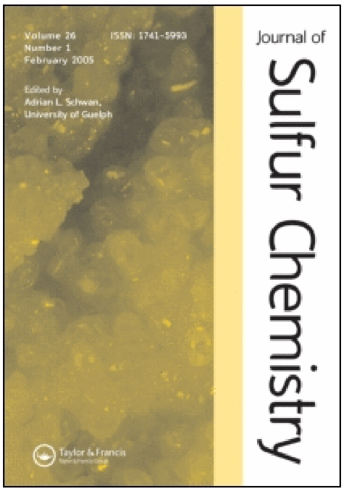

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RESEARCH ARTICLE

A novel method for the preparation of unsymmetrical bis (di- and trisulfides) via trityl sulfenyl chlorides: Precursors for cyclic polysulfides

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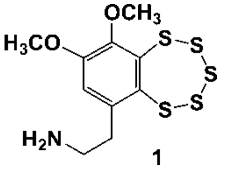
When triphenylmethanesulfenyl chloride **6** [$(C_6H_5)_3CSCl$] and its thio homologue **7** [$(C_6H_5)_3CSSCl$] were treated with dithiols, unsymmetrical bis(di- and trisulfides) **11** and **12** were produced in high yields. Final products were determined by ^1H and ^{13}C NMR as well as by elemental analysis. The X-ray crystallographic structures of *p*-di(methylenedithiotrityl)benzene **11a** and *p*-di(methylenetrithiotrityl)benzene **12a** were obtained. In addition, cyclic polysulfide **13** was produced in high isolated yield via **11c**.

Keywords: Methylenedithiotrityl; Methylenetrithiotrityl; Dimethanethiol; Methanesulfenyl chloride; Methanethiosulfenyl chloride; Cyclic polysulfides; Disulfides; Trisulfides

1. Introduction

Our interest in the chemistry of organosulfides arises from their diverse roles in living organisms and their occurrence in natural sources [1, 2].

Pentasulfides possess significant importance as they are naturally occurring compounds in a variety of plants and animals [3]. For instance, diallylpentasulfide [4] and varacin **1** [5] have antitumor, antifungal and cytotoxic properties. Lithium batteries have pentasulfides as one of its components [6]; in addition, pentasulfides are used as antifouling agents [7].



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The reaction of dithiols and elemental sulfur in the presence of an ammonia catalyst [8–13] produces benzotrithiepin **2** and benzopentathiepin **3**. The latter two occur naturally and belong

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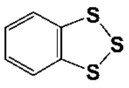
to the class of cyclic polysulfides.



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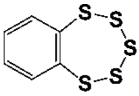
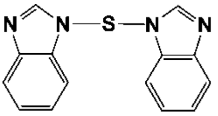


3

Other methods have been reported for the preparation of cyclic polysulfides. In 1981, Harpp and co-workers explored the reaction of dithiols by the use of N,N-dibenzimidazolyl sulfide **4** for the synthesis of cyclic trisulfide **2** [14]. The thermal decomposition of 1,2,3-benzothiadiazoles **5** with elemental sulfur [15, 16] produces pentathiepin **3**.



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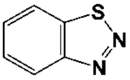

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Alkyl and aryl mono and disulfides are easily prepared and are generally stable and easily characterized compounds. The difficulty arises in the isolation, purification and characterization of the individual trisulfides as polysulfides are often obtained. Clean-cut separations might sometimes be impossible as a series of sulfides have similar properties. Therefore, several methods were developed for the synthesis of both symmetrical and unsymmetrical trisulfides [17–21].

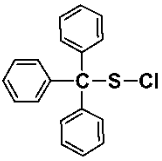
The chemistry of organic polysulfanes R-S$_x$-R (x > 2) has been reviewed by Steudel [22]. In addition, the synthesis, properties, and uses of industrial application of polysulfide polymers have been reported [23–27].

