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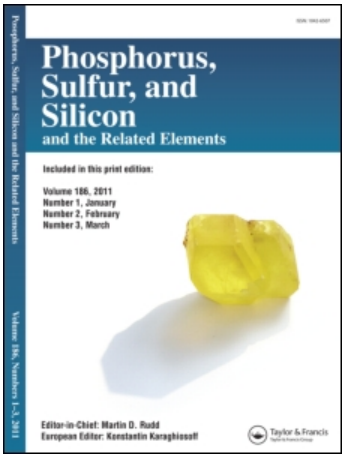

Phosphorus, Sulfur, and Silicon and the Related Elements

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N,S-Substituted Dienes from Mono(arylthio)substituted- and S-, S,S-Substituted Dienes

Cemil İbiş[a]; Zeliha Gökmen[a]; Nihal Yilmaz Bozkurt[a]
[a] Istanbul University, Avcılar-Istanbul, Turkey

Online publication date: 27 October 2010

To cite this Article İbiş, Cemil , Gökmen, Zeliha and Bozkurt, Nihal Yilmaz(2002) 'N,S-Substituted Dienes from Mono(arylthio)substituted- and S-, S,S-Substituted Dienes', Phosphorus, Sulfur, and Silicon and the Related Elements, 177: 12, 2907 — 2914

To link to this Article: DOI: 10.1080/10426500214874

URL: http://dx.doi.org/10.1080/10426500214874

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Phosphorus, Sulfur and Silicon, 2002, Vol. 177:2907–2914
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1042-6507/02 $12.00 + .00
DOI: 10.1080/10426500290146956

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N,S-SUBSTITUTED DIENES FROM MONO(ARYLTHIO)SUBSTITUTED- AND S-, S,S-SUBSTITUTED DIENES

Cemil İbiş, Zeliha Gökmen, and Nihal Yilmaz Bozkurt
Istanbul University, Avcılar-Istanbul, Turkey

(Received March 26, 2002)

*Mono(thio)substituted dienes **1a–1b** gave compounds **3a–c** and **5d–g** with piperazine and piperidine derivatives in dichloromethane. Compounds **8**, **9**, and **10** were obtained from the reactions of perchlorobutadiene (**6**) with 1,4-butanedithiol (**7**) in ethanol in the presence of sodium hydroxide. Compounds **12a–b**, **13a–b** were obtained from the reactions of perchlorobutadiene (**6**) with allylmercaptan (CH_2═CH─CH_2─SH) and mercaptoethanol (HO─CH_2─CH_2─SH).*

Keywords: Disulphid; dithiol; hexachlorobutadiene; mono- and disubstituted halodienes; mono(thio)substituted nitrodiene; N,S-thiosubstituted nitrodiene; piperazine; piperidine; thioether; thiol

The reactions of nitrodiene compounds with some thiols and amines are known.[1–8] Previously, we have obtained N,S-substituted dienes from the reaction of mono(aryl- and alkylthio) substituted dienes with some amines (primary amine, piperazine, and piperidine, etc.).[9–11]

The reactions of perchlorobutadiene with thiols in DMF, DMSO, and EtOH have been reported previously.[12–20] Thiosubstituted halodienes were obtained from the reactions of perchlorobutadiene with methane thiol in EtOH.[21]

The aim of this study was to synthesize new S-, S,S- and N,S-substituted 1,3-diene compounds and to determine their structures.

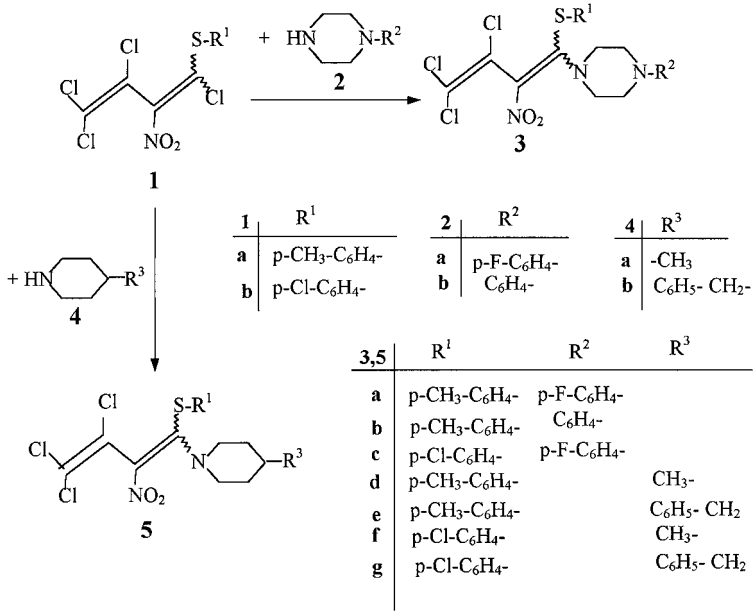
The piperazine compounds are important for therapeutical use, also some piperazine compounds were used in gen transfer reactions.[22,23] The piperidinyl derivatives show an excellent biological activity.[24]

We thank the Research Fund of the University of Istanbul for financial support of this work.

