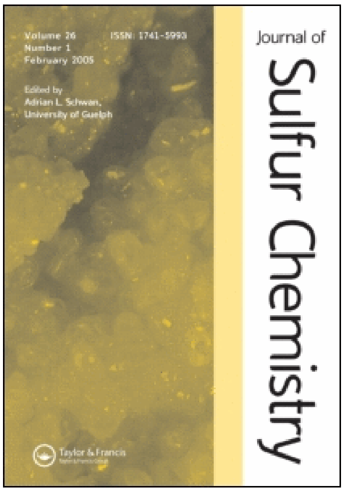

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Synthesis, Reactions, and Selected Physico-Chemical Properties of 1,3- and 1,2-Tetrachalcogenafulvalenes

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SYNTHESIS, REACTIONS, AND SELECTED PHYSICO-CHEMICAL PROPERTIES OF 1,3- AND 1,2-TETRACHALCOGENAFULVALENES

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(Received August 21, 1995)

This review describes the synthesis of 1,3- and 1,2-tetrachalcogenafulvalenes, the chemical transformation of substituents, reactions of the ring systems and the synthesis of polymers with 1,3-tetrathiafulvalene units in the backbone and in the side chain. For selected charge-transfer complexes of tetrachalcogenafulvalenes the relation between molecular structure and solid-state structure, respectively, and electric conductivity are discussed. Superconducting properties of some charge-transfer complexes with tetrachalcogenafulvalenes as donor are reported.

Preparations of important starting compounds for the synthesis of tetrachalcogenafulvalenes (like 1,2- and 1,3-dithiole, 1,3-thiaselenole, and 1,3-diselenole derivatives) have been considered in this review.

Selected properties (melting point, UV-VIS absorption data and oxidation potentials) of 1,2- and 1,3-tetrachalcogenafulvalenes are summarized in appropriate tables.

Key words: Tetrachalcogenafulvalenes, synthesis, TTF, polymeric TTF.

CONTENTS

INTRODUCTION

Tetrachalcogenafulvalenes are electron donors which form charge-transfer complexes with electron acceptors like tetracyanoquinodimethane (TCNQ), or halogens. They can also easily be oxidized electrochemically to form radical salts. Among them those are of special interest which possess a segregated stack structure. They are so-called organic metals.

Systematic investigation of the relation between the structure and the properties of tetrachalcogenafulvalene complexes, as well as the discovery of radical salts with superconductivity and an increase of their critical temperature has strongly stimulated efforts to find efficient preparations for tetrachalcogenafulvalenes and also for the corresponding starting materials. Especially heteroatoms have been introduced at peripheral sites in the donors, and conjugated tetrachalcogenafulvalenes with extended π-systems have been prepared. It is known that a strong inter-stack as well as intra-stack interaction in CT complexes leads to an increased dimensionality and inhibits Peierls transitions at low temperatures. Therefore many efforts have been made to synthesize donors for organic superconductors with heteroatoms in the outer sphere of the tetrachalcogenafulvalenes framework.

Interest in the preparation of vinylogous derivatives of tetrachalcogenafulvalenes with conjugation between dichalcogenole rings is founded on the fact that increased separation of the dichalcogenole rings decreases the Coulomb repulsion in the dicationic state and improves the electrical transport properties of organic conductors.

These relations between structures of donors and properties of conducting CT complexes as well as other aspects have been the object of international conferences,[1-16] reviews,[17-33] and books.[34-37]

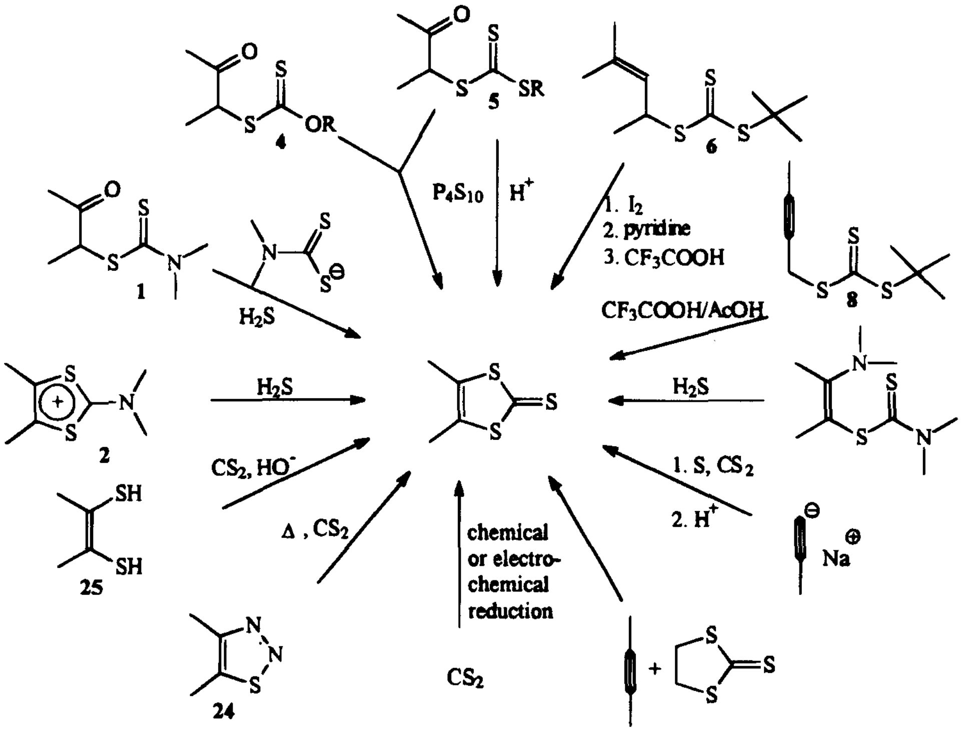
In this review a survey of methods for the preparation of tetrachalcogenafulvalenes and their properties is given.

1. SYNTHESIS OF TETRATHIAFULVALENES (TTF)

1.1. *1,3-Dithiol-2-ones, -thiones and -selones as Starting Materials for TTF*

Some methods for the synthesis of TTF have been developed. The most important methods start with 1,3-dithiole derivatives which are converted into TTF in different ways. Therefore the synthesis of 1,3-dithiole-2-thiones, the precursors of 1,3-dithiole derivatives, is important. Possibilities for their synthesis are summarized in Scheme 1.

With a large variety of substituents the cyclization of β-keto *N,N*-dialkyldithiocarbamates **1** with concentrated sulfuric acid or 70% perchloric acid and subsequent treatment of the so formed 2-amino-1,3-dithiolium salts **2** with hydrogen sulfide in pyridine or methanol gives high yields.[38-40]



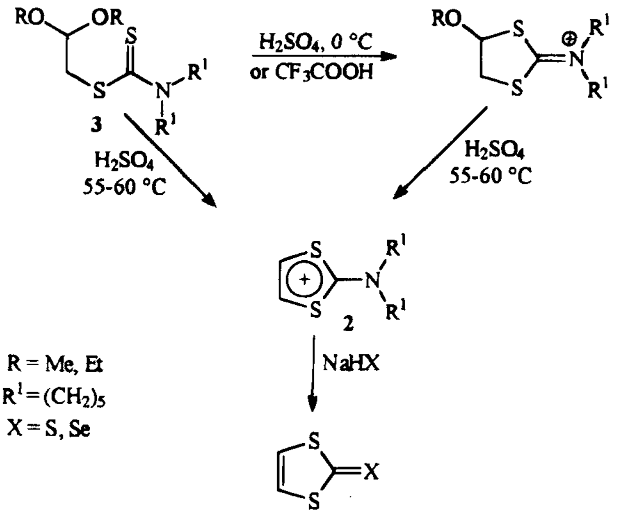
SCHEME 1

The corresponding β-keto *N,N*-dialkyldithiocarbamate **1** is cyclized directly to e.g. 4,5-tetramethylene-1,3-dithiole-2-thione with hydrogen sulfide in methanol in the presence of dimethylammonium *N,N*-dimethyldithiocarbamate as catalyst in satisfactory yield (66%), too.[41]

Cyclization of β-keto *N,N*-dialkyldithiocarbamates **1** with concentrated sulfuric acid to 2-(*N,N*-dialkylamino)- 1,3-dithiolium salts is also successful in the case of β-formyl-*N,N*-dialkyldithiocarbamates **3**.[42] In the acetalized form cyclization in sulfuric acid at 55–60 °C affords the 2-(*N,N*-dialkylamino)- 1,3-dithiolium salts **2**, which can be converted to the 1,3-dithiole-2-thione or 1,3-dithiole-2-selone by treatment with sodium hydrogen sulfide or sodium hydrogen selenide, respectively. If the cyclization is accomplished at 0 ° or with refluxing trifluoroacetic acid, only intermediate alkoxy substituted dithiolium salts are formed. These compounds provide the desired 2-(*N*-*N*-dialkylamino)- 1,3-dithiolium salts upon treatment with sulfuric acid at 55–60 °C.

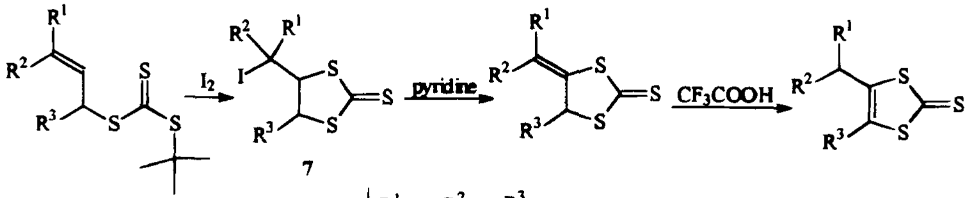
The cyclization of β-keto *O*-alkyldithiocarbonates **4** or β-keto *S*-alkyl trithiocarbonates **5** succeeds with phosphorus pentasulfide in boiling decaline.[43–46] In the case of β-keto *S*-*t*-butyl trithiocarbonates **5** the cyclization takes place in higher yields with a mixture of trifluoroacetic acid and acetic acid.[47]

The readily available allyl halides can be easily converted with high yields into variously substituted 1,3-dithiole-2-thiones with sodium *t*-butyl trithiocarbonate in a multistep reaction. The corresponding allyl *t*-butyl trithiocarbonate **6** is cyclized with iodine with con-



SCHEME 2

comitant loss of isobutylene and hydrogen iodide. In the next steps elimination of hydrogen iodide with pyridine from the 1,3-dithiolane-2-thione **7** and isomerization of the double bond with trifluoroacetic acid takes place.[48]



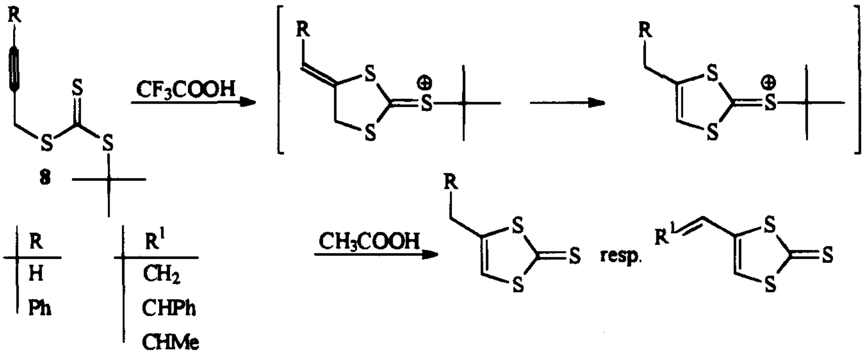
R^1	R^2	R^3
H	H	H
Me	Me	H
Ph	H	H

SCHEME 3

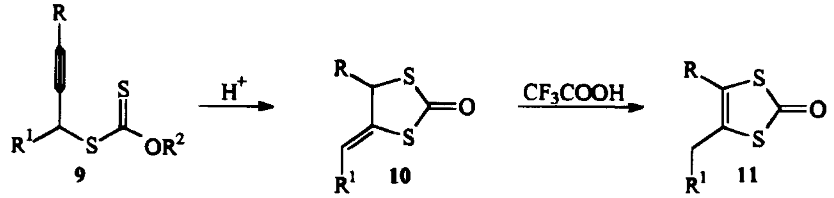
In a similar manner, 1,3-dithiole-2-thiones are formed in high yield in a one-step reaction by ring closure of propargyl *t*-butyl trithiocarbonate **8** with trifluoroacetic acid in glacial acetic acid. When acetylenic alcohols (R = R'CHOH) are employed in the reaction sequence dehydration occurs, resulting in the formation of vinyl-1,3-dithiole-2-thiones.[49]

Heating of propargyl xanthates **9** in the presence of a carboxylic acid in chlorobenzene yields the corresponding 1,3-dithiol-2-ones **10** and/or **11**. The 1,3-dithiol-2-ones **10** can be easily isomerized to **11** with hot trifluoroacetic acid.[50]

Enamines can be substituted in the 2-position by tetramethylthiuram disulfide in the presence of triethylamine and the products cyclized by passing hydrogen sulfide through the



SCHEME 4



R = H, Ph, CH_2=CH—CH(Me), 1-cyclohexenyl
R^1 = H, Me, n-C_5H_{11}
R^2 = Me, Et

SCHEME 5

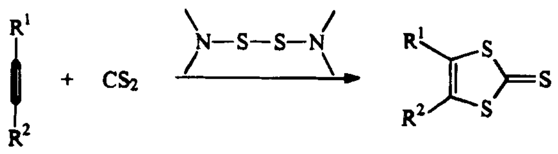
reaction mixture. Enamines of cyclic or aryl substituted ketones give 1,3-dithiole-2-thiones in good yields (35–90%). Enamines of aliphatic aldehydes do not react. With ketimines of acyclic dialkyl ketones the reaction occurs. However, the yields of 1,3-dithiole-2-thiones are moderate (10–35%).[41]

The reaction between alkynes and carbon disulfide in the presence of bisamino disulfides in a Parr bomb reactor at 140 °C provides the corresponding 1,3-dithiole-2-thiones.[51] The unsubstituted 1,3-dithiole-2-thione is formed in 25% yield in a one-step synthesis upon treatment of sodium acetylide with sulfur and carbon disulfide.[52]

Higher yields of unsubstituted 1,3-dithiole-2-thione are obtained in the reaction of dimethyl acetylenedicarboxylate with 1,3-dithiolane-2-thione, followed by hydrolysis and decarboxylation of the 4,5-bis(carbomethoxy)-1,3-dithiolane-2-thione formed.[53] The reaction of alkynes with 1,3-dithiolane-2-thione proceeds only in the case of alkynes with electron-withdrawing substituents (COOR, CF_3, CN, CHO).[54,55]

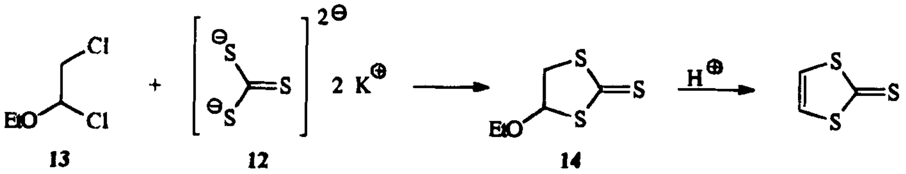
A good yield of unsubstituted 1,3-dithiole-2-thione is obtained in a two-step synthesis, starting from potassium trithiocarbonate **12** and 1,2-dichloroethyl ethyl ether **13**. The 4-ethoxy-1,3-dithiolane-2-thione **14** formed is converted into the 1,3-dithiole-2-thione by elimination of ethanol with *p*-toluenesulfonic acid.[56]

The electrochemical reduction of carbon disulfide on a mercury or platinum electrode in *N,N*-dimethylformamide provides the 4,5-dimercapto-1,3-dithiole-2-thione dianion **15**,



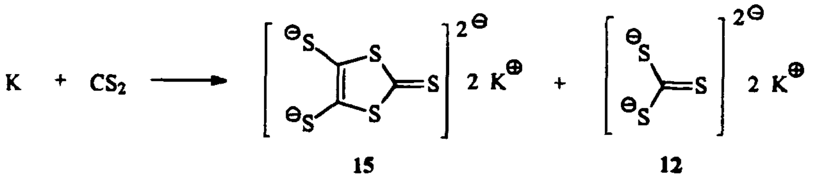
R^1	Ph	*t*-Bu	Ph	H	COOMe
R^2	H	H	COOEt	COOMe	COOMe

SCHEME 6



SCHEME 7

which with alkylating agents can form 4,5-bis(alkylthio)-1,3-dithiole-2-thiones.[57–59] On a preparative scale it is better to reduce carbon disulfide with chemical reducing agents. Reduction with potassium in *N,N*-dimethylformamide gives the highest yields.[60]

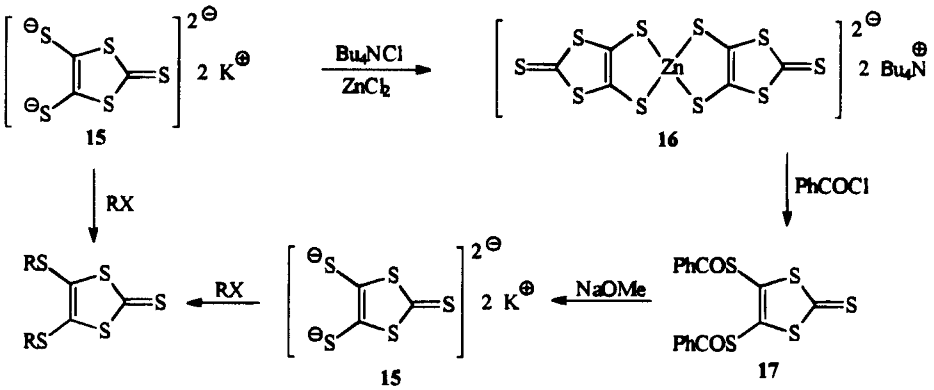


SCHEME 8

In the electrochemical as well as in the chemical reduction appreciable amounts of trithiocarbonate **12** are formed. The 4,5-dimercapto-1,3-dithiole-2-thione dianion **15** can be separated from the trithiocarbonate **12** as the tetrabutylammonium zinc chelate **16**.[61,62] Compound **16** is converted into 4,5-bis(benzoylthio)-1,3-dithiole-2-thione **17**, then to 4,5-dimercapto-1,3-dithiole-2-thione dianion **15** by cleavage with sodium methoxide and afterwards by alkylation to 4,5-bis(alkylthio)-1,3-dithiole-2-thiones.[63] After the reduction the reaction mixture can be alkylated directly, too, so that a separation of **15** from **12** is unnecessary.[60]

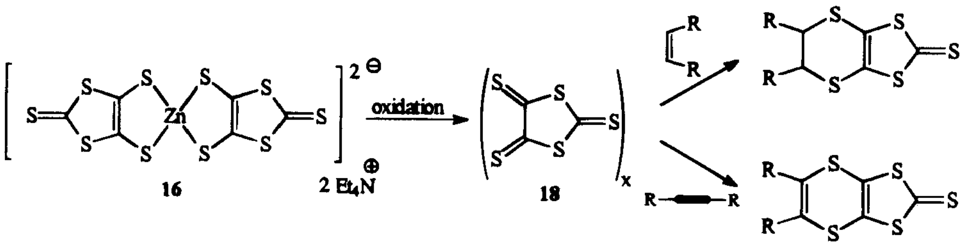
Incorporation of chalcogen atoms into the TTF skeleton involves lithiation of TTF, followed by reaction with elemental chalcogen. Then the chalcogenide anion can be capped with dihaloalkanes according to several methods (see also Chapter 6.1).

Oxidation of bis(tetraethylammonium) bis(2-thioxo-1,3-dithiole-4,5-dithiolato)zincate(II)**16**[62] produces an oligomeric 1,3-dithiole-2,4,5-trithione **18**, which presumably de-



SCHEME 9

polymerizes on heating and can be added as well to cyclic olefins as to alkynes and provides substituted 1,3-dithiole-2-thiones.[64-66]

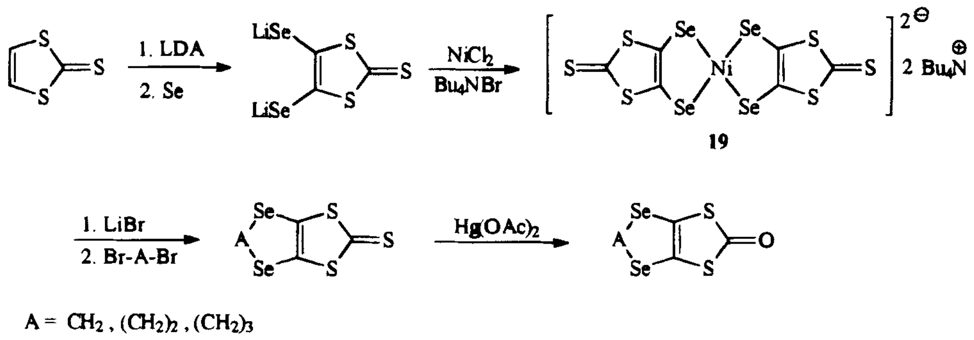


SCHEME 10

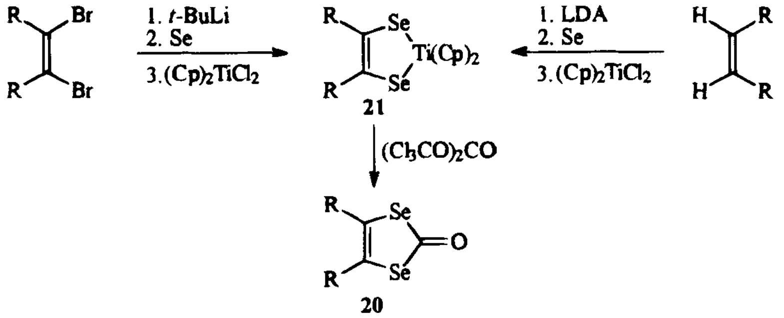
An alternative synthesis of selenium substituted TTF starts from the air and moisture stable organometallic complex bis(tetrabutylammonium) bis(2-thioxo-1,3-dithiole-4,5-diselenolato)-nickelate(II) **19**.[67-69] This complex is formed by lithiation of 1,3-dithiole-2-thione, followed by treatment with powdered selenium, nickel chloride and tetrabutylammonium bromide. Upon reflux of the nickel complex **19** with lithium bromide and dibromoalkanes the corresponding (alkylenediseleno)-1,3-dithiole-2-thiones are formed, which can be converted to alkylenediseleno-1,3-dithiol-2-ones with mercuric acetate in a refluxing mixture of chloroform and acetic acid.

1,3-Diselenol-2-ones **20** (obviating the use of highly toxic CSe_2 or H_2Se) are also formed by lithiation of 1,2-dibromoalkanes with *t*-butyllithium or substituted alkenes with lithium diisopropylamide (LDA), followed by treatment with Se powder. To the reaction mixture titanocene dichloride is added at –78 °C. The titanocene complex **21** provides, upon treatment with triphosgene, the corresponding 1,3-diselenol-2-one.[70]

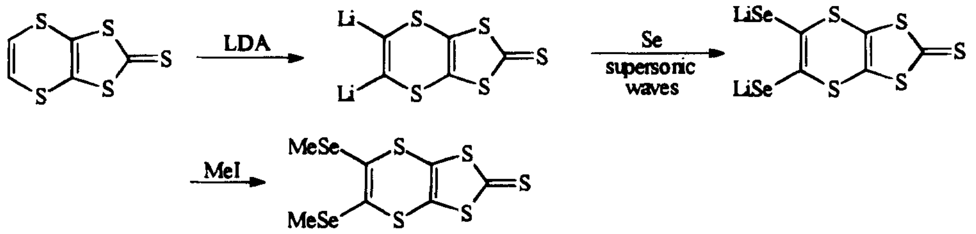
Also (vinylenedithio)-1,3-dithiole-2-thione has been lithiated with LDA and substituted, the selenium insertion being facilitated by supersonic waves.[71]



SCHEME 11



SCHEME 12

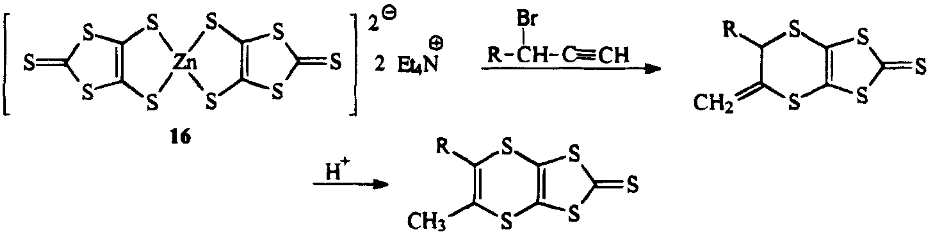


SCHEME 13

Dialkyl substituted (vinylenedithio)-1,3-dithiole-2-thiones have been prepared by reflux of the complex bis(tetraethylammonium) bis-(2-thioxo-1,3-dithiole-4,5-dithiolato)zincate(II) **16** with propargyl halides in acetone and subsequent isomerization with *p*-toluenesulfonic acid.[72]

This reaction is assumed to be analogous to the formation of dimethylenetetrahydro-TTF **51** by cleavage of 4,5-bis(propargylthio)-1,3-dithiol-2-ones in alkaline solution (see Chapter 1.10.).[73]

1,3-Dithiol-2-ones can be prepared directly by cyclization of β-keto *O*-alkyl dithiocarbonates with concentrated sulfuric acid. 4,5-(Ethylenedithio)-1,3-dithiol-2-one has been



16

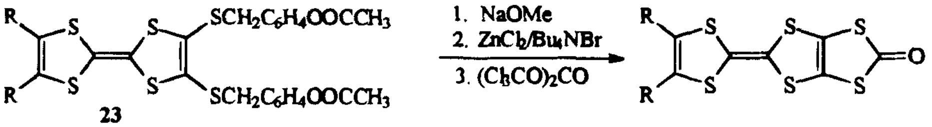
SCHEME 14

synthesized in 50% yield by treatment of 2-oxo-3-(isopropoxythiocarbonylthio)-1,4-dithiane **22** with sulfuric acid at 0 °C.[74,75]

22

SCHEME 15

A method for the synthesis of fused 1,3-dithiol-2-ones has been developed in the shape of reaction of **23** with excess sodium methoxide in dichloromethane-methanol at room temperature, subsequent treatment with zinc chloride and tetra-*n*-butylammonium bromide, followed by excess triphosgene in THF at −78 °C.[76]



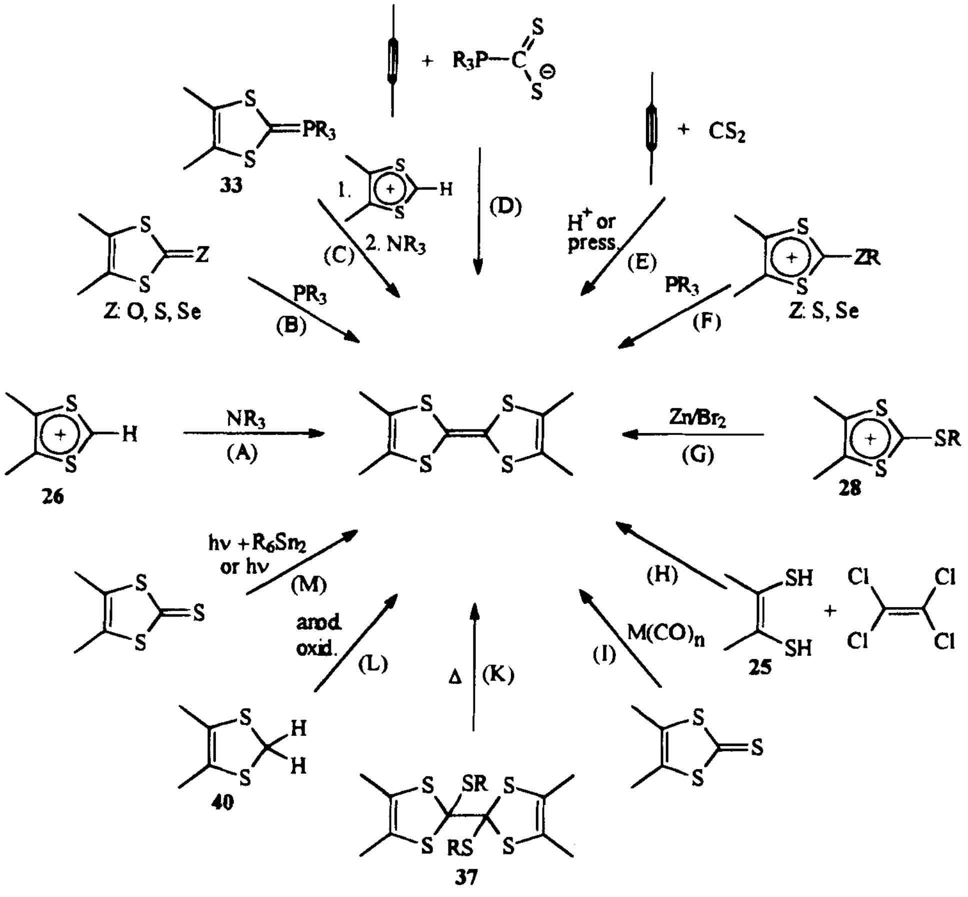
23

SCHEME 16

Thermolysis of 1,2,3-thiadiazole **24** under pressure at 220 °C in carbon disulfide allows the synthesis of trimethylene- and tetramethylene-1,3-dithiole-2-thione in moderate yields (41 resp. 43%) and of benzo-1,3-dithiole-2-thione in good yield (84%).[77,78]

Benzo-annelated and heterocyclo substituted 1,3-dithiole-2-thiones have been formed by treatment of the 1,2-dimercapto compounds **25** with carbon disulfide in alkaline solution or with thiocarbonyldiimidazole in glacial acetic acid/tetrahydrofuran.[79–82]

The 1,3-dithiole-2-thiones can be converted into TTF in different ways. The most important methods are summarized in Scheme 17.



