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Interaction of 4-oxoalkane-1,1,2,2-tetracarboxitriles with Lawesson's reagent – a new approach to the synthesis of 2,2'-disulfanediybis(1*H*-pyrroles). The synthesis of photochromic diarylethene with disulfide bridge[†]

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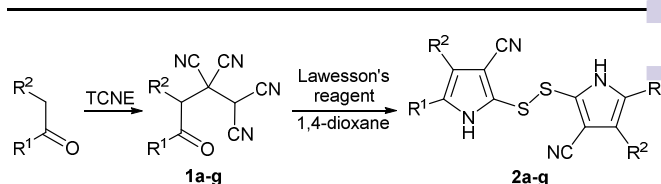
Transformation of 4-oxoalkane-1,1,2,2-tetracarboxitriles under the action of Lawesson's reagent leads to 2,2'-disulfanediybis(1*H*-pyrrole-3-carbonitriles) in good yields. The developed method allowed to synthesize photochromic 2,2'-disulfanediybis(4,5-bis(2,5-dimethylthiophen-3-yl)-1*H*-pyrrole-3-carbonitrile).

Disulfide bond (S–S) plays an important role in the living organisms. It protects bacteria as a reversible switch that turns a protein ON or OFF when bacterial cells are exposed to oxidation reactions.^{1a} The reversible formation of disulfide bonds is utilized biologically in enzyme catalytic mechanisms, in the transport of reducing equivalents, in metabolic regulation, and as a cellular defense system.^{1b–f} Many naturally occurring compounds also include such disulfide bridges.^{1g–k} The combination of pyrrole moiety and disulfide bond in one molecule has a specific importance. There are only a few methods for preparation of 2,2'-disulfanediybis(1*H*-pyrroles).² Despite of their rarity, they are useful as a pharmaceutical product, particularly an acid secretion inhibitor (proton pump inhibitor).^{2d}

Therefore, the development of convenient methods for the construction of bispyrroles with disulfide bridge is actual problem due to the potentially practical properties of synthesized products.

We have developed a new approach to the synthesis of 2,2'-disulfanediybis(1*H*-pyrroles), basing on the interaction of readily available 4-oxoalkane-1,1,2,2-tetracarboxitriles³ **1a–g** and Lawesson's reagent (LR). As a result 2,2'-disulfanediybis(1*H*-pyrrole-3-carbonitriles) **2a–g** have been synthesized in 53–74% yields (Scheme 1).

Structures of disulfides **2** are considered with IR-, ¹H NMR-spectroscopy and mass-spectrometry. The IR spectra characterized with the intensive absorption bands of conjugated C≡N in the area of 2218–2232 cm⁻¹ and bands of pyrrolic N–H bond in the area of 3169–3256 cm⁻¹. The ¹H NMR spectra characterized with the signals



Substrate	R ¹	R ²	Product	Yield [*] , %
1a	CH ₃	CH ₃	2a	53
1b	CH ₃	C ₂ H ₅	2b	57
1c		–(CH ₂) ₄ –	2c	62
1d		–(CH ₂) ₅ –	2d	67
1e		–(CH ₂) ₆ –	2e	59
1f	Ph	CH ₃	2f	67
1g	Ph	Ph	2g	74

^{*}Yield has been reported for isolated crude product

Scheme 1 Synthesis of 2,2'-disulfanediybis(1*H*-pyrrole-3-carbonitriles) **2a–g**.

of NH-protons in the area of 12.00–12.98 ppm and the signals of appropriate alkyl and aryl substituents. Because the molecules of disulfides **2** are symmetrical, the signals of equivalent protons overlap and manifest as a single in the ¹H NMR spectra. For the mass spectra the presence of the fragment ion with m/z=M⁺/2, emerging after S–S bond breaking, is usual.

For an unambiguous proof of the structure of the compounds **2** X-ray diffraction study for the single crystal of disulfide **2e** was carried out (Figure 1). It can be seen that the two-fold axis passes through the middle of the S–S bond. The symmetry code of the two symmetrical parts of the molecule is i = x, 0.5–y, 1–z; Distances and angles in the molecule are unexceptional and match those found in similar molecules. The crystal structure is stabilized by a N1–H1...N2ⁱⁱ (–x, 0.5+y, z) hydrogen bond interaction so to form infinite chain running along b – axis.