Address correspondence to Cemil İbiş, Istanbul University, Engineering Faculty, Department of Chemistry, Avcilar-Istanbul, Turkey.

The occurence of sulphur as a donor atom for transition metals is a well-known phenomenon. It acts as a very good ligating atom when in the form of the sulfide ion (S^{2-}) or as a mercaptide ion (RS^{-}), but complexes of sulphur as a thioether (RSR) are much less abundant.[25]

Compounds **1a**[26] and **1b**[27] gave new compounds **3a–c** with piperazine derivatives. Compounds **5d**, **5e**, **5f**, and **5g** were obtained from the reactions of compounds **1a** and **1b** with piperidine derivatives (Scheme 1). The obtained products are yellow and stable crystals.



3,5	R^1	R^2	R^3
a	p-CH_3-C_6H_4-	p-F-C_6H_4-	
b	p-CH_3-C_6H_4-	C_6H_4-	
c	p-Cl-C_6H_4-	p-F-C_6H_4-	
d	p-CH_3-C_6H_4-		CH_3-
e	p-CH_3-C_6H_4-		C_6H_5- CH_2
f	p-Cl-C_6H_4-		CH_3-
g	p-Cl-C_6H_4-		C_6H_5- CH_2

SCHEME 1

The novel compounds **8**, **9**, and **10** were obtained from the reaction of perchlorobutadiene with 1,4-butanedithiol. Compound **8** is a thioether, compound **9** is a disulphids, compound **10** is an interesting compound with a cyclic structure. The ^{1}H-NMR spectrum of compound **10** shows multiplet signals at $\delta = 1.6$–2.2 and 2.4–3.4 ppm for the protons of the CH_2 groups.

Perchlorobutadiene (**6**) gave new unknown **12a–b** and **13a–b** compounds with allylthiol and 2-mercaptoethanol in EtOH in the presence of NaOH (Scheme 2).

The ^{1}H-NMR spectra of **12a–b** and **13a–b** showed the expected signals. These products are viscouse oil and stable compounds. The structures of S-, S,S-, and N,S-substituted diene compounds were determined by microanalysis and spectroscopic data.

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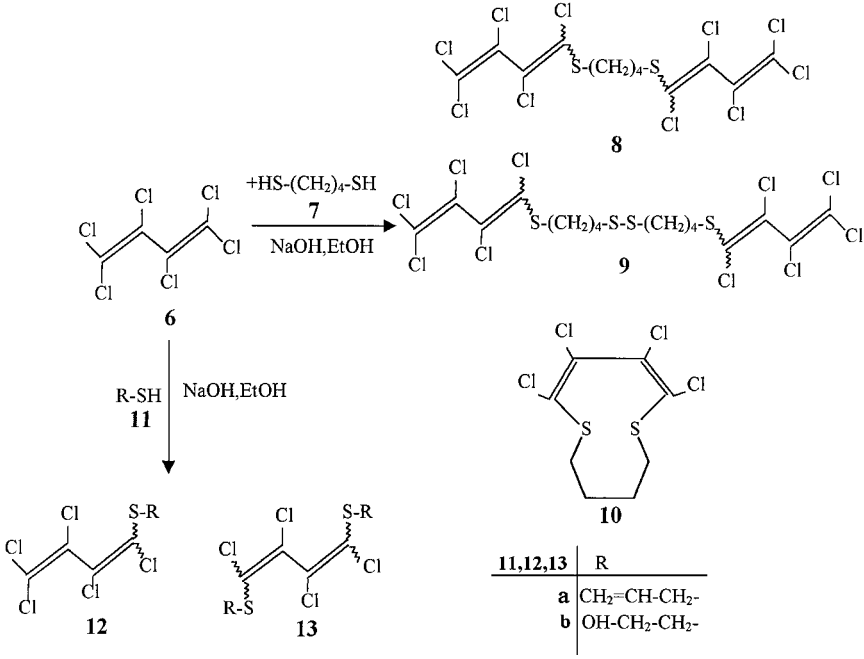

