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New Approach to the Synthesis of Cyclic 1,3-Dithioacetals from Thiophene-2-carbaldehydes

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Abstract—A highly selective method was developed for preparation of previously unknown 1,3-dithiolanes and 1,3-dithianes by reactions of thiophene-2-carbaldehyde and its analogs with alkanedithiols under the action of trimethylchlorosilane.

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The first representative of cyclic 1,3-dithioacetals, 1,3-dithiolane, was obtained as early as 1893 [1]. Nowadays a great number of publications are known treating the studies of this class compounds for they are valuable from the theoretical and practical viewpoint.

The cyclic 1,3-dithioacetals are known as highly reactive precursors in the organic sysnthesis [2–6]. They are often used as an equivalent of an acyl anion [7, 8] or a methylene function in reactions proceeding with a C–C bond formation [9, 10]. Because of their high stability both in acid and alkaline media (pH 2–12) [11] they are widely applied to protection of the carbonyl function in chemical processes. The cyclic 1,3-dithioacetals are used as the initial building blocks in preparation of liquid crystalline materials [12] and in construction of fragments of biologically active substances, for instance, of immunosuppressant (–) – FK-506 [13]. Various 1,3-dithiolane derivatives are efficient biostimulants and fungicides [14] they possess properties of radioprotectors [15] and semi-conductors [16].

Especially interesting are cyclic 1,3-dithioacetals containing a thiophene fragment for presumably the introduction of a thiophene ring into the molecule should increase its biological action. However the chemistry of this class compounds is poorly documented. A single representative of the cyclic 1,3-dithioacetals from the thiophene series, 2-(1,3-dithiolan-2-yl)thiophene was obtained, but its physical constants were not published [7, 17].

The classic way of building up the 1,3-dithiolane and 1,3-dithiane rings consisted in condensation of carbonyl compounds with dithols in the presence of dry hydrogen chloride [18], inorganic [19–21] and organic acids [22–24], Lewis acids [3, 13, 17, 25], ion-exchange resin Amberlyst-15 [11], sulfur(II) oxide [26], molten tetrabutylammonium bromide [7], and bromodimethylsulfonium bromide [10]. Alongside these methods exist also other procedures, based on the reaction of dithols with ethyl diethoxyacetate [27], (organylsulfanyl)chloroacetylenes [28], substituted propene-2-nitriles [29], and acetals [3, 10, 22]. Regretfully, some of these procedures require stringent reaction conditions, use of expensive catalysts and reagents, inert atmosphere, prolonged process time, and additional workup of the reaction mixture for separation of the product. Besides in some cases water liberated in the course of the process should be trapped by special reagents or in the Dean-Stark trap.

Therefore the numerous existing methods for preparation of the cyclic dithioacetals do not contradict the necessity of searching for new alternative procedures occurring under milder and simpler conditions. This is especially important for building up the 1,3-dithiolane and 1,3-dithiane systems containing thiophene structures for the stringent reaction conditions and aggressive environment cause the degradation of the thiophene ring.

The target of this study was development of a synthetic procedure for 1,3-dithiolanes and 1,3-dithianes containing the thiophene fragment. We recently carried out a chemoselective reaction catalyzed by trimethylchlorosilane (Me_3SiCl) between thieno[2,3-*b*]thiophene-2-carbaldehyde with ethanedithiol affording previously unknown heterocyclic compound, 2-(1,3-dithiolan-2-yl)thieno[2,3-*b*]thiophene [30]. In extension of these investigations we

carried out condensations of aldehydes from the thiophene series with aliphatic dithiols applying trimethylchlorosilane both as solvent and as catalyst.

We established that the Me_3SiCl is an efficient catalyst for aldehydes thioacetalization. Under its action aldehydes **Ia–Id** cleanly reacted with ethane- and 1,3-propanedithiols at –5...0°C within 30 min furnishing 1,3-dithiolanes **IIa** and **IId** and dithianes **IIIa–IIId** in virtually quantitative yields (Scheme 1). Inasmuch as the reaction is performed at low temperature and at 4–8-fold excess of the silane the side reaction of dithiols into disulfides and tarring are avoided.