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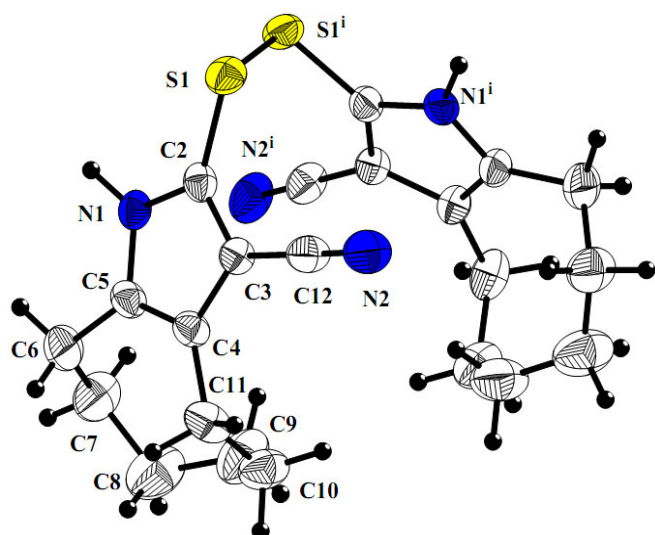
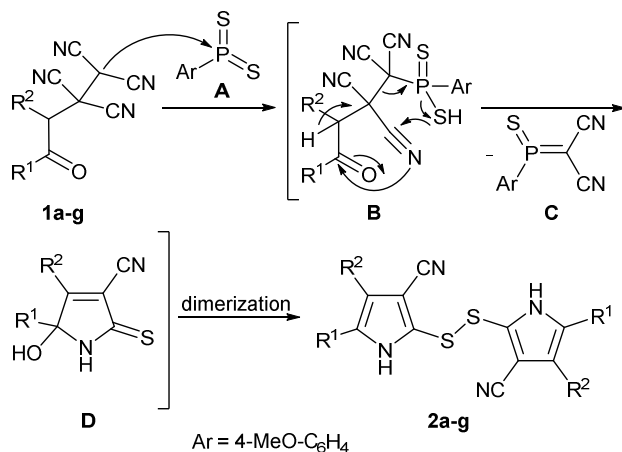


Figure 1 The molecular structure of **2e** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii.

To explain such unusual conversion of starting 4-oxoalkane-1,1,2,2-tetracarbonitriles **1** the following sequence of transformations is proposed (Scheme 2). Probably, the attaching of the nitrile **1** to the P=S double bond of methoxyphenylphosphine disulfide **A** is occurred at first stage. The latter, as described, have been formed from LR by heating.⁴ In the resulting adduct **B** the elimination process of 2-((4-methoxyphenyl)(thio)-λ⁵-phosphanylidene)malononitrile **C** is initiated by the transfer of the mobile proton from α-position of carbonyl group. It leads to the formation of pyrrole derivatives **D**. This process can be considered as an actual addition of hydrogen sulfide to the cyano group, initiated by the attaching of CH-acid **1** to the electron deficient P-atom of LR. Then pyrrole **D** likely undergoes the dimerization process leading to the final disulfide **2**. It should be noted, that similar dimerization of intermediate **D** analogs to disulfides is described in literature.^{2c}



Scheme 2 Proposed mechanism for the synthesis of 2,2'-disulfanediybis(1*H*-pyrrole-3-carbonitrile)s **2a-g**.

The developed method for the synthesis of 4,4',5,5'-tetrasubstitued 2,2'-disulfanediybis(1*H*-pyrroles) is universal, it is demonstrated by involvement the various 4-oxoalkane-1,1,2,2-

tetracarbonitriles **1** in the interaction with LR. The method allowed to prepare derivatives **2** with alkyl, cycloalkyl and aryl substituents in the pyrrole rings.

Therefore, it was decided to synthesize compound **2** with a specific potentially useful feature. As we think, the developed approach to the synthesis of disulfides **2** is promising for the creation of photochromic compounds of diarylethene (DAE) series, containing two potentially photoswitchable fragments in the structure (Figure 2).

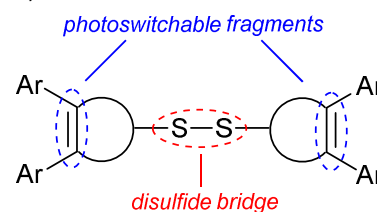


Figure 2 The general structure of a potential photochrome with S-S bond

It is known,⁵ that DAE are molecular switches with characteristic transition from the colorless form **A** into the colored form **B** under the action of UV light. The inverse transformation occurs by the visible light irradiation or thermally (figure 3).