SCHEME 2

EXPERIMENTAL SECTION

- ^{1}H-NMR: Bruker AC 200 L.
- IR: Shimadzu FTIR-8101.
- Microanalyses: Carlo-Erba 1106 Elemental Analyser.
- UV: HP 8453.
- Melting Points: Büchi SMP 20.
- Products were isolated by column chromatography on silica gel (Fluka Kieselgel 60, particle size 63–200 μm).
- TLC plates silica 60 F_{254} (Merck, Darmstadt), detection with ultraviolet light (254 nm).

Preparation of S-Substituted Polyhalonitrodienes

General Procedure I

Equimolar amounts of 1,1,3,4,4-pentachloro-2-nitro-1,3-butadiene in 10 ml of ethanol and thiols in 10 ml ethanol were mixed and NaOH (in 8 ml of water) was added at room temperature. The mixture was stirred for 24 h. Chloroform was added to the reaction mixture. The organic layer was separated and washed with water (4 × 30 ml), and dried with

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$MgSO_4$. The solvent was evaporated and the residue was purified by column chromatography on silica gel.

Preparation of N,S-Substituted Polyhalonitrodienes

General Procedure II

Equimolar amounts of S-substituted polyhalonitrodienes and piperazine or piperidine derivatives were stirred in dichloromethane until completion of the reaction (TLC). Chloroform was added to the reaction mixture. The organic layer was separated and washed with water (4×30 ml), and dried with $MgSO_4$. The solvent was evaporated and the residue was purified by column chromatography on silica gel.

*1,3,4,4-Tetrachloro-mono(p-methylphenylthio)-2-nitro-1,3-butadiene (**1a**).* Compound **1a** was synthesized from 1,1,3,4,4-pentachloro-2-nitro-1,3-butadiene (2 g, 7.37 mmol) and p-methylphenylthiol (0.92 g, 7.37 mmol) according to the general procedure I. The mixture was purified by column chromatography with carbon tetrachloride as eluent.

1a: Yield: 2.4 g (90%); m.p. 112–113°C. UV (chloroform): $\lambda_{max} = 234$, 344 nm.

*1,3,4,4-Tetrachloro-mono(p-chlorophenylthio)-2-nitro-1,3-butadiene (**1b**).* Compound **1b** was synthesized from 1,1,3,4,4-pentachloro-2-nitro-1,3-butadiene (2 g, 7.37 mmol) and p-chlorophenylthiol (1 g, 7.37 mmol) according to the general procedure I. The mixture was purified by column chromatography with carbon tetrachlorid as eluent.

1b: Yield: 2.2 g (79%); m.p. 113–115°C. UV (chloroform): $\lambda_{max} = 245$, 339 nm.

*3,4,4-Trichloro-1-(4-fluorophenylpiperazine)-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (**3a**).* Compound **3a** was synthesized from 1,3,4,4-tetrachloro-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (0.119 g, 0.33 mmol) and 4-(p-fluorophenyl)piperazine (0.059 g, 0.33 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

3a: Yield: 0.12 g (73%); m.p. 156–157°C. $R_f = 0.6923$ ($CHCl_3$/Petroleumether 2:1). IR (KBr): $\nu = 2980, 3010\ cm^{-1}$ (C—H), 1600 (C=C), 1290, 1520 (C—NO_2). ^1H NMR ($CDCl_3$): $\delta = 6.9$–7.5 ppm (m, 8H, 8 Ar—H), 2.8–3.1, 3.6–3.9 (m, 8H, $4CH_2$), 2.4 (m, 3H, CH_3). UV (chloroform): $\lambda_{max} = 230$, 388 nm. $C_{21}H_{19}Cl_3N_3O_2SF$ (502.825): calcd. C, 50.16; H, 3.80; N, 8.35; found C, 50.50; H, 3.63; N, 8.23. MS: 503.

