Synthesis of 5-Alkyl-1,3,5-triazinan-2-ones and 5-Alkyl-1,3,5-triazinane-2-thiones Using Cu- and Sm-Containing Catalysts

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Abstract—Efficient procedures have been developed for the synthesis of 5-alkyl-1,3,5-triazinan-2-ones and 5-alkyl-1,3,5-triazinane-2-thiones by reaction of urea (thiourea) with primary alkylamines and N,N,N',N'-tetramethylmethylenediamine and by reaction of primary amines with N,N'-bis(dimethylaminomethyl)urea(thiourea) in the presence of Cu- and Sm-containing catalysts.

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Interest in compounds of the 1,3,5-triazinane series originates from their potential applications as pesticides [1] and bioprotective agents for fuels and lubricants [2]. Substituted 1,3,5-triazinane-2-thiones and 1,3,5-triazinan-2-ones exhibit antiviral and antibacterial activity [3] and are also promising as intermediate products in the synthesis of antitumor alkaloids [4, 5].

One of the most popular procedures for the synthesis of 5-substituted 1,3,5-triazinan-2-ones and 1,3,5-triazinane-2-thiones is based on classical Mannich aminomethylation of ureas (thioureas) with primary amines and formaldehyde [6–8]; a modification of this procedure, thermal condensation of primary amines with bis(hydroxymethyl)urea preliminarily prepared from formaldehyde and urea, is also known [6, 9]. These methods are characterized by some limitations related to the use of aqueous formaldehyde [6–8], elevated reaction temperature (90–100°C), and low selectivity [9].

With a view to develop a new efficient procedure for the synthesis of potentially practically important 5-alkyl-1,3,5-triazinan-2-ones and 5-alkyl-1,3,5-triazinane-2-thiones [3–5] we examined the reaction of urea, thiourea, and N-allylthiourea with primary alkylamines (Alk = i-Pr, Bu, Pr, t-Bu, cyclohexyl, 2-hydroxyethyl)and N, N, N', N'-tetramethylmethylenediamine, catalyzed by transition and rare-earth metal salts and complexes. N, N, N', N'-Tetramethylmethylenediamine was selected as aminomethylating agent, taking into account its efficiency in C-N bond-forming reactions with primary and secondary amines [10–13]. We presumed that geminal diamines can be used in intermolecular aminomethylation of ureas (thioureas) and primary amines for the synthesis of 1,3,5-triazinan-2-ones and 1.3.5-triazinane-2-thiones.

Urea reacted with N,N,N',N'-tetramethylmethylenediamine and cyclohexylamine to give 5-cyclohexyl-1,3,5-triazinan-2-one (**Ia**) [molar ratio C₆H₁₁NH₂-





la-lc, lla-llf, llla, lllb

 $\begin{bmatrix} Cu \end{bmatrix} = CuCl_2 \cdot 2H_2O; I, X = O, R = H; II, X = S, R = H; III, X = S, R = CH_2 = CHCH_2; \\ R' = cyclo-C_6H_{11} (a), t-Bu (b), HO(CH_2)_2 (c), i-Pr (d), Bu (e), Pr (f).$

Me₂NCH₂NMe₂-urea-catalyst 10:20:10:0.5, 80°C, CHCl₃-EtOH (3:1), 8 h] in 44 (CuCl₂·2H₂O), 32 (SmCl₃·6H₂O), 29 [Sm(NO₃)₃·6H₂O], and 27% yield (FeCl₃·6H₂O). In the absence of a catalyst the yield of **Ia** did not exceed 10%. Using CuCl₂·2H₂O as catalyst under the above conditions, by reactions of urea (thioureas) with N,N,N',N'-tetramethylmethylenediamine and alkylamines we obtained triazinan-2-ones and triazinane-2-thiones **Ia–Ic**, **IIa–IIf**, **IIIa**, and **IIIb** in 24– 46% yield (Scheme 1).