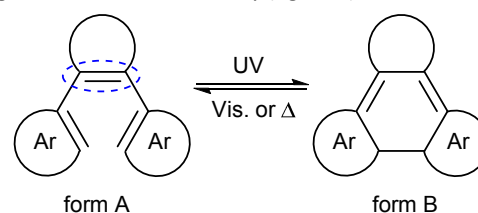


Figure 3 The general scheme for photochemical transformations of photochromic compounds of diarylethene series

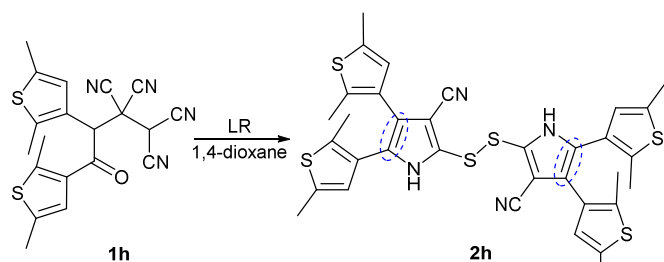
The investigations of synthesis of photochromic compounds with several photosensitive centers in the structure are very significant. It is related with applicability of such compounds in the different areas⁵, e.g. for the process management in biological systems.^{5f-i}

The presence of disulfide bridge is the key structural element allowing to create at once two diarylethene fragments in one molecule according to the developed approach.

It should be noted, that disulfide bonds were not used as bridges for covalent binding of two potentially photoswitchable fragments until this investigation.^{5b} This fact undoubtedly increases the scientific significance of the research work.

For the synthesis of photochromic compound dithienyl ketone **1h**, early described by us,^{3c} was involved in the interaction with LR. Ketone **1h** is specific, because it is substituted with two 2,5-dimethylthiophenes. This choice is caused by the fact that photochromic diarylethenes with thienyl substituents are the most promising for practical use in various fields.⁵

It was found, that 2,2'-disulfanediybis(4,5-bis(2,5-dimethylthiophen-3-yl)-1*H*-pyrrole-3-carbonitrile) **2h** in 79% yield is the result of interaction between ketone **1h** and LR (Scheme 3).



Scheme 3 Synthesis of the photochromic compound **2h**

Structure of compound **2h** is similar to disulfides **2a-g** described in the Scheme 1. It was also considered with IR-, ^1H NMR-spectroscopy and mass-spectrometry.

The resulting compound **2h** contains two potentially photoswitchable fragments (marked with dashed line on the Scheme 3).

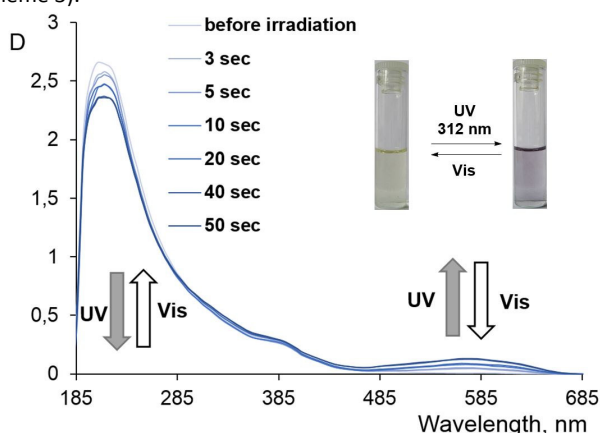


Figure 4 Photochromism of disulfide **2h** and the absorption spectrum changes for ethanol solution ($C\ 1 \cdot 10^{-4}\ \text{M}$) with subsequent irradiation with 312 nm UV light

Therefore, a preliminary studying of photochromic properties in the ethanol solution of compound **2h** was carried out. The absorption spectrum of compound **2h** before and after irradiation with 312 nm UV light is shown in Figure 4. It was found, that disulfide **2h** is photochromic, a new absorption band appeared at 570 nm after irradiation with UV light, indicating the formation of pale purple colored photoinduced form of the **2h**. The reverse transformation to the nearly colorless form was induced by the short time action of full-spectrum visible light. In addition, it was found that photochrome **2h** is thermally unstable (T-type). So bleaching of colored solution is also possible at room temperature in the darkness with $\tau_{1/2} = 129\ \text{s}$ (time required for a 50% decrease of a maximum value of optical density at the maximum of the long-wave absorption band in the dark at 298K) (for details see \dagger ESI). It should be noted, that thermally unstable photochromic diarylethenes were early described in literature for thiophene, oxazole, thiazole and imidazole ethene bridges.^{5k-n}

Considering the polyfunctionality of the resulting **2h**, namely the presence of cyano group, mobile NH-proton, easily reduced S–S bond, it could be assumed a great prospect of its applicability in the directed synthesis of photochromic diarylethenes with the pyrrolic ethene bridge.