*3,4,4-Trichloro-1-(p-methylphenylthio)-1-(N-phenylpiperazine)-2-nitro-1,3-butadiene (**3b**).* Compound **3b** was synthesized from 1,3,4,4-tetrachloro-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (0.12 g,

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0.33 mmol) and N-phenylpiperazine (0.054 g, 0.33 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

3b: Yield: 0.123 g (76%); m.p. 178–179°C. R_f = 0.3947 ($CHCl_3$/Petroleumether 2:1). IR (KBr): $\nu = 2970, 3000\ cm^{-1}$ (C—H), 1600 (C=C), 1230, 1510 (C—NO_2). ^1H NMR ($CDCl_3$): $\delta = 6.8–7.4$ ppm (m, 9H, Ar—H), 2.6–3.1, 3.4–4.0 (m, 8H, $4CH_2$), 2.4 (m, 3H, CH_3). UV (chloroform): λ_{max} = 233, 387 nm. $C_{21}H_{20}Cl_3N_3O_2S$ (484.835): calcd. C, 52.02; H, 4.15; N, 8.66; found C, 52.07; H, 3.99; N, 8.51.

*3,4,4-Trichloro-1-(p-chlorophenylthio)-1-(4-fluorophenylpiperazine)-2-nitro-1,3-butadiene (**3c**).* Compound **3c** was synthesized from 1,3,4,4-tetrachloro-1-(p-chlorophenylthio)-2-nitro-1,3-butadiene (0.1 g, 0.26 mmol) and 4-(p-fluorophenyl)piperazine (0.047 g, 0.26 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

3c: Yield: 0.095 g (70%); m.p. 179–180°C. R_f = 0.6428 ($CHCl_3$/Petroleumether 2:1). IR (KBr): $\nu = 2980, 3010\ cm^{-1}$ (C—H), 1580 (C=C), 1280, 1500 (C—NO_2). ^1H NMR ($CDCl_3$): δ = 6.7–7.5 ppm (m, 8H, 8 Ar—H), 2.8–3.2, 3.4–3.95 (m, 8H, $4CH_2$). UV (chloroform): $\lambda_{max} = 251$, 385 nm. $C_{20}H_{16}Cl_4N_3O_2SF$ (523.294): calcd. C, 45.91; H, 3.08; N, 8.03; found C, 45.92; H, 3.02; N, 7.93.

*3,4,4-Trichloro-1-(p-methylphenylthio)-1-(4-methylpiperidino)-2-nitro-1,3-butadiene (**5d**).* Compound **5d** was synthesized from 1,3,4,4-tetrachloro-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (0.2 g, 0.55 mmol) and 4-methylpiperidine (0.055 g, 0.55 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

5d: Yield: 0.212 g (92%); m.p. 149–150°C. R_f = 0.4375 ($CHCl_3$/Petroleumether 1:1). IR (KBr): $\nu = 2890, 2900\ cm^{-1}$ (C—H), 1600 (C=C), 1290, 1550 (C—NO_2). ^1H NMR ($CDCl_3$): $\delta = 7.2–7.5$ ppm (m, 4H, Ar—H), 3.5–4.1 (m, H, CH), 3.4–3.1 (m, 4H, $2CH_2$), 1.4–1.8 (m, 4H, $2CH_2$), 2.4 (m, 3H, S—CH_3), 0.5–0.9 (m, 3H, CH_3). UV (chloroform): λ_{max} = 240, 390 nm. $C_{17}H_{19}Cl_3N_2O_2S$ (421.776): calcd. C, 48.41; H, 4.54; N, 6.64; S 7.60; found C, 48.13; H, 4.20; N, 6.39; S, 7.50.

*3,4,4-Trichloro-1-(4-benzylpiperidino)-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (**5e**).* Compound **5e** was synthesized from 1,3,4,4-tetrachloro-1-(p-methylphenylthio)-2-nitro-1,3-butadiene (0.2 g, 0.55 mmol) and 4-benzylpiperidine (0.097 g, 0.55 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

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5e: Yield: 0.145 g (52%); m.p. 122–123°C. R_f = 0.6310 ($CHCl_3$/Petroleumether 1:1). IR (KBr): $\nu = 2970, 3000\ cm^{-1}$ (C—H), 1525 (C=C), 1240, 1510 (C—NO_2). ^1H NMR ($CDCl_3$): $\delta = 6.9–7.5$ ppm (m, 9H, Ar—H), 3.2–4.1 (m, H, CH), 3.0–3.3 (m, 4H, $2CH_2$), 1.4–1.9 (m, 4H, $2CH_2$), 2.2–2.3 (m, 2H, CH_2), 2.4 (m, 3H, S—CH_3). UV (chloroform): $\lambda_{max} = 231, 390$ nm. $C_{23}H_{23}Cl_3N_2O_2S$ (497.844): calcd. C, 55.49; H, 4.65; N, 5.62; S 6.44; found C, 55.20; H, 4.35; N, 5.37; S, 6.13.