Due to the property of the trityl moiety as an excellent leaving group, we have investigated its chemistry to prepare unsymmetrical bis(di- and trisulfides) hoping that these reagents can be used to develop a useful pathway for preparing cyclic polysulfides.

2. Results and discussion

In 1994, Harpp and co-workers [28] prepared a series of $(C_6H_5)_3CS_XBu$ and $(C_6H_5)_3CS_xC(C_6H_5)_3$ ($x = 1$ to 4) polysulfides using sulfenyl chloride chemistry.

It was found [29–31] that tri-, tetra-, and pentasulfides RS$_x$R ($x = 2, 3, 4$), could be prepared easily in good to excellent yield by the reaction of disulfides with sulfenyl chloride **6** (and its thio **7** and dithio homologue **8**), respectively.



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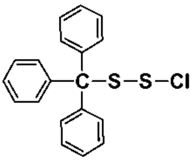

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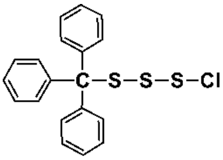

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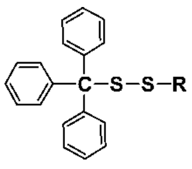
Recently, we reported [32] the reaction of sulfenyl chlorides **6** and **7** with thiols. Unsymmetrical disulfide **9** and trisulfide **10** were prepared and isolated in high yields. Trisulfide **10** showed an interesting reactivity with electrophiles to give polysulfides RS_xR ($x = 2$ to 6).



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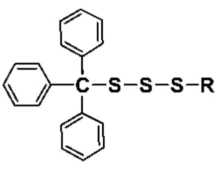

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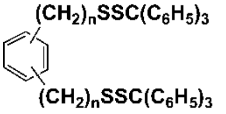
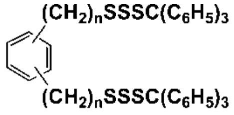
Also, we discovered that triphenylmethanesulfenyl chloride (**6**) and thiosulfenyl chloride **7** reacted with various dithiols at room temperature. Unsymmetrical bis(disulfides) **11** and bis(trisulfides) **12** were formed, respectively, in high isolated yields.



11a: *para*, n = 1
11b: *meta*, n = 1
11c: *ortho*, n= 1
11d: *ortho*, n = 0



12a: *para*, n = 1
12b: *meta*, n = 1
12c: *ortho*, n = 1
12d: *ortho*, n = 0

Although trityl polysulfides **9** and **10** were previously reported [32] to be rapidly formed from benzyl and benzenethiols, however bisthiols, especially *ortho*-substituted isomers, are shown to react slowly. Addition of a stoichiometric or larger amount of Et_3N resulted in a dramatic increase of the reaction rate where the corresponding products are formed in 10 minutes. Apparently, the removal of HCl from the reaction medium by forming ammonium salt shifted the equilibrium towards the product. Precipitation of $Et_3N \cdot HCl$ occurs, as Et_2O has no influence on the reaction rate. On the other hand it is advantageous to remove the ammonium salt since this limits the chromatographic column load. In addition, acidification of the crude product with acetic acid before purification on the column eliminates any trace of Et_3N that might be still present and could result in equilibration of the main product due to nucleophilic properties of the amine.

The yields obtained from the synthetic approach provided herein were high, up to *ca*. 93% except for the *para*-isomers where only 80% of the product was observed. This low yield is due to a low solubility of *para* derivatives and consequently a loss of product will take place during filtration.

The structures of **11** and **12** were established on the basis of their ^{1}H- and ^{13}C-NMR spectra as well as by elemental analysis.

The X-ray crystallographic structure of *p*-bis(methylenedithiotrityl)benzene (**11a**) was obtained (see section 5). The ORTEP drawing is shown in figure 1 and selected bond lengths, bond angles, and dihedral angles are shown in table 1.