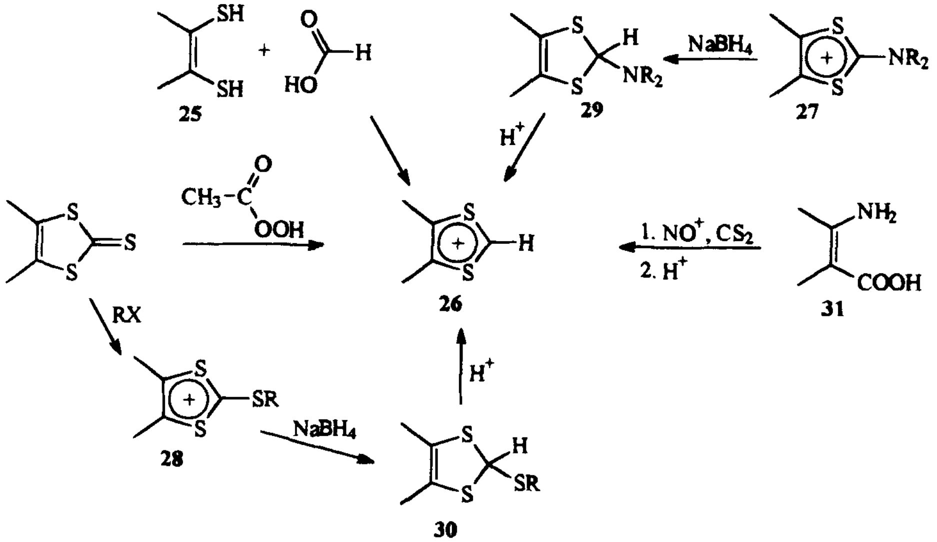
SCHEME 17

1.2. *TTF by Deprotonation of 1,3-Dithiolium Salts (Method A)*

Treatment of 2*H*-1,3-dithiolium salts **26** with tertiary aliphatic amines (*N*-ethyldiisopropylamine, triethylamine) in acetonitrile or acetone provides TTF in large quantities. 1,3-Dithiolium salts are stable compounds because the 1,3-dithiolium cation is resonance stabilized. They can be prepared in different ways (Scheme 18).[83,84]

1,3-Dithiolium salts **26** with donor substituents or one acceptor substituent and the unsubstituted 1,3-dithiolium salt can be formed by oxidizing the corresponding 1,3-dithiole-2-thione with peracetic acid in acetone or with hydrogen peroxide in glacial acetic acid.[40,43] 1,3-Dithiole-2-thiones with acceptor substituents in the 4- and 5-position cannot be converted into 1,3-dithiolium salts **26** in this way.[85] TTF with electron-withdrawing substituents are formed in high yields by method B. 2-(*N*,*N*-Dialkylamino)- **27** and 2-(alkylthio)- 1,3-dithiolium salts **28** are converted into 1,3-dithiolium salts **26** by reduction with sodium borohydride, followed by treatment with acid.[39,86,87]

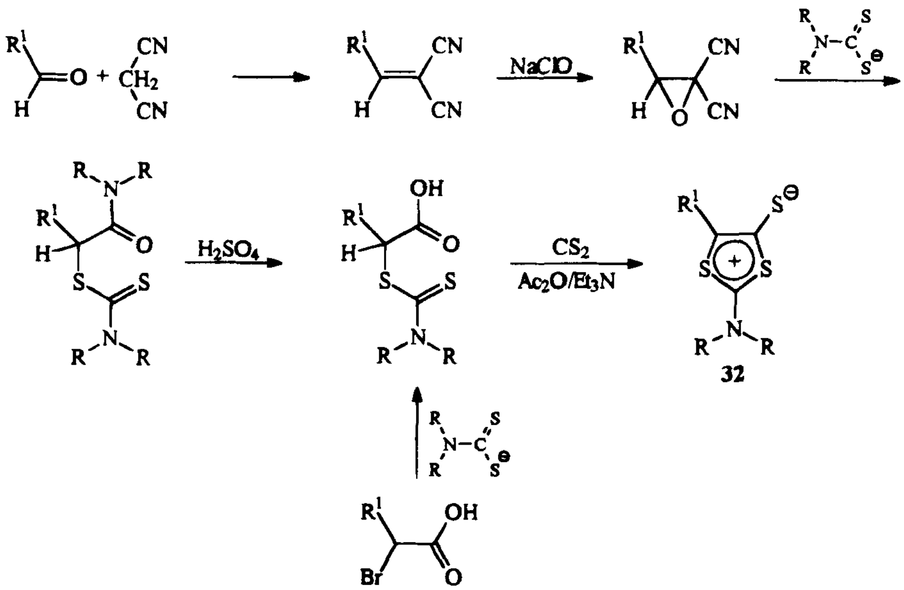
Benzo- or naphtho-annelated 1,3-dithiolium salts **26** are obtained by reaction of the 1,2-dimercapto compounds **25** with formic acid[88–91] or by that of 2-amino carboxylic acids **31** with isopentyl nitrite in 1,2-dichloroethane in the presence of carbon disulfide and treatment of the thus obtained 2-isopentyl-1,3-dithiole with acid.[92] Naphtho-annelated or heterocyclo substituted 1,3-dithiolium salts **26** are also formed in large quantities by alkylation of the corresponding 1,3-dithiole-2-thiones, reduction of the so prepared 2-(alkylthio)-1,3-dithiolium salts **28** with sodium borohydride and reaction of the 2-(alkylthio)-1,3-dithioles **30** with acid.[80,93]



SCHEME 18

Intermediates for 2*H*-1,3-dithiolium salts are also the mesoionic 1,3-dithioles **32** which can be obtained in a multi-step reaction.[94,95] An improved synthesis of **32** involves reaction of a 2-halo carboxylic acid with excess *N*,*N*-dialkyldithiocarbamate in ethanol. The crude product is treated (without further purification) with acetic acid anhydride and triethylamine, followed by reaction with excess carbon disulfide.[96] After alkylation of **32** the 1,3-dithiolium salts **26** can be obtained via the known route, i.e. by reduction with sodium borohydride and subsequent deamination with strong acid (Scheme 18).

1,3-Dithiolium salts **26** with the anions tetrafluoroborate, hexafluorophosphate, and perchlorate are well crystallizing salts, convenient for further reaction with tertiary aliphatic amines. Perchlorates must be handled with care. These salts are explosive and their handling in large quantities constitutes a potential hazard. Because of their high solubility and hygroscopic properties hydrogen sulfates are inconvenient.



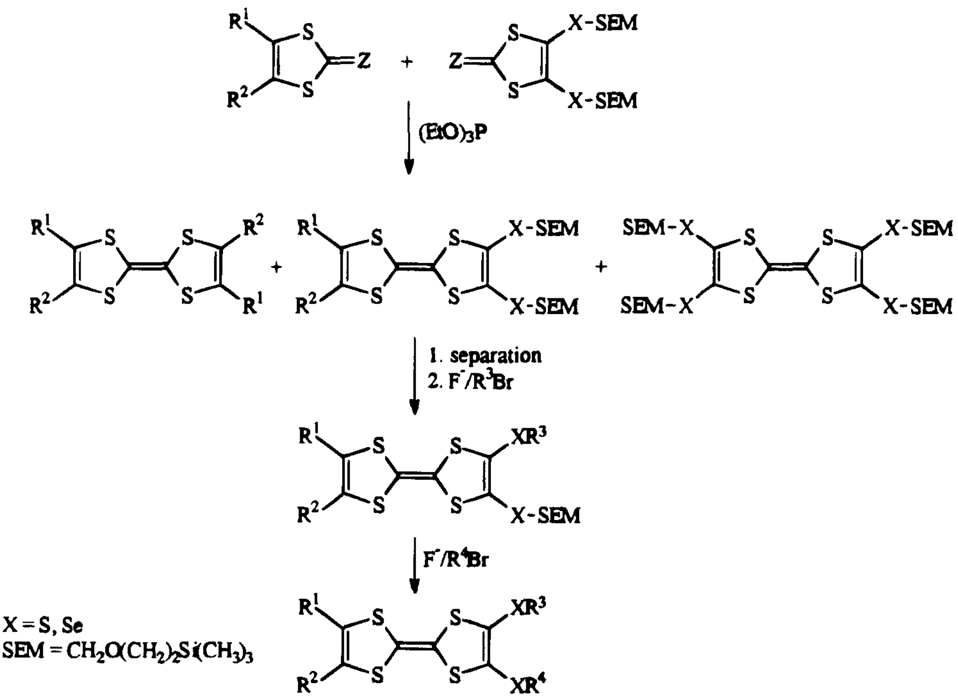
SCHEME 19

1.3. *Dechalcogenization of 1,3-Dithiol-2-ones, -thiones, and -selones to TTF with Trivalent Phosphorus Compounds (Method B)*

After the conversion of 1,3-dithiolane-2-thiones with phosphites to tetrathioethylenes,[97] the formation of TTF by desulfurization of 1,3-dithiole-2-thiones with trivalent phosphorus compounds was also successful.[98] 1,3-Dithiole-2-thiones undergo the desulfurization reaction only when they are substituted by electron-withdrawing groups (COOR, CF_3,CN),[98–101] by alkylthio groups,[46,102–104] by heterocycles,[80,103] or if they are benzo-annelated.[90,105] As trivalent phosphorus compounds trialkyl or triaryl phosphites as well as triphenylphosphine are convenient.[99,101] Often the yields of TTF are higher with 1,3-dithiol-2-ones or -selones than with 1,3-dithiole-2-thiones.[46,80,99,103,106] 1,3-Dithiole-2-thiones can easily be converted into 1,3-dithiol-2-ones by reaction with mercury (II) acetate in chloroform/glacial acetic acid[107] or into 1,3-dithiole-2-selones after alkylation and subsequent treatment with hydrogen selenide.[108]

Cross-coupling of the appropriate 1,3-dithiole derivatives with triethyl phosphite usually leads to a mixture of the three possible TTF. The difficulty of their separation can be overcome by use of trimethylsilylethoxymethyl (SEM) as a protecting group for the 1,3-dithiole derivatives. The three protected compounds thus formed can easily be separated by column chromatography. In a subsequent reaction the protecting group is removed under mild conditions with tetrabutylammonium fluoride and the free anion alkylated with an alkyl bromide.[109]

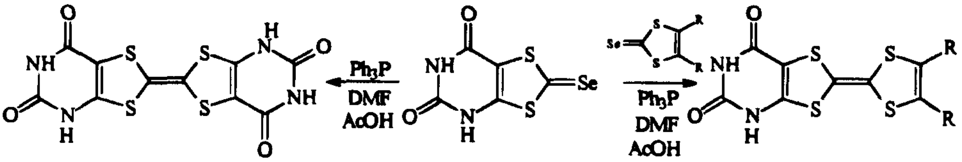
The yield of alkylthio substituted TTF by coupling of 1,3-dithiole-2-thiones with trimethyl or triethyl phosphite is increased by high pressure (4–5 kbar).[110]



X = S, Se
SEM = $CH_2O(CH_2)_2Si(CH_3)_3$

SCHEME 20

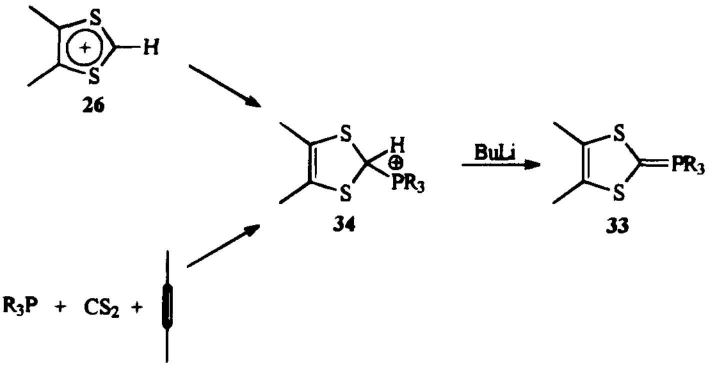
Pyrimido substituted TTF are formed by coupling of the appropriate dithiole-2-selones with triphenylphosphine in *N,N*-dimethylformamide in the presence of acetic acid at room temperature. The selones do not react with triethyl phosphite in benzene at 80 °C.[111]



SCHEME 21

1.4. *Treatment of 1,3-Dithiolium Salts with Phosphoranes or Phosphonates (Method C)*

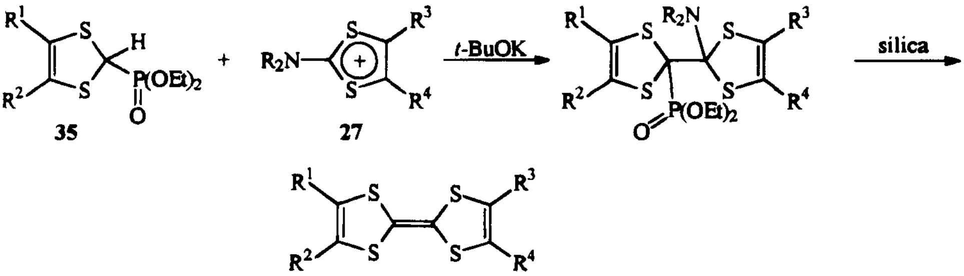
1,3-Dithiol-2-ylidenephosphoranes **33** are prepared, as unstable compounds, by deprotonation of 1,3-dithiolylphosphonium salts **34** with butyllithium at −78 °C in tetrahydrofuran.[112-117] 1,3-Dithiolylphosphonium salts **34** are obtained in the case of alkyl substituted, the unsubstituted, and benzo-annelated compounds by reaction of the 1,3-dithiolium salts **26** with a phosphine. Because 1,3-dithiolium salts **26** with electron-withdrawing substituents in the 4- and 5-position can only be formed with difficulty, these 1,3-dithiolylphosphonium salts **34** are better synthesized by treatment of alkynes with an adduct of carbon disulfide and a phosphine in acidic solution at –65 °C in ether.[115,116,118]



SCHEME 22

The thus prepared 1,3-dithiol-2-ylidenephosphoranes **33** react in the presence of triethylamine with 1,3-dithiolium salts **26** to form TTF.[113,115,119] This method has the great advantage that unsymmetrical TTF derivatives can be prepared. The instability of the 1,3-dithiol-2-ylidenephosphoranes **33** under the reaction conditions leads to the formation of variable amounts of symmetrical TTF. Therefore, purification of the mixture by column chromatography is necessary.[120] Other methods for the synthesis of unsymmetrical TTF by coupling of two different 1,3-dithiole derivatives require separation from symmetrical coproducts by fractional recrystallization[79,121,122] or chromatography.[123,124]

This side reaction and the formation of symmetrical TTF can be avoided by coupling of phosphonate esters of various substituted 1,3-dithioles **35** with 2-amino-1,3-dithiolium salts **27** under Wittig-like conditions. Yields of pure, unsymmetrical TTF are typically 30–50%.[96,125–127]

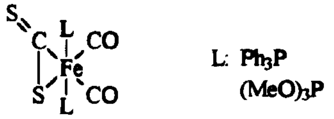


SCHEME 23

The required phosphonate esters of 1,3-dithioles **35** are synthesized by reaction of 1,3-dithiolium salts **26** with sodium iodide.[112,114] Some substituted **35** can also be prepared in a one-step reaction by heating of the corresponding 1,3-dithiole-2-thiones or -2-ones in the presence of a trialkyl phosphite at high dilution.[126,128]

1.5. *TTF by Reaction of Carbon Disulfide with Alkynes (Methods D and E)*

The reaction between the adduct of carbon disulfide and tributylphosphine with methyl propiolate at –30 °C in tetrahydrofuran in the absence of acid and with excess carbon disulfide gives the corresponding TTF and not the 1,3-dithiolylphosphonium salt **34**.[53] Electron-deficient alkynes form with carbon disulfide, activated by iron complex **36**, TTF in moderate yield (25–30%).[129,130] The formation of TTF derivatives from complexes of electron-deficient alkynes and iron complexes **36** can be initiated either by electrochemical oxidation or by chemical oxidation with $AgCF_3SO_3$, I_2, or $[Fe(C_5H_5)_2]PF_6$.[131]



36

SCHEME 24

Alkynes with electron-withdrawing substituents and carbon disulfide react at 100 °C in a sealed tube to form TTF derivatives in very low yield (2%).[98] The preparation of TTF in high yield is achieved by carrying out the reaction under high pressure (5–6 kbar)[132–134] or in the presence of strong acids (trifluoroacetic acid).[98,135] Cyclalkynes provide with carbon disulfide at room temperature the corresponding TTF.[136–138]

1.6. *TTF by Treatment of 2-(Alkylthio)-, 2-(Arylthio)-, and 2-(Alkylseleno)-1,3-dithiolium Salts with Trivalent Phosphorus Compounds (Method F)*

TTF are generated by treatment of 2-(alkylthio)- or 2-(arylthio)-1,3-dithiolium salts with triethyl phosphite or triphenylphosphine in higher yields and with short reaction time than by treatment of the corresponding 1,3-dithiol-2-thiones. By use of 2-(alkylseleno)-1,3-dithiolium salts the highest yields are obtained. In this case also tributylphosphine or hexamethylphosphorous triamide can be used as the trivalent phosphorus compound.[139]

1.7. *TTF by Treatment of 2-(Methylthio)-1,3-dithiolium Salts with Zinc and Bromine (Method G)*

In a convenient way and with easily varied substituents (alkyl, aryl, benzo) TTF are prepared by treatment of 2-(methylthio)-1,3-dithiolium salts **28** (R = Me) with zinc dust in ethanol in the presence of an oxidizing agent or an electrophilic reagent. Suitable oxidizing agents are bromine, iodine, peracetic acid, lead dioxide, or hydrogen peroxide, and electrophilic reagents, proton acids, methyl iodide, or dimethyl sulfate. The yield reaches values between 35 and 65%.[140–142]

1.8. *TTF by Reaction of 1,2-Dimercapto Compounds with Tetrachloroethene (Method H)*

By treating 1,2-dimercaptobenzene derivatives **25** with tetrachloroethene under basic conditions (*N*,*N*-diisopropylethylamine, triethylamine, pyridine, *N*,*N*-dimethylformamide) one can obtain TTF in modest yields (5–35%).[79,143] This method was already used in 1926 for the synthesis of the first tetrathiafulvalene.[144]

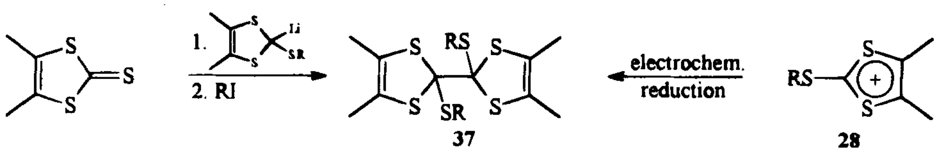
1.9. *Miscellaneous Methods*

A photochemical method for the synthesis of TTF is the irradiation of 1,3-dithiole-2-thiones with UV light in benzene. The yields are modest (5–25%), better in the presence of triethylamine or triethyl phosphite. Only phenyl substituted and benzo-annelated compounds react, but cycloalkyl substituted thiones are inactive and donor or acceptor substituted derivatives are decomposed in an obscure way.[145]

The irradiation of 1,3-dithiole-2-thiones in the presence of hexabutyldistannane produces the corresponding TTF in appreciably higher yields (50–80%). The method is also generally applicable for the preparation of TTF substituted either by electron-donating or electron-withdrawing groups (method M).[146–148]

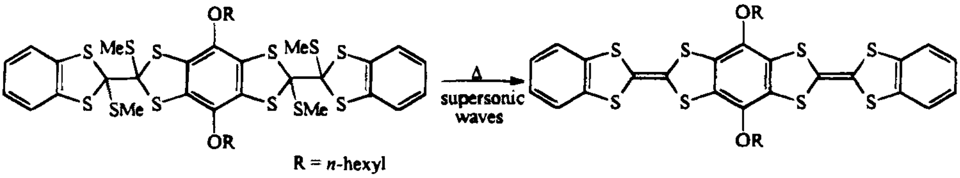
In a one-step synthesis TTF are afforded directly from 1,3-dithiole-2-thiones, substituted with electron-donating or electron-withdrawing groups, in 10–60% yield by heating with dicobaltoctacarbonyl or triirondodecacarbonyl in benzene or toluene solution (method I).[55,99,149]

Thermolysis of the orthothiooxalates **37** in carbon tetrachloride gives TTF in high yield.[58,150] The addition of catalytic amounts of *p*-toluenesulfonic acid accelerates the reaction.[150] The orthothiooxalates **37** are formed by electrochemical reduction of the 2-(alkylthio)-1,3-dithiolium salts **28**[58,151,152] or by treatment of 1,3-dithiole-2-thiones with 2-lithio-2-(alkylthio)-1,3-dithioles and alkylation of the intermediates formed. The 2-lithio-2-(alkylthio)-1,3-dithioles are prepared by deprotonation of 2-(alkylthio)-1,3-dithioles with butyllithium at −78 °C in tetrahydrofuran.[153] This last method for the synthesis of orthothiooxalates **37** allows the formation of unsymmetrical TTF in their pure form (method K).



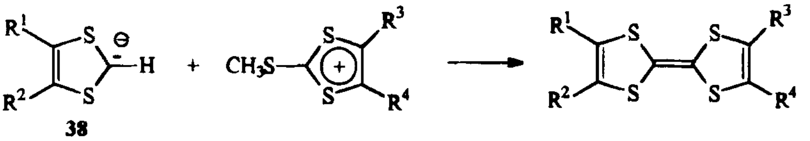
SCHEME 25

Addition of catalytic amounts of *p*-toluenesulfonic acid does not influence the thermolysis of bis(orthothiooxalates) in 1,1,2,2-tetrachloroethane at 130 °C.[154,155] However, upon supersonic irradiation the reaction proceeds at lower temperatures (70 °C) in a yield as high as 80%.[156,157]



SCHEME 26

A coupling method leading to unsymmetrical TTF involves treatment of 1,3-dithiole anions **38** with electrophilic 1,3-dithiolium salts. In addition to the expected condensation products, symmetrical TTF, derived from **38**, probably via carbenes, are formed.[158]



38

SCHEME 27

The reaction of organoaluminium reagents with the ester of the 1,3-dithiole **39** gives also the possibility of the synthesis of unsymmetrical substituted TTF.[159]

39

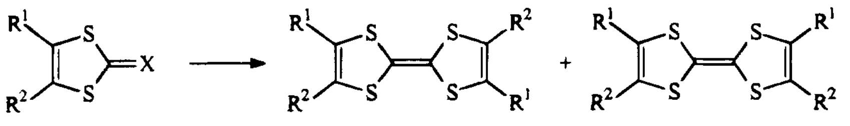
SCHEME 28

Thermolysis of 2-alkoxy-1,3-dithioles also produces TTF.[160] The yield increases if the thermolysis is performed in benzene in the presence of trichloroacetic acid.[95,161,162] Electrochemical oxidation of 1,3-dithioles **40** in acetonitrile, in the presence of pyridine, provides TTF directly (method L).[163]

TTF and their data are summarized in Table 11.

1.10. The Formation of cis- and trans-*Isomers of TTF*

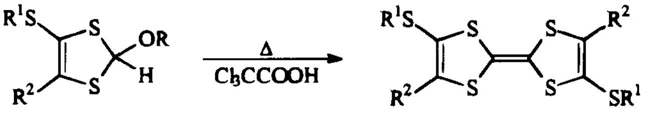
Coupling of unsymmetrically substituted 1,3-dithiole derivatives to TTF by various methods normally provides a mixture of *cis*- and *trans*-isomers.



trans *cis*

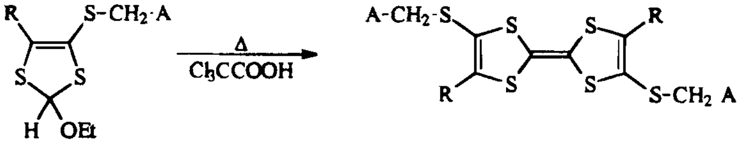
SCHEME 29

Only in the case of thermolysis of 2-alkoxy-1,3-dithioles in the presence of trichloroacetic acid in refluxing benzene the formation of *trans*-isomers could be demonstrated by X-ray analysis.[164]



SCHEME 30

By thermolysis of 2-alkoxy-1,3-dithioles in the presence of trichloroacetic acid trans-TTF with covalently bonded acceptors on the donor unit can be prepared.[162]

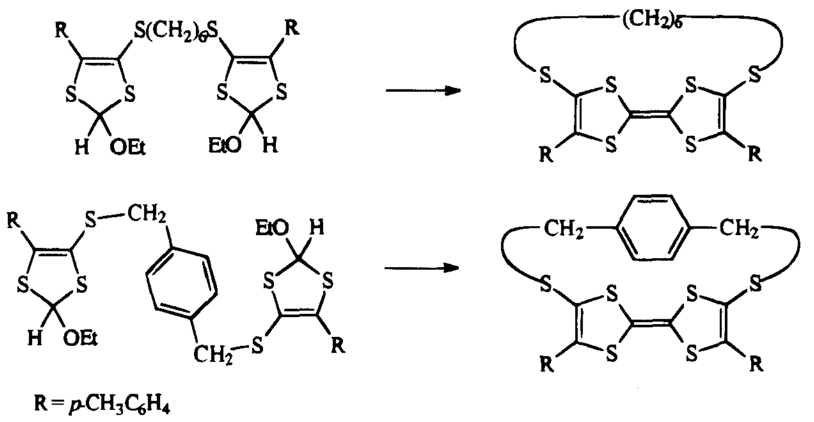


SCHEME 31

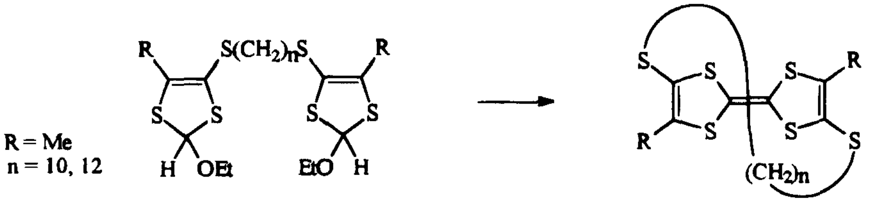
In two cases, however, bridged 2-alkoxy-1,3-dithioles form the *cis*-isomers in nearly quantitative yield.[165]

However, in two other cases the bridge was long enough for the formation of the *trans*-isomers ($(CH_2)_n$ with n = 10 or 12). During the electrochemical oxidation the *trans*-isomer is converted to the *cis*-isomer.[166]

Mostly the properties of *cis* and *trans* isomers of TTF are very similar and prevent a distinction or separation. In a ^{1}H NMR study for the first time evidence of an acid catalysed *trans-cis* isomerization was obtained in the case of bis[4-(4-chlorophenyl)-5-(methylthio)]-TTF.[167] The *cis-trans* equilibrium was too rapidly established to permit



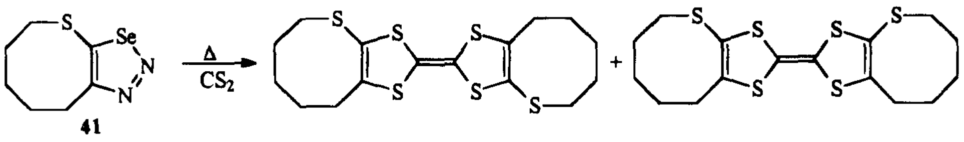
SCHEME 32



SCHEME 33

isolation of the pure *cis* form. Only the *trans* isomer could be recovered after concentration of the solution.

By means of ${}^{13}C$ NMR measurements the formation of a 1:1 mixture of *cis*- and *trans*-isomer by thermolysis of a substituted 1,2,3-selenadiazole **41** in carbon disulfide could be proved.[168]

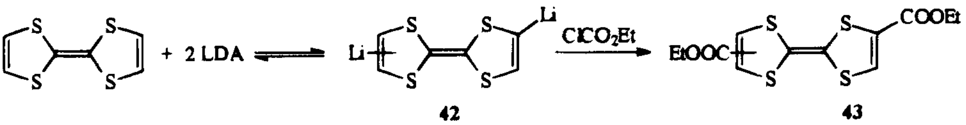


SCHEME 34

Dilithiation of TTF gave an approximate 1:1 mixture of the *cis*- and *trans*-isomers **42** as evident from the ${}^{1}H$ NMR spectrum of the reaction products **43**, formed by reaction of **42** with ethyl chloroformate.[169]

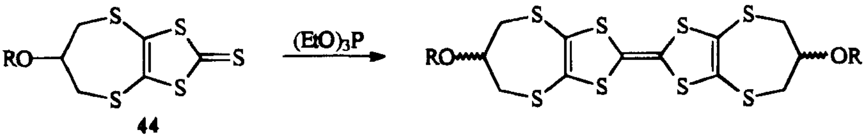
The self-coupling of the thiones **44** with triethyl phosphite provides a mixture of diastereoisomers. This was confirmed by ${}^{1}H$ NMR data but it was not necessary to separate the diastereoisomers.[170]

In some cases the *cis/trans* mixture could be separated.