The structure of compounds **I–III** was proved by their ¹H and ¹³C NMR (including 2D), IR, and mass spectra. The ¹³C and ¹H NMR spectra of **I–III** contained signals from methylene carbon atoms and protons at δ_C 57.18–63.39 and δ 3.99–4.30 ppm. In the 2D HMBC spectra of these compounds we observed cross peaks corresponding to interactions of C² with 4-H and 6-H.

Presumably, the reaction involves initial aminomethylation of urea (thiourea) with N,N,N',N'-tetramethylmethylenediamine to produce N,N'-bis[(dimethylamino)methyl]urea(thiourea) **IV** or **V**, and the latter reacts with primary alkylamines, yielding 1,3,5-triazinan-2-ones or -thiones. In order to verify this assumption we synthesized N,N'-bis[(dimethylamino)methyl]urea (**IV**) and N,N'-bis[(dimethylamino)methyl]thiourea (**V**) by aminomethylation of urea and thiourea with N,N,N',N'-tetramethylmethylenediamine by analogy with catalytic aminomethylation of compounds having labile hydrogen atoms (terminal acetylenes and thiols) with geminal diamines; these reactions were reported to afford propargylamines [14, 15] or amino sulfides [16, 17].

In the model aminomethylation of urea with N,N,N',N'-tetramethylmethylenediamine in the absence of a catalyst (EtOH, 20°C, 1.5 h) N,N'-bis(dimethyl-

aminomethyl)urea (**IV**) was formed in no more than 30% yield. To improve the yield of **IV**, various catalysts based on metal halides and complexes were tested [SmCl₃·6H₂O, ZnCl₂·2H₂O, Sm(NO₃)₃·6H₂O, YbF₃, EuCl₃·6H₂O, CuCl₂·2H₂O, FeCl₃·6H₂O, CoCl₂, MnCl₂·2H₂O, Cp₂ZrCl₂, Cp₂TiCl₂, PdCl₂], and the effects of temperature, reaction time, and solvent nature on the yield of **IV** were studied. Among the above catalysts, the most active were SmCl₃·6H₂O and Sm(NO₃)₃·6H₂O: they ensured formation of **IV** in 98 and 97% yield, respectively (Scheme 2). When the reaction was carried out in C₆H₁₄–EtOH or DMF–EtOH, the yield of **IV** decreased to 60% against 98% in CHCl₃–EtOH or EtOAc–EtOH.

The structure of compounds IV and V was proved by spectral data. The NCH₂N fragment in IV and V was represented by the following signals in the ¹H and ¹³C NMR spectra: δ_C 63.56, δ 4.34–4.44 (IV), δ_C 68.00, δ 3.65 ppm (V). The MALDI TOF mass spectra contained the molecular ion peaks $[M + K]^+$ with m/z 213.554 (IV) and 229.354 (V).

Compounds IV and V reacted with primary alkylamines to give the corresponding 1,3,5-triazinan-2ones and 1,3,5-triazinane-2-thiones Ia–Ic and IIa–IId (Scheme 2). The reactions were carried under the conditions optimized for the reaction with cyclohexylamine [amine–IV–SmCl₃·6H₂O ratio 10:10:0.5, EtOH, 80°C, 8 h]. The yield of 5-cyclohexyl-1,3,5-triazinan-2-one (Ia) was ~39%, and the yields of the other compounds I and II ranged from 23 to 41%. If no catalyst was added, the yield of I and II did not exceed 5%.

Taking into account published data [18–21], the aminomethylation of primary amines with N,N'-bis-[(dimethylamino)methyl]urea(thiourea) is likely to begin with coordination of molecule **IV** or **V** to the



Scheme 2.

I, IV, X = O; II, V, X = S; R' = cyclo-C₆H₁₁ (a), t-Bu (b), HO(CH₂)₂ (c); i-Pr (d).