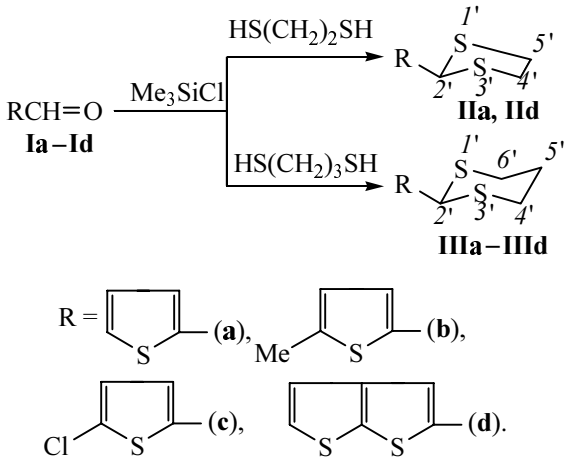
The reaction occurred as an intramolecular condensation of two thiol groups with the aldehyde function. The role of Me_3SiCl consists in assisting the protonation of the carbonyl group (Scheme 2).

The hydrogen chloride forms *in situ* by reaction of dithiol with Me_3SiCl and then rapidly generates a proton for the chloride anion is bound by the silyl cation. This process excludes the formation of aggressive reagents capable to deteriorate the thiophene ring. In the absence of Me_3SiCl, for instance, the thiophene-2-carbaldehyde with ethanedithiol afford 2-(1,3-dithiolan-2-yl)thiophene (**IIa**) only in 13% yield, and the conversion of the initial aldehydes reaches 17%. In contrast, the introduction into the reaction of Me_3SiCl increases the yield of dithioacetal **IIa** to 78%, and the conversion of thiophene-2-carbaldehyde, to 100%. The other valuable quality of Me_3SiCl is that its presence excludes the use of water-consuming agents since the water liberating in the course of the synthesis hydrolyzes Me_3SiCl into HMDS [31]. The arising HMDS alongside the Me_3SiCl is easily evaporated from the reaction mixture on completion of the reaction and warming the reaction mixture to the room temperature, and the reaction products are isolated further by distillation or recrystallization of the residue.

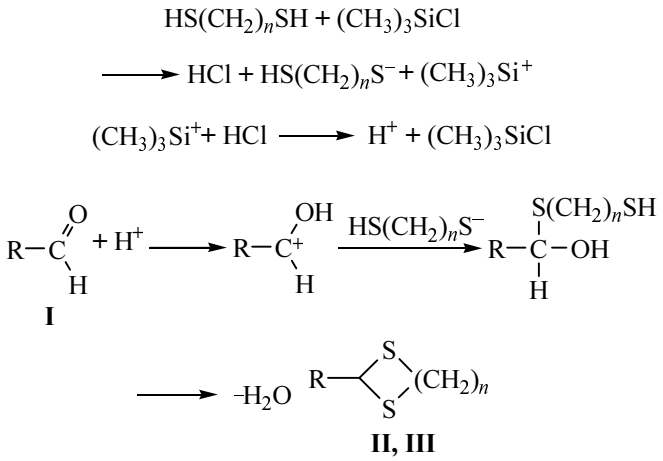
The structure of the newly obtained compounds **IIa** and **IId**, **IIIa–IIId** was unambiguously proved by spectral methods. In the ^1H NMR spectra in the appropriate regions appear the signals from protons of the 1,3-dithiolane and 1,3-dithiane rings with the corresponding multiplicity and integral intensity. Therewith the 1,3-dithiane moieties of compounds **IIIa–IIId** have the *chair* conformation. The ^{13}C NMR spectra also confirm the structure of compounds synthesized. In the mass spectra of 1,3-dithioacetals **II** and **III** the peaks of molecular ions are observed, and the elemental analysis data are well consistent with their composition.

Thus a new way of synthesis was developed for previously unknown 1,3-dithiolanes and 1,3-dithianes

Scheme 1.



Scheme 2.



containing thiophene fragments. It was established that thioacetalization of the thiophene-2-carbaldehydes with alkanedithiols occurred chemoselectively in the presence of a 4–8-fold excess of Me_3SiCl. A series of new 1,3-dithioacetals was prepared, promising as biologically active compounds, practically useful materials, and synthons for organic synthesis.