The X-ray crystallographic structure of *p*-bis(methylenetrithiotrityl)benzene (**12a**) was also obtained (see section 5). The ORTEP drawing is shown in figure 2 and selected bond lengths, bond angles, and dihedral angles are shown in table 2.

Trityl polysulfides are prone to react with electrophiles (scheme 1). The trityl group plays here a crucial role due to its positive charge-stabilizing properties. The stabilization renders

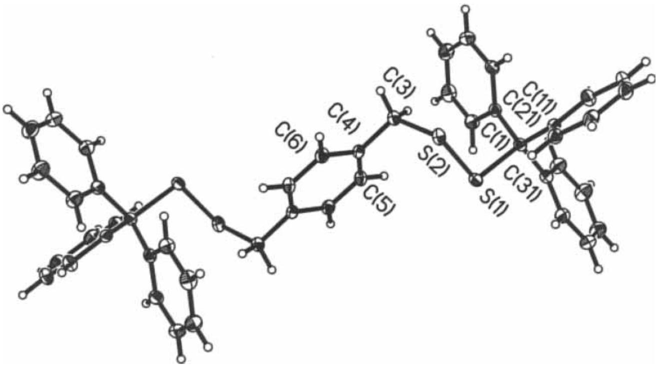


Figure 1. ORTEP Representation of *p*-bis(methylenedithiotrityl)benzene (**11a**).

Table 1. Selected bond lengths, bond angles, and dihedral angles from the X-ray structure of *p*-bis(methylenedithiotrityl)benzene (**11a**).

	Bond Lengths (Å)		
Bond		Bond	
S(1)-S(2)	2.031(6)	C(3)-C(4)	1.503(2)
S(1)-C(1)	1.883(2)	C(1)-C(11)	1.539(2)
S(2)-C(3)	1.835(2)	C(1)-C(21)	1.527(2)
	Bond Angles (deg)		
Angle		Angle	
S(1)-S(2)-C(3)	105.43(6)	S(1)-C(1)-C(21)	105.23(1)
C(1)-S(1)-S(2)	104.05(6)	S(1)-C(1)-C(31)	104.52(1)
S(1)-C(1)-C(11)	112.66(1)	S(2)-C(3)-C(4)	113.71(1)
	Dihedral Angles (deg)		
Angle		Angle	
C(1)-S(1)-S(2)-C(3)	92.88(8)	S(2)-S(1)-C(1)-C(31)	179.71(1)
S(2)-S(1)-C(1)-C(21)	−59.38(1)	S(1)-S(2)-C(3)-C(4)	75.42(1)
S(2)-S(1)-C(1)-C(11)	64.94(1)	S(2)-C(3)-C(4)-C(5)	−96.20(2)

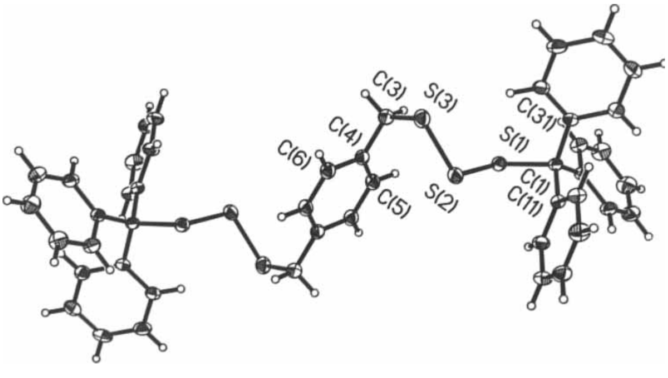


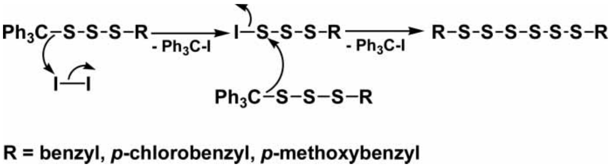
Figure 2. ORTEP Representation of *p*-bis(methylenetrithiotrityl)benzene (**12a**).

the adjacent sulfur atom nucleophilicity, resulting in a high rate of cleavage even at room temperature.