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central metal ion of the catalyst. The subsequent nucleophilic addition of alkylamine molecule to the carbocation thus formed generates new C–N bonds and leads to the formation of 1,3,5-triazinane ring (Scheme 3).

We can conclude that the aminomethylation of primary alkylamines with N,N'-bis[(dimethylamino)methyl]urea(thiourea) in the presence of Sm-containing catalyst ensures successful selective synthesis of 5-alkyl-1,3,5-triazinan-2-ones and 5-alkyl-1,3,5-triazinane-2-thiones.

EXPERIMENTAL

The progress of reactions was monitored by TLC on Sorbfil plates. The ¹H and ¹³C NMR spectra were recorded on a Bruker Avance-400 spectrometer at 400.13 and 100.62 MHz, respectively, using DMSO-d₆ as solvent and reference (δ 2.50, δ_C 39.50 ppm). The IR spectra were measured on a Bruker Vertex 70v spectrometer from samples dispersed in mineral oil. The mass spectra were obtained on a Bruker Autoflex III MALDI TOF spectrometer (Germany) using α -cyano-4-hydroxycinnamic acid and 2,5-dihydroxybenzoic acid as matrices; samples were prepared by the dried drop technique in chloroform (1:10). The elemental compositions were determined on a Carlo Erba 1106 analyzer, and the melting points were measured on an PHMK 80/2617 melting point apparatus. Individual substances were isolated by column chromatography on KSK silica gel (50-160 µm).

5-Alkyl-1,3,5-triazinan-2-ones Ia–Ic and 5-alkyl-1,3,5-triazinane-2-thiones IIa–IIf, IIIa, and IIIb (general procedures). a. Three-component condensation of alkylamine with urea and N,N,N',N'-tetrameth-

ylmethylenediamine. A mixture of 10 mmol of alkylamine, 20 mmol of N,N,N',N'-tetramethylmethylenediamine, and 0.5 mmol of CuCl₂·2H₂O was stirred for 5 min. Urea, 10 mmol, was then added, and the mixture was heated to 80°C and stirred for 8 h at that temperature. The product was isolated by column chromatography on silica gel.

b. Aminomethylation of alkylamines with N,N'-bis-[dimethylaminomethyl]urea(thiourea). A mixture of 10 mmol of N,N'-bis[(dimethylamino)methyl]urea (thiourea), 3 ml of ethanol, 0.5 mmol of $SmCl_3 \cdot 6H_2O$, and 10 mmol of alkylamine was stirred for 8 h at 80°C. The product was isolated by column chromatography on silica gel.

5-Cyclohexyl-1,3,5-triazinan-2-one (Ia). Yield 44% (*a*), 39% (*b*); colorless crystals, mp 204–205°C (205°C [6]), $R_{\rm f}$ 0.63 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ , ppm: 1.17 m (5H, 8-H, 9-H, 10-H, 11-H, 12-H), 1.55 s (1H, 10-H), 1.70 s (2H, 9-H, 11-H), 1.89 br.s (2H, 8-H, 12-H), 2.54 br.s (1H, 7-H), 4.28 s (4H, 4-H, 6-H), 6.21 s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 25.08 (C⁹, C¹¹), 25.96 (C¹⁰), 31.05 (C⁸, C¹²), 54.20 (C⁷), 58.48 (C⁴, C⁶), 155.69 (C²). Mass spectrum: *m*/*z* 184.444 (*I*_{rel} 100%) [*M* + H]⁺. Calculated: *M* 183.251.

5-tert-Butyl-1,3,5-triazinan-2-one (Ib). Yield 37% (*a*), 34% (*b*); colorless crystals, mp 182–184°C (181– 182°C [22]), $R_{\rm f}$ 0.66 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ , ppm: 1.12 s (9H, *t*-Bu), 4.10 s (4H, 4-H, 6-H), 6.23 br.s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 28.71 [C(CH₃)₃], 53.50 [C(CH₃)₃], 57.18 (C⁴, C⁶), 156.12 (C²). Mass spectrum: *m*/*z* 158.446 (*I*_{rel} 100%) [*M* + H]⁺. Calculated: *M* 157.214.