EXPERIMENTAL

The monitoring of reaction progress and checking the purity of compounds obtained was carried out by GLC on a chromatograph LKhM-8MD-3 in the linear programming mode of the oven temperature, column 2000×3 mm, stationary phase silicone XE-60, 5%, on Chromaton N-AW-HMDS, carrier gas helium. ^1H and ^{13}C NMR spectra were registered from solutions of compounds in $CDCl_3$ on a spectrometer Bruker DPX-

400 (operating frequencies 400.1 and 100.6 MHz for ^{1}H and ^{13}C respectively), internal reference HMDS. It should be noted that in the ^{1}H NMR spectra of 1,3-dithiolanes **IIa** and **IId** the protons of the SCH_2 group constitute a four-spin system AA^1BB^1 and appear in the spectrum as two multiplets. The nonequivalence of the protons originates from the spatial orientation of the heteroaromatic fragment. For the assignment of carbon atoms of CH group in thieno[2,3-*b*]thiophene system of compound **IId** a two-dimensional C–H correlation HMQC was applied with the *z*-gradient and detection of the proton spectrum, whereas the assignment of the quaternary carbon atoms was done on the ^{13}C NMR spectra registered without decoupling from protons. The mass spectra were obtained on a GC-MS instrument Shimadzu GCMS-QP5050A in the mode of electron impact ionization at the electrons energy 60 eV; capillary column 60 m. stationary phase SE-54, injector temperature 250°C, oven temperature programmed at the rate 10 deg/min in the range 70–260°C.

Reaction of thiophene-2-carbaldehyde (Ia) with ethanedithol in the absence of trimethylchlorosilane. To 1.12 g (0.01 mol) of thiophene-2-carbaldehyde (**Ia**) cooled to –5°C was added dropwise 1.41 g (0.015 mol) of ethanedithol. The reaction mixture was vigorously stirred at –5...5°C. According to GLC in 1.5 h the conversion of the initial aldehyde was 17%, and the yield of 2-(1,3-dithiolan-2-yl)-thiophene (**IIa**), 13%.

General procedure for the synthesis of cyclic 1,3-dithioacetals (II, III). To aldehyde **Ia–Id** dissolved in Me_3SiCl and cooled to –5°C the corresponding dithiol was added dropwise. Because of high volatility the dithiols were taken in a slight excess over the equimolar amount. The reaction mixture was vigorously stirre at –5...0°C. According to GLC data the reaction was completed within 0.5 h. Then the reaction mixture was warmed to the room temperature. On evaporating the silane the reaction products were isolated either by vacuum distillation or by recrystallization.

2-(1,3-Dithiolan-2-yl)thiophene (IIa) was obtained from 1.68 g (0.015 mol) of aldehyde **Ia** and 1.88 g (0.02 mol) of ethanedithiol in 12.96 g (0.12 mol) of Me_3SiCl. By vacuum distillation was separated 1.47 g (78%) of oily substance of light-lemon color, bp 132–134°C (2 mm Hg), n_D^{18} 1.6542. ^{1}H NMR spectrum, δ, ppm: 3.26 m (2H, SCH_2), (AA^1-part of AA^1BB^1-spin system of 1,3-dithiolane ring), 3.39 m (2H, SCH_2), (BB^1-part of AA^1BB^1-spin system of 1,3-dithiolane ring), 5.87 s (1H, $H^{2'}$), thiophene ring: 6.83 d.d [1H, H^4, $^3J(H^4–H^5)$ 3.54, $^3J(H^4–H^5)$ 5.14 Hz], 7.0 d.d.d [1H, H^3, $^4J(H^3–H^{2'}) \approx 1.0$, $^4J(H^3–H^5)$ 1.2 Hz], 7.15 d.d (1H, H^5). ^{13}C NMR spectrum, δ, ppm: 39.62 ($C^{4'}$, $C^{5'}$), 50.45 ($C^{2'}$), thiophene ring: 125.23 (C^5), 125.32 (C^3), 126.27 (C^4), 146.72 (C^2). Found, %: C 44.64; H 4.50; S 50.50. $[M]^+$ 188. $C_7H_8S_3$. Calculated, %: C 44.68; H 4.25; S 51.00. *M* 188.

2-(1,3-Dithiolan-2-yl)thieno[2,3-*b*]thiophene (IId) was obtained from 2.52 g (0.015 mol) of aldehyde **Id** and 1.41 g (0.015 mol) of ethanedithiol in 6.8 g (0.063 mol) of Me_3SiCl. Yield of crude substance 3.66 g (100%). On recrystallization from a mixture benzene–ethanol, 1:2, was isolated 3.3 g (90%) of crystalline substance of light-pink color, mp 81–83°C. ^{1}H NMR spectrum, δ, ppm: 3.29 m and 3.44 m (4H, SCH_2CH_2S), (AA^1-part and BB^1-part of AA^1BB^1-spin system of 1,3-dithiolane ring), 5.90 s (1H, $H^{2'}$), thieno[2,3-*b*]thiophene system: 7.07 d [1H, H^4, $^3J(H^4–H^5)$ 5.2 Hz], 7.17 s (1H, H^3), 7.24 d (1H, H^5). ^{13}C NMR spectrum, δ, ppm: 39.95 ($C^{4'}$, $C^{5'}$), 51.67 ($C^{2'}$), thieno[2,3-*b*]thiophene system: 118.32 (C^3), 119.92 (C^4), 127.42 (C^5), 137.34 (C^8), 145.64 (C^2), 150.32 (C^7). Found, %: C 45.16; H 3.56; S 51.94. $[M]^+$ 244. $C_9H_8S_4$. Calculated, %: C 44.26; H 3.27; S 52.45. *M* 244.