42 43

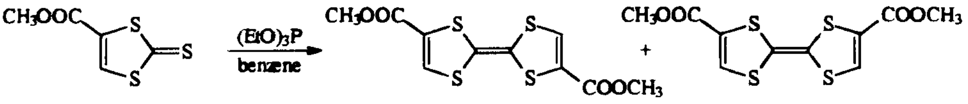
SCHEME 35



44

SCHEME 36

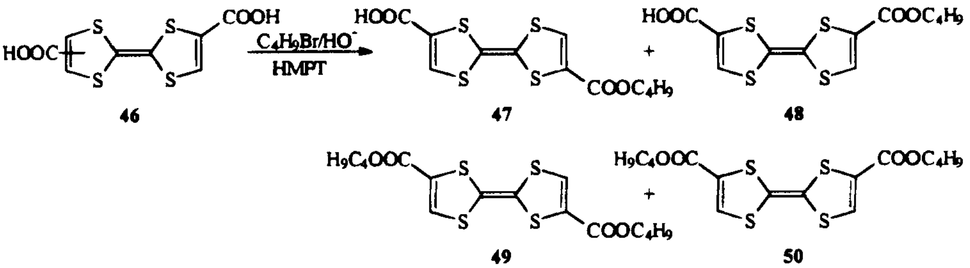
Treatment of 4-carbomethoxy-1,3-dithiole-2-thione **45** with triethyl phosphite in refluxing benzene resulted in the formation of two products which could be separated by their different solubility in benzene.[100] The two products are distinguished by different melting points and small differences in their IR spectra but an assignment to *cis*- and *trans*-isomers has not been given.



45

SCHEME 37

Esterification of tetrathiafulvalene-2,6(7)-dicarboxylic acid **46** with *n*-butyl bromide in hexamethylphosphoric triamide (HMPT) at room temperature provides a mixture of two monoesters **47, 48** and two diesters **49, 50**. Also by their different solubility in acetonitrile, benzene and hexane the *cis*- and the *trans*-isomer as well from the monoester as from the diester could be separated. In each case the *cis*- and the *trans*-isomer show few differences in the ^{1}H NMR, UV-VIS, and IR spectra.[171] By irradiation with sun light the *trans*-diester **49** isomerizes to the *cis*-isomer **50**.[172]

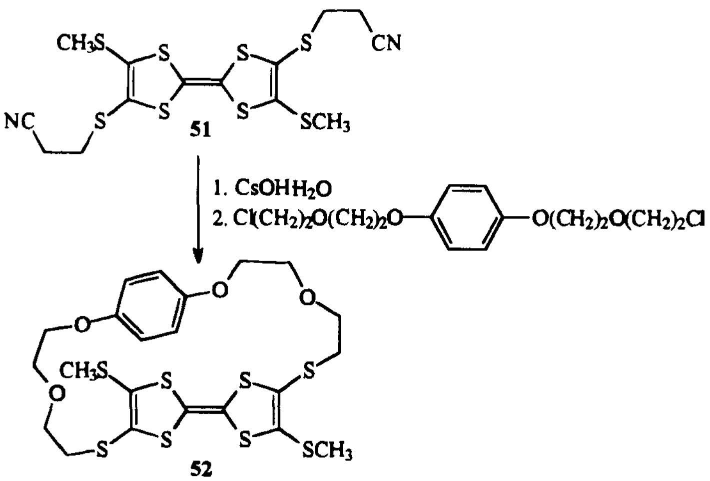


46 47 48

49 50

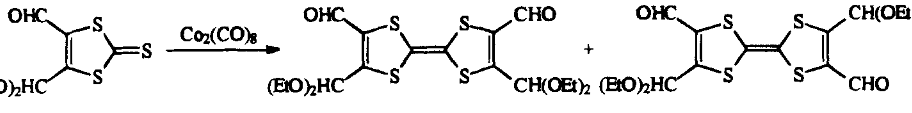
SCHEME 38

Deprotection of a *cis/trans* mixture of **51** with caesium hydroxide, followed by realkylation with a bisalkylating agent provides a new TTF, where the *trans*-isomer **52** could be isolated.[173]



SCHEME 39

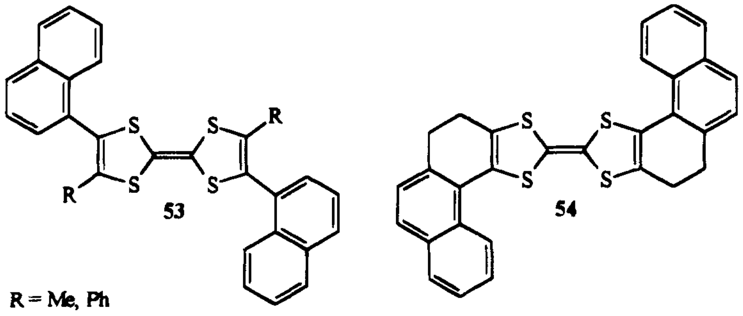
The diacetalized tetraformyl-TTF, prepared by desulfurative coupling of the corresponding thione with dicobaltoctacarbonyl, is obtained as a mixture of essentially equal amounts of the *cis*- and *trans*-isomers.[174] The *cis*-isomer is separated by selective solubilization in ether and subsequent column chromatography. The *trans* structure has been confirmed by X-ray analysis. No *cis-trans* isomerization is observed under the influence of acids on the *cis*-isomer. This is in contrast to the above bis[4-(4-chlorophenyl)-5-(methylthio)]-TTF.



SCHEME 40

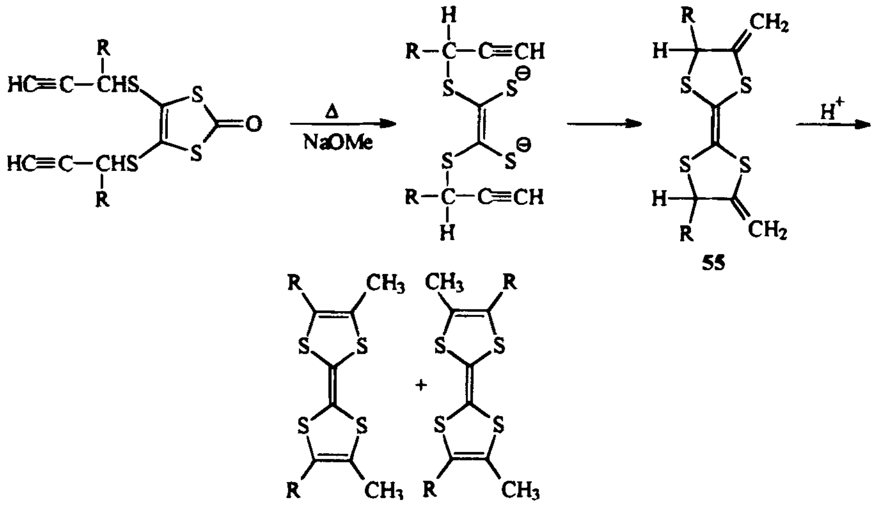
Bulky substituents modify the properties of *cis*- and *trans*-isomers so much that a separation can be carried out based on different solubilities in the case of 1-naphthylmethyl **53**[175] or 1-naphthylphenyl **53**, 2-naphthylphenyl substituted and dihydrophenanthreno-annelated TTF **54**.[176]

In an alternative synthesis by stereospecific cyclization of 4,5-bis(propargylthio)-1,3-dithiol-2-one due to the *cis* relationship of the sulfide groups in the intermediate the *cis*-isomer should be formed. However, the resulting dimethylenetetrahydro-TTF **55** are oils, making X-ray analysis unfeasible. After isomerization with *p*-toluenesulfonic acid in refluxing benzene a mixture of *cis*- and *trans*-isomers is formed, as an X-ray structure de-



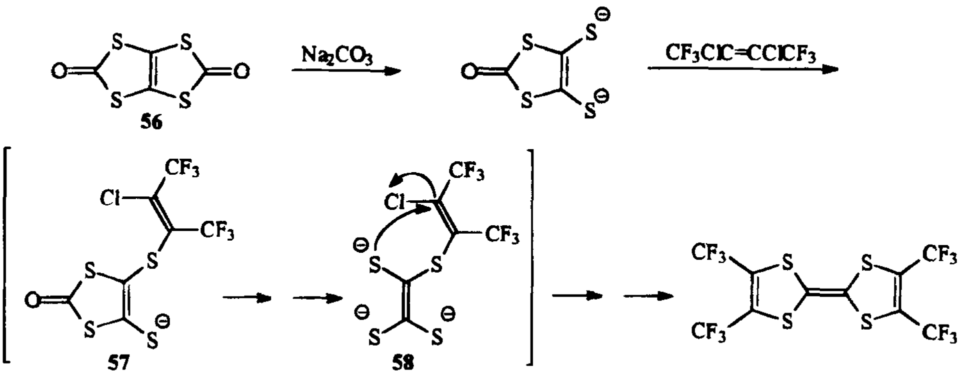
SCHEME 41

termination proves.[73] The *cis-trans* mixture is probably formed by protonation of the central double bond, followed by isomerization.



SCHEME 42

In a nucleophilic substitution reaction tetrakis(trifluoromethyl)-TTF is formed by alkaline hydrolysis of 1,3,4,6-tetrathiapentalene-2,5-dione **56** in the presence of 2,3-dichlorohexafluoro-2-butene under phase transfer conditions.[75,177] It is proposed that the intermediate **57** is formed after initial attack of the alkyl halide by the dianion. The extremely electron-withdrawing substituent could facilitate the opening of the second ring and stabilize the resulting trianion **58**. After intramolecular cyclization and reaction with a second mol of alkyl halide tetrakis(trifluoromethyl)-TTF would be formed.



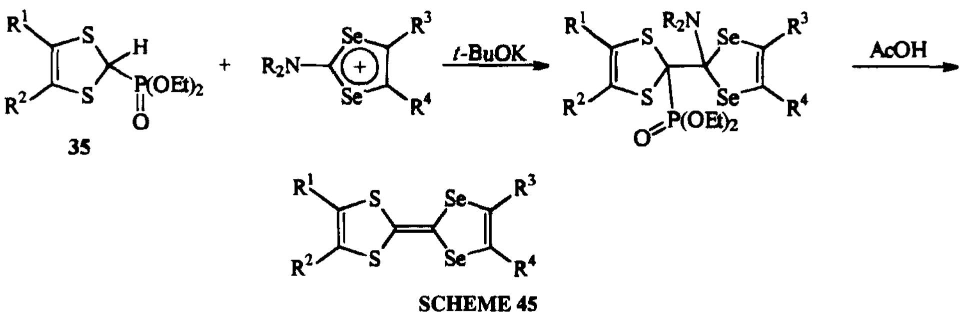
SCHEME 43

2. SYNTHESIS OF DISELENADITHIAFULVALENES (DSDTF), 1,3-THIASELENOLE-2-THIONES AND -SELONES

DSDTF exist in two isomeric forms; in the isomer **59** each ring contains identical heteroatoms (1,4-diselena-5,8-dithiafulvalenes), in the isomer **60** each ring contains different heteroatoms (1,5-diselena-4,8-dithiafulvalenes) with the possibility of a *cis-* and a *trans-*form.

SCHEME 44

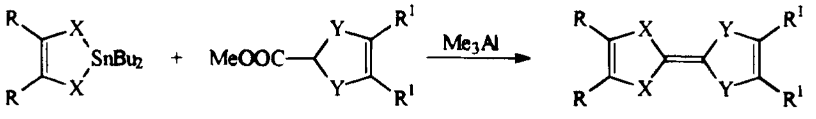
The 1,4-diselena-5,8-dithiafulvalenes **59** are prepared by cross-coupling of the appropriate 1,3-dithiole and 1,3-diselenole derivatives with trimethyl or triethyl phosphite. In addition to the desired DSDTF the symmetrical self-coupling products are formed. Separation of the symmetrical self-coupling products is accomplished by column chromatography on silica, followed by repeated preparative gel permeation chromatography.[178,179]



SCHEME 45

In analogy to the synthesis of unsymmetrical TTF derivatives[125] the DSDTF **59** are obtained in pure form and without by-product (TTF and TSF) by reaction of (1,3-dithiol-2-yl)ethyl phosphonate **35** with 2-amino-1,3-diselenolium salts in the presence of strong bases, followed by addition of acetic acid.[180-182]

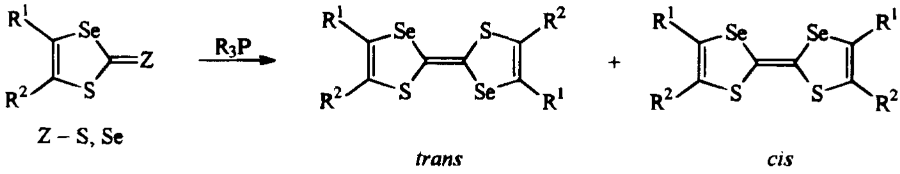
In an analogous manner to organoaluminium reagents (see Chapter 1.9.) organotinthiolates or -selenolates react with the esters of 1,3-dithioles or 1,3-diselenoles in the presence of trimethylaluminium and provide the unsymmetrical substituted DSDTF **59**.[183]



R = Me, X = Se; $R^1 = S(CH_2)_2S$, Y = S
R = $S(CH_2)_2S$, X = S; $R^1 = (CH_2)_3$, Y = Se

SCHEME 46

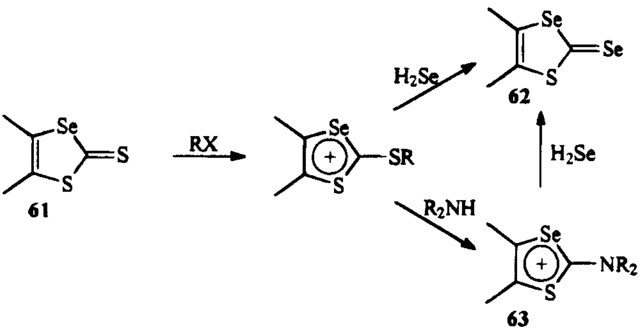
The synthesis of symmetrical DSDTF **60** is successful only by treatment of 1,3-thiaselenole-2-thiones or -selones with trivalent phosphorus compounds (trimethyl or triethyl phosphite, triphenylphosphine). The coupling reaction affords a *cis*- and *trans*-mixture of isomers. The mixture cannot be separated at all.[184,185] The existence of both isomers is proved by ^{1}H NMR spectra.[108,176,184,186]



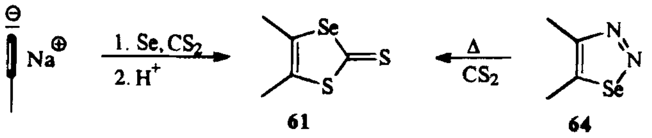
SCHEME 47

Among the unsubstituted compounds the reaction works only with 1,3-thiaselenole-2-selone.[184,185] Selones normally give higher yields than the corresponding thiones.[176,187] Therefore 1,3-thiaselenole-2-thiones **61** are converted into the 1,3-thiaselenole-2-selones **62**. This occurs in the case of the methyl substituted compound **61** by alkylation and treatment of the **50** formed thiaselenolium salt with hydrogen selenide,[108] or in the case of cycloalkyl substituted and benzo-annelated derivatives[187] as well as unsubstituted 1,3-thiaselenole-2-thione[185] by conversion to a 2-amino-1,3-thiaselenolium salt **63** and following treatment with hydrogen selenide.[185,187] Methyl-, phenyl- and unsubstituted 1,3-thiaselenole-2-thione **61** is prepared in a one-step synthesis by addition of selenium and carbon disulfide to the corresponding acetylide.[188,189]

Thermolysis of the corresponding 1,2,3-selenadiazoles **64** in excess carbon disulfide affords the unsubstituted,[185] the cycloalkyl substituted[46,187,190] and the benzo-annelated[187] 1,3-thiaselenole-2-thione **61**.

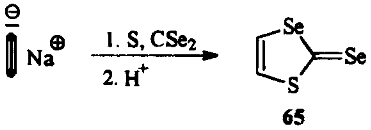


SCHEME 48



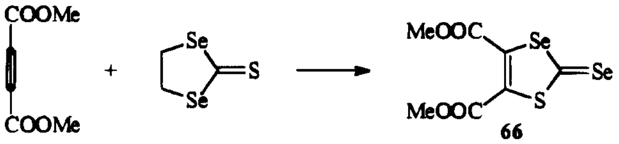
SCHEME 49

The 1,3-thiaselenole-2-selones **62** can also be obtained directly. The unsubstituted compound **65** is formed in low yield (8%) from sodium acetylide, sulfur and carbon diselenide.[189]



SCHEME 50

Treatment of 1,3-diselenolane-2-thione with dimethyl acetylenedicarboxylate gives the corresponding selone ester **66** in good yield (70%).[185,191]

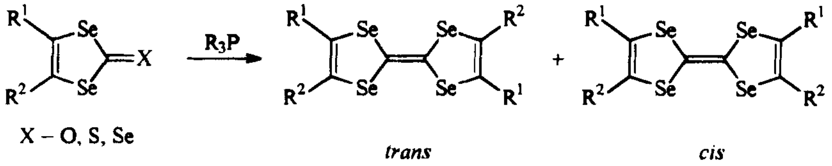


SCHEME 51

DSDTF and their data are summarized in Table 12.

3. SYNTHESIS OF TETRASELENAFULVALENES (TSF)

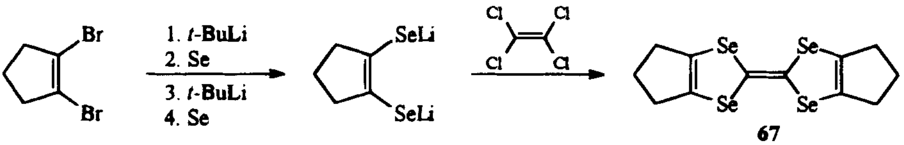
TSF are obtained by coupling of 1,3-diselenole-2-thiones, -selones, or -ones with trivalent phosphorus compounds.



SCHEME 52

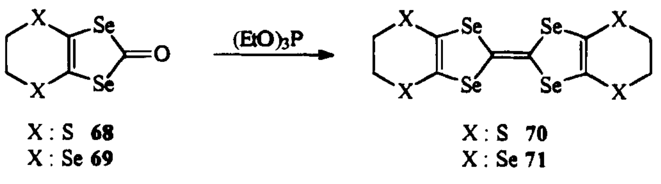
If differently substituted 1,3-diselenole derivatives are used, a mixture of *cis/trans*-isomers is formed. Only in one case could such a mixture be separated on the basis of solubility differences of the isomers.[192]

Only tetrakis(trimethylsilyl)tetraselenafulvalene has been synthesized by reaction under high pressure (5–6 kbar) between bis(trimethylsilyl)acetylene and carbon diselenide in moderate yield (20%).[134] Hexamethylenetetraselenafulvalene **67** was obtained by a synthesis analogous to that of the tetratellurafulvalenes.[193]



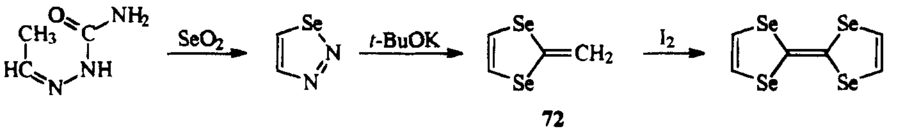
SCHEME 53

In the dechalcogenation reaction of 1,3-diselenole-2-thiones, -selones, or -ones with trivalent phosphorus compounds the 1,3-diselenole-2-selones give mostly higher yields of TSF than the corresponding 1,3-diselenole-2-thiones.[106,194] With 1,3-diselenole-2-thiones an unexpected selenium-sulfur scrambling can occur, so that triselenathiafulvalenes are formed as by-products.[194] The synthesis of bis(ethylenedithio)- and bis(ethylenediseleno)tetraselenafulvalene **70** and **71**, respectively, is successful only with 4,5-ethylenedithio- or 4,5-ethylenediseleno-1,3-diselenol-2-one **68** and **69**, respectively.[103,195] The corresponding selones do not react with triethyl phosphite.



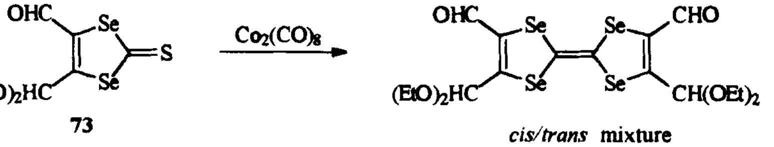
SCHEME 54

In a yield of about 30% TSF is obtained without use of the toxic CSe_2 in a three-step reaction. Acetaldehyde semicarbazone forms with selenium dioxide 1,2,3-selenadiazole which in turn reacts with potassium *t*-butoxide in DMF/*t*-BuOH and provides fulvene **72**. Addition of a mixture of iodine and morpholine in DMF to a solution of **72** furnishes TSF.[196]



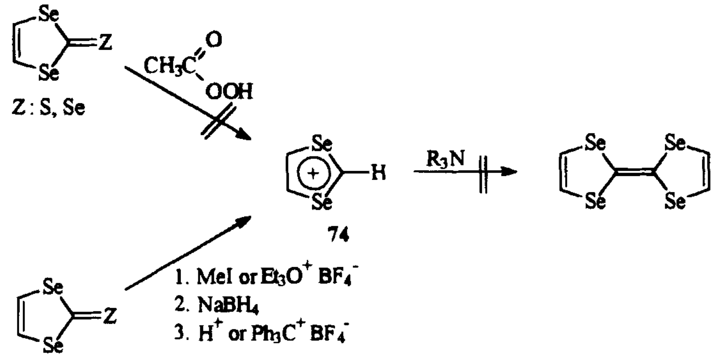
SCHEME 55

In analogy to the synthesis of TTF[197] the corresponding TSF are formed upon reflux of 1,3-diselenole-2-selones in toluene with dicobaltoctacarbonyl. In the case of the monoacetalized **73** a mixture of practically equal amounts of the *cis*- and *trans*-isomer of the TSF is obtained and separated by silica thick layer chromatography.[198] The stereochemistry was assigned by comparison with X-ray structures of the corresponding TTF.[199]



SCHEME 56

Contrary to TTF synthesis, an analogous formation of TSF by oxidation of 1,3-diselenole-2-thiones or -selones with peracids and the following reaction with trialkylamines could not be realized.[106] The oxidation of 1,3-diselenole-2-thiones or -selones fails to give the desired 1,3-diselenolium salts **74**.[106,200,201] If they are available by alkylation of 1,3-diselenole-2-thiones or -selones, reduction with sodium borohydride and treatment with tetrafluoroboric acid[202] or trityl tetrafluoroborate,[203] the reaction of **74** with trialkylamines provides only elemental selenium as precipitate.[106]



SCHEME 57

TSF and their data are summarized in Table 13.

3.1. *1,3-Diselenole-2-selones as Starting Materials for TSF*

The required 1,3-diselenole-2-selones can be prepared by various methods (Scheme 58). In analogy to 1,3-dithiole-2-thiones,[38–40] 1,3-diselenole-2-selones are synthesized by ring closure of β-keto *N,N*-dialkyldiselenocarbamates **75** with concentrated sulfuric acid at room temperature and subsequent treatment of the **50** formed 2-(*N,N*-dialkylamino)-1,3-diselenolium salts **76** with hydrogen selenide in 70% methanol.[201,204] Before reaction with hydrogen selenide, the 2-(*N,N*-dialkylamino)-1,3-diselenolium hydrogen sulfates **76** are converted to well crystallizing, but explosive perchlorates.[201,204,205] Less dangerous is the handling of tetrafluoroborates[206] or hexafluorophosphates.[207,208] Alkyl,[201,204] aryl,[201] and aralkyl substituted[205] β-keto *N,N*-dialkyldiselenocarbamates **75** are generated by treatment of the corresponding α-bromo ketones with *N,N*-dialkyldiselenocarbamates **77**. In the synthesis of alkyl derivatives a convenient solvent is methylene chloride[201,209,210] or *N,N*-dimethylformamide[207] and for aralkyl substituted compounds methanol.[211]

The *N,N*-dialkyldiselenocarbamate **77** is prepared by the reaction of carbon diselenide with a dialkylamine (e.g. piperidine, morpholine, pyrrolidine).[201,212] Since carbon diselenide is an extremely fetid reagent and only produced in 30% yield via the reaction of methylene chloride with selenium at 600 °C,[213] it is better to treat hydrogen selenide with dichloromethylene-*N,N*-dimethylammonium chloride **78**[214] in the presence of triethylamine[210,215] or piperidine.[209] The disadvantages of handling gaseous hydrogen selenide are avoided by the use of sodium hydrogen selenide, which can be generated by reducing black selenium with sodium borohydride *in situ*.[207]

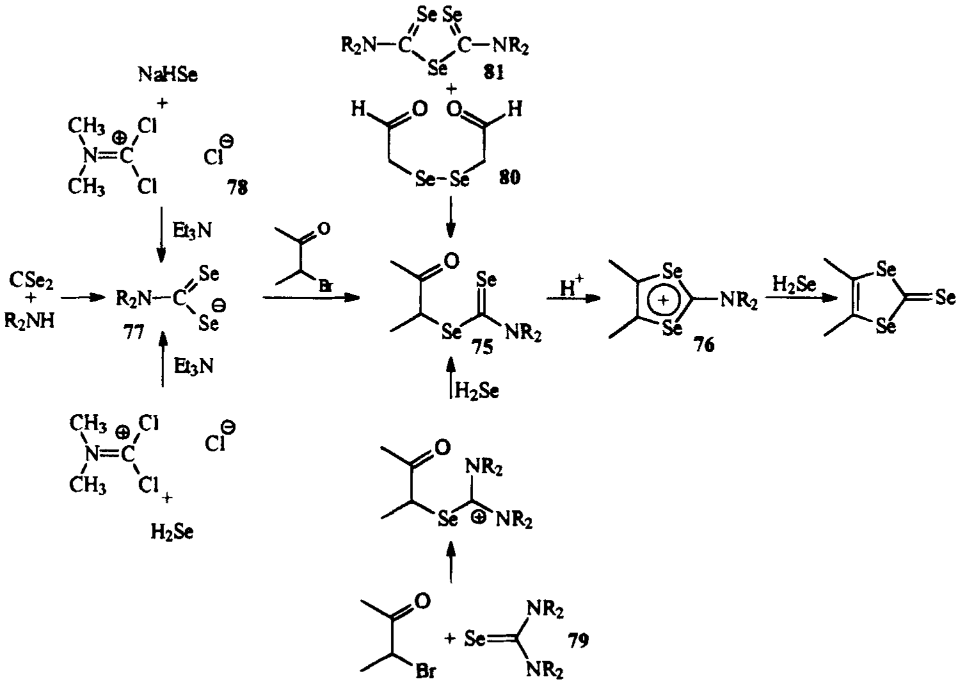
Synthesis of β-keto *N,N*-dialkyl diselenocarbamates **75** without use of carbon diselenide is possible by reaction of *N,N,N′,N′*-tetramethylselenourea **79** with α-bromo ketones, followed by treatment with hydrogen selenide.[216] The disadvantage of this method is that only fair yields of **75** are obtainable. The reaction between the corresponding diselenide **80** and bis(*N,N*-dialkylselenocarbamoyl) selenides **81** in the presence of *p*-toluenesulfonic acid in chloroform leads to the β-formyl-*N,N*-dialkyldiselenocarbamates **75**, which permit access to the unsubstituted 1,3-diselenole-2-selone.[208]

The unsubstituted and 4-phenyl substituted 1,3-diselenole-2-selone is prepared more easily in analogy to 1,3-dithiole-2-thione[52] by addition of selenium and carbon diselenide to the corresponding sodium acetylides.[189,217]

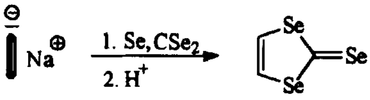
In a multi-step synthesis the parent compound is obtained, also in analogy to 1,3-dithiole-2-thione,[53] in high yield (each step over 60%) by reaction of 1,3-diselenolane-2-selone with dimethyl acetylenedicarboxylate, followed by hydrolysis and decarboxylation.[218]

Cycloalkyl substituted[46,219] and benzo-annelated[220] 1,3-diselenole-2-selones are formed in moderate (10–35%) resp. good (70%) yields by thermolysis of the corresponding 1,2,3-selenadiazoles **64** in the presence of excess carbon diselenide. Thermolysis of 1,2,3-selenadiazoles **64** takes place under appreciably milder conditions (reflux in toluene, xylene, carbon diselenide) than the thermolysis of 1,2,3-thiadiazoles **24**. In the presence of potassium *t*-butoxide thermolysis of the unsubstituted 1,2,3-selenadiazole **64** already occurs at 0 °C and gives 1,3-diselenole-2-selone in 40% yield.[203]

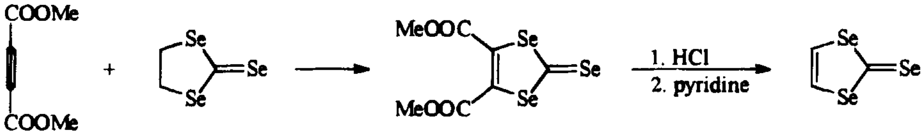
Benzo-annelated[221] and heterocyclo substituted[80–82] 1,3-diselenole-2-selones are prepared in analogy to the corresponding sulfur compounds[79–82] from the 1,2-dibromo derivatives **82**. These are lithiated with *t*-butyllithium or via an *o*-phenylenemercury compound[222] with lithium, then treated with selenium powder; subsequently the corresponding 1,3-diselenole-



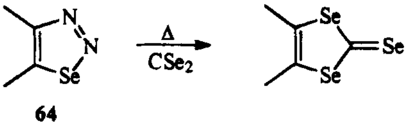
SCHEME 58



SCHEME 59

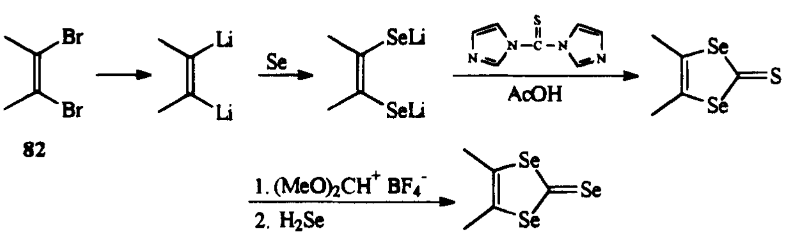


SCHEME 60



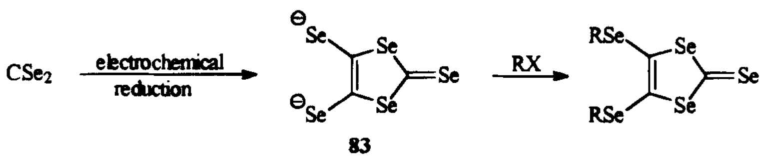
SCHEME 61

2-thione is formed with thiocarbonyldiimidazole in glacial acetic acid/tetrahydrofuran. By alkylation of the thione and reaction with hydrogen selenide the 1,3-diselenole-2-selone is obtained.