5-(2-Hydroxyethyl)-1,3,5-triazinan-2-one (Ic). Yield 25% (*a*), 23% (*b*); colorless crystals, mp 173– 176°C (176–177°C [6]), R_f 0.45 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ, ppm: 2.65 t (2H, 5-CH₂), 3.50 br.s (2H, CH₂OH), 4.00 s (4H, 4-H, 6-H), 4.78 br.s (1H, OH), 6.23 s (2H, NH). ¹³C NMR spectrum, δ_C, ppm: 52.34 (5-CH₂), 59.95 (CH₂OH), 61.87 (C⁴, C⁶), 155.74 (C²). Mass spectrum: *m/z* 146.404 (I_{rel} 100%) [*M* + H]⁺. Calculated: *M* 145.160.

5-Cyclohexyl-1,3,5-triazinane-2-thione (IIa). Yield 46% (*a*), 41% (*b*); colorless crystals, mp 172–174°C (172–176°C [6]), $R_{\rm f}$ 0.76 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ , ppm: 1.15 m (5H, 8-H, 9-H, 10-H, 11-H, 12-H), 1.53 s (1H, 10-H), 1.70 s (2H, 9-H, 11-H), 1.85 br.s (2H, 8-H, 12-H), 2.45 br.s (1H, 7-H), 4.09 s (4H, 4-H, 6-H), 7.94 s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 25.01 (C⁹, C¹¹), 25.89 (C¹⁰), 30.88 (C⁸, C¹²), 55.22 (C⁷), 58.62 (C⁴, C⁶), 176.56 (C²). Found, %: C 54.17; H 8.86; N 20.99; S 15.98. C₉H₁₇N₃S. Calculated, %: C 54.23; H 8.60; N 21.08; S 16.09.

5-tert-Butyl-1,3,5-triazinane-2-thione (IIb). Yield 35% (*a*), 28% (*b*); colorless crystals, mp 169–172°C (175–177°C [7]), $R_{\rm f}$ 0.79 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ, ppm: 1.10 s (9H, *t*-Bu), 4.11 s (4H, 4-H, 6-H), 7.98 br.s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 28.44 [C(CH₃)₃], 53.71 [C(CH₃)₃], 57.19 (C⁴, C⁶), 176.78 (C²). Mass spectrum: *m*/*z* 174.380 (*I*_{rel} 100%) [*M* + H]⁺. Calculated: *M* 173.280.

5-(2-Hydroxyethyl)-1,3,5-triazinane-2-thione (IIc). Yield 29% (*a*), 25% (*b*); colorless crystals, mp 160–162°C (162°C [6]), $R_{\rm f}$ 0.71 (ethanol–acetone, 1:1). ¹H NMR spectrum, δ , ppm: 2.59 t (2H, 5-CH₂), 3.51 m (2H, CH₂OH), 4.00 s (4H, 4-H, 6-H), 4.57 br.s (1H, OH), 8.00 s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 53.04 (5-CH₂), 60.01 (CH₂OH), 62.12 (C⁴, C⁶), 175.89 (C²). Mass spectrum: *m*/*z* 162.416 (*I*_{rel} 100%) [*M* + H]⁺. Calculated: *M* 161.226.