Earlier, we prepared a series of linear tetra- and hexasulfides [32]. It was suggested a parallel approach is useful in the preparation of cyclic polysulfides. It is known that the preparation of

Table 2. Selected bond lengths, bond angles, and dihedral angles from the X-ray structure of *p*-bis(methylenetrithiotrityl)benzene (**12a**).

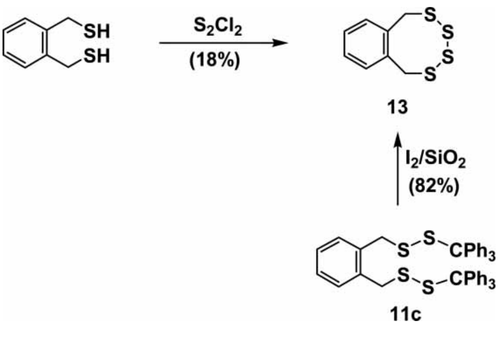
Bond Lengths (Å)			
Bond		Bond	
S(1)-S(2)	2.051(8)	C(1)-C(11)	1.530(3)
S(2)-S(3)	2.040(8)	C(1)-C(31)	1.540(2)
S(1)-C(1)	1.884(2)	C(3)-C(4)	1.491(3)
S(3)-C(3)	1.828(2)	C(4)-C(5)	1.387(3)
Bond Angles (deg)			
Angle		Angle	
S(1)-S(2)-S(3)	107.81(3)	C(11)-C(1)-S(1)	107.73(1)
C(1)-S(1)-S(2)	107.10(7)	C(21)-C(1)-S(1)	100.78(1)
C(3)-S(3)-S(2)	102.37(8)	C(31)-C(1)-S(1)	112.70(1)
C(11)-C(1)-C(31)	113.18(2)	C(4)-C(3)-S(3)	114.38(2)
Dihedral Angles (deg)			
Angle		Angle	
C(1)-S(1)-S(2)-S(3)	109.28(7)	S(2)-S(1)-C(1)-C(31)	−86.40(1)
S(1)-S(2)-S(3)-C(3)	75.47(8)	S(2)-S(3)-C(3)-C(4)	48.91(2)
S(2)-S(1)-C(1)-C(11)	39.16(1)	S(3)-C(3)-C(4)-C(5)	−109.30(2)
S(2)-S(1)-C(1)-C(21)	158.10(1)	S(3)-C(3)-C(4)-C(6)	70.90(2)



SCHEME 1

polysulfides is frequently hampered by the co-formation of their homologues with a varying number of sulfur atoms. In general, purification of polysulfides is difficult due to their similar properties resulting not only in lower yields but also inhibiting the crystallization of the main product from the oily mixture.

As there is interest in finding new clean approaches to these sulfur-rich systems, we have applied the iodine-induced coupling of trityl di- and trisulfides for the preparation of the previously reported cyclic tetrasulfide **13** [33] that was obtained in 18% from corresponding dithiol and sulfur monochloride (S_2Cl_2) as shown in scheme 2.



SCHEME 2

Application of the conditions that we have reported earlier followed by purification by column chromatography (silica gel) resulted in an isolation of a white solid with the same spectral properties as reported by Weigand [33] but in a much higher yield (82%). Compound **13** was pure and no other homologues were detected.

3. Conclusion

A simple new route for the preparation of the unsymmetrical bispolysulfides **11** and **12** is provided by the reaction of various dithiols with trityl sulfenyl chlorides **6** and **7**, respectively. A successful cyclization of *ortho*-substituted bis(methylenedithiotrityl)- benzene (**11c**) to the corresponding tetrasulfide **13** suggests that other bispolysulfides **11** and **12** also have the potential to form cyclic polysulfides. We are currently working on the scope of this reaction.