*3,4,4-Trichloro-1-(p-chlorophenylthio)-1-(4-methylpiperidino)-2-nitro-1,3-butadiene (**5f**).* Compound **5f** was synthesized from 1,3,4,4-tetrachloro-1-(p-chlorophenylthio)-2-nitro-1,3-butadiene (0.2 g, 0.52 mmol) and 4-methylpiperidine (0.052 g, 0.55 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

5f: Yield: 0.153 g (66%); m.p. 172–173°C. R_f = 0.7330 ($CHCl_3$/Petroleumether 1:1). IR (KBr): ν = 2970, 2985 cm^{-1} (C—H), 1580, 1600 (C=C), 1290, 1545 (C—NO_2). ^1H NMR ($CDCl_3$): δ = 7.3–7.5 ppm (m, 4H, Ar—H), 3.5–4.1 (m, H, CH), 3.1–3.4 (m, 4H, $2CH_2$), 1.5–1.8 (m, 4H, $2CH_2$), 0.6–1.1 (m, 3H, CH_3). UV (chloroform): $\lambda_{max} = 253, 389$ nm. $C_{16}H_{16}Cl_4N_2O_2S$ (442.194): calcd. C, 43.40; H, 3.65; N, 6.33; S 7.25; found C, 43.09; H, 3.32; N, 6.22; S, 6.91.

*3,4,4-Trichloro-1-(4-benzylpiperidino)-1-(p-chlorophenylthio)-2-nitro-1,3-butadiene (**5g**).* Compound **5g** was synthesized from 1,3,4,4-tetrachloro-1-(p-chlorophenylthio)-2-nitro-1,3-butadiene (0.2 g, 0.52 mmol) and 4-benzylpiperidine (0.092 g, 0.52 mmol) according to the general procedure II. The mixture was purified by crystallization in methanol.

5g: Yield: 0.147 g (54%); m.p. 129–130°C. R_f = 0.40 ($CHCl_3$/Petroleumether 1:1). IR (KBr): $\nu = 2980, 3000\ cm^{-1}$ (C—H), 1585 (C=C), 1290, 1520 (C—NO_2). ^1H NMR ($CDCl_3$): $\delta = 6.9–7.5$ ppm (m, 9H, Ar—H), 3.4–4.2 (m, H, CH), 3.0–3.3 (m, 4H, $2CH_2$), 1.4–2.0 (m, 4H, $2CH_2$), 2.3–2.5 (m, 2H, CH_2). UV (chloroform): λ_{max} = 250, 389 nm. $C_{22}H_{20}Cl_4N_2O_2S$ (513.292): calcd. C, 50.98; H, 3.88; N, 5.40; S 6.18; found C, 50.68; H, 3.51; N, 5.10; S, 5.92.

*1,2,3,4,4-Pentachloro-1-(2-hydroxyethylthio)-1,3-butadiene (**12b**).* Compound **12b** was synthesized from 1,1,2,3,4,4-hexachloro-1,3-butadiene (4 g, 15.32 mmol) and 2-mercaptoethanol (2.394 g, 30.64 mmol) according to the general procedure I. The mixture was purified by column chromatography with petroleumether and chloroform as eluent.

12b: Yield: 1.8 g (76%); oil. $R_f = 0.48$ ($CHCl_3$). IR (film): ν = 2970, 2990 cm^{-1} (C—H), 1550, 1600 (C=C), 3480 (OH). ^1H NMR ($CDCl_3$):

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$\delta = 3.6–3.9$ ppm (m, 2H, OH—CH_2), 2.6–3.1 (m, 2H, S—CH_2). $C_6H_5Cl_5SO$ (302.435): calcd. C, 23.82; H, 1.66; found C, 24.75; H, 1.77. MS: 302.

*1,4-Bis(2-hydroxyethylthio)-1,2,3,4-tetrachloro-1,3-butadiene (**13b**).* Compound **13b** was synthesized from 1,1,2,3,4,4-hexachloro-1,3-butadiene (4 g, 15.32 mmol) and 2-mercaptoethanol (7.18 g, 91.89 mmol) according to the general procedure I. The mixture was purified by column chromatography with ethylacetate and petroleumether as eluent.

13b: Yield: 1.373 g (26%); oil. R_f = 0.3333 (ethylacetate/petroleumether 1:1). IR (film): $\nu = 2970$, 2985 cm^{-1} (C—H), 1530, 1620 (C=C), 3490 (OH). $C_8H_{10}Cl_4S_2O_2$ (344.103): calcd. C, 27.92; H, 2.92; found C, 27.82; H, 3.01.