2-(1,3-Dithian-2-yl)thiophene (IIIa) was obtained from 1.12 g (0.01 mol) of aldehyde **Ia** and 1.44 g (0.013 mol) of 1,3-propanedithiol in 5.4 g (0.05 mol) of Me_3SiCl. Yield of crude substance 2.02 g(100%). On recrystallization from a mixture benzene–ethanol, 1:2, was isolated 1.71 g (84.7%) of crystalline substance of light-brown color, mp 74–76°C. ^{1}H NMR spectrum, δ, ppm: 1.96 d.t.t [1H, $H_a^{5'}$, $^2J(H_a^{5'}–H_e^{5'})$ 13.98, $^3J(H_a^{5'}–H_a^{4'(6')})$ 10.21, $^3J(H_a^{5'}–H_e^{4'(6')})$ 3.66 Hz], 2.13 d.t.t [1H, $H_e^{5'}$, $^3J(H_e^{5'}–H_e^{4'(6')})$ 2.79, $^3J(H_e^{5'}–H_a^{4'(6')})$ 5.66 Hz], 2.93 d.d.d [2H, $H_e^{4'(6')}$, $^2J(H_a^{4'(6')}–H_e^{4'(6')})$ 14.22 Hz], 2.97 d.d.d (2H, $H_a^{4'(6')}$), 5.38 s (1H, $H^{2'}$), thiophene ring: 6.94 d.d [1H, H^4, $^3J(H^4–H^3)$ 3.74 Hz, $^3J(H^4–H^5)$ 4.91 Hz], 7.14 d (1H, H^3), 7.24 d (1H, H^5). ^{13}C NMR spectrum, δ, ppm: 25.06 ($C^{5'}$), 31.04 ($C^{4'(6')}$), 44.74 ($C^{2'}$), thiophene ring: 125.72 (C^5), 126.25 (C^3), 126.82 (C^4), 142.50 (C^2). Found, %: C 47.65; H 5.15; S 47.15. $[M]^+$ 202. $C_8H_{10}S_3$. Calculated, %: C 47.52; H 4.95; S 47.52. *M* 202.

2-(1,3-Dithian-2-yl)-5-methyl thiophene (IIIb) was obtained from 1.26 g (0.01 mol) of aldehyde **Ib** and 1.19 g (0.011 mol) of 1,3-propanedithiol in 8.64 g (0.08 mol) of Me_3SiCl. The vacuum distillation afforded 1.81 g (84%) of oily substance of yellow color, bp 180–186°C (5 mm Hg) that crystallized on standing. After washing with cold hexane we obtained light-yellow crystalline compound, mp 38–39°C. ^{1}H NMR spectrum, δ, ppm:

1.88 d.t.t [1H, $H_a^{5'}$, $^2J(H_a^{5'}–H_e^{5'})$ 14.07, $^3J(H_a^{5'}–H_a^{4'(6')})$ 10.74, $^3J(H_a^{5'}–H_e^{4'(6')})$ 3.45 Hz], 2.05 d.t.t [1H, $H_e^{5'}$, $^3J(H_e^{5'}–H_e^{4'(6')})$ 2.74, $^3J(H_e^{5'}–H_a^{4'(6')})$ 5.64 Hz], 2.85 d.d.d [2H, $H_e^{4'(6')}$, $^2J(H_a^{4'(6')}–H_e^{4'(6')})$ 14.20 Hz], 2.92 d.d.d (2H, $H_a^{4'(6')}$), 5.30 s (1H, $H^{2'}$), 2.40 s (3H, CH_3), thiophene ring: 6.54 d.q [1H, H^4, $^3J(H^4–H^3)$ 3.50, $^4J(H^4–CH_3)$ 1.10 Hz], 6.88 d (1H, H^3). ^{13}C NMR spectrum, δ, ppm: 24.70 ($C^{5'}$), 30.76 ($C^{4'(6')}$), 44.65 ($C^{2'}$), 15.04 (CH_3), thiophene ring: 124.44, 125.63 (C^3, C^4), 139.52, 139.68 (C^2, C^5). Found, %: C 50.42; H 5.91; S 44.80. $[M]^+$ 216. $C_9H_{12}S_3$. Calculated, %: C 50.00; H 5.56; S 44.44. *M* 216.