SCHEME 62

Electrochemical reduction of carbon diselenide at 40 °C provides the 4,5-diselenolate-1,3-diselenole-2-selone **83**, which can be alkylated with alkylating agents.[195] The same product is also afforded by reduction with potassium naphthalenide in tetrahydrofuran.[195]



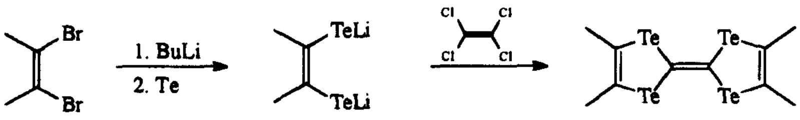
SCHEME 63

The 4,5-diselenolate-1,3-diselenole-2-selone **83** is formed not only by electrochemical reduction of carbon diselenide but also by reduction of carbon diselenide with sodium metal in organic solvents. 4,5-Diselenolate-1,3-diselenole-2-selone is isolated as bis(tetraphenylphosphonium) bis(2-selenoxo-1,3-diselenole-4,5-diselenolato)zincate(II) in a yield of 15% in diglyme and 2% in dimethyl sulfoxide.[223] This complex is also formed by reaction of 1,3-diselenole-2-selone with LDA and selenium and subsequent treatment with zinc chloride and tetraphenylphosphonium bromide.[224]

4. SYNTHESIS OF TETRATELLURAFULVALENES (TTeF)

Tetratellurafulvalenes can be synthesized in the following way: 1,2-Dibromo derivatives **82** are lithiated with *t*-butyllithium, then treated with tellurium powder and finally with tetrachloroethene.[81,82,225-227] Yields vary between 10 and 75%.

The synthesis of TTeF has been improved by the development of a five-step, one pot reaction starting from *cis*-1,2-bis(trimethylstannyl)ethene.[228,229] After two consecutive metallations the 1,2-(dilithioditelluro)ethene formed (trapping with *cis*-Pt$(PPh_3)_2Cl_2$ gives

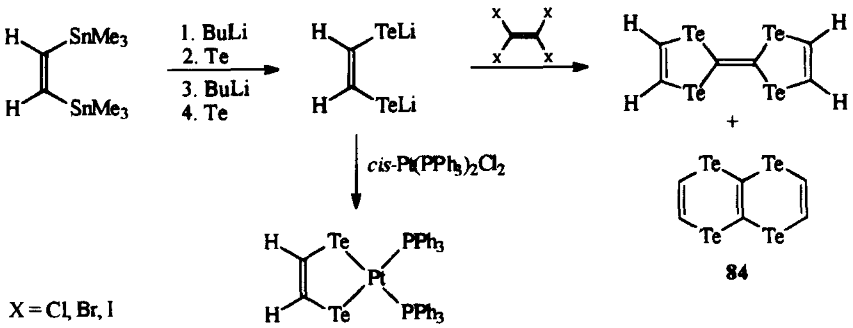


82

SCHEME 64

$\mathrm{Pt(Te_2C_2H_2)(PPh_3)_2}$ in 28% yield) was treated not with tetrachloroethene,[228] but with tetrabromoethene.[229] This greatly improves the ease of isolation, lowers the yield of undesirable by-products, and increases the purified yield of TTeF to 12% (tetrachloroethene gives a 5% yield of pure TTeF and tetraiodoethene <1% yield of pure TTeF).

One by-product in the reaction of 1,2(dilithioditelluro)ethenes with tetrachloroethene is the corresponding 1,4,5,8-tetratelluratetralin **84**.[228,230]



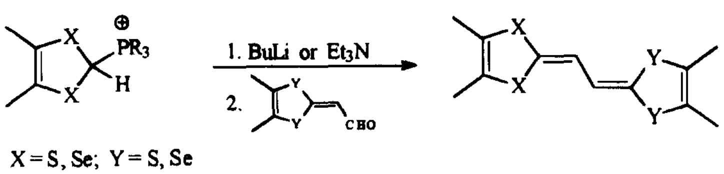
84

SCHEME 65

TTeF and their data are summarized in Table 14.

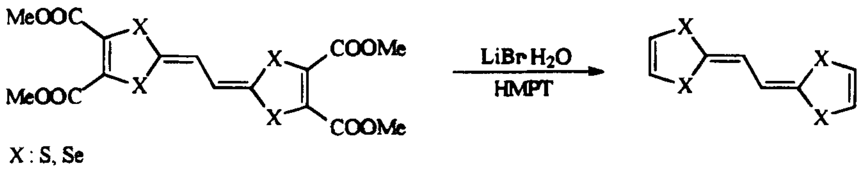
5. SYNTHESIS OF CONJUGATED TETRATHIA- AND TETRASELENAFULVALENES

Essentially the synthesis of the ethanediylidene derivatives is based on the Wittig-type reaction of a phosphorane with the appropriate aldehyde.[117,231]



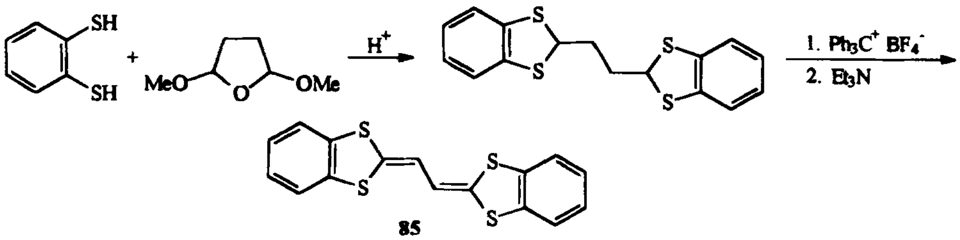
SCHEME 66

By this reaction it is possible to obtain unsymmetrically substituted derivatives. The unsubstituted compound is formed by decarbomethoxylation of the corresponding tetraester with lithium bromide monohydrate in hexamethylphosphoramide at 160 °C.[117,231]



SCHEME 67

The benzo-annelated compound **85** is prepared by treatment of 1,2-benzodithiole with 2,5-dimethoxytetrahydrofuran in acidic solution, followed by hydride abstraction and deprotonation.[232,233]



SCHEME 68

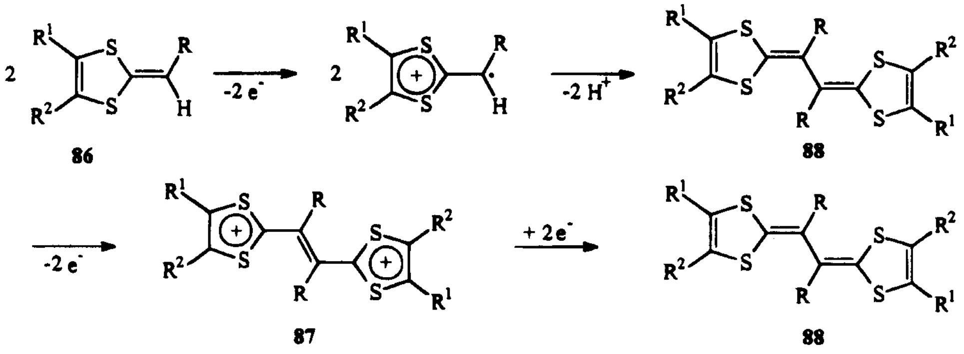
The oxidative dimerization of 1,4-dithiafulvenes **86** with *p*-benzoquinones or electrochemically provides the corresponding dications **87**, which can be reduced to the ethanediylidene derivatives **88** with zinc dust, with mercury or electrochemically. The first step in this reaction is the formation of the radical cation with subsequent dimerization to the dimer **88**. The dimer **88** has a lower oxidation potential than the 1,4-dithiafulvene **86** and is oxidized to the dication **87**. The dication **87** can be reduced to the ethanediylidene derivatives **88**.[234–239]

Some TTF analogs have been synthesized with two or more 1,3-dithiol-2-ylidene moieties separated by olefinic bonds.[240,241]

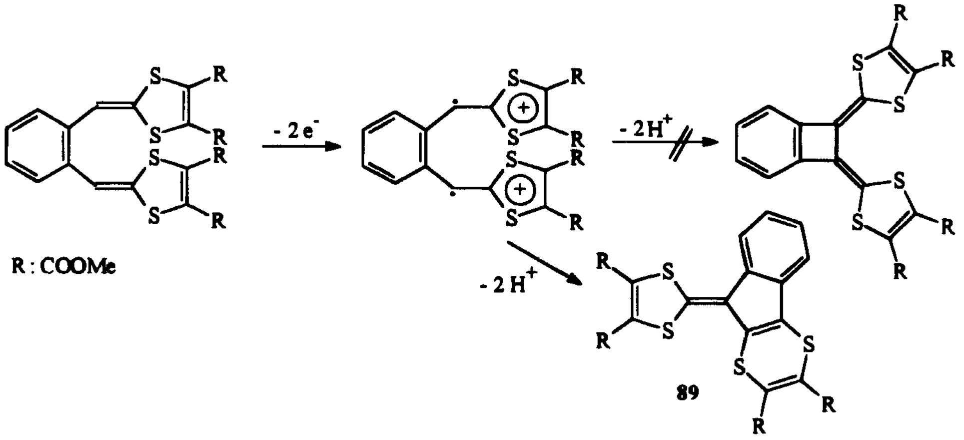
Bromination of 1,2-xylene-α,α′-diylidenebis(4,5-dicarbomethoxy-1,3-dithiole) with bromine in carbon tetrachloride does not lead analogously to oxidation of diphenyldithiafulvene **86** to a benzocyclobutene-derived molecule, but provides the dithiinofulvenetetraester **89**.[242]

Oxidation of bis(methylthio)acetylene with bromine leads to the dication of the corresponding ethanediylidene-2,2′-bis(1,3-dithiole) which can be reduced with zinc to the parent compound.[243]

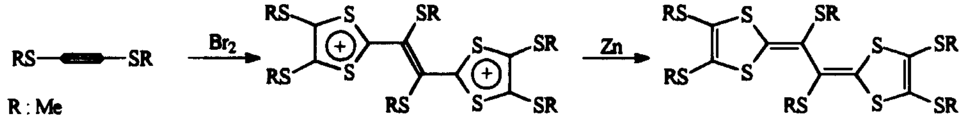
Starting from ethanediylidene-2,2′-bis(1,3-dithioles) [5]- and [6]-radialene **90** and **91** (R = COOMe, $(CH{=}CH)_2$) are obtained in reasonable yields by bromination with *N*-bro-



SCHEME 69



SCHEME 70

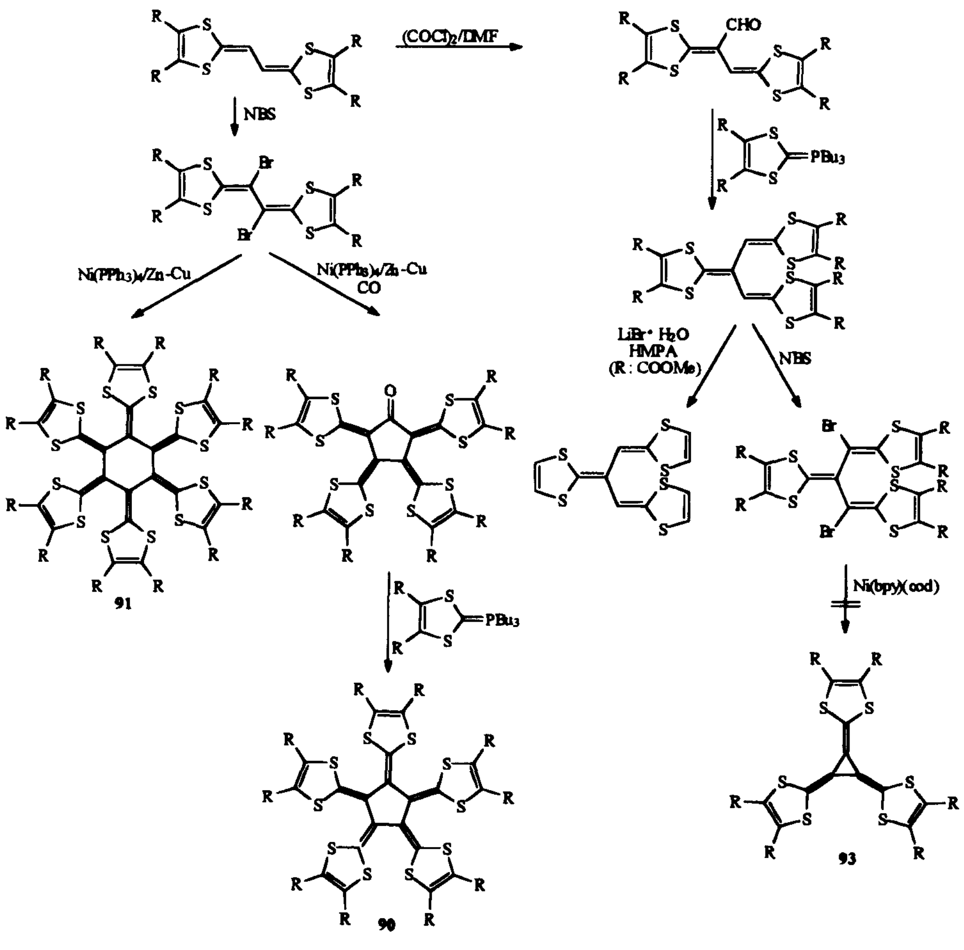


SCHEME 71

mosuccinimide, followed by treatment with a $Ni(PPh_3)_4$ complex and Zn-Cu coupling in the presence or absence of carbon monoxide, respectively.[244–246] Reaction of the substituted tris(1,3-dithiol-2-ylidene)propane and tetrakis(1,3-dithiol-2-ylidene)butane (R = COOMe) with $LiBr \cdot H_2O$ in HMPA afforded the unsubstituted derivatives, both in 80% yield (Schemes 72 and 73).[247]

The [4]-Radialene **92** (R = COOMe) is obtained in very low yield (3%) from ethanediylidene-2,2′-bis(1,3-dithioles) by two consecutive Vilsmeier reactions with oxalyl chloride/DMF and Wittig reactions with a phosphorane, then bromination with *N*-bromosuccinimide and treatment with a zerovalent Ni(bpy)(cod) complex.[247] The [3]-Radialene **93** (R = COOMe, $(CH{=}CH)_2$) has not yet been prepared in this way.[247]

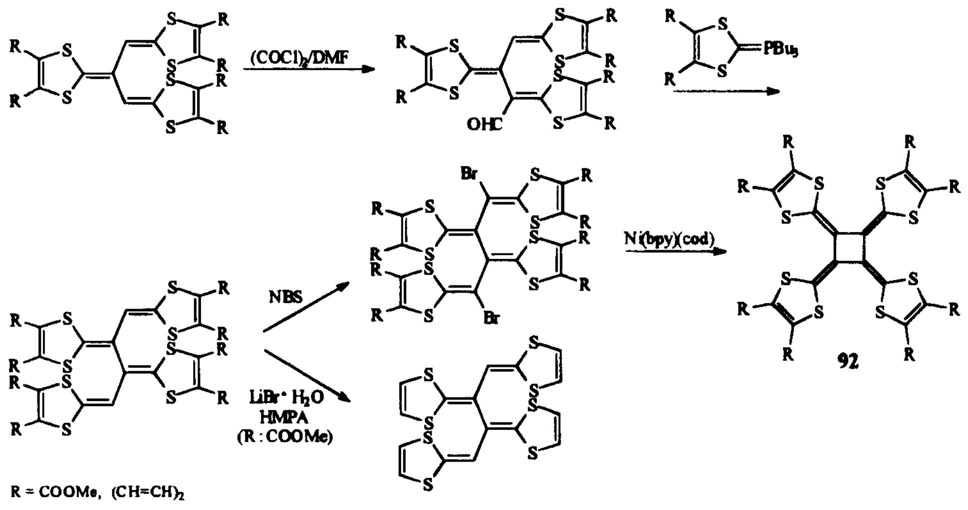
X-Ray structure analyses of the [4]-, [5]- and [6]-radialenes indicated not a planar, but rather a three-dimensional structure.[244]



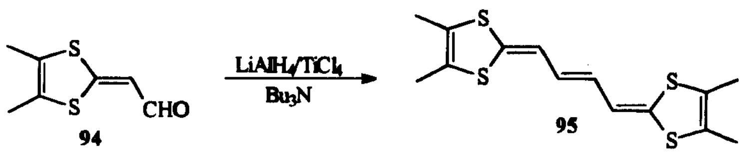
SCHEME 72

The unsubstituted and the benzo-annelated compound in the series of 1,4-butenediylidene derivatives **95** are obtained by reduction of the corresponding 2-formylmethylene-1,3-dithiole **94** with lithium aluminium hydride in the presence of titanium tetrachloride and tributylamine in tetrahydrofuran.[248]

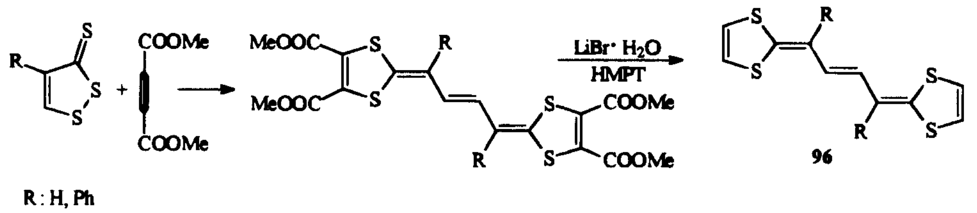
The unsubstituted compound **96** (R = H) is also formed by treatment of 1,2-dithiole-3-thione with dimethyl acetylenedicarboxylate in benzene, followed by decarbomethoxylation with lithium bromide monohydrate in hexamethylphosphoramide.[248] In the same way substituted derivatives can be obtained by reaction of substituted 1,2-dithiole-3-thiones with acetylenedicarbaldehyde.[249,250]



SCHEME 73



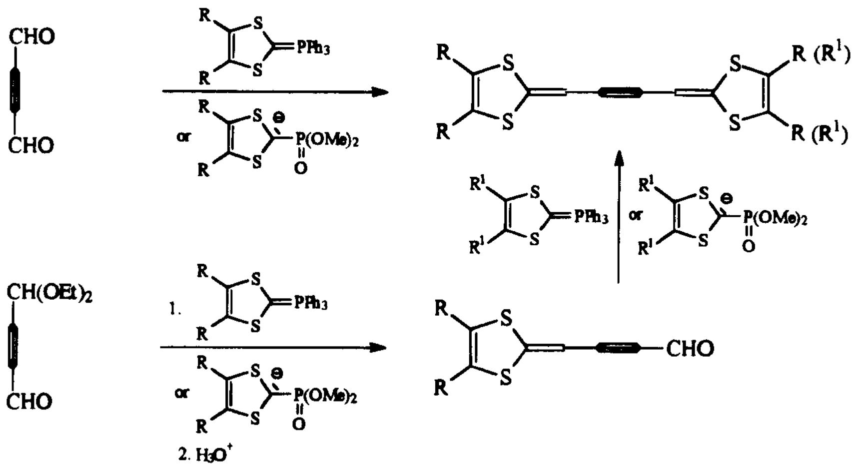
SCHEME 74



SCHEME 75

An extension of the π-system between the two 1,3-dithiol-2-ylidene moieties can also be achieved with olefinic and acetylenic bonds.

The Wittig or Wittig-Horner reaction of butynedial with phosphoranes or phosphonate anions, respectively, generates the symmetrical acetylenic analogs of TTF.[251,252] From the mono(diethyl acetal) the unsymmetrical acetylenic analogs of TTF can be obtained. The yields are increased when complexes of the dialdehyde or the mono(diethyl acetal) with dicobalthexacarbonyl are used as starting material instead of the corresponding free alkynes. The final decomplexation is achieved by treatment with trimethylamine oxide.[253]



R = H, Me, $(CH_2)_3$, $(CH_2)_4$, $(CH=CH)_2$, COOMe
R^1 = H, Me, $(CH_2)_3$, $(CH_2)_4$, $(CH=CH)_2$

SCHEME 76

A cumulene skeleton has been inserted between the 1,3-dithioles in **97** and **98**. These TTF derivatives are formed by treatment of 2-morpholino-1,3-dithiolium salts with (trimethylsilyl)ethynyl magnesium bromide, followed by hydrolytic removal of the trimethylsilyl group. The dithiole thus obtained can be coupled with the starting 2-morpholino-1,3-dithiolium salts or dimerized in the presence of copper (II) acetate. Removal of the morpholino substituent by perchloric acid provides the cumulenic TTF.[234] So far only the dications **97** and **98** could be isolated in fairly good yields (70–90%). The neutral form is extremely unstable.

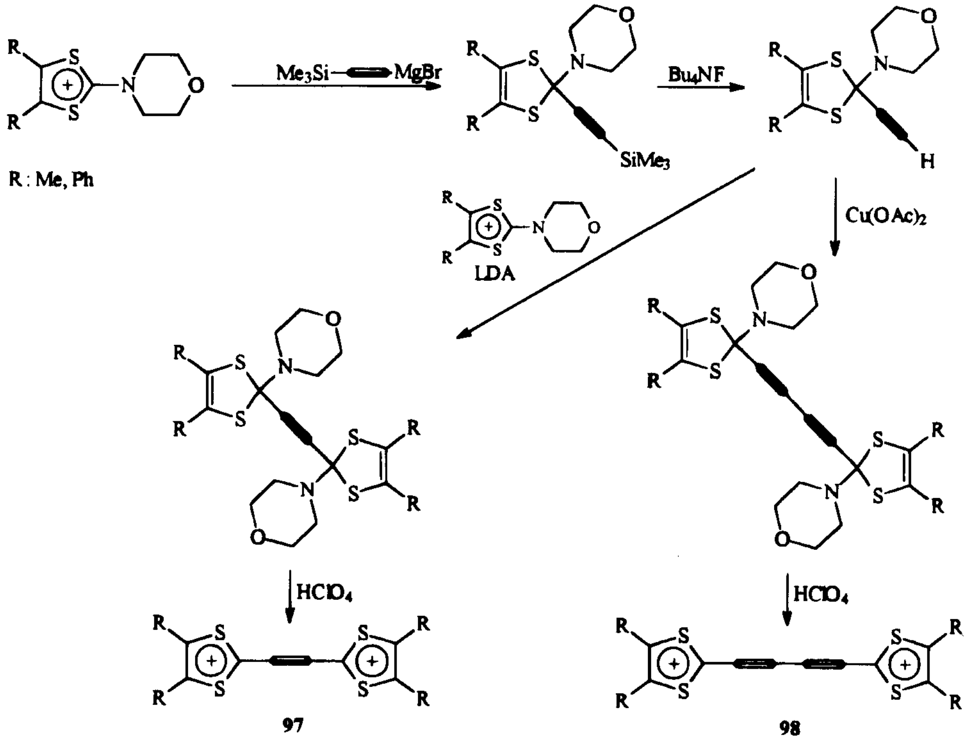
Both positive charges are more located in the two 1,3-dithiolium units than in the corresponding ethanediylidene units. Probably π-conjugation between the 1,3-dithiolium units is less effective through an acetylenic than through an olefinic bond.

Via a Wittig or Wittig-Horner reaction, TTF derivatives are obtained with double bonds of olefinic and aromatic character between the two 1,3-dithiol-2-ylidene moieties in yields of 40–90%.[255]

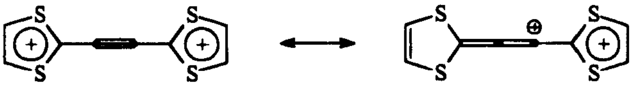
The *p*-derivative **99** with n = 2 (R = $(CH=CH)_2$) is also formed by reaction of benzene-1,2-dithiole with *p*-phenylene diacetaldehyde, subsequent hydride abstraction with trityl tetrafluoroborate and deprotonation with triethylamine.[241]

Conjugated TTF derivatives with incorporated five-membered heterocycles are synthesized by Wittig or Wittig-Horner reaction of the corresponding heterocyclic dicarbaldehyde with phosphoranes or phosphonate anions, respectively.[235,256,257]

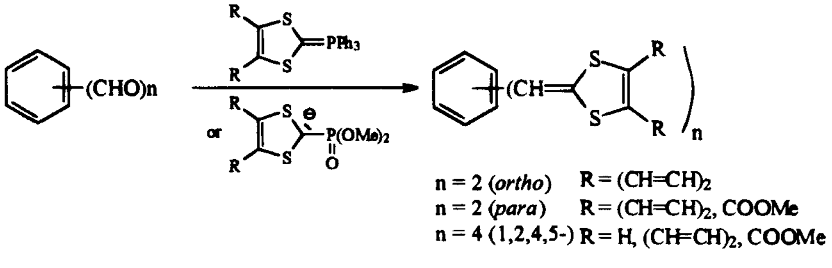
The 1,3-dithiolylidene units have also been connected by a *p*-benzoquinoid system, but these compounds are rather unstable. Both the tetramethyl[258] and the diphenyl derivative[259] **100** ($R^1 = R^2$ = Me resp. R^1 = Ph, R^2 = H) could be isolated as dications. Polarographic reduction or reduction with zinc dust only lead to unstable products. With lithium iodide the



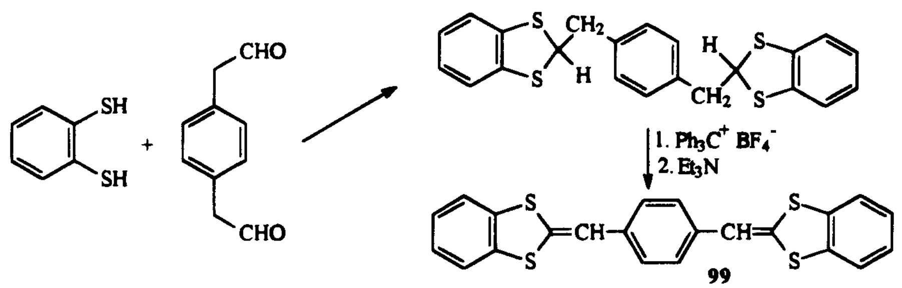
SCHEME 77



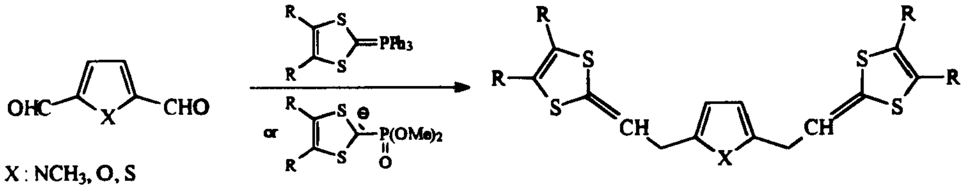
SCHEME 78



SCHEME 79

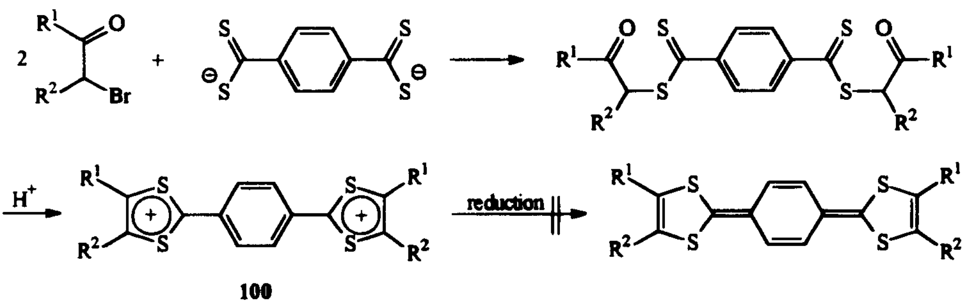


SCHEME 80



SCHEME 81

dication of the diphenyl derivative could be converted into a stable iodine complex.[259] In the same way a coupling to a 2,6-naphthoquinoid system was also successful.[260,261]

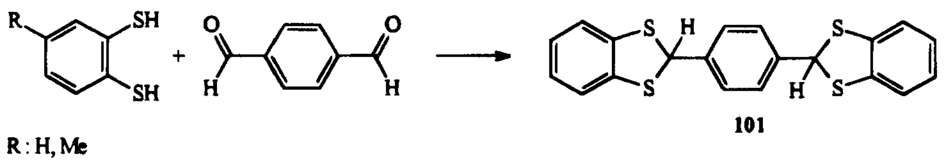


$R^1 = R^2$: H, Me
R^1: Ph R^2 : H

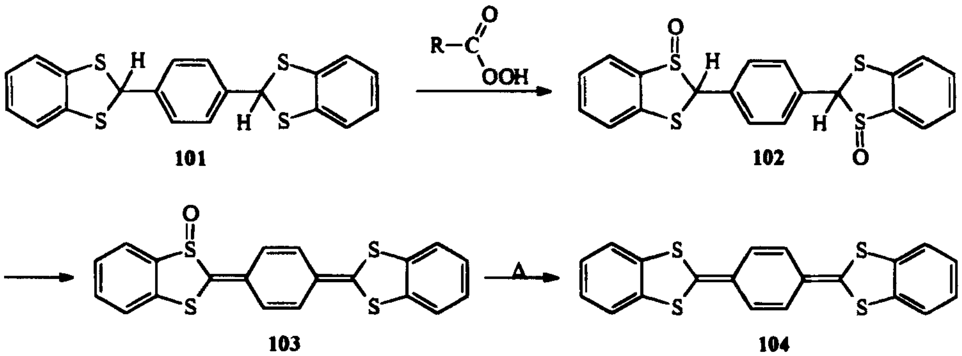
SCHEME 82

Benzo-annelated derivatives are formed by reaction of the corresponding 1,2-dimercapto compounds with terephthaldehyde.