5-Isopropyl-1,3,5-triazinane-2-thione (IId). Yield 25% (*a*), 23% (*b*); colorless crystals, mp 153–155°C, $R_f 0.74$ (ethanol–acetone, 1:1). IR spectrum, v, cm⁻¹: 3205, 2854–2924, 1555, 1539, 1384, 1302, 1203, 1038, 721, 666. ¹H NMR spectrum, δ , ppm: 1.02 s and 1.04 s (3H each, CH₃), 2.78 m (1H, 5-CH), 4.08 s (4H, 4-H, 6-H), 7.88 br.s (2H, NH). ¹³C NMR spectrum, δ_C , ppm: 21.23 (CH₃), 47.49 (5-CH), 59.08 (C⁴, C⁶), 176.19 (C²). Mass spectrum: *m*/*z* 160.360 (*I*_{rel} 100%) [*M* + H]⁺. Found, %: C 45.22; H 8.04; N 26.40; S 20.34. C₆H₁₃N₃S. Calculated, %: C 45.25; H 8.25; N 26.38; S 20.12. *M* 159.254.

5-Butyl-1,3,5-triazinane-2-thione (IIe). Yield 25%, colorless crystals, mp 166–170°C, $R_{\rm f}$ 0.64

(acetone). IR spectrum, v cm⁻¹: 3188, 2854–2925, 1609, 1556, 1456, 1391, 1301, 1206, 992, 666. ¹H NMR spectrum, δ , ppm: 0.85 m (3H, CH₃), 1.26 m (2H, CH₃CH₂), 1.37 m (2H, 5-CH₂CH₂), 2.47 m (2H, 5-CH₂), 3.99 s (4H, 4-H, 6-H), 7.06 br.s (2H, NH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 14.20 (CH₃), 20.32 (CH₃CH₂), 29.64 (5-CH₂CH₂), 49.85 (5-CH₂), 61.32 (C⁴, C⁶), 175.57 (C²). Mass spectrum: *m/z* 174.342 (*I*_{rel} 100%) [*M* + H]⁺. Calculated: *M* 173.280.

5-Propyl-1,3,5-triazinane-2-thione (IIf). Yield 24%, colorless crystals, mp 135–137°C, R_f 0.61 (acetone). IR spectrum, v, cm⁻¹: 3187, 2854–2925, 1609, 1556, 1376, 1301, 1239, 1063, 955, 667. ¹H NMR spectrum, δ, ppm: 0.83 t (3H, CH₃), 1.41 q (2H, CH₃CH₂, J = 6.8 Hz), 2.44 t (2H, 5-CH₂), 3.99 s (4H, 4-H, 6-H), 7.88 br.s (2H, NH). ¹³C NMR spectrum, δ_C , ppm: 11.96 (CH₃), 20.79 (CH₃CH₂), 52.08 (5-CH₂), 61.33 (C⁴, C⁶), 175.55 (C²). Mass spectrum: m/z 160.405 (I_{rel} 100%) [M + H]⁺. Found, %: C 45.24; H 8.02; N 26.33; S 20.41. C₆H₁₃N₃S. Calculated, %: C 45.25; H 8.25; N 26.36; S 20.14. *M* 159.254.

1-Allyl-5-cyclohexyl-1,3,5-triazinane-2-thione (IIIa). Yield 43%, colorless crystals, mp 107–110°C, R_f 0.33 (ethyl acetate–hexane, 2:1). IR spectrum, v, cm⁻¹: 3243, 2780–2958, 1641, 1520, 1463, 1303, 1212, 1113, 993, 645. ¹H NMR spectrum, δ , ppm: 1.11 m (5H, 8-H, 9-H, 10-H, 11-H, 12-H), 1.53 s (1H, 10-H), 1.67 s (2H, 9-H, 11-H), 1.83 br.s (2H, 8-H, 12-H), 2.43 br.s (1H, 7-H), 4.11 s (2H, 4-H), 4.22 s (2H, 6-H), 4.27 s (2H, 1-CH₂), 5.18 m (2H, =CH₂), 5.77 m (1H, CH=), 7.91 s (1H, NH). ¹³C NMR spectrum, δ_C , ppm: 24.92 (C⁹, C¹¹), 25.74 (C¹⁰), 30.80 (C⁸, C¹²), 52.20 (1-CH₂), 55.63 (C⁷), 58.27 (C⁴), 63.39 (C⁶), 118.18 (=CH₂), 133.45 (=CH), 177.11 (C²). Mass spectrum: *m/z* 240.435 (*I*_{rel} 100%) [*M* + H]⁺. Found, %: C 60.12; H 8.91; N 17.54; S 13.43. C₁₂H₂₁N₃S. Calculated, %: C 60.21; H 8.84; N 17.55; S 13.40. *M* 239.381.