2-(1,3-Dithian-2-yl)-5-chlorothiophene (IIIc) was obtained from 1.18 g (0.008 mol) of aldehyde **Ic** and 1.08 g (0.01 mol) 1,3-propanedithiol in 4.35 g (0.04 mol) of Me_3SiCl. Yield of crude substance 1.88 g (98.9%). On recrystallization from a mixture benzene–ethanol, 1:2, was isolated 1.5 g (80%) of crystalline substance of light-brown color, mp 36–37.5°C. ^1H NMR spectrum, δ, ppm: 1.95 d.t.t [1H, H_a5', $^2J(H_a^{5'}–H_e^{5'})$ 14.10, $^3J(H_a^{5'}–H_a^{4'(6')})$ 9.14, $^3J(H_a^{5'}–H_e^{4'(6')})$ 3.76 Hz], 2.10 d.t.t [1H, $H_a^{5'}$ $^3J(H_e^{5'}–H_e^{4'(6')})$ 3.35, $^3J(H_e^{5'}–H_a^{4'(6')})$ 6.32 Hz], 2.87 d.d.d [2H, $H_e^{4'(6'}$, $^2J(H_a^{4'(6')}–H_e^{4'(6')})$ 14.23 Hz], 2.93 d.d.d (2H, $H_a^{4'(6')}$), 5.19 C (1H, $H^{2'}$), thiophene ring: 6.75 d [1H, H^4, $^3J(H^4–H^3)$ 3.76 Hz], 6.93 br.d (1H, H^3). ^{13}C NMR spectrum, δ, ppm: 24.54 ($C^{5'}$), 29.52 ($C^{4'(6')}$), 43.57 ($C^{2'}$), thiophene ring: 125.43, 125.50 (C^3, C^4), 129.69 (C^5), 141.46 (C^2). Found, %: C 40.77; H 3.59; Cl 15.15; S 40.38. $[M]^+$ 236 (calculated on Cl^{35}). $C_8H_9ClS_3$. Calculated, %: C 40.59; H 3.81; Cl 15.01; S 40.59. *M* 236.5.

2-(1,3-Dithian-2-yl)thieno[2,3-*b*]thiophene (IIId) was obtained from 1.68 g (0.01 mol) of aldehyde **Id** and 1.44 g (0.013 mol) of 1,3-propanedithiol in 5.4 g (0.05 mol) of Me_3SiCl. Yield of crude substance 2.41 g (93%). On recrystallization from a mixture benzene–ethanol, 1:2, was isolated 2.0 g (77%) of crystalline substance of light-brown color, mp 85–87.5°C. ^1H NMR spectrum, δ, ppm: 1.96 d.t.t [1H, $H_a^{5'}$, $^2J(H_a^{5'}–H_e^{5'})$ 14.11, $^3J(H_a^{5'}–H_a^{4'(6')})$ 8.96, $^3J(H_a^{5'}–H_e^{4'(6')})$ 7.20 Hz], 2.11 d.t.t [1H, $H_e^{5'}$, $^3J(H_e^{5'}–H_e^{4'(6')})$ 3.60, $^3J(H_e^{5'}–H_a^{4'(6')})$ 3.20, $^3J(H_e^{5'}–H_e^{4'(6')})$ 3.80 Hz], 2.91 d.d.d [2H, $H_a^{4'(6')}$, $^2J(H_a^{4'(6')}–H_e^{4'(6')})$ 14.40 Hz], 2.95 d.d.d (2H, $H_e^{4'(6')}$), 5.34 C (1H, $H^{2'}$), thieno[2,3-*b*]thiophene system: 7.13 d [1H, H^4, $^3J(H^4–H^5)$ 5.2 Hz], 7.28 d (1H, H^5), 7.32 d [1H, H^3, $^4J(H^3–H^{2'})$ 1.04 Hz]. ^{13}C NMR spectrum, δ, ppm: 25.06 ($C^{5'}$), 30.21 ($C^{4'(6')}$), 44.92 ($C^{2'}$), thieno[2,3-*b*]thiophene system: 119.46 (C^3), 120.05 (C^4), 127.64 (C^5). Found, %: C 46.53; H 4.05; S 49.40. $[M]^+$ 258. $C_{10}H_{10}S_4$. Calculated, %: C 46.51; H 3.87; S 49.61. *M* 258.

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