The unsubstituted derivative **101** (R = H) could not be dehydrogenated with chloranil or 2,3-dichloro-5,6-dicyano-*p*-benzoquinone.[262,263] Instead an unusual method is used. The dihydro compound **101** is oxidized with *m*-chloroperbenzoic acid to the disulfoxide **102**, converted to the monosulfoxide **103** with diazabicyclononene and pyridine, the pyrolysis of which gives cyclohexa-2,5-diene-1,4-diylidenebis-1,3-benzodithiole **104**.[262,263]

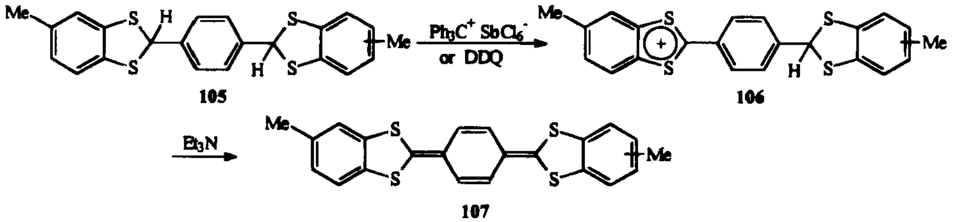


SCHEME 83



SCHEME 84

The dimethyl derivative **107** could be synthesized by treatment of the corresponding dihydro compound **105** with trityl hexachloroantimonate or 2,3-dichloro-5,6-dicyano-*p*-benzoquinone to form the monocation **106**, followed by deprotonation with triethylamine.[264]



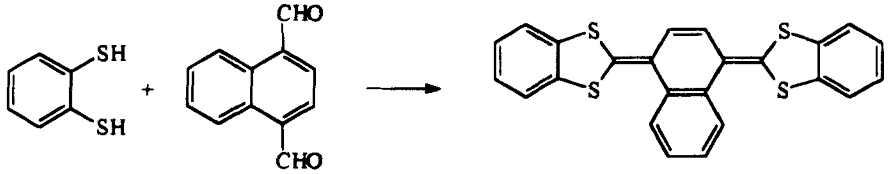
SCHEME 85

Both the unsubstituted and the dimethyl derivative **104** resp. **107** are slightly air sensitive and decompose. In the same way the 1,4-naphthoquinoid compound **108** is formed.[285]

For the unsubstituted compound **100** ($R^1 = R^2 = H$) no details have been reported.[266] Somewhat later the unsubstituted quinodimethane analog of TTF could be prepared by a retro-Diels-Alder reaction of the corresponding cyclopentadiene adduct in 54% yield.[269]

Pyrazine fused derivatives **109** are quantitatively obtained by this retro-Diels-Alder reaction of cyclopentadiene adducts which were synthesized described in Scheme 88.[267,268]

Also other benzo- and naphtho-condensed quinodimethane analogues of TTF can be obtained by this retro-Diels-Alder reaction.[269]



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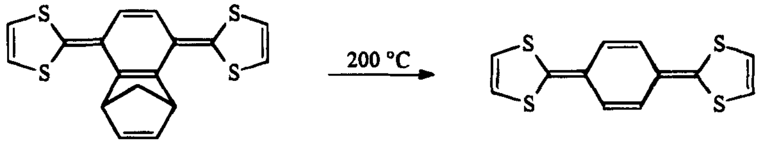
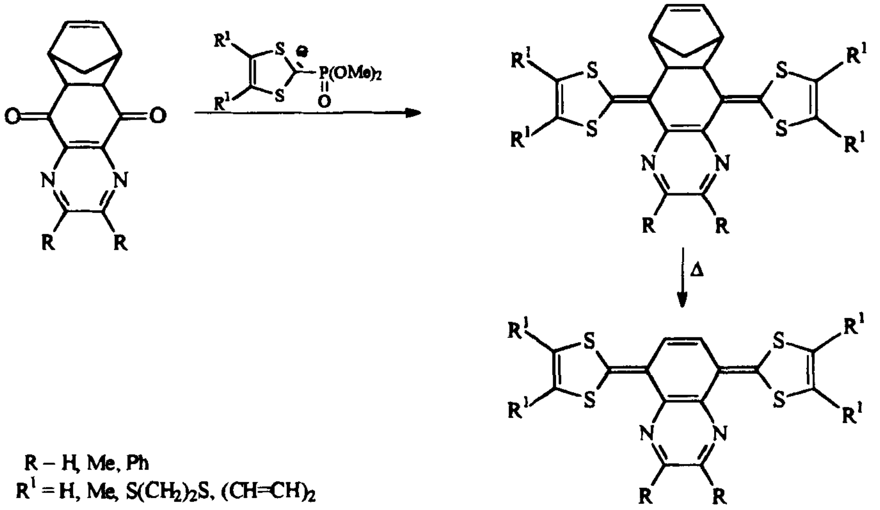
SCHEME 86



SCHEME 87



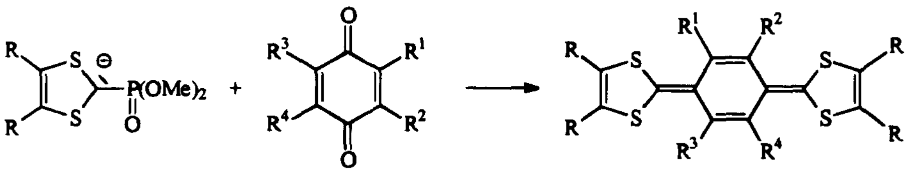
109

SCHEME 88

Quinodimethane analogs **110** of TTF have been synthesized by Wittig or Wittig-Horner reaction of quinones with phosphoranes or phosphonate anions, respectively. A number of annelated or heterocyclic fused quinoid structures have also been prepared in this way.[270–273]

The Wittig-Horner reaction is also successful in the case of treatment of the (1,3-diselenol-2-yl)phosphonate anion **111** with the ketone **112** and provides, in quantitative yield, the vinylogous DSDTF **113**.[274]

Asymmetrically substituted quinoid analogs of TTF **114** have been obtained (yield 55–70%) by treatment of 2-(alkylthio)-1,3-dithiolium salts with anthrone under basic conditions, followed by Wittig-Horner reaction with phosphonate anions.



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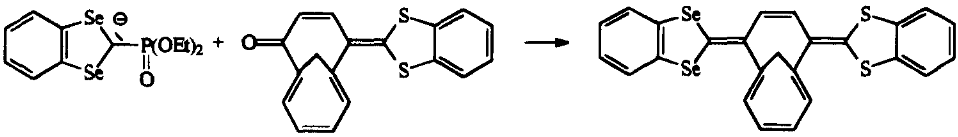
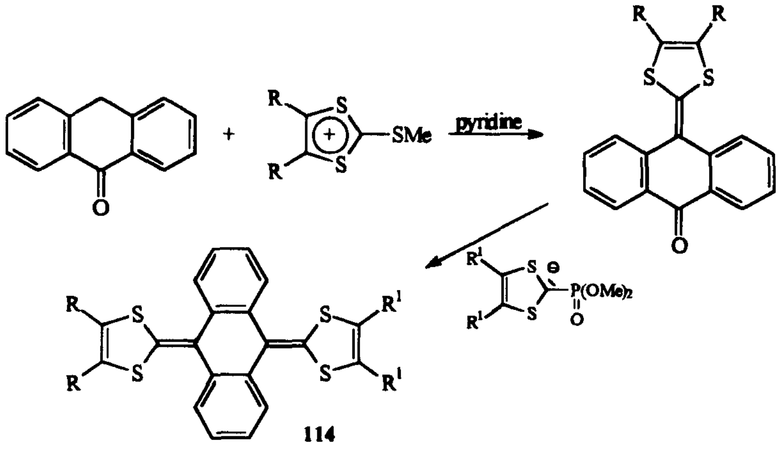
SCHEME 89



111 112 113

SCHEME 90



114

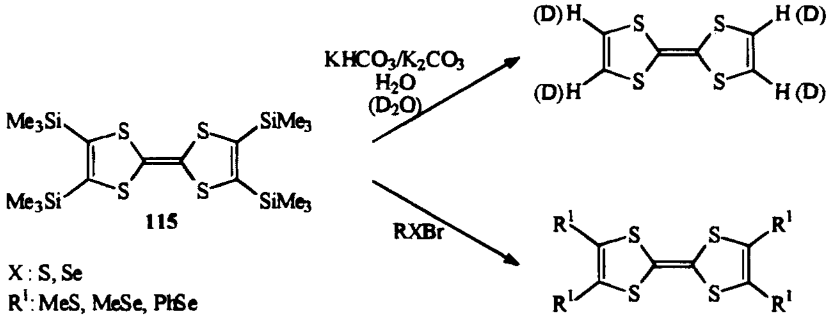
SCHEME 91

Use of a 1,3-thiaselenolephosphonate anion as a starting material afforded selenatrithiafulvalenes.[275] Vinylogous, cumulenic, acetylenic, quinoid TTF and TSF and their data are summarized in Table 15.

6. PROPERTIES OF TETRACHALCOGENAFULVALENES

6.1. *Reactions of Tetrathia- and Tetraselenafulvalenes*

The tetrathia- and tetraselenafulvalene moiety is rather stable under basic and weakly acidic conditions so that a conversion of substituents can be accomplished without destroying the tetrathia- and tetraselenafulvalene structure. In ethanol/tetrahydrofuran (1:1) at room temperature with an aqueous buffer containing potassium carbonate and potassium hydrogen carbonate the trimethylsilyl groups of tetrakis(trimethylsilyl)tetrathiafulvalene **115** are readily replaced by hydrogen. Deuterated solvents (D_2O, EtOD) under the same conditions afford deuterated tetrathiafulvalene. With sulfenyl or selenyl bromides the corresponding tetrakis(organylthio) or tetrakis(organylseleno)tetrathiafulvalenes are obtained.[134]



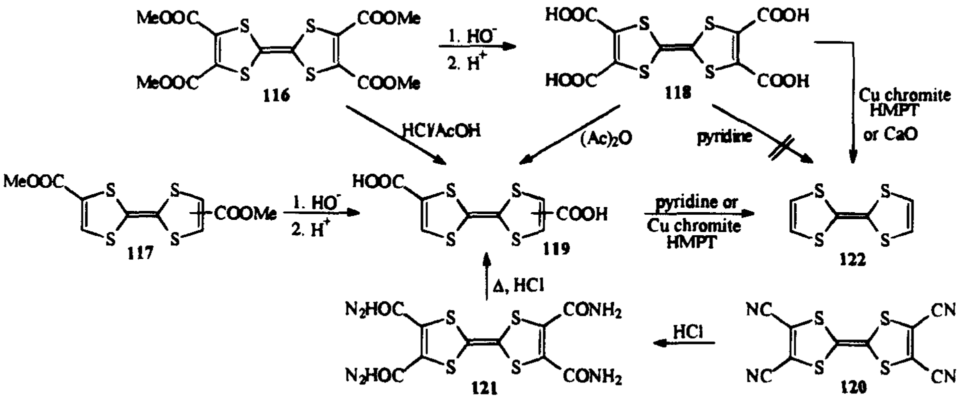
SCHEME 92

The tetrathiafulvalene tetraester **116** and the diester **117** can be hydrolyzed in aqueous or ethanolic alkaline solution[53,100,101,135] as well as in half-concentrated hydrochloric acid/glacial acetic acid[101] to the corresponding tetraacid **118** or diacid **119**, respectively, where the hydrolysis is accompanied by decarboxylation under acidic conditions. Hydrolysis of tetracyanotetrathiafulvalene **120** in concentrated hydrochloric acid at room temperature gives tetrathiafulvalene-tetracarboxamide **121**. Hydrolysis with reflux does not provide the amide **121**, but generates the diacid **119** by simultaneous decarboxylation.[101] The diacid **119** is also formed upon heating of the tetraacid **118** in acetic anhydride.[101] The diacid **119** decarboxylates in pyridine at 250 °C in a sealed tube to give the parent tetrathiafulvalene **122**.[53,101] Under the same conditions the tetraacid **118** does not decarboxylate.[101] The decarboxylation of the diacid **119** as well as of the tetraacid **118** in hexamethylphosphoramide at 150 °C in the presence of copper chromite gives the parent tetrathiafulvalene **122**. Also copper(II) sulfate or copper powder in quinoline induces the decarboxylation of acid derivatives.[278] The tetraacid **118** can be decarboxylated by heating at 300 °C in the presence of calcium oxide.[135]

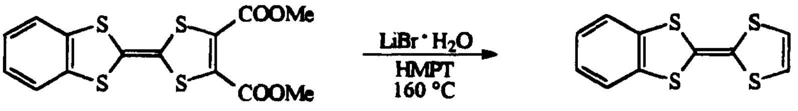
Hydrolysis and decarboxylation can be performed in one step by treatment of the corresponding methyl ester with lithium bromide monohydrate in hexamethylphosphoramide.[123,170]

Under the same conditions the *sym*-diselenadithiafulvalene **123**[185] and tetraselenafulvalene ester **124**[277] decarbomethoxylate, too. The *sym*-diselenadithiafulvalene **125** is also formed by hydrolysis of *sym*-diselenadithiafulvalenetetracarboxylic acid tetraester **123** in alkaline solution, followed by decarboxylation in hexamethylphosphoramide in the presence of copper bronze.[185,191]

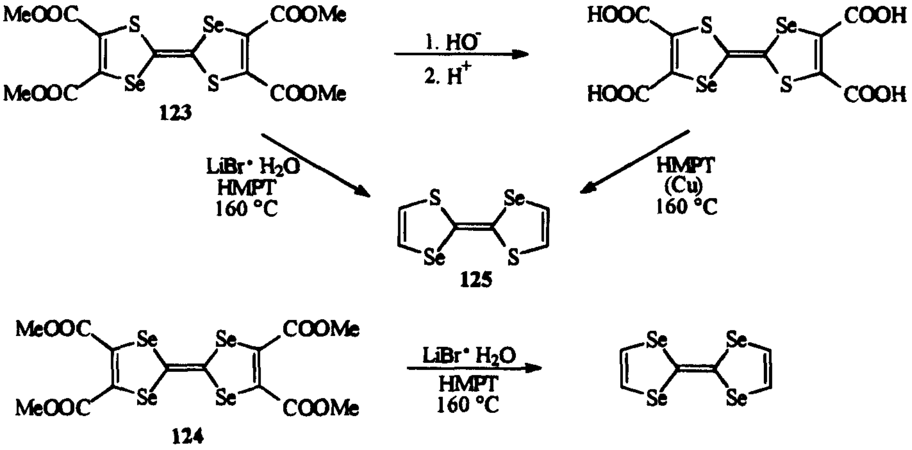
For the hydrolysis of the substituted aryltetrathiafulvalenes **126** hydrazine in methanol is a convenient reagent.[100,278] After treatment with long-chain acid chlorides in pyridine the



SCHEME 93



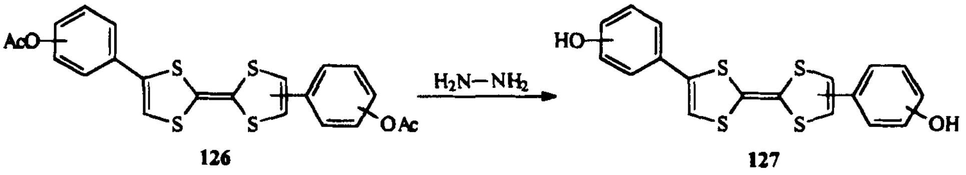
SCHEME 94



SCHEME 95

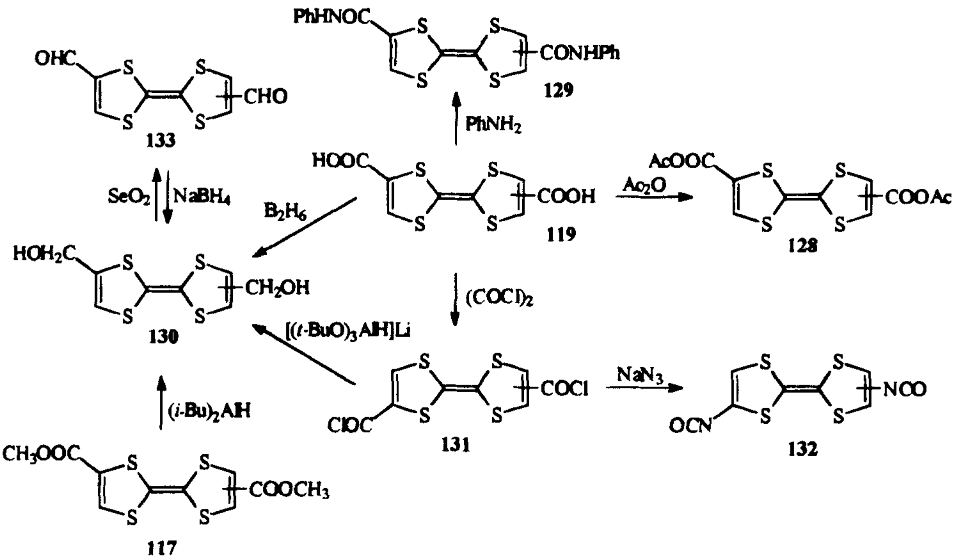
2,6(7)-bis(4-hydroxyphenyl)tetrathiafulvalene **127** constitutes a TTF with liquid crystal properties.[279,280]

The tetrathiafulvalene diacid **119** can be converted to various other acid derivatives. With acetic anhydride in tetrahydrofuran the bis-anhydride **128** is formed,[100] with aniline in *N,N*-dimethylformamide at 70 °C, with diphenyl phosphite-pyridine as dehydrating reagent, the bis-anilide **129**,[100] and with diborane in diglyme the bis(hydroxymethyl)tetrathiafulvalene **130**[100] is obtained. The acid function is converted to the acid chloride **131** with oxalyl chloride in methylene chloride[281] or in benzene/acetonitrile.[282] The tetrathiafulvalenedicarbonyl



SCHEME 96

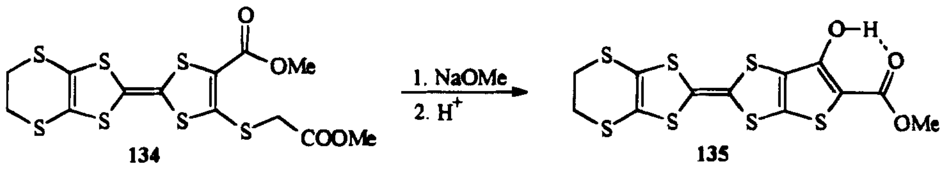
chlorides **131** yield, with activated sodium azide in benzene or toluene, in a Curtius reaction, the corresponding isocyanates **132**.[281,282] Tetrathiafulvalenedicarbonyl chlorides **131** are reduced with lithium tri-*t*-butoxyaluminium hydride in tetrahydrofuran to bis(hydroxymethyl)tetrathiafulvalenes **130**.[281] With diisobutylaluminium hydride in methylene chloride or tetrahydrofuran at lower temperatures or with sodium borohydride/zinc chloride the ester group in tetrathiafulvalenes **117** is reduced to the alcohol group. The aldehyde function can be reduced to the alcohol group by use of sodium borohydride.[283-285] The dialcohol **130** or monoalcohols are oxidized with selenium dioxide in refluxing dioxane to the corresponding aldehydes **133**.[286,287]



SCHEME 97

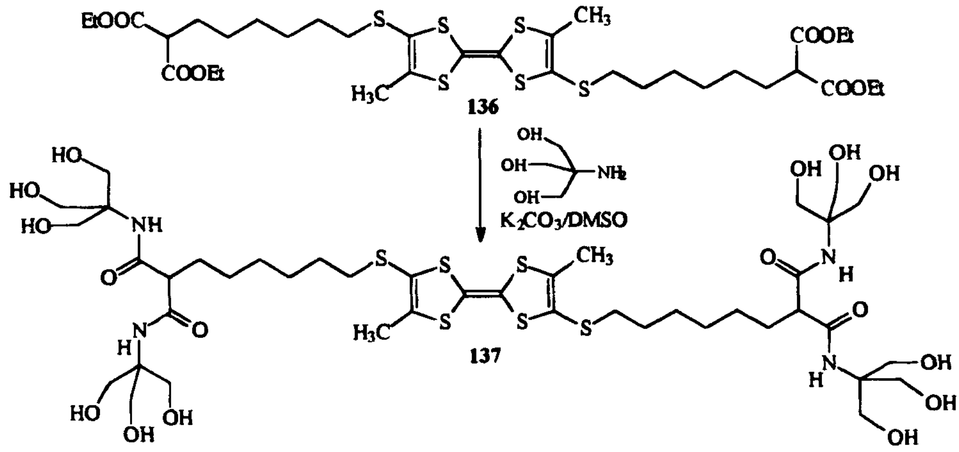
Reaction of sodium methoxide with a TTF derivative **134**, substituted by a methoxycarbonylmethylthio group and a neighbouring methoxycarbonyl group, yields by cyclization a [2,3-*d*]thieno-condensed TTF **135**.[288]

Amidation of a long chain substituted TTF ester **136** with tris(hydroxymethyl)-aminomethane provides a new TTF derivative **137** which forms a gel. Structure investigations prove string-like superstructures with lengths of the order of microns and diameters ranging from about 30 to several hundred nanometers. In connection with the



SCHEME 98

conductivity properties of the TTF moiety, such a compound could be a candidate for "molecular" wires.[289]

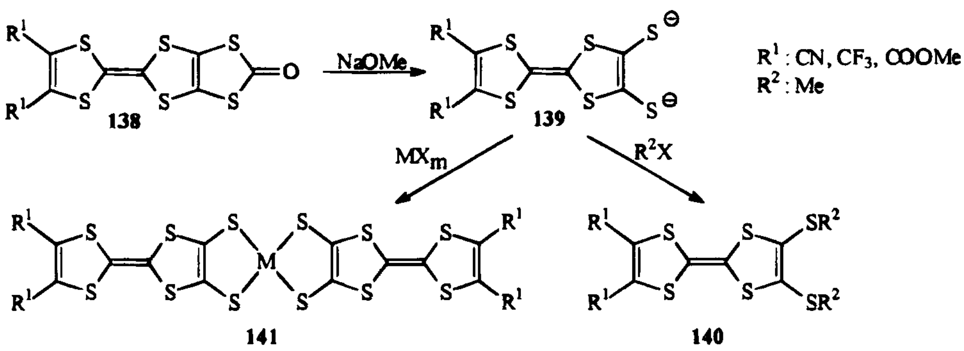


SCHEME 99

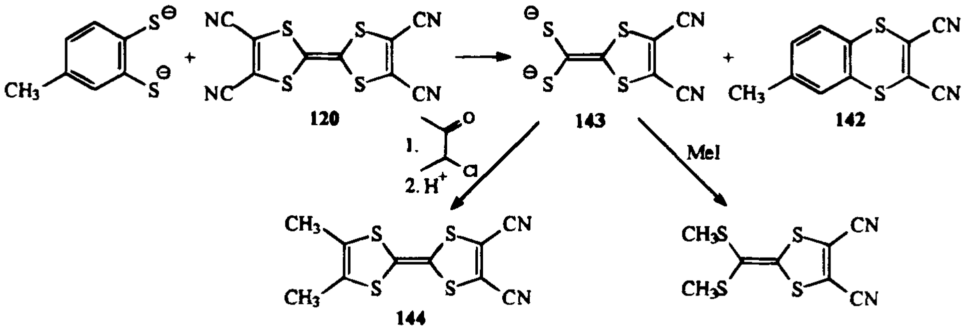
1,3-Dithiol-2-ono-annelated TTF **138** are degraded by strong bases like sodium methoxide or methyllithium.[109,290,291] The dithiolate salts **139** so formed can be converted with alkylating agents to different substituted tetrathiafulvalenes **140**[291] and with transition metal salts to the corresponding metal dithiolene complexes **141**.[292]

In the same manner bis(1,3-dithiol-2-oxo)tetrathiafulvalene[59,63,292-294] and tetrakis(benzoylthio)tetrathiafulvalene[295] are cleaved. The air-sensitive tetrathiolate salt thus obtained affords, upon addition of alkylating agents, the corresponding TTF[59,293,296] and with transition metal salts the polymeric dithiolene complexes.[63,292,294] When only two equivalents of base are used and subsequently treated with methyl iodide, the half-opened bis(methylthio) derivative is formed.[293]

The nucleophilic attack of the salt of 3,4-toluenedithiol upon tetracyanotetrathiafulvalene **120** provides 2,3-dicyano-5,6-toluo-1,4-dithiene **142** and the air-sensitive geminal dithiolate intermediate **143** which can be converted with methyl iodide or 3-chloro-2-butanone and subsequently with concentrated sulfuric acid to a new tetrathiafulvalene derivative **144**. Also alkane-, arene-, acylthiols, -selenol and -tellurol salts react with tetracyanotetrathiafulvalene **120** in analogous manner to give the geminal dithiolate intermediate **143**. Tetracyanotetraselenafulvalene reacts in a similar fashion.[297]



SCHEME 100



SCHEME 101

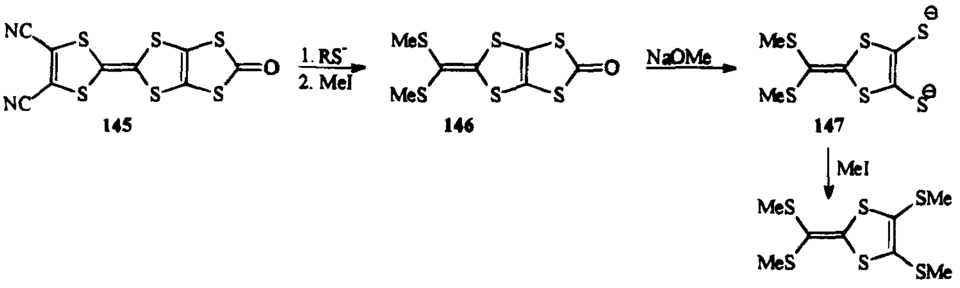
The reaction of the unsymmetrically substituted tetrathiafulvalene **145** with thiolates leads to attack preferentially at the cyano substituted double bond. After alkylation with methyl iodide **146** is formed which can be cleaved at the dicarbonate group with a stronger base (NaOMe, MeLi) to give a 1,2-dithiolate intermediate **147** which is then alkylated.[297]

Sodium hydroxide in DMSO hydrolyzes the 1,3-dithiol-2-one system also in the benzo-annelated TTF **148**.[298,299] The tetrathiolate formed can be alkylated with alkyl iodides.

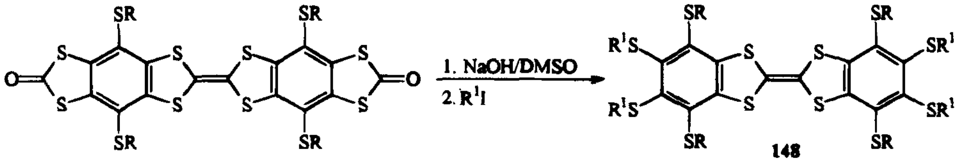
Refluxing of the thiazoloethylenedioxo-TTF **149** in alkaline solution, followed by heating of the acid in DMF with decarboxylation and simultaneous rearrangement, provides the isothiazoloethylenedioxo-TTF **150**.[300]

Halides or tosyl chloride can also be used as nucleophilic agents, so that substituents can be modified without conversion of the TTF moiety.[301,302] Hydrolysis of tetrakis(benzoyloxyethylthio)-TTF **151** takes place in alkaline solution to form tetrakis(hydroxyethylthio)-TTF **152**. This compound reacts with tosyl chloride to the corresponding tosylate **153**, which can be further converted with sodium halides and sodium methanethiolate to the tetrakis(haloethylthio)-TTF **154** and tetrakis(methylthioethylthio)-TTF **155**, respectively, in almost quantitative yield.