1-Allyl-5*-tert*-butyl-1,3,5-triazinane-2-thione (IIIb). Yield 32%, colorless crystals, mp 65–68°C, R_f 0.38 (ethyl acetate–hexane, 2:1). IR spectrum, v, cm⁻¹: 3204, 2866–3004, 1695, 1638, 1538, 1310, 1217, 1137, 996, 648. ¹H NMR spectrum, δ, ppm: 1.17 s (9H, *t*-Bu), 4.30 m (4H, 4-H, 6-H), 4.41 d (2H, 1-CH₂, J = 6 Hz), 5.26 m (2H, =CH₂), 5.91 m (1H, =CH), 7.25 br.s (1H, NH). ¹³C NMR spectrum, δ_C, ppm: 28.42 [C(CH₃)₃], 53.22 (1-CH₂), 54.47 [C(CH₃)₃], 57.21 (C⁴), 61.68 (C⁶), 118.60 (=CH₂), 132.60 (=CH), 177.76 (C²). Mass spectrum: *m*/*z* 212.377 (*I*_{rel} 100%) [*M* – H]⁺. Found, %: C 56.23; H 9.07; N 19.64; S 15.06. C₁₀H₁₉N₃S. Calculated, %: C 56.30; H 8.98; N 19.70; S 15.02. *M* 213.344.

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Aminomethylation of urea and thiourea with N,N,N',N'-tetramethylmethylenediamine. A mixture of 22 mmol of N,N,N',N'-tetramethylmethylenediamine and 0.5 mmol of SmCl₃·6H₂O was stirred for 5 min, a solution of 10 mmol of urea or thiourea in CHCl₃– EtOH (1:1) was added, and the mixture was stirred for 1 h at 20°C.

N,*N*'-Bis[(dimethylamino)methyl]urea (IV) [23]. Yield 99%, viscous resinous material, $n_D^{20} = 1.4770$, $R_f 0.33$ (ethanol–acetone, 1:1). ¹H NMR spectrum, δ, ppm: 2.67 br.s (12H, CH₃), 4.34 br.s and 4.44 br.s (2H each, CH₂), 8.16 br.s (2H, NH). ¹³C NMR spectrum, δ_C , ppm: 41.79 (CH₃), 63.56 (CH₂), 159.83 (C=O). Mass spectrum: *m*/*z* 213.554 (*I*_{rel} 100%) [*M* + K]⁺. Found, %: C 48.53; H 10.24; N 31.98. C₇H₁₈N₄O. Calculated, %: C 48.25; H 10.42; N 32.15. *M* 174.244.

N,N'-Bis[(dimethylamino)methyl]thiourea (V). Yield 99%, colorless amorphous substance, mp 77– 80°C, R_f 0.46 (ethanol–acetone, 1:1). IR spectrum, v, cm⁻¹: 3437, 2923–2853, 1619, 1463, 1383, 1095–1072, 845–738. ¹H NMR spectrum, δ , ppm: 2.26 br.s (12H, CH₃), 3.65 br.s (4H, CH₂), 8.58 br.s (2H, NH). ¹³C NMR spectrum, δ_C , ppm: 41.81 (CH₃), 68.00 (CH₂), 184.89 (C=S). Mass spectrum: *m*/*z* 229.354 (*I*_{rel} 100%) [*M* + K]⁺. Found, %: C 44.02; H 9.76; N 29.49; S 16.73. C₇H₁₈N₄S. Calculated, %: C 44.18; H 9.53; N 29.44; S 16.85. *M* 190.311.

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