Conclusions

Therefore, a new approach to the synthesis of 2,2'-disulfanediybis(1*H*-pyrroles) basing on the interaction between 4-oxoalkane-1,1,2,2-tetracarboxylic nitriles **1** and Lawesson's reagent was developed. Considering the availability of ketones **1** and sulfiding agent this approach is perspective for preparing pyrroles **2** with different alkyl, cycloalkyl, aryl and heterocyclic substituents. The universality of this method was shown by the involvement in the interaction various types of 4-oxoalkane-1,1,2,2-tetracarboxylic nitriles **1**, including dithienyl substituted ketone **1h** which led to the formation of disulfide **2h** with photochromic properties.

Experimental

General experimental methods

The progress of reactions and the purity of the products were monitored by TLC on Sorbfil plates (spots were visualized under UV light, by treatment with iodine vapor or by heating). The IR spectra were recorded on an FSM-1202 spectrometer with Fourier transform from samples dispersed in mineral oil. The NMR spectra were measured in DMSO- d_6 on a Bruker DRX-500 spectrometer using tetramethylsilane as an internal reference. The elemental compositions were determined on a CHN-analyzer vario Micro cube. The mass spectra (electron impact, 70 eV) were obtained on a Finnigan MAT INCOS-50 spectrometer. The UV spectra were recorded from solutions in ethanol on an SF-2000 spectrophotometer. Melting points were determined on the device M-560. Crystals of compounds **2e** suitable for X-ray analysis were grown at room temperature from the mixture of acetone and ethyl acetate (1:1). The data of **2e** were collected by using STOE diffractometer Pilatus 100K detector, focusing mirror collimation Cu K α (1.54086 Å) radiation, rotation method mode. STOE X-AREA software was used for cells refinement and data reduction. Cell parameters: $a = 9.6655(3)$, $b = 13.8908(6)$, $c = 16.1834(7)$ Å, $V = 2172.81(15)$ Å³, space group P_{bna} , $Z=4$. Absorption correction was applied. The structures were solved and refined with SHELX⁶ program. The non-hydrogen atoms were refined by using the anisotropic full matrix least-square procedure. The hydrogen atoms were located from a difference Fourier map and refined freely or were placed in the calculated positions and allowed to ride on their parent atoms [C-H 0.93-0.98, U_{iso} 1.2 U_{eq} (parent atom)]. Refinement was made against 2154 reflections. The final R 0.040 against 1692 $F^2 > 2\sigma$ (F^2) respectively. Molecular geometry calculations were performed with the SHELX program, and the molecular graphics were prepared by using DIAMOND⁷ software.

Starting 4-oxoalkane-1,1,2,2-tetracarboxylic nitriles **1a-h** were prepared according to the known method – acid-catalyzed interaction of TCNE with ketones.^{3c}

Typical procedure for the preparation of 2,2'-disulfanediybis(1*H*-pyrrole-3-carbonitriles) **2**.

To the solution of 0.1 g (0.3 mmol) of appropriate 4-oxoalkane-1,1,2,2-tetracarboxylic nitrile **1** in 1,4-dioxane (3 mL) 0.12 g (0.3 mmol) of Lawesson's reagent was added. The reaction mixture stirred at heating until the complete dissolution. After the reaction was completed (TLC), water (50 mL) was poured into reaction mixture, then extracted with ethyl acetate (5 x 2 mL). In the extract 0.5 g of the activated carbon was added, then mixture was refluxed for 5 min and filtered. The solvent was evaporated, to the residue BuOH (0.5 mL) and water (20 mL) were added, precipitated product

filtered, washed with water and mixture of *t*-BuOH : H₂O = 1 : 40 (2 x 1 mL). Dried in a vacuum desiccator over CaCl₂.

2,2'-disulfanediybis(4,5-dimethyl-1H-pyrrole-3-carbonitrile)

2a. M.p. 119-120 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3251 (NH), 2215 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 2.00 (6H, s, 2CH₃), 2.14 (6H, s, 2CH₃), 12.03 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 302 (M⁺, 6), 151 (M⁺/2, 100); Elemental analysis (%): Found: C, 55.64; H, 4.671; N, 18.50, calculated for C₁₄H₁₄N₄S₂: C, 55.60; H, 4.67; N, 18.53.