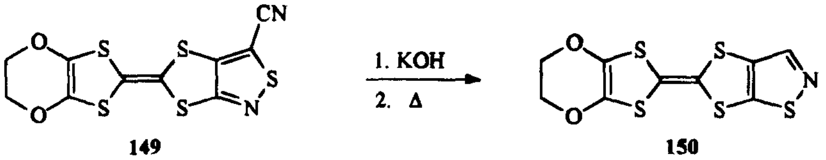
Some functional groups in 1,3-dithiol derivatives do not survive the reaction conditions in the coupling of 1,3-dithiole derivatives (e.g. hydroxy, keto or aldehyde groups). This problem can be circumvented by protection of these groups and subsequent deprotection



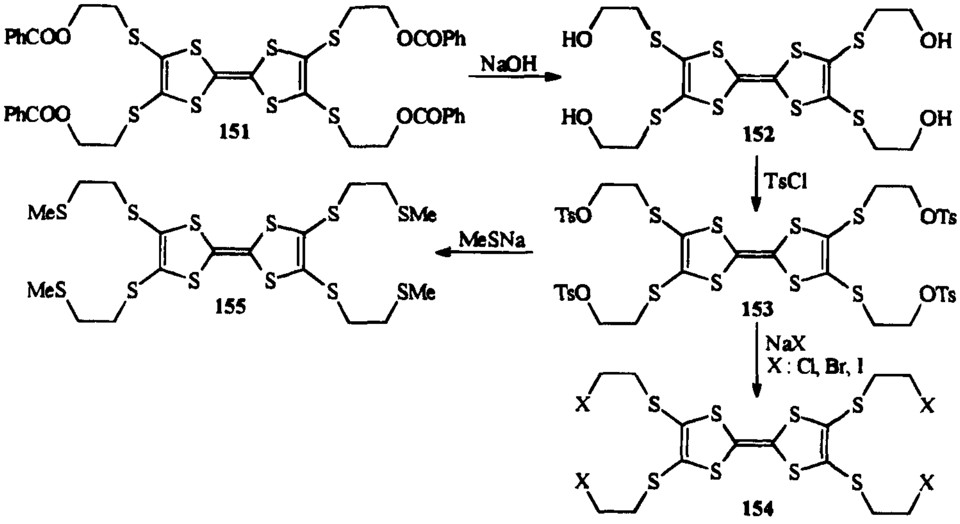
SCHEME 102



SCHEME 103

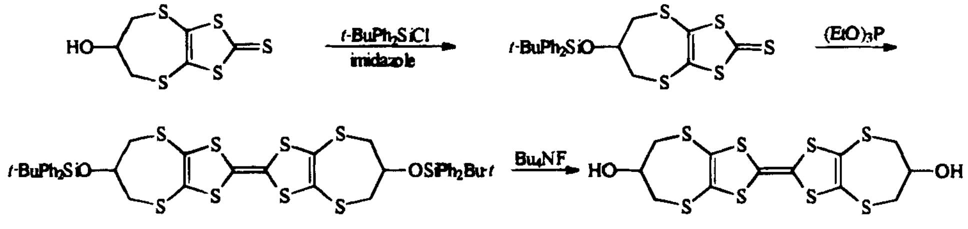


SCHEME 104



SCHEME 105

after the TTF moiety has been formed. Thus the hydroxy group is protected as a *t*-butyldiphenylsilyl ether[303,304] and the aldehyde group as an acetal.[149] After coupling the *t*-butyldiphenylsilyl protecting group is removed with fluoride ions.



SCHEME 106

Metallation of TTF with different lithiation agents (LDA-lithium diisopropylamide, LiHMDS-lithium hexamethyldisilazane, PhLi, MeLi, BuLi) provides tetrathiafulvalenyllithium **156** as a reactive intermediate.[169]

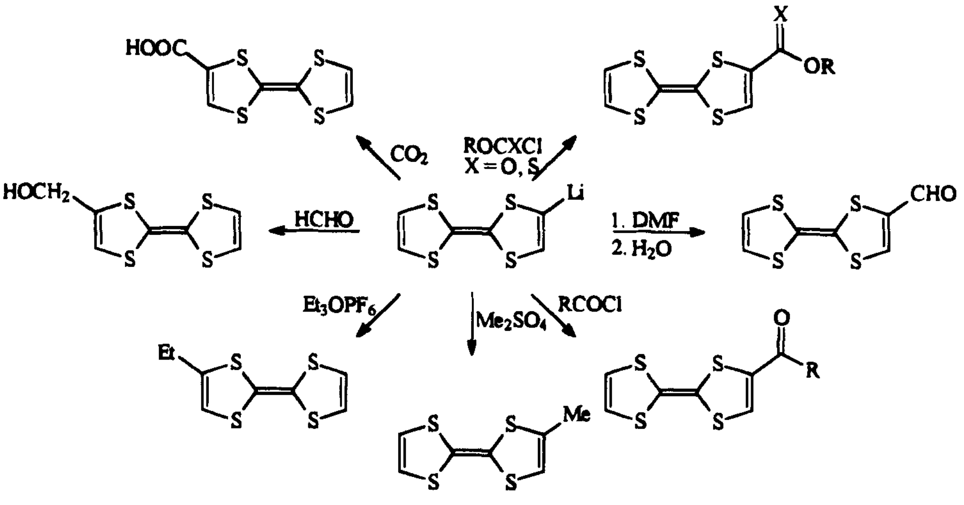
156

SCHEME 107

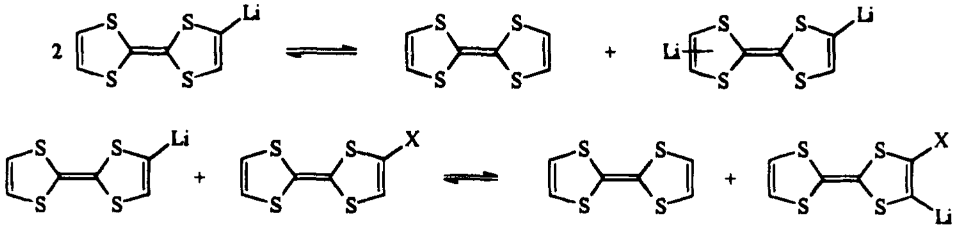
This key intermediate can be converted with electrophiles to form monosubstituted TTF (Schemes 108 and 110). This reaction constitutes a general method for the synthesis of unsymmetrical substituted TTF.[305–307]

In most cases the reaction of tetrathiafulvalenyllithium **156** with electrophiles leads to disubstituted derivatives as by-products. This is probably due to the tendency of tetrathiafulvalenyllithium to undergo disproportionation. Substituents on the TTF skeleton direct the second lithiation and hence the second substitution. Electron-donating substituents, such as lithium or methyl groups, decrease the acidity of the adjacent proton, and thereby direct substitution to the opposite ring. Halogens, formyl groups or acyl substituents as electron-withdrawing substituents increase the acidity of the adjacent proton and direct the second substitution to the same ring.[169]

The first *N,N*-dialkylaminomethyl substituted TTF **157** were obtained in yields of 35–50% by reaction of tetrathiafulvalenyllithium **156** with Eschenmoser's salts.[308] The best yields were obtained with LDA as the metallation agent. Lithiation of TTF with LDA and subsequent treatment with isocyanates or isothiocyanates yields the amido- or thioamido-TTF **158**.[307,309] The metallated TTF **156** provides with the halides PR_2X or PRX_2 or PR_3 the corresponding monomeric PR_2TTF **159**, the dimers PR(TTF)$_2$ **160** or the trimers P(TTF)$_3$ **161**.[310–313] Bridged dimeric TTF **162** can also be formed by reaction of main-group element



SCHEME 108

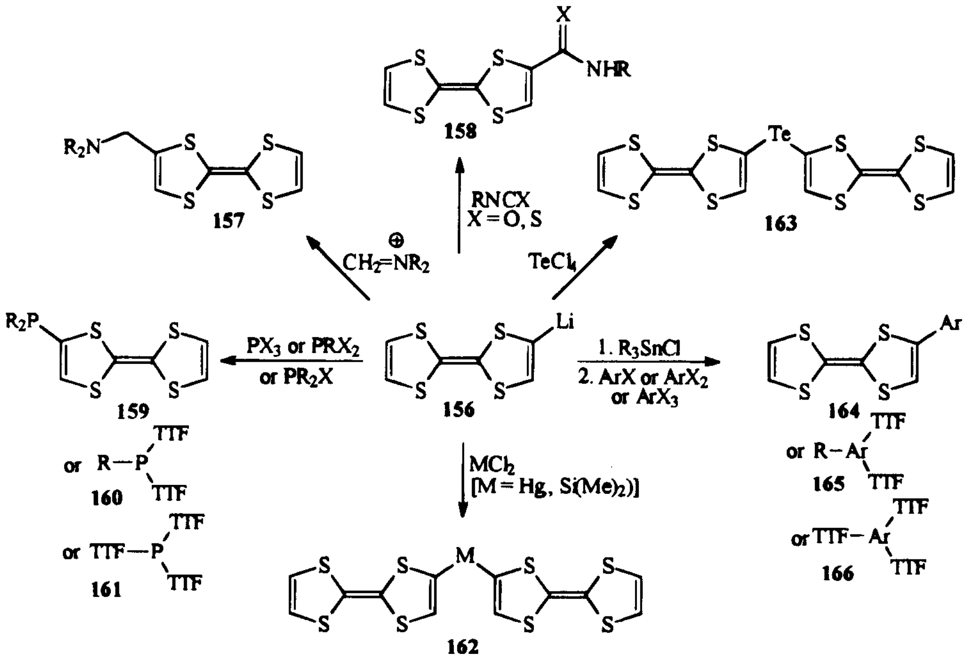


SCHEME 109

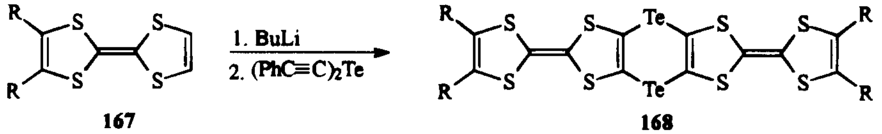
halides with lithiated TTF **156**.[311] With $TeCl_4$ the monoanion yields only the dimer Te(TTF)$_2$ **163**.[314,315] After reaction of tetrathiafulvalenyllithium **156** with an trialkylstannyl chloride the corresponding trialkylstannyl derivative is formed. The corresponding mono-, bi-, or tritetrathiafulvalenes **164–166** can be prepared by palladium catalysed coupling of the trialkylstannyl derivative with mono-, di- or trihaloarenes.[316,317] Halomethanoannulenes can also be used.[318]

Two TTF moieties can be linked to each other by two tellurium atoms if the 2,3-positions in the TTF derivative **167** are substituted. After lithiation with two equivalents of BuLi and reaction with bis(phenylethynyl) telluride a fused system **168** containing a ditellurine ring is formed.[319,320]

Halogenation of the lithiated species **156** (generated with LDA) with the appropriate *p*-toluenesulfonyl halide affords the monohalogenated TTF derivatives **169** in good yields (35–50%).[321] The yield of bromotetrathiafulvalene **170** is decreased when 1,2-dibromotetrachloroethane is used as halogenating agent. Beside the 2,3-dibromo-TTF, also tri- and tetrabromo derivatives are formed. The yield of by-products depends on the kind of metallation agent.[322] Use of *N*-chlorosuccinimide or bromine as halogenating agents provides chloro- or bromo-TTF **169** in moderate yields (11% and 34%, respectively). In both cases the corresponding 2,6(7)-dihalo-TTF can be found as by-product.[323] Monoiodo-TTF **171** is



SCHEME 110



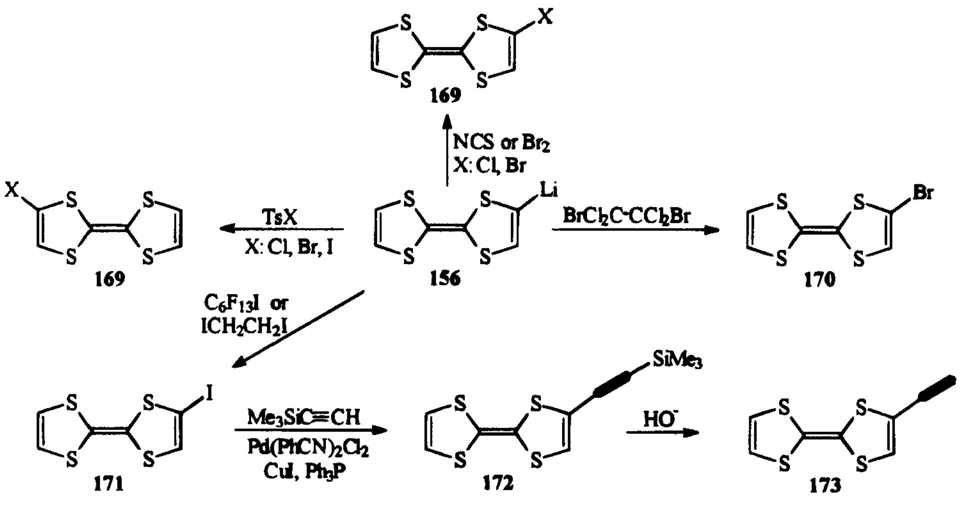
SCHEME 111

prepared in good yields (70–78%) by treatment of TTF with LDA, followed by reaction with perfluorohexyl iodide or 1,2-diiodoethane.[324,325] 2-Iodo-TTF reacts with trimethylsilylacetylene in the presence of a palladium and copper compound as catalyst to afford 2-(trimethylsilylethynyl)-TTF **172** in 70% yield, which can be hydrolysed under alkaline conditions to produce 2-ethynyl-TTF **173** (Scheme 112).[325]

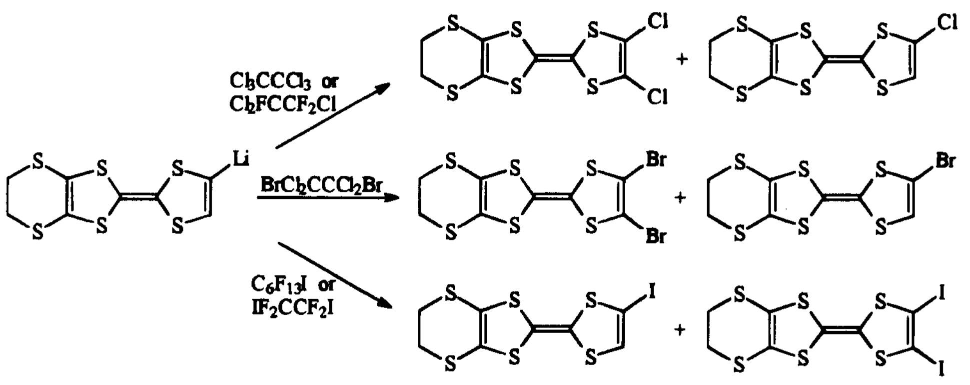
Monolithiated ethylenedithiotetrathiafulvalene can be halogenated with different halogenating reagents. With two equivalents of hexachloroethane or 1,2-dibromotetrachloroethane mainly the corresponding dihalides besides small amounts of the monohalides are formed. A better yield of the monochloride is obtained by use of 1,1,2-trichloro-1,2,2-trifluoroethane as chlorinating reagent. In contrast to chlorination and bromination, iodination with perfluorohexyl iodide or 1,2-diiodotetrafluoroethane gives the monoiodide as main product.[326]

Tetrathiafulvalenyllithium **156** forms with benzeneselenenyl chloride phenylseleno-TTF **174** (X = Se) and with diphenylditelluride phenyltelluro-TTF **174** (X = Te).[327,328]

Reaction of the monoanion **156** with bis(phenylsulfonyl) sulfide, bis(phenylsulfonyl) disulfide or bis(phenylsulfonyl) diselenide produces the corresponding sulfur or selenium



SCHEME 112



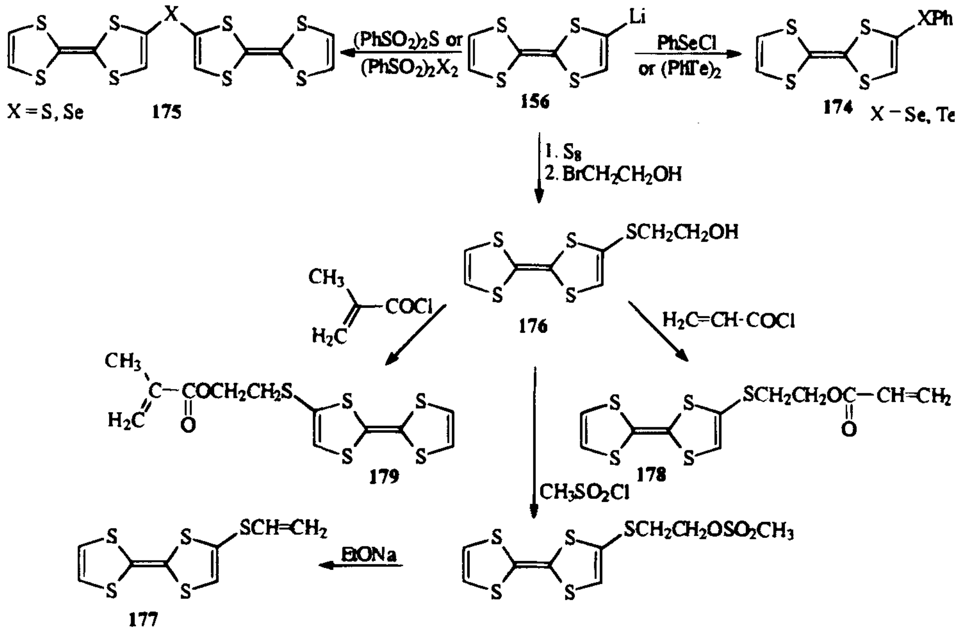
SCHEME 113

bridged dimer bis(tetrathiafulvalenyl) sulfide **175** (X = S) or bis(tetrathiafulvalenyl) selenide **175** (X = Se) in moderate yield.[329]

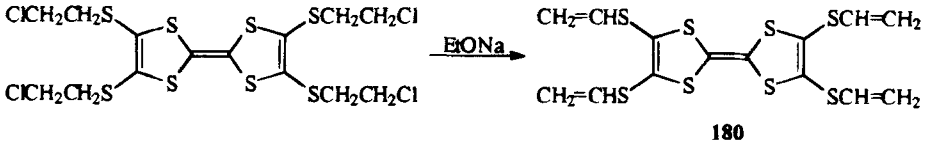
Reaction of monolithio-TTF **156** with elemental sulfur provides the thiolate anion, which can be alkylated with bromoethanol to hydroxyethylthio-TTF **176**. Subsequent mesylation and treatment with sodium ethoxide gives vinylthio-TTF **177**.[330] Hydroxyethylthio-TTF **176** can be converted with acryloyl or methacryloyl chloride under basic conditions to the acrylate **178** or the methacrylate **179**, respectively (Scheme 114).

In an analogous manner the base-induced elimination of hydrogen chloride from tetrakis(chloroethylthio)tetrathiafulvalene affords tetra(vinylthio)tetrathiafulvalene **180**.

Hydroxyethylthio-TTF **176**, formed by the reaction of monolithio-TTF **156** with elemental sulfur and subsequent reaction with 2-bromoethanol, can be used as intermediate for further reactions (Scheme 116). Conversion of the alcohol **176** with acid chlorides leads to bis- and tris(tetrathiafulvalenes) **181–184**, joined by ester linkages.[331] The TTF-thiolate



SCHEME 114



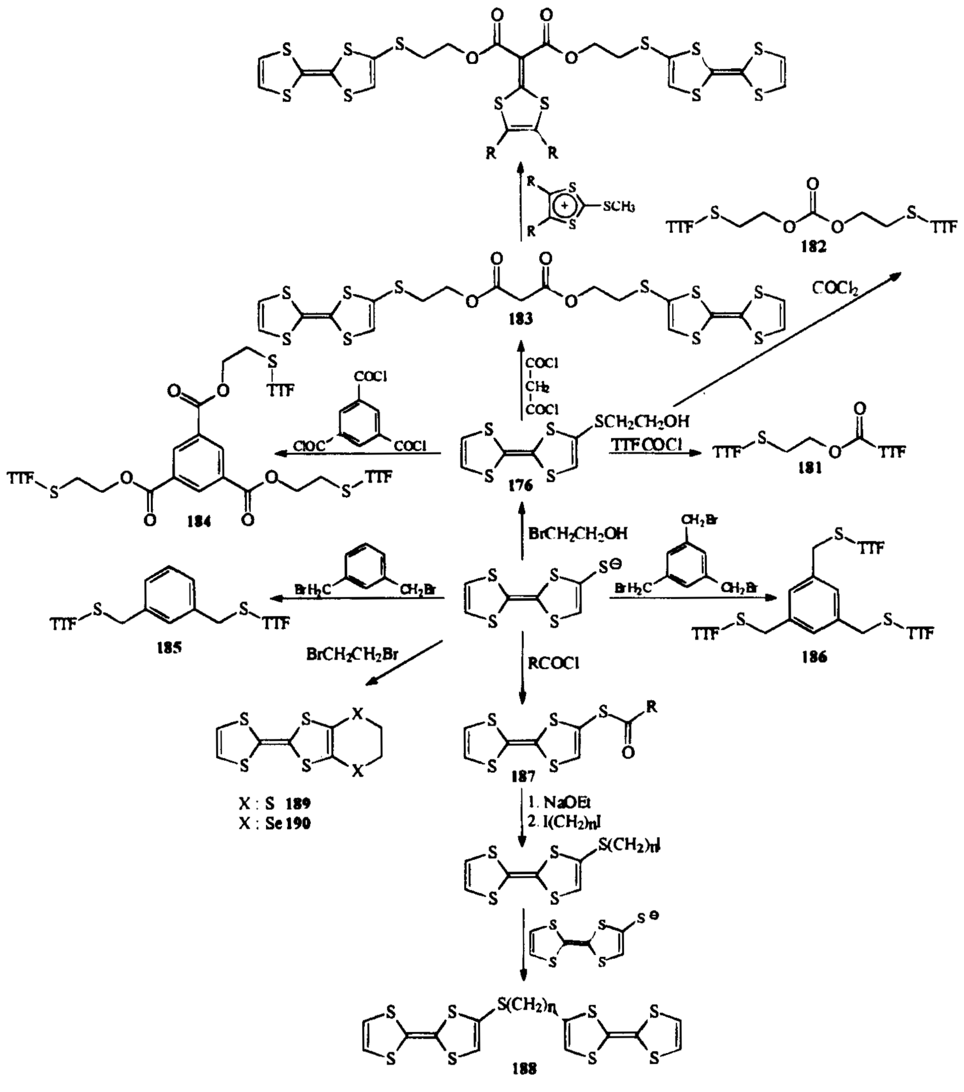
SCHEME 115

anion can also be trapped with bis- and trishalides to yield the bis- and tris(tetrathiafulvalenes) **185** and **186**.[331] With acid halides the TTF-thiolate forms thioesters **187**, from which the thiolate anion can be regenerated with sodium ethoxide in ethanol at −10°C. Reaction of the regenerated TTF-thiolate with diiodoalkanes provides bis-TTF **188**.[331]

The alkylthio substituent in TTF can be modified by reaction of hydroxyethylthio-TTF **176** with alkyl- or acyl halides under basic conditions or by conversion of the methanesulfonyloxyethylthio-TTF with nucleophiles.[332,333]

Surprisingly, the reaction of the TTF-monoanion with elemental sulfur or selenium (1.5 equivalents) and subsequent addition of 1,2-dibromoethane (0.5 equivalents) yielded ethylenedithio-TTF **189** and ethylenediseleno-TTF **190** in yields of 10–20%.[332]

After development of an improved synthesis of formyl-TTF **191**, this reactive TTF has been used as key intermediate for the synthesis of new TTF derivatives. In the reaction of monolithio-TTF **156** with formylating agents (*N*,*N*-dimethylformamide, *N*-formylpiperidine, *N*-formylmorpholine, *N*-methylformamide, *N*-methyl-*N*-phenylformamide) with *N*-methylformamide[236] or *N*-methyl-*N*-phenylformamide[334] the highest yields (80%) of formyl-TTF are obtained. Metallation of TTF with four equivalents of LDA and subsequent



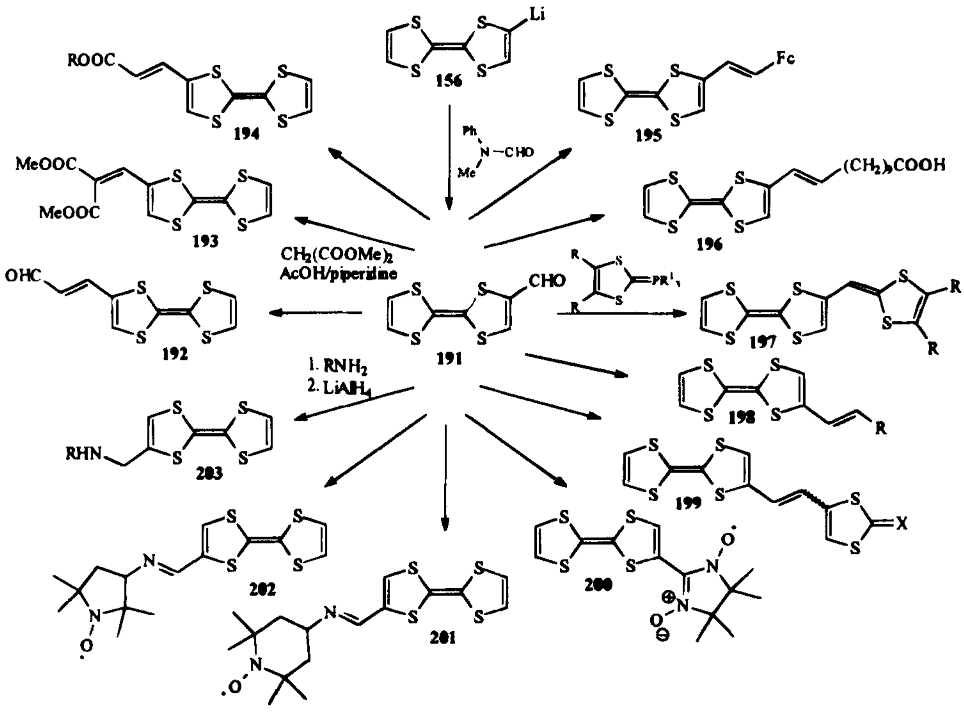
SCHEME 116

addition of 2.5 equivalents *N*-methyl-*N*-phenylformamide yields tetrathiafulvalene-2,6(7)-dialdehyde.[286]

Formyl-TTF **191** can be converted, by Wittig reactions or condensation reactions, to the new functionalised TTF-derivatives **192–194**,[287,334] **195**,[333] **196**,[335] **197**,[336] **198**,[335,337] **199**,[338] **200**,[339] **201**,[340] **202**,[341] and **203**[342] (Scheme 117).

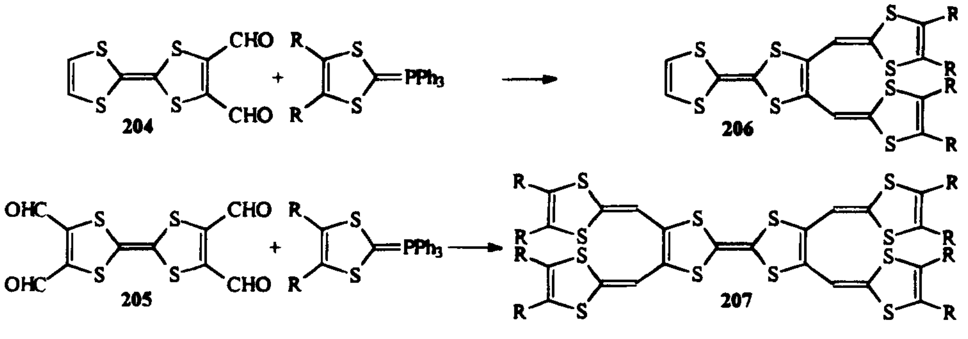
The TTF derivatives **200–202** contain a radical substituent and in CT complexes of these compounds an interaction of the spins of the radical substituent with the spins in the donor and acceptor stacks is expected, resulting in materials with ferromagnetic properties.[339–341]

In an analogous manner multi-formyl-TTF upon treatment with phosphoranes form olefinic bonds. Wittig reaction of 1,2-diformyl-TTF **204** and tetraformyl-TTF **205** with



SCHEME 117

phosphoranes produces bis- and tetrakis(1,4-dithiafulvenyl)-TTF **206** and **207** in yields of 70–80%.[343]

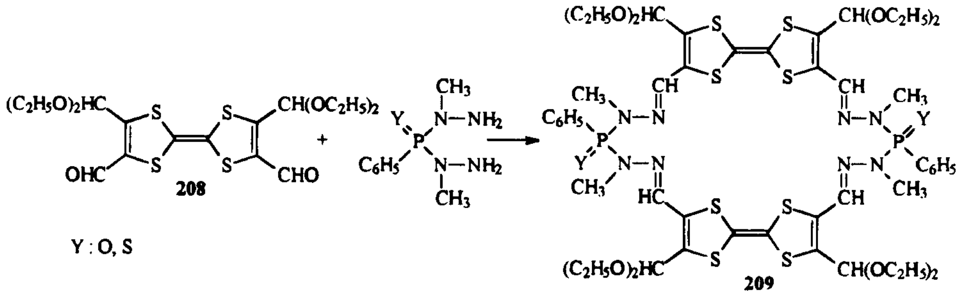


R = H, COOMe, $(CH{=}CH)_2$

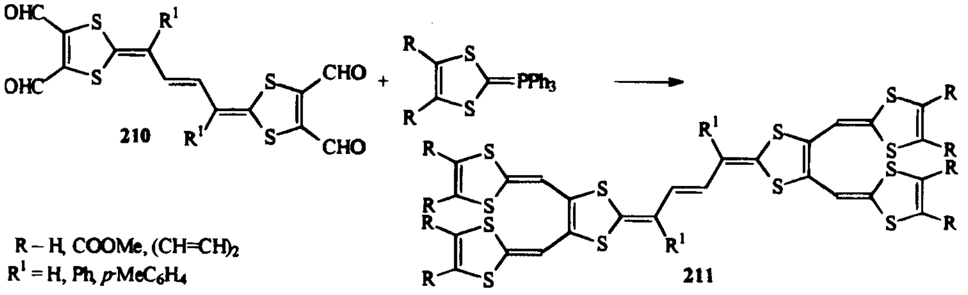
SCHEME 118

Treatment of phosphodihydrazides (Y = O, S) with the *cis*-isomer of the diacetalized tetraformyl-TTF **208** leads to macrocycles **209** in yields of 80% and 60%, respectively.[344]

Also tetraformyl substituted vinylogues of TTF **210** react with phosphoranes or phosphonate anions to TTF derivatives **211** with a more extended π-system.[345]

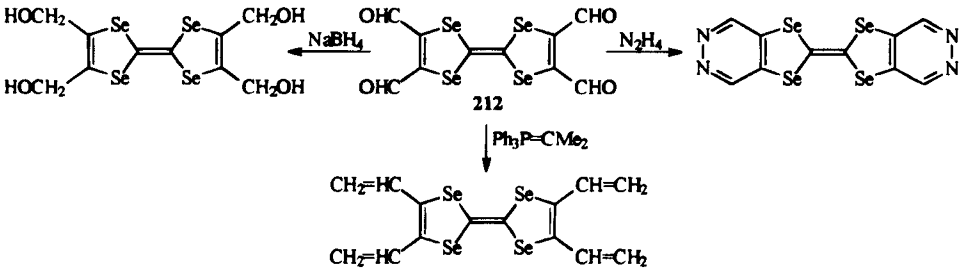


SCHEME 119



SCHEME 120

Tetraformyl-TSF **212** is an efficient precursor for heteroannelated and tetrasubstituted TSF.[198]



SCHEME 121

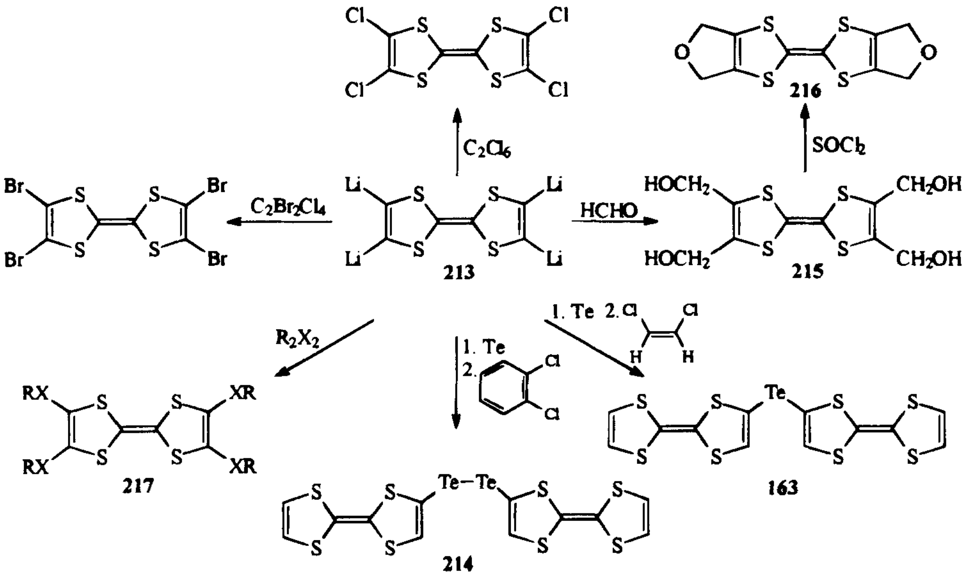
Multilithiated TTF are formed when the lithiation is carried out at −20 °C or when excess lithiation reagent is used.[346] The treatment of multilithiated TTF with reagents provides multisubstituted TTF (Scheme 122).