4. Experimental

Experiments do not require nitrogen atmosphere neither strictly dried solvents. Commercially available solvents were used without any treatment. Nuclear magnetic resonance spectra were recorded at 400 MHz (Varian Mercury 400) spectrometer. Deuteriochloroform ($CDCl_3$) was used as solvent and an internal standard. Dithiols were obtained from Aldrich Chemical Company (Milwaukee, Wl 53233 USA) and used directly.

4.1 *Preparation of bis trityl polysulfides*

To a stirred solution of 0.63 mmol of **6** or **7** and 0.30 mmol of the dithiol in 30 mL of anhydrous diethyl ether, 0.90 mmol (90 μL) of triethylamine was added in one portion. Immediately a white precipitate of ammonium chloride formed. Once yellow color due to sulfenyl chloride disappeared the mixture was filtered and dried. The residue was transferred to a chromatographic column with a small amount of benzene acidified with 2–3 drops of acetic acid. Elution was carried out with 0–2% of ethyl acetate in hexanes. A white solid formed upon solvent removal (ortho-derivatives crystallized even in test tubes during chromatographic separation).

4.2 *Preparation of tetrasulfide 13*

To a stirred suspension of 2 g of silica gel in a solution of 224 mg (1.76 mmol) of iodine in 7.5 mL of diethyl ether, 158 mg (0.22 mmol) of bis trityldisulfide **11c** in 12 mL of methylene chloride was added. The reaction mixture was stirred for 10 min filtered (precipitate was washed with methylene chloride) and the iodine was destroyed with excess of concentrated sodium thiosulfate aqueous solution (intense stirring). The reaction mixture was washed with water, then with a dilute solution of hydrochloric acid, dried with magnesium sulfate and filtered. Solvent was removed and the residue was transferred to the chromatographic column with a small amount of carbon disulfide acidified with a few drops of acetic acid to prevent any equilibration on the silica. Elution was carried out with hexanes; 42 mg (0.18 mmol, 82%) of white tetrasulfide **13** was isolated. ^{1}H NMR ($CDCl_3$): δ 4.184 (s, 4H), 7.287 (s, 4H) (lit.: 4.20 (s, 4H), 7.30 (s, 4H)) ppm. ^{13}C NMR ($CDCl_3$): δ 39.77 (lit. 39.68), 128.37, 130.66, 137.17 ppm.

4.3 *p-Bis(methylenedithiotrityl)benzene 11a*

Yield: 81%; mp: 158-160 °C. ^{1}H NMR (CDCl$_3$): δ 2.74 (s, 4H), 6.78–7.45 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 41.21, 71.14, 126.80, 127.74, 129.09, 130.04, 135.55, 143.60 ppm. Anal. Calc'd for $C_{46}H_{38}S_4$: C, 76.88; H, 5.29%. Found C, 76.65; H, 5.49%.

4.4 *m-Bis(methylenedithiotrityl)benzene 11b*

Yield: 92%; mp: 104-106 °C. ^{1}H NMR (CDCl$_3$): δ 2.77 (s, 4H), 6.59–7.46 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 41.53, 71.16, 126.83, 127.76, 128.00, 128.22, 129.86, 130.05, 136.66, 143.61 ppm. Anal. Calc'd for $C_{46}H_{38}S_4$: C, 76.88; H, 5.29%. Found C, 77.30; H, 5.41%.

4.5 *o-Bis(methylenedithiotrityl)benzene 11c*

Yield: 95%; mp: 74–76 °C. ^{1}H NMR (CDCl$_3$): δ 2.84 (s, 4H), 6.75–7.44 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 39.19, 71.20, 126.58, 126.86, 127.45, 129.32, 130.07, 130.68, 134.88, 143.60 ppm. Anal. Calc'd for $C_{46}H_{38}S_4$: C, 76.88; H, 5.29%. Found C, 77.07; H, 5.30%.

