S$ (311)	12.84 (12.81	10.28 10.26)
6j	Et	H	Br	> 360	DMF/ethanol	$C_{15}H_{10}N_3BrO_2S$ (376)	11.17 (11.14	8.51 8.50)
6k	Et	Br	Br	> 360	n-butanol	$C_{15}H_9N_3Br_2O_2S$ (455)	9.23 (9.20	7.00 6.96)
6l	Et	H	Cl	> 360	DMF/ethanol	$C_{15}H_{10}N_3ClO_2S$ (331.5)	12.66 (12.62	9.65 9.62)
6m	Et	5,6-benzo-		> 360	Aq./DMF	$C_{19}H_{13}N_3O_2S$ (347)	12.10 (12.10	9.22 9.21)
6n	Et	H	CH_3	> 360	DMF/ethanol	$C_{16}H_{13}N_3O_2S$ (295	13.50 (13.47	10.28 10.26)

[a]All compounds were obtained in 70-80% yield
Satisfactory C, H analysis have been obtained

acetylcoumarin (0.05 mole) and to this was added slowly bromine (0.05 mole in CCl_4, 20 mL) or dioxane (20 mL) while stirring the reaction mixture in the presence of light. After the addition of bromine, the temperature was raised to 50°C to facilitate the completion of the reaction. To the reaction mixture 3-alkyl-5-mercapto-*s*-triazole (0.05 mole) was added and the reaction completed in 5 min. (TLC). The solvent was removed and the product obtained was filtered, washed with ethanol and then with water and recystallized from suitable solvents (**Table I**).

3-Alkyl-5-coumarinylthiazolo[3, 2-*b*]-*s*-triazole 4. A mixture of **3** (1 g), phosphorous pentoxide (4 g) and orthophosphoric acid (3 mL) was heated in an oil-bath at 150°C for 3 hr. The reaction mixture was poured into ice cold water and neutralized with sodium carbonate. The solid, thus obtained was crystallized from suitable solvents.

4h : IR : 1606 (C=N), 1717 cm^{-1} (ketone C=O); ^{1}H-NMR ($CDCl_3$) : δ 1.40 (t, 3H, CH_3), 2.90 (2H, q, CH_2, ethyl), 7.2 - 7.75 (m, 4H, Ar-H), 8.2 (s, C_4-H) and 9.6 (s, 1H, C_6); MS : m/z 297 (M^+, 70%).

The properties and analysis of other ketones **3** and triazoles **4** are recorded in **Table I**.

N^2-Acylhydrazino-coumarinylthiazoles 5: Method I. Lanthanum complex (5 mg) was suspended in carbon tetrachloride (100 mL) or in dioxan (100 mL) containing the corresponding 6,8-disubstituted-3-acetyl-coumarin (0.01 mole) and to this was added slowly bromine (0.01 mole) in carbon tetrachloride (20 mL) with stirring in the presence of light. After the addition of bromine, the temperature was raised to 50°C and *N*-acyl-thiosemicarbazide added to the reaction mixture. After 5 min., the solvent was distilled off. The product so obtained was treated with ethanol,

filtered, washed with water and crystallized from appropriate solvent (**Table I**).

Method II. A mixture of **1** (0.01 mole) and 3-(2-bromoacetyl)coumarin (2.67 g; 0.01 mole) is abs. ethanol (25 mL) was heated under reflux for 30 min. The reaction mixture was cooled to room temperature, the solid that separated was filtered and crystallized from suitable solvents (**Table I**).

5a : IR : 1607 (C=N), 1668 (–CO–NH), 1717 (lactone C=O) and 3201 cm^{-1} (-NH-); ^{1}H-NMR (DMSO-d_6): δ 2.50 (s, 3H, CH_3), 7.39 - 7.89 (m, 4H, Ar-H and 1H, NH), 7.68 (s, 1H thiazole), 8.54 (s, 1H, C-4) and 9.5 (s, 1H, –NH–CO–); MS : m/z 301 (M^+, 66.6%).

3-Alkyl-5-coumarinylthiazolo[2,3-*c*]-*s*-triazole 6. Compound **5** (1g) in $POCl_3$ (8.0 mL) was heated on an oil-bath at 130-40°C for 4 hr. The reaction mixture was cooled, poured into water and neutralized with sodium carbonate. The solid so obtained was filtered and washed with water and recrystallized from suitable solvents viz. Table I.

6a : IR : 1606 (C=N) and 1720 cm^{-1} (lactone C=O); ^{1}H NMR (DMSO-d_6) : δ 2.51 (s, 3H, CH_3), 7.56-7.75 (m, 4H, Ar-H), 7.8 (s, 1H, thiazole) and 8.49 (s, 1H, C_4-H); MS: m/z 283 (M^+, 66.6%)

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