Tetralithiated TTF **213**[347] has been chlorinated and brominated with hexachloroethane and 1,2-dibromotetrachloroethane, respectively (yield 15%). When iodine was used as the halogenating agent the desired product was not obtained.[348]

After treatment of tetralithiated TTF **213** with tellurium and subsequent reaction with *cis*-dichloroethylene the bis(tetrathiafulvalenyl)telluride **163** is formed in 20% yield and not the unsaturated telluro analog of bis(ethylenedithio)-TTF.[314] With 1,2-dichlorobenzene the TTF tetratellurolate anion yielded the unexpected ditelluride **214** instead of the anticipated fused system.[349]

The intermediate tetralithio-TTF **213** reacts with formaldehyde and provides tetrakis(hydroxymethyl)-TTF **215** which can be converted with thionyl chloride to bis(oxydimethylene)-TTF **216**.[350]

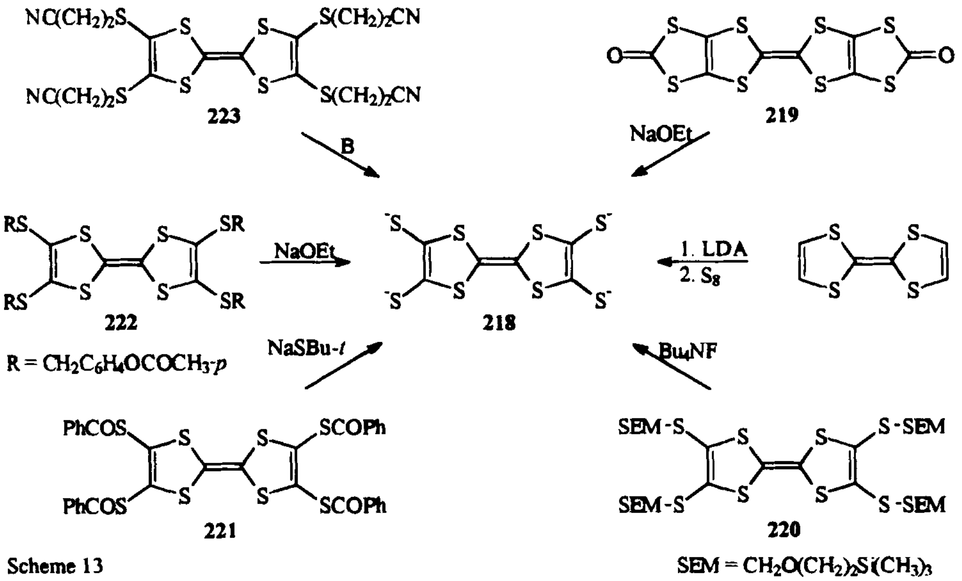
The tetrakis(alkylchalcogeno)-TTF **217** (X = S, Se, Te) can be prepared in good to excellent yield (64–90%) by reaction of tetralithio-TTF **213** with various dialkyl or diaryl disulfides, diselenides, or ditellurides as electrophiles.[328,351] When the lithiation and the subsequent reaction are performed with four equivalents of LDA and benzeneselenenyl chloride the corresponding di, tri-, and tetrasubstituted TTF can be isolated after chromatographic separation.[327]



SCHEME 122

TTF-tetrathiolate **218** is a key intermediate in the synthesis of thio substituted TTF. This intermediate has been prepared in different ways. Firstly the dithiapendione **219** is hydrolyzed with sodium ethoxide in refluxing ethanol or methyllithium in tetrahydrofuran at room temperature.[293] Later TTF-tetrathiolate **218** is formed by treatment of TTF with 4 equivalents of LDA, followed by 4 equivalents of sulfur.[347] This method was improved by use of excess base (10 equiv. LDA). In that way a higher yield and a purer product could be obtained.[346]

Furthermore some precursors with protecting groups can be deprotected by treatment with nucleophiles and afford TTF-tetrathiolate **218**. Tetrakis(trimethylsilylethoxymethylthio) TTF **220** is converted to TTF-tetrathiolate **218** by reaction with tetrabutylammonium fluoride in tetrahydrofuran at room temperature.[109] Sodium *t*-butylthiolate in tetrahydrofuran reacts at –10°C with tetrakis(benzoylthio)-TTF **221** and provides TTF-tetrathiolate **218**.[63,352] The 4-acetoxybenzylthio protecting group in the corresponding TTF **222** can be removed with sodium ethoxide in ethanol.[353,354] Strong bases such as sodium ethoxide in ethanol or sodium hydride in DMF or tetrabutylammonium hydroxide, methyllithium, LDA, potassium *t*-butoxide or caesium hydroxide in tetrahydrofuran remove the cyanoethyl group in the TTF **223**.[355] The advantage of the latter procedure is the synthesis of the precursor **223** on a large scale (>20 g) in high yield from inexpensive starting materials.



SCHEME 123

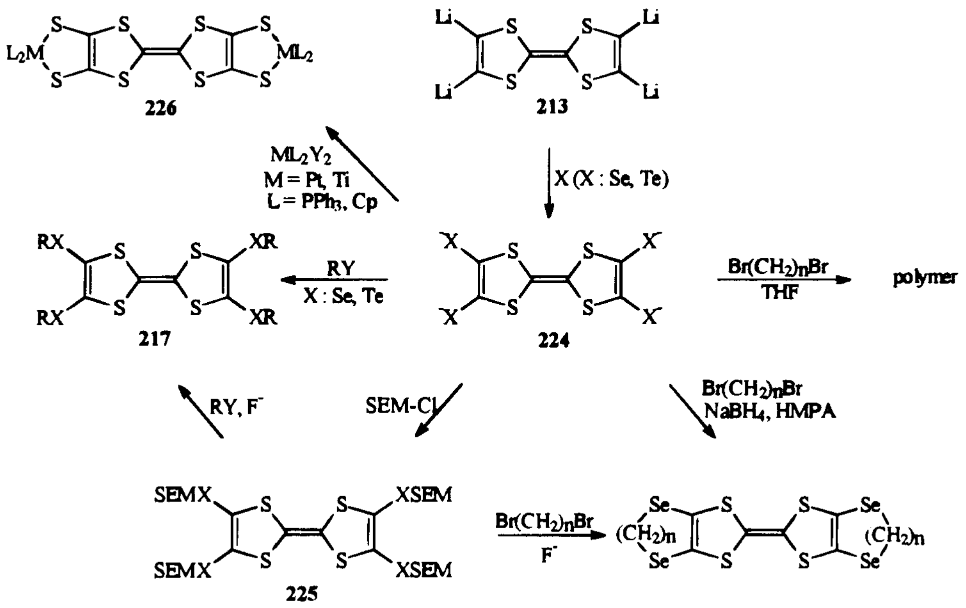
TTF-tetrathiolate **218** can react with alkyl halides or inorganic salts to form alkylthio substituted TTF or coordination complexes of TTF.

TTF-tetraselenolate or TTF-tetratellurolate is prepared by tetralithiation of TTF and subsequent insertion of selenium or tellurium into the carbon-lithium bonds of tetralithio-TTF. Attempts at intramolecular alkylative ring closure of the selenium and tellurium derivatives **224** (X = Se, Te) with dibromoalkanes only lead to polymeric products. Obviously, these are formed by predominantly intermolecular alkylation, even at high dilution or at lower temperatures.[356] In order to solve this problem, the TTF-tetrachalcogenolate anions **224** have been converted to stable intermediates by introduction of protective groups, a convenient blocking reagent being 2-(trimethylsilyl)ethoxymethyl chloride (SEM-Cl). Removal of the SEM groups proceeds with tetrabutylammonium fluoride at a lower rate compared to the alkylation of selenolate anions with dibromoalkanes, thus promoting cyclization over polymerization. With 1,2-dibromoethane or -dibromomethane as alkylating agent alkylseleno-TTF are obtained in good yields (30–35%).

In the case of the TTF-tetratellurolate anion tetrakis(SEM-telluro)-TTF **225** (X = Te) is produced in good yield (63%), but reaction with dibromoalkanes in the presence of tetrabutylammonium fluoride fails to provide the desired products.[356] The formation of polymers in the reaction of TTF-tetraselenolate anion **224** (X = Se) with dibromoalkanes is attributed to the poor solvation of the TTF-tetraselenolate anions in the solvent used so far (THF). It has been speculated that if the TTF-tetraselenolate anion is sufficiently solvated, the dibromoalkane molecule enters the solvent sphere of the solvated TTF-tetraselenolate anion and reacts with the anion within the solvent sphere faster than with one outside the solvent sphere. In fact replacement of THF by hexamethylphosphoramide (HMPA), a dipolar aprotic solvent, allows the bis(alkylseleno)-TTF to be formed in good yields (25–65% after recrystallization). Addition of sodium borohydride eliminates the formation of some polymeric by-products by cleavage of Se-Se linkages formed by oxidation of the TTF-tetraselenolate anions.[357]

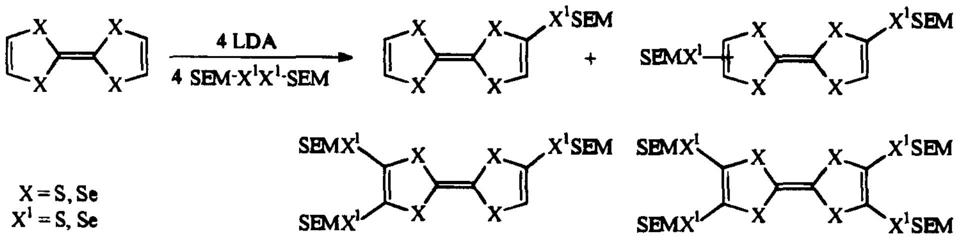
Reaction of the TTF-tetrachalcogenolate anions **224** as well as a reaction of tetrakis(SEM-chalcogeno)-TTF **225** after removal of the protecting group by tetrabutylammonium fluoride with alkyl halides yields the tetrakis(alkylchalcogeno)-TTF **217**.[322,358–361]

Transition metal homobimetallic coordination complexes **226** of TTF-tetrathiolate are formed by reaction of TTF-tetrathiolate **224** (X = S) with titanocene dichloride or bis(triphenylphosphine)platinum(II) chloride.[362]



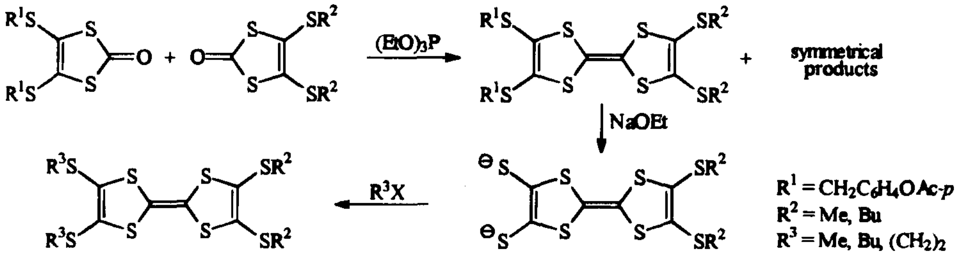
SCHEME 124

The removal of the protecting SEM-group with tetrabutylammonium fluoride opened up the possibility of the synthesis of unsymmetrically substituted tetrachalcogenafulvalenes. The lithiated TXF is treated with SEM disulfide or SEM diselenide and affords all four types of the X^1-SEM substituted TXF in an overall yield of 66–75%.[363] This product mixture is easily separated on a silica gel column. Reaction of the separated products with anhydrous tetrabutylammonium fluoride in THF in the presence of alkyl bromides provides the desired compounds in a very pure form.



SCHEME 125

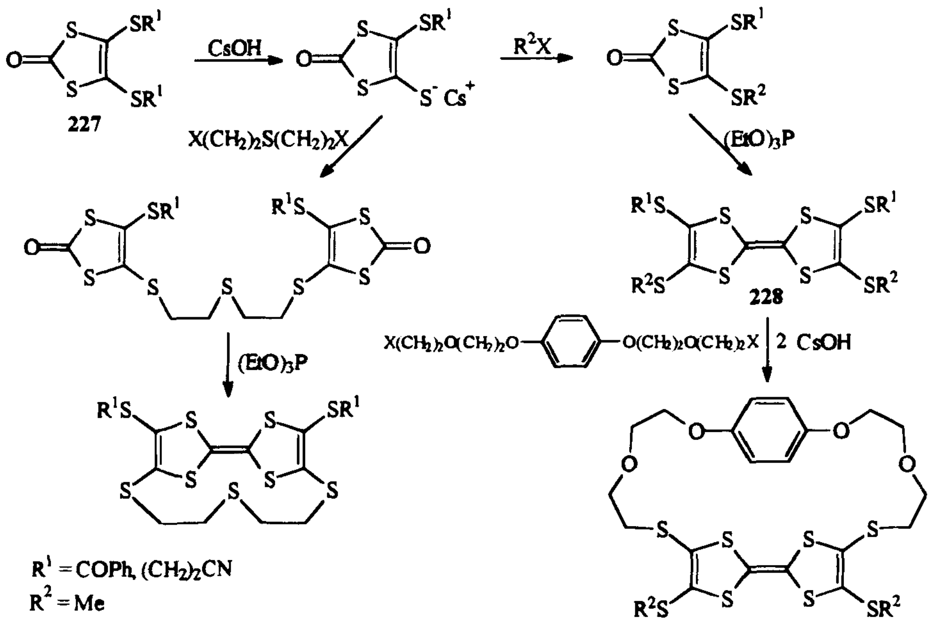
Unsymmetrical TTF have also been synthesized with 4-acetoxybenzyl as the blocking group.[353,354] After cross-coupling of two 1,3-dithiol-2-ones the resulting product mixture is separated by flash column chromatography. The protecting group is then removed with sodium ethoxide in ethanol or sodium *t*-butylthiolate in tetrahydrofuran at −10 °C. The dithiolate thus formed is then quenched with alkyl halides.



SCHEME 126

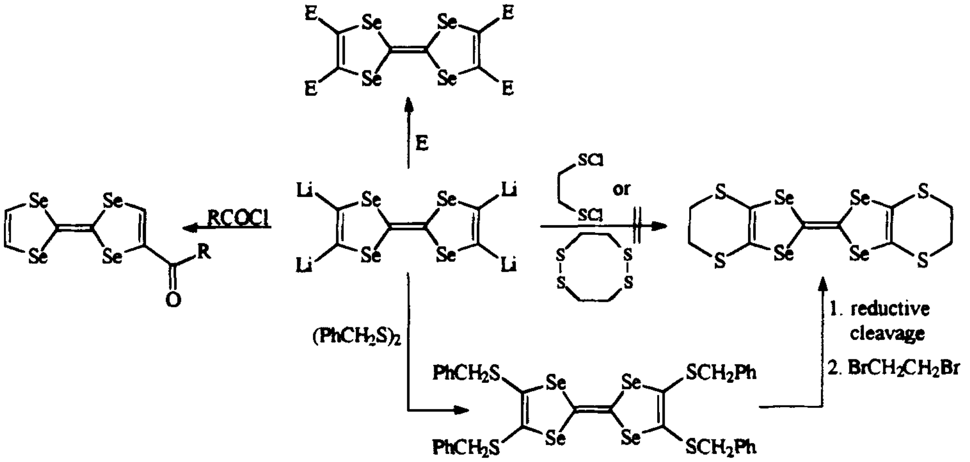
Treatment of protected 1,3-dithiole-2-thione-4,5-dithiolates **227** or protected TTF **228** with one equivalent of caesium hydroxide selectively and in high yields provides the corresponding monocaesium salts. The caesium salts are more air and moisture stable than the other alkali metal salts and can subsequently be alkylated. Combination of deprotecting reactions of **227** and **228** followed by realkylation with various mono- or bisalkylating agents offers a possibility for the synthesis of unsymmetrically substituted TTF.[173,364,365]

TSF also can be converted with LDA to the tetralithio derivative. Treatment of TSF with *n*-butyllithium under conditions employed with TTF led to total destruction of the TSF framework.[366] Reaction of tetralithio-TSF with excess electrophile (Me_2S_2, Ph_2S_2, Me_2Se_2, Ph_2Se_2, ClCOOMe, CO_2) affords the corresponding tetrasubstituted TSF in moderate yield (30–70%).[360,366,367] The reaction with 1,2-disulfenyl chlorides or 1,2,5,6-tetrathiocyclooctane does not provide bis(ethylenedithio)-TSF. Promising is the route via the tetrabenzylthio in-



SCHEME 127

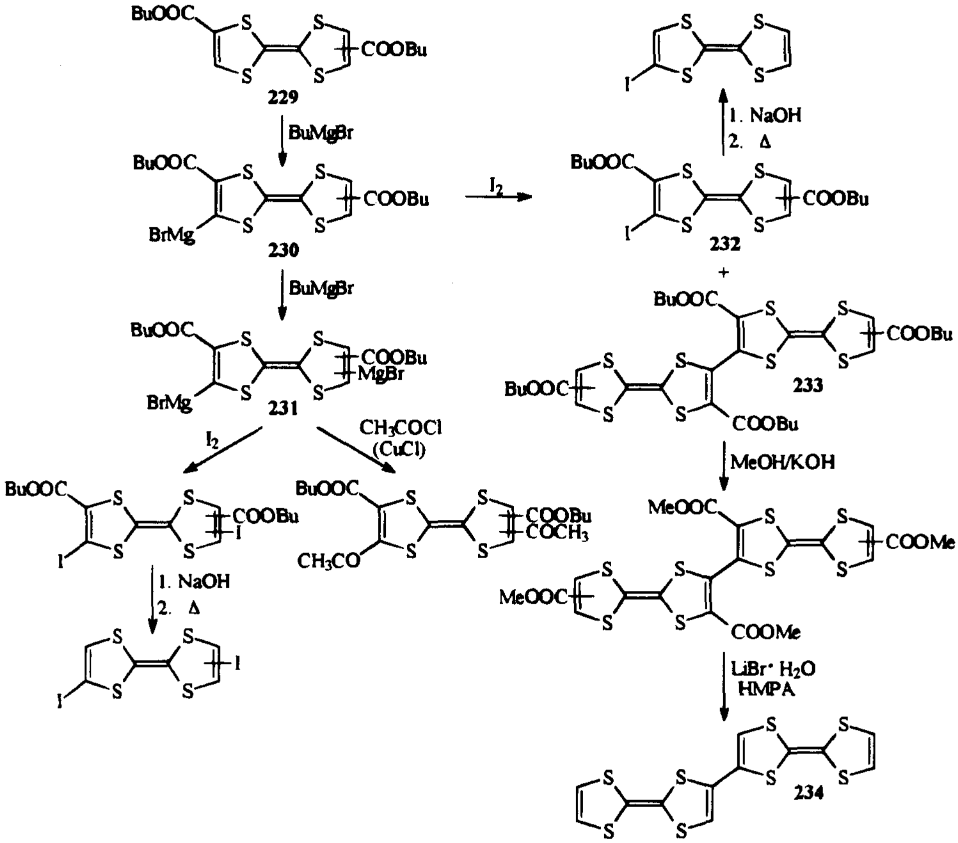
termediate (yield over 50%) via subsequent reductive cleavage and alkylation with 1,2-dibromoethane.[368] By addition of less than one equivalent acid chloride to tetralithiated TSF the monoacyl-TSF derivative is the only isolated product (14–20% yield) besides regenerated TSF.[369]



SCHEME 128

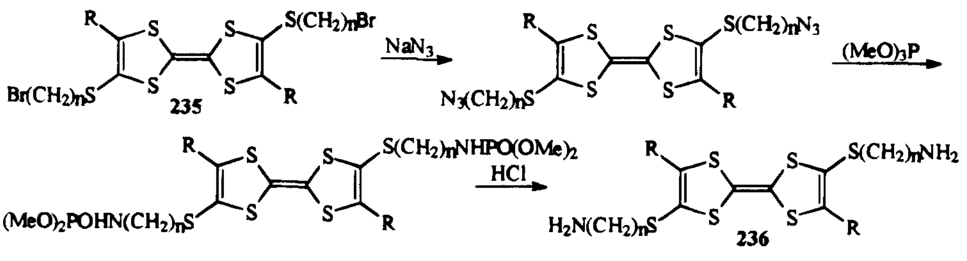
Metallation of TTF derivatives can also be achieved with Grignard agents.[370] Treatment of bis(butoxycarbonyl)-TTF **229** with butylmagnesium bromide gives the monometallated derivative **230**, and further reaction with butylmagnesium bromide the dimetallated derivative **231**. Both react readily with electrophiles, e.g. acetyl chloride, 2-chloroethyl formate or iodine and provide the corresponding derivatives (Scheme 129).

In the reaction of bis(butoxycarbonyl)tetrathiafulvalenylmagnesium bromide **230** with iodine a mixture of two products is formed, a monoiodo substituted TTF **232** (37%) and a dimerized TTF derivative **233** (25%). The butoxycarbonyl group in the TTF derivatives can be hydrolyzed in a refluxing alkaline mixture of water and dioxan. Upon heating in diglyme the acid decarboxylates. With methanol in alkaline solution the butoxycarbonyl group is converted to a methoxycarbonyl group. Demethoxycarbonylation with lithium bromide monohydrate at 150 °C provides the unsubstituted dimerized TTF **234**.



SCHEME 129

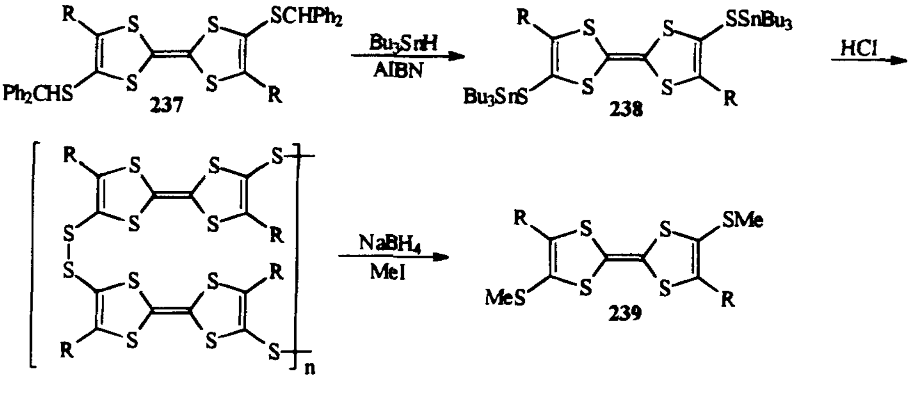
The synthetic route to TTF via mesoionic 1,3-dithioles made it possible to prepare many substituted TTF derivatives which in turn can be converted to new derivatives.[95] Thus, the bis(aminoalkylthio)-TTF **236** (n = 3, 6) are obtained by nucleophilic substitution of the bis(bromoalkylthio)-TTF **235** with sodium azide in DMSO, followed by treatment with trimethyl phosphite and subsequent hydrolysis with hydrochloric acid.



$R = CH_3(CH_2)_{10}, p\text{-}MeC_6H_4, p\text{-}ClC_6H_4$

SCHEME 130

In a radical initiated reaction the bis(benzhydrylthio)-TTF **237** are treated with tributylstannane and provide bis(tributylstannylthio)-TTF **238**. Further reaction with gaseous hydrogen chloride leads to S-S bond formation and formation of polymeric TTF. The S-S linkages of polymeric TTF can be cleaved with sodium borohydride and alkylated with methyl iodide to form the bis(methylthio)-TTF **239**.[95]



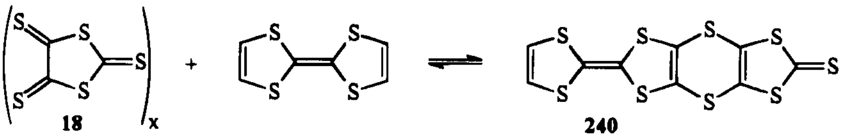
$R = CH_3(CH_2)_{10}, p\text{-}MeC_6H_4, p\text{-}ClC_6H_4$

SCHEME 131

The oligomeric 1,3-dithiole-2,4,5-trithione **18**, which presumably depolymerizes on heating, can be added to TTF to form the cycloadduct **240** in 30–50% yield. The reaction is reversible and in boiling toluene 60–65% TTF can be recovered.[371]

With $TiCl_4/Et_3N$ as the condensation reagent, chalcones with TTF as a substituent can be prepared (Scheme 133).[372]

The dicyano(ethynyl)ethenes **241** and TTF do not form charge transfer complexes, but new covalent compounds.[373] Probably the reaction begins with a [2+2] cycloaddition of the

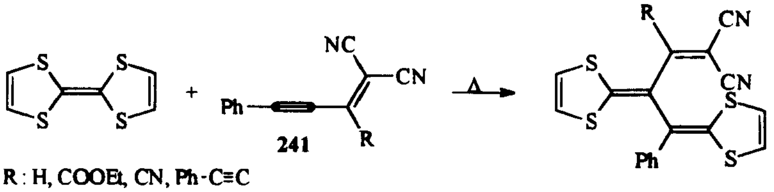


SCHEME 132

R : TTF, $C_5H_4FeC_5H_5$, $C_6H_5Cr(CO)_3$
R^1 : TTF, $C_5H_4FeC_5H_5$, $C_6H_5Cr(CO)_3$, C_6H_5
R^2 : CH_3

SCHEME 133

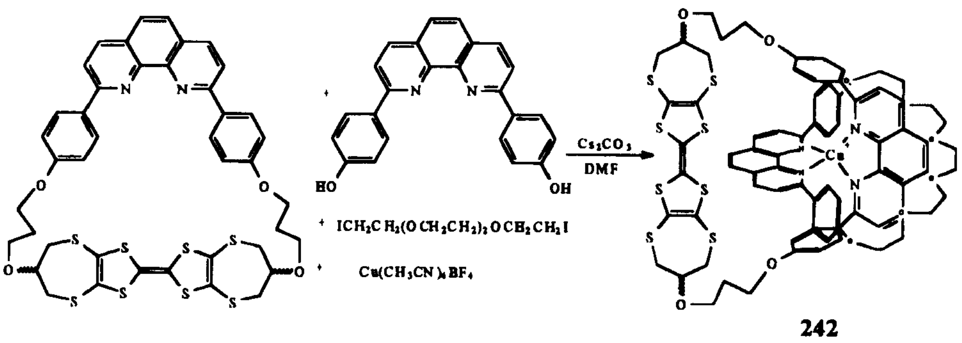
triple bond to the central double bond of the TTF and formation of a cyclobutene derivative which is stabilized by ring opening.