2,2'-disulfanediybis(4-ethyl-5-methyl-1H-pyrrole-3-carbonitrile) 2b. M.p. 161-162 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3256 (NH), 2218 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 1.09 (6H, t, *J*=7.5 Hz, 2CH₃), 2.16 (6H, s, 2CH₃), 2.16 (4H, q, *J*=7.5 Hz, 2CH₂), 12.02 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 330 (M⁺, 3), 165 (M⁺/2, 34); Elemental analysis (%): Found: C, 58.21; H, 5.44; N, 16.88, calculated for C₁₆H₁₈N₄S₂: C, 58.15; H, 5.49; N, 16.95.

2,2'-disulfanediybis(4,5,6,7-tetrahydro-1H-indole-3-carbonitrile) 2c. M.p. 192-193 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3229 (NH), 2224 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 1.65-1.78 (8H, m, 4CH₂), 2.41-2.45 (4H, m, 2CH₂), 2.51-2.54 (4H, m, 2CH₂), 12.00 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 354 (M⁺, 18), 177 (M⁺/2, 100); Elemental analysis (%): Found: C, 61.03; H, 5.10; N, 15.77; calculated for C₁₈H₁₈N₄S₂: C, 60.99; H, 5.12; N, 15.81.

2,2'-disulfanediybis(1,4,5,6,7,8-hexahydrocyclohepta[b]pyrrole-3-carbonitrile) 2d. M.p. 218-219 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3231 (NH), 2226 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 1.57-1.66 (8H, m, 4CH₂), 1.73-1.80 (4H, m, 2CH₂), 2.45-2.53 (4H, m, 2CH₂), 2.64-2.68 (4H, m, 2CH₂), 12.03 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 191 (M⁺/2, 100); Elemental analysis (%): Found: C, 64.39; H, 6.33; N, 13.62, calculated for C₂₀H₂₂N₄S₂: C, 64.36; H, 6.38; N, 13.65.

2,2'-disulfanediybis(4,5,6,7,8,9-hexahydro-1H-cycloocta[b]pyrrole-3-carbonitrile) 2e. M.p. 226-227 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3223 (NH), 2229 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 1.32-1.44 (8H, m, 4CH₂), 1.51-1.65 (8H, m, 4CH₂), 2.52-2.56 (4H, m, 2CH₂), 2.64-2.68 (4H, m, 2CH₂), 12.03 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 205 (M⁺/2, 100); Elemental analysis (%): Found: C, 62.84; H, 5.77; N, 14.61, calculated for C₂₂H₂₆N₄S₂: C, 62.80; H, 5.80; N, 14.65.

2,2'-disulfanediybis(4-methyl-5-phenyl-1H-pyrrole-3-carbonitrile) 2f. M.p. 236-237 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3169 (NH), 2232 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 2.25 (6H, s, 2CH₃), 7.36-7.56 (10H, m, 2C₆H₅), 12.60 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 213 (M⁺/2, 4); Elemental analysis (%): Found: C, 67.34; H, 4.22; N, 13.05, calculated for C₂₄H₁₈N₄S₂: C, 67.58; H, 4.25; N, 13.13.

2,2'-disulfanediybis(4,5-diphenyl-1H-pyrrole-3-carbonitrile) 2g. M.p. 138-140 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3197 (NH), 2226 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 7.16-7.33 (20H, m, 4C₆H₅), 12.98 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 275 (M⁺/2, 11); Elemental analysis (%): Found: C, 74.23; H, 3.99; N, 10.13, calculated for C₃₄H₂₂N₄S₂: C, 74.16; H, 4.03; N, 10.17.

2,2'-disulfanediybis(4,5-bis(2,5-dimethylthiophen-3-yl)-1H-pyrrole-3-carbonitrile) 2h. M.p. 116-117 °C (dec.); IR $\nu_{\max}/\text{cm}^{-1}$ (mineral oil): 3184 (NH), 2223 (C≡N); ¹H NMR (500.13 MHz, DMSO-d₆): δ 1.86 (6H, s, 2CH₃), 1.92 (6H, s, 2CH₃), 2.32 (6H, s, 2CH₃), 2.34 (6H, s, 2CH₃), 6.40 (2H, s, 2CH), 6.55 (2H, s, 2CH), 12.74 (2H, s, 2NH) ppm; MS, (EI, 70 eV), m/z (%): 343 (M⁺/2, 74); Elemental analysis (%): Found: C, 59.49; H, 4.35; N, 8.10, calculated for C₃₄H₃₀N₄S₆: C, 59.44; H, 4.40; N, 8.16.

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