*1-Mono-(allylthio)-1,2,3,4,4-pentachloro-1,3-butadiene (**12a**), 1,4-bis-(allylthio)-1,2,3,4-tetrachloro-1,3-butadiene (**13a**).* Compounds **12a** and **13a** were synthesized from 1,1,2,3,4,4-hexachloro-1,3-butadiene (2 g, 7.66 mmol) and allylmercaptan (0.57 g, 7.66 mmol) according to the general procedure I. The mixture was purified by column chromatography with petroleumether as eluent. Column chromatography gave the compounds **12a**, **13a** as oils.

12a: Yield: 1.5 g (68%); oil. $R_f = 0.7142$ (petroleumether). IR (film): ν 2900, 3000 cm^{-1} (C—H), 1550 (C=C), 655 (C—Cl). ^1H NMR ($CDCl_3$): $\delta = 5.7–6.1$ ppm (m, H, CH=), 5.0–5.5 (m, 2H, CH_2=), 3.3–4.9 (m, 2H, CH_2). $C_7H_5Cl_5S$ (298.447): calcd. C, 28.17; H, 1.68; S, 10.74; found C, 28.36; H, 1.39; S, 11.02.

13a: Yield: 0.5 g (20%); oil. $R_f = 0.2381$ (petroleumether). IR (film): $\nu = 2900$, 3020 cm^{-1} (C—H), 1550, 1610 (C=C), 650 (C—Cl). ^1H NMR ($CDCl_3$): $\delta = 5.6–6.1$ ppm (m, 2H, 2CH=), 4.9–5.4 (m, 4H, $2CH_2$=), 2.4–3.6 (m, 4H, $2CH_2$). $C_{10}H_{10}Cl_4S_2$ (336.132): calcd. C, 35.73; H, 2.99; S, 19.07; found C, 36.04; H, 2.99; S, 19.41.

*4,4′-Butanedithio-bis(1,1,2,3,4-pentachloro-1,3-butadiene) (**8**), 1,6,7,12-tetrathio-bis-(1,2,3,4,4-pentachloro-1,3-butadienyl)-icosan (**9**), 2,3,4,5-tetrachloro-1,6-dithia-cyclodeca-2,4-dien (**10**).* Compounds **8**, **9**, and **10** were synthesized from 1,1,2,3,4,4-hexachloro-1,3-butadiene (2 g, 7.66 mmol) and butandithiol (0.935 g, 7.66 mmol) according to the general procedure I. The mixtures were purified by column chromatography with petroleumether as eluent. Column chromatography gave the compounds **8**, **9**, and **10**.

8: Yield: 0.6 g (14%); m.p: 77–78°C. $R_f = 0.7659$ (petroleumether). IR (film): $\nu = 2850$, 2950 cm^{-1} (C—H), 1550, 1600 (C=C). ^1H NMR ($CDCl_3$): $\delta = 1.6–2.0$ ppm (m, 4H, CH_2—CH_2), 2.8–3.2 (m, 4H, $2CH_2S$). $C_{12}H_8Cl_{10}S_2$ (570.856): calcd. C, 25.25; H, 1.41; S, 11.23; found C, 25.22; H, 1.19; S, 10.98.

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9: Yield: 0.01 g (1%); oil. $R_f = 0.5500$ (petroleumether). IR (film): $\nu = 2860, 2980, 3000\ cm^{-1}$ (C—H), 1550, 1600 (C=C). ^1H NMR ($CDCl_3$): $\delta = 1.6–2.2$ ppm (m, 8H, $4CH_2$), 2.8–3.2 (m, 8H, $4CH_2S$). $C_{16}H_{16}Cl_{10}S_4$ (691.092): calcd. C, 27.80; H, 2.33; S, 18.56; found C, 27.53; H,1.99; S, 18.37.

10: Yield: 0.05 g (2%); oil. $R_f = 0.4500$ (petroleumether). IR (film): $\nu = 2930, 2860\ cm^{-1}$ (C—H), 1550, 1600 (C=C). ^1H NMR ($CDCl_3$): $\delta =$ 1.6–2.2 ppm (m, 4H, $2CH_2$), 2.4–3.4 (m, 4H, $2CH_2S$). $C_8H_8Cl_{14}S_2$ (310.093): calcd. C, 3099; H, 2.60; S, 20.68; found C, 30.68; H, 2.40; S, 20.41.

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