4.6 *o-Bis(dithiotrityl)benzene 11d*

Yield: 93%; mp: 65-67 °C. ^{1}H NMR (CDCl$_3$): δ 6.95–7.34 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 126.73, 127.05, 127.16, 129.23, 130.16, 131.10, 134.79, 143.83 ppm. Anal. Calc'd for $C_{44}H_{34}S_4$: C, 76.48; H, 4.96%. Found C, 76.80; H, 5.12%.

4.7 *p-Bis(methylenetrithiotrityl)benzene 12a*

Yield: 80%; mp: 140-142 °C. ^{1}H NMR (CDCl$_3$): δ 3.75 (s, 4H), 7.08–7.29 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 43.45, 73.20, 127.03, 127.75, 129.37, 130.23, 135.70, 143.25 ppm. Anal. Calc'd for $C_{46}H_{38}S_6$: C, 70.59; H, 4.86%. Found C, 70.15; H, 4.92%.

4.8 *m-Bis(methylenetrithiotrityl)benzene 12b*

Yield: 91%; mp: 56-58 °C. ^{1}H NMR (CDCl$_3$): δ 3.74 (s, 4H), 6.99–7.45 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 39.19, 71.20, 126.86, 127.45, 127.86, 129.32, 130.03, 130.07, 134.88, 143.60 ppm Anal. Calc'd for $C_{46}H_{38}S_6$: C, 70.59; H, 4.86%. Found C, 70.99; H, 5.10%.

4.9 *o-Bis(methylenetrithiotrityl)benzene 12c*

Yield: 90%; mp: 60-62 °C. ^{1}H NMR (CDCl$_3$): δ 3.85 (s, 4H), 7.08–7.44 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 41.01, 73.14, 126.63, 127.04, 127.79, 129.37, 130.24, 131.27, 134.68, 143.24 ppm. Anal. Calc'd for $C_{46}H_{38}S_6$: C, 70.59; H, 4.86%. Found C, 70.72; H, 4.62%.

4.10 *o-Bis(trithiotrityl)benzene 12d*

Yield: 91%; mp: 54-56 °C. ^{1}H NMR (CDCl$_3$): δ 7.05–7.24 (m, 34H) ppm. ^{13}C NMR (CDCl$_3$): δ 126.93, 127.24, 127.34, 129.41, 130.24, 131.29, 134.88, 143.94 ppm. Anal. Calc'd for $C_{44}H_{34}S_6$: C, 69.98; H, 4.54%. Found C, 70.32; H, 4.42%.

5. Supplementary information

X-ray data for **11a**: $C_{46}H_{38}S_4$, Mr = 719.00, monoclinic, C2/c, a = 23.2107(4), b = 10.2783(2), c = 15.4757 (3) Å, $\beta = 91.107(1)°$, V = 3691.29 (12) $Å^3$, Z = 4, $D_x = 1.294\,Mgm^{-3}$, Cu Kα radiation, $\lambda = 1.54178\,Å$, $\mu = 2.605\,mm^{-1}$, $\theta = 3.81–72.92°$, $wR(F^2) = 0.1039$, S = 1.005, T = 220(2) K, extinction correction=none for 3588 independent reflections and 14965 measured reflections.

X-ray data for **12a**: $C_{46}H_{38}S_6$, Mr = 783.12, triclinic, $P\bar{1}$, a = 7.5760(3), b = 8.8481 (4), c = 15.8671 (6) Å, $\alpha = 84.720(3)°$, $\beta = 83.792\ (3)°$, V = 973.75 (7) $Å^3$, Z = 1, $D_x = 1.335\,Mgm^{-3}$, Cu Kα radiation, $\lambda = 1.54178\,Å$, $\mu = 3.490\,mm^{-1}$, $\theta = 2.81–70.78°$, $wR(F^2) = 0.1376$, S = 1.098, T = 220 (2) K, extinction correction=none for 3710 independent reflections and 11735 measured reflections.

Acknowledgements

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