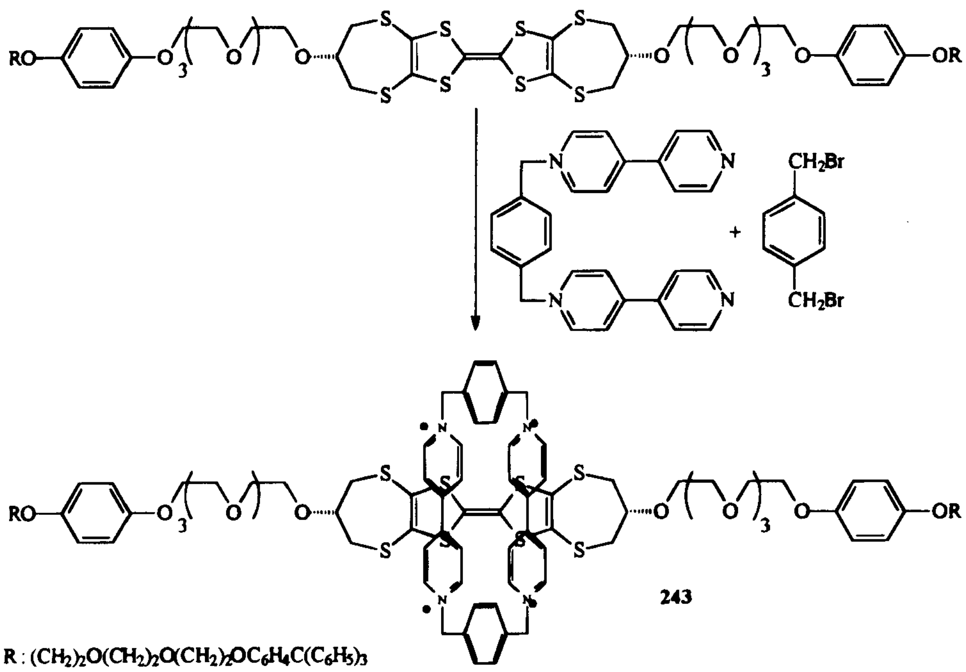
R : H, COOEt, CN, Ph-C≡C

SCHEME 134

Two interesting ring systems, incorporated the same TTF unit, have been synthesized. A catenate **242**[374] with additional copper coordination and a rotaxane **243**[375] are afforded.



SCHEME 135



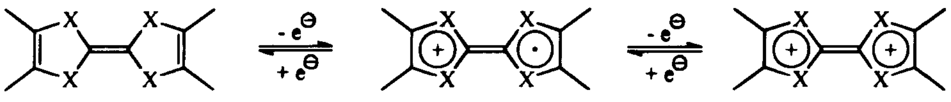
243

R : $(CH_2)_2O(CH_2)_2O(CH_2)_2OC_6H_4C(C_6H_5)_3$

SCHEME 136

6.2. *Redox Reactions and Spectroscopic Behaviour of Tetrathia-, Tetraselena-, and Tetratellurafulvalenes*

Tetrathia-, tetraselena-, and tetratellurafulvalenes are good electron donors which can be oxidized in two successive one-electron steps to their mono- and dications.



X : S, Se, Te

SCHEME 137

Polarographically it has been proven that these electron transfers are reversible for the tetrathia-[90,376] as well as for the tetraselena-[377] and tetratellurafulvalenes.[225]

The half-wave oxidation potentials E_1^{Ox} and E_2^{Ox} show that the donor strength decreases from tetrathiafulvalene to the corresponding tetraselenafulvalene and increases again for the corresponding tetratellurafulvalene (Table 1).

This is caused by two opposite effects. The π-orbital interaction between the carbon framework and the heteroatoms decreases as a result of increasing atom size in the series

TABLE 1 Oxidation potentials of substituted tetrathia-, tetraselena-, and tetratellurafulvalenes (vs. sce)[225,226]

Compound	E_1^{Ox} (V)	E_2^{Ox} (V)	ΔE (V)
TMTTF	0.24[a]	0.73	0.49
TMTSF	0.42[a]	0.81	0.39
HMTTeF	0.40[a]	0.69	0.29
DBTTF	0.71[b]	1.14	0.43
DBTSF	0.78[b]	1.17	0.39
DBTTeF	0.71[b]	1.05	0.34

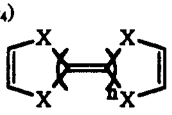
[a] benzonitrile, Bu_4NAsF_6; TMTTF and HMTTF as well as TMTSF and HMTSF have practically identical redox properties.[46,378]

[b] methylene chloride, 0.2 M Bu_4NBF_4; TMTTF, HMTTF: tetramethyl-, hexamethylenetetrathiafulvalene, respectively, TMTSF, HMTSF: tetramethyl-, hexamethylenetetraselenafulvalene, respectively, HMTTeF: hexamethylenetetratellurafulvalene, DBTTF, DBTSF, DBTTeF: dibenzotetrathia-, dibenzotetraselena-, dibenzotetratellurafulvalene, respectively.

S, Se, Te. Therefore in this series the resonance stabilization of the positive charge after removal of an electron also decreases and higher energy is required for the removal of this electron. On the other hand the ionization potential decreases in the series S, Se, Te. Hence the ionization of TTF should primarily involve the π-bonded network and in TTeF the tellurium lone pairs.[225,226]

Conjugated tetrathia- and tetraselenafulvalenes are stronger donors than the corresponding tetrathia- and tetraselenafulvalenes. With increasing extension of the conjugated electron system the oxidation potential becomes smaller than that of the parent compound. By the same token the Coulomb repulsion between the charges of the dication is decreased. This is apparent in the decrease of the difference between the two oxidation potentials ($E_2 - E_1$). In the tetrathiafulvalene derivative with three double bonds the difference is so small that only one wave, representing a two-electron oxidation, appears (Table 2).[248]

TABLE 2 Oxidation potentials of vinylogous tetrathia- and tetraselenafulvalenes and their parent compounds[231,248] in acetonitrile (vs. Ag/AgCl electrode, 0.1 M Et_4NClO_4)



n	X	E_1^{Ox} (V)	E_2^{Ox} (V)	ΔE (V)
1	S	0.34	0.71	0.37
2	S	0.20	0.36	0.16
3	S	0.22		
1	Se	0.49	0.77	0.28
2	Se	0.33	0.47	0.14

As mentioned above and demonstrated in Tables 1 and 2, TTF derivatives with an extended π-system between the two 1,3-dithiole moieties are mostly stronger donors than the parent TTF. The donor ability is increased by cumulenic and quinoid structures (Table 3). In the last case the two single-electron redox waves coalesce to a single, two-electron redox wave. This means that in the radical cation and dication states of these donors the one-site Coulomb repulsion is greatly reduced. However, irreversible redox reactions indicate that cumulenic TTF are unsuitable donors for the preparation of organic conductors because of the extreme instability of the neutral species, radical cations or dications.

The donor strength is also influenced by substituents. It is increased by electron-donating and lowered by electron-withdrawing substituents. In diaryl substituted TTF the first half-wave potential correlates with the Hammett values, where the σ_p^+ values understandably provide a better correlation than the σ_p values (Figure 1)[379]

If two or three TTF moieties are connected, the corresponding "dimeric" or "trimeric" TTF should be able to donate four or six electrons, respectively. Depending on the kind of connection between the TTF moieties (every TTF unit bears the same substituents) one can find in the cyclic voltammogram of "dimeric" TTF two, three, or four redox couples.[32]

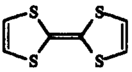
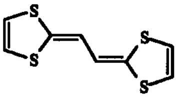
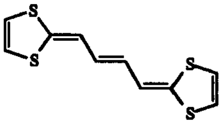
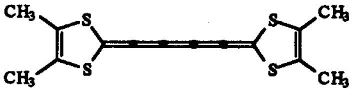
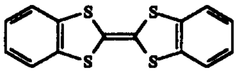
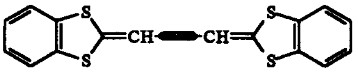
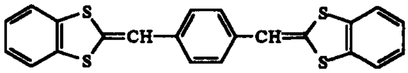
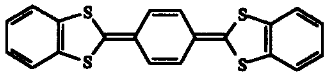
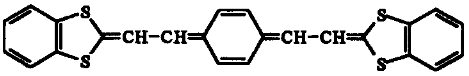
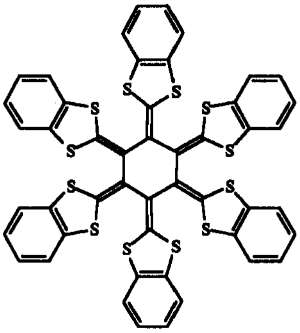
Group 1. The TTF moieties are conjugatively bridged or annelated to form a new donor with extension of the π-system. In this case the oxidation can take place in four one-electron steps. These four waves in the cyclic voltammetry correspond to the oxidation to the mono-, di-, tri-, and tetracation. The "trimeric" TTF provides six one-electron oxidation waves (Table 4).

Group 2. The TTF moieties are linked by a bridging group, which provides conjugation between the TTF subunits, but there is no coplanarity between the TTF subunits and the bridging group. These dimers or trimers show two redox waves due to simultaneous oxidation of the two or three TTF units at the same potential (two two-electron or two three-electron oxidation waves). There is no interaction between the TTF units (Table 5).

Group 3. The TTF moieties are linked by a spacer group, with the individual TTF units electronically isolated, but the arrangement of the TTF units enables a Coulomb interaction through space. Such dimers provide, by oxidation, three waves in the cyclic voltammogram. The "sandwich" structure of the dimer (Scheme 138) lowers the first oxidation potential by interaction of the two TTF π-systems, resulting in stabilization of the mono radical cation. At slightly higher potential the second electron is removed. As a result of the Coulomb repulsion between the two positively charged TTF subunits in the dimer, the two TTF subunits unfold. The two TTF units now behave like individual molecules and the third and fourth electrons are removed at the same potential. By the same token in tris(tetrathiafulvalenyl)phosphine there are observed three close-lying one-electron oxidation waves, followed by a three-electron wave (Table 6).[312]

Group 4. The TTF moieties are linked by spacer groups and there is no interaction between the TTF units (Scheme 139). The individual TTF units are sterically and electronically isolated. In such dimers or trimers the TTF units are oxidized at the same potential and only two two-electron or two three-electron oxidation waves are seen, respectively (Table 7).

TABLE 3 Oxidation potentials of vinylogous, cumulenic, acetylenic, and quinoid tetrathiafulvalenes in acetonitrile (vs. Ag/AgCl electrode, 0.1 M Et_4NClO_4 or 0.1 M Bu_4NClO_4)

Compound	E_1^{Ox}	(V)	E_2^{Ox}	reference
	0.34		0.71	240
	0.20		0.36	240
		0.22		240
	(0.07)		(0.31)a)	254
	(−0.12)		(0.25)a)	254
	0.72		1.06	241
	0.67		1.02b)	252
	0.64		0.78	241
		0.27		241
		0.40		241
	0.43		(0.65)c)	244

Parentheses indicate an irreversible redox reaction, a) BF_4 salts of the corresponding dications, b) peak potentials vs. sce, c) in benzonitrile

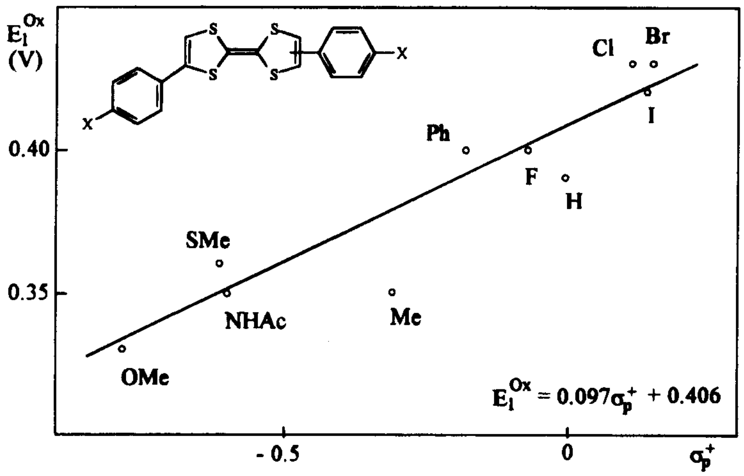
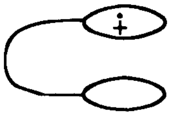


FIGURE 1 Relation of half-wave potentials vs. sce for oxidation of 2,6(7)-diaryl substituted TTF in methanol/benzene (4:1, v:v) to Hammett substituent constants.



SCHEME 138

SCHEME 139

Sometimes in polar solvents in group 1 or group 3 dimeric TTF only a broadening of the oxidation wave is observed in the cyclic voltammogram. A change to a less polar solvent leads to separation of the waves.[312,383]

TABLE 4 Oxidation potentials of group 1 dimeric or trimeric tetrathiafulvalenes in methylene chloride (vs. saturated calomel electrode, 0.1 M Bu_4NPF_6 or 0.1 M Bu_4NClO_4)

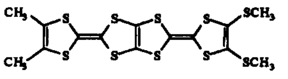
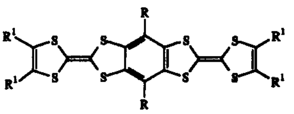
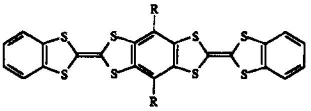
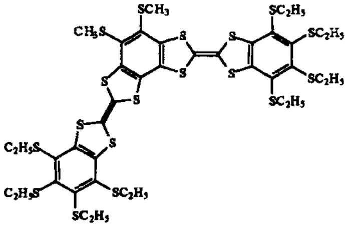
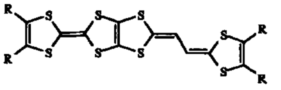
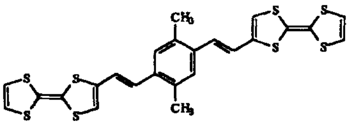
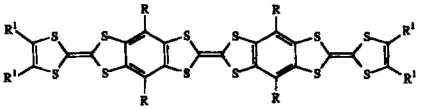
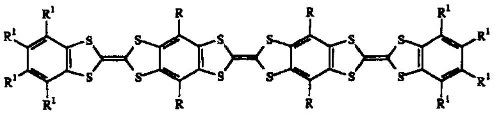
Compound		E_1^{Ox}	E_2^{Ox}	E_3^{Ox} (V)	E_4^{Ox}	E_5^{Ox}	E_6^{Ox}	reference
		0.43	0.68	0.98	1.17$^{a)}$			380
								
R: *O*-hex	R': $S(CH_2)_2S$	0.38	0.59	0.95	(1.16)			156
R: *O*-hex	R': *S*-hex	0.36	0.53	0.86	0.96			381

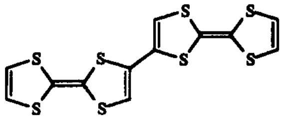
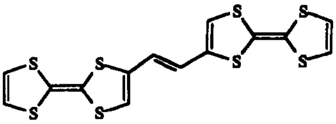
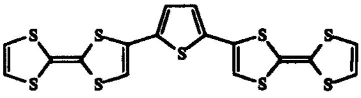
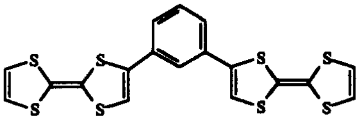
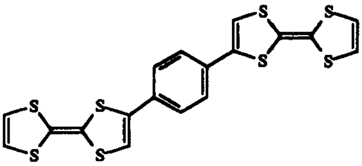
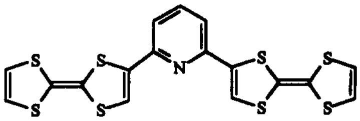
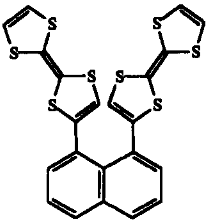
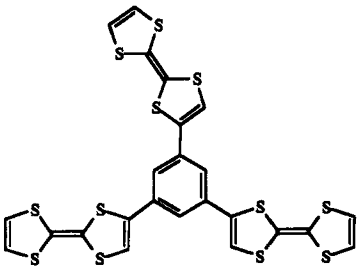
table continues

TABLE 4 *(continued)*

Compound	E$_1^{Ox}$	E$_2^{Ox}$	E$_3^{Ox}$ (V)	E$_4^{Ox}$	E$_5^{Ox}$	E$_6^{Ox}$	reference
R: OC_2H_4Cl R': *S*-hex	0.41	0.56	0.90	1.00			381
R: *S*-isoamyl R': *S*-hex	0.38	0.59	0.90	1.02			384
							
R: *O*-3,5-di-*t*-butylbenzyl	0.46	0.68	1.13	1.29			157
R: *S*-isoamyl	0.44	0.68	1.10	1.29			157
	0.54	0.69	1.06	1.22			157
							
R: H	0.37	0.50	0.81	1.05[a]			382
R: $COOCH_3$	0.56	0.70	1.07	1.30[a]			382
	(0.44) 0.51	(0.69) 0.80[c]	(0.92)[b]				383 383
							
R: *S*-isoamyl R': *S*-hex	0.34	0.44	0.69	0.78	1.10	1.33[d]	384
R: *O*-hex R': *S*-Bu	0.34	0.45	0.78[e]		1.07	1.31[f]	384
R: *O*-hex R': $-SCH_2CH_2S-$	0.28	0.42	0.68	(0.87)	1.08[f]		384
							
R: *O*-hex R': H	0.31		0.61[g]	0.81	1.01	1.28[f]	155, 384
R: *S*-isoamyl R': *S*-Et	0.44	0.61	0.81	1.01	1.28[h]		384

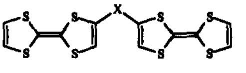
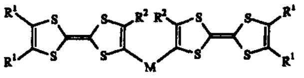
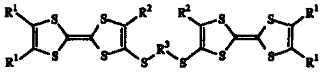
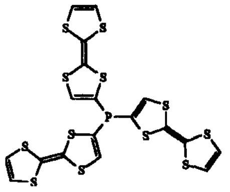
Parentheses indicate an irreversible redox reaction, a) in benzonitrile, b) 0.05 M TBAT, vs. Ag/AgCl, in benzonitrile, c) 0.05 M TBAT, vs. Ag/AgCl, in dimethylformamide, d) in 1,1,2-trichloroethane at 70 °C, e) two-electron transfer step, f) in 1,1,2-trichloroethane, g) poorly separated, h) in 1,1,2-trichloroethane at 90 °C

TABLE 5 Oxidation potentials of group 2 dimeric or trimeric tetrathiafulvalenes in benzonitrile (vs. Ag/AgCl electrode, 0.1 M Bu_4NClO_4)

Compound	E_1^{ox}	(V)	E_2^{ox}	reference
	0.43		0.84	316
	0.41		0.71	325
	0.38		0.81	316
	0.40		0.81[a]	317
	0.35		0.82	316
	0.29		0.87[a]	317
	0.30		0.91[a]	318
	0.29		0.77[a]	317

a) vs. sce

TABLE 6 Oxidation potentials of group 3 dimeric or trimeric tetrathiafulvalenes in methylene chloride (vs. Ag/AgCl electrode, 0.1 M Et_4NPF_6 or 0.05 M resp. 0.1 M Bu_4NPF_6 or 0.1 M Bu_4NClO_4)

Compound			E_1^{Ox}	E_2^{Ox} (V)	E_3^{Ox}	E_4^{Ox}	reference
							
X: S			0.49	0.61	(0.86)		329
X: Se			0.49	0.61	(0.86)		329
X: Te			0.31	0.65	$(0.8)^{a)}$		314
							
R^1: H	R^2: H	M:Si$(CH_3)_2$	0.29	0.38	$0.77^{b)}$		311
R^1: CH_3	R^2: H	M:Si$(CH_3)_2$	0.22	0.32	$0.82^{b)}$		311
R^1: CH_3	R^2: CH_3	M:Si$(CH_3)_2$	0.24	0.34	$0.88^{b)}$		311
R^1: H	R^2: H	M:P(C_6H_5)	0.37	0.47	$0.84^{b)}$		311
R^1: CH_3	R^2: H	M:P(C_6H_5)	0.28	0.38	$0.88^{b)}$		311
R^1: CH_3	R^2: CH_3	M:P(C_6H_5)	0.23	0.36	0.76	$0.87^{b)}$	311
							
R^1: CH_3	R^2: CH_3	R^3:CH_2	0.22	0.36	$0.80^{b)}$		96
R^1: CH_3	R^2: CH_3	R^3:$(CH_2)_2$	0.23	0.36	$0.74^{b)}$		96
R^1: CH_3	R^2: $COOCH_3$	R^3:$(CH_2)_3$	0.40	0.56	$0.94^{c)}$		385
R^1: $S(CH_2)_2S$	R^2: SCH_3	R^3:CH_2	0.47	0.55	(0.81)		386
			0.35	0.47	0.54	$0.86^{b)}$	312
			0.47	$0.86^{b,c)}$			312

Parentheses indicate an irreversible redox reaction, a) in acetonitrile/tetrahydrofuran (5:1), b) vs. sce c) in acetonitrile

TABLE 7 Oxidation potentials of group 4 dimeric or trimeric tetrathiafulvalenes in methylene chloride (vs. Ag/AgCl electrode, 0.1 M Bu_4NClO_4 or 0.05 M resp. 0.1 M Bu_4NPF_6)

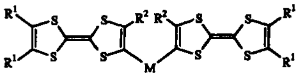
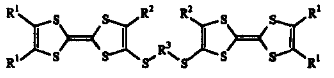
Compound			E_1^{Ox} (V)	E_2^{Ox}	reference
					
R^1: H	R^2: H	M: Hg	0.35	$0.68^{a,b)}$	311
R^1: CH_3	R^2: H	M: Hg	0.27	$0.66^{a,b)}$	311
R^1: CH_3	R^2: CH_3	M: Hg	0.28	$0.63^{a,b)}$	311
					
R^1: H	R^2: H	R^3:$(CH_2)_3$	0.44	0.78	331
R^1: H	R^2: H	R^3:$(CH_2)_4$	0.44	0.81	331
R^1: CH_3	R^2: H	R^3:$(CH_2)_2$	0.53	$0.87^{c)}$	385
R^1: CH_3	R^2: H	R^3:$(CH_2)_3$	0.46	$0.78^{c)}$	385

TABLE 7 *(Continued)*

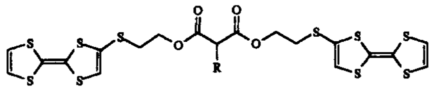
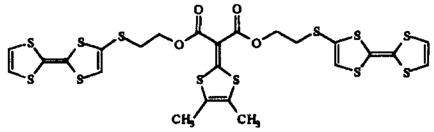
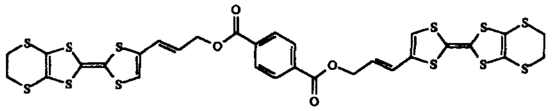
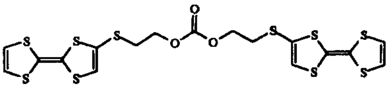
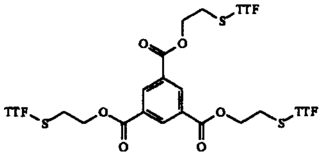
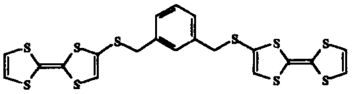
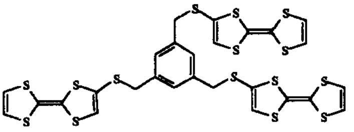
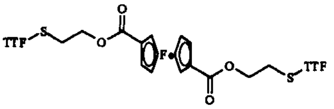
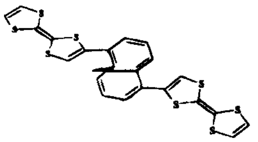
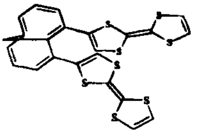
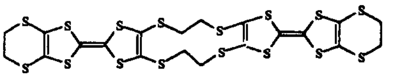
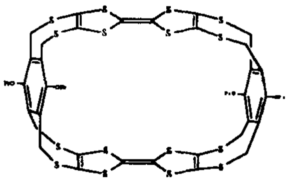
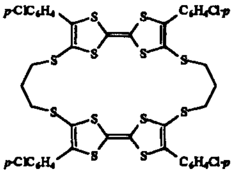
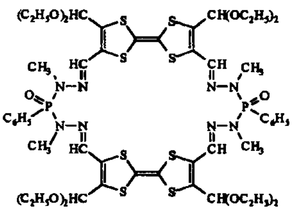
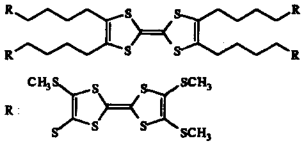
R^1: CH_3	R^2: H	R^3:$(CH_2)_4$	0.44	$0.78^{c)}$	385
R^1: CH_3	R^2: CH_3	R^3:$(CH_2)_3$	0.28	$0.75^{b)}$	96
R^1: CH_3	R^2: CH_3	R^3:$(CH_2)_{10}$	0.27	$0.77^{b)}$	96
R^1: CH_3	R^2: CH_3	R^3:$CH_2C_6H_4CH_2$(*o*)	0.29	$0.79^{b)}$	96
R^1: CH_3	R^2: CH_3	R^3:$CH_2C_6H_4CH_2$(*m*)	0.26	$0.75^{b)}$	96
R^1: CH_3	R^2: CH_3	R^3:$CH_2C_6H_4CH_2$(*p*)	0.28	$0.76^{b)}$	96
R^1: CH_3	R^2: $COOCH_3$	R^3:$(CH_2)_2$	0.60	$0.94^{c)}$	385
R^1: CH_3	R^2: $COOCH_3$	R^3:$(CH_2)_4$	0.56	$0.91^{c)}$	385
					
R: H			0.47	0.78	331
R: $C_{18}H_{37}$			0.47	0.79	331
			0.44	0.88	331
			0.49	$0.90^{b)}$	287
			0.46	0.81	331
			0.45	0.78	331
			0.45	0.79	331
			0.47	0.81	331
			0.37	$0.71^{c)}$	333
			0.40	$0.79^{b,d)}$	318

table continues

TABLE 7 *(continued)*

Compound	E_1^{Ox}	(V)	E_2^{Ox}		reference
	0.29		$0.89^{b,d)}$		318
	0.59		$(0.84)^{c)}$		387
	0.36		1.01	$1.06^{b,e)}$	388
	0.18		0.45		165
	0.61		$1.05^{b,f)}$		344
	0.52		$0.82^{g,h)}$		365

Parentheses indicate an irreversible redox reaction, a) in *N,N*-dimethylformamide, b) vs. sce c) in acetonitrile, d) in benzonitrile, e) the small potential difference reflects a small Coulomb interaction, f) in 1,1,2-trichloroethane, g) in methylene chloride/acetonitrile (3:1), h) broadened first wave indicates a non-simultaneous loss of electrons from the central and peripheral TTF

After removal of an electron the radical cations or dications formed are stable and can be isolated as the corresponding salts in most cases. The formation constants K of the radical cations are calculable from the redox potentials.[389]

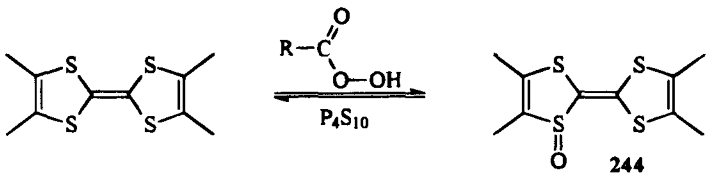
$$\log K = \frac{E_2^{Ox} - E_1^{Ox}}{0.059}$$

The radical cations give ESR spectra the resolution of which depends on the solvent and the formation conditions. The line width increases from tetrathiafulvalene[90,376,390–395] to tetraselenafulvalene radical cations.[396,397] The low solubility of TTeF has so far thwarted the generation of radical cations and the recording of their ESR spectra.[225] The oxidation of TTF[398,399] and TSF[399,400] can take place electrochemically. With a low current single crystals are generated.[400]

Also the application of oxidation agents affords radical cations or dications. TTF can be oxidized e.g. with halogens,[90,390,401,402] thiocyanogen,[403] metal salts,[404–406] hydrogen peroxide,[407,408] aryldiazonium salts,[409,410] lead(IV) acetate,[90] lead(IV) oxide,[379,411] or halocarbons in the presence of light[412,413] and TSF e.g. with iodine[192,414] or lead(IV) oxide.[205]

Radical cations are also formed in comproportionation reactions between the dications and the parent tetrathia- or tetraselenafulvalenes, respectively.[205,379,411]

Reduction of the monocations and dications with sodium bisulfite regenerates the corresponding TTF or TSF.[192,390] When TTF are oxidized with *m*-chloroperbenzoic acid the corresponding tetrathiafulvalene *S*-oxides are formed.[415,416] The tetrathiafulvalene *S*-oxides **244** are also formed under the influence of air oxygen on TTF.[416] Therefore TTF should be synthesized under exclusion of oxygen. With phosphorus pentasulfide the tetrathiafulvalene *S*-oxides **244** can be converted back to the corresponding TTF.[415]



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TTF, TSF, and TTeF have several bands in the ultraviolet-visible spectra. The energy of the longest-wavelength band is strongly influenced by substituents. This band can be found in TTF between 400 and 500 nm, in TSF between 490 and 510 nm, and in TTeF at up to 610 nm. Alkyl substituents shift this band in TTF and TSF bathochromically and aryl substituents hypsochromically compared to the unsubstituted compound. Aryl substituted TTF have a higher extinction coefficient (factor 20) than alkyl substituted TTF. Whereas with increasing ring size in cycloalkyl substituted TTF the longest-wave visible absorption is clearly shifted bathochromically, it is only slightly influenced by *p*-substituents in the aryl group.[379] This band is shifted to higher energy when the TTF and TSF are benzo-annelated. By changing the TTF to the corresponding TSF and TTeF one can find a bathochromic shift of the longest-wavelength band. This is consistent with its assignment as a $\pi \rightarrow \pi^*$ transition.[106,417–419] From a comparison of quantum-chemical calculations with experimental absorption spectra an assignment as an $n \rightarrow \pi^*$ transition[419] and a $\pi \rightarrow \sigma^*$ transition[376] is also possible.

As a rule the monocations and dications of TTF and TSF have a long-wavelength band at much lower energy than the corresponding TTF and TSF. Of extreme magnitude is the bathochromic shift in aryl substituted compounds which have donor substituents in the aryl group. Only the dications of the unsubstituted and of the aryl substituted TTF have a absorption at shorter wavelength than the corresponding TTF. In the series of the tetrathiafulvalene monocations and dications the longest-wavelength band is shifted bathochromically by alkyl as well as by aryl substituents as compared to the unsubstituted compound. This band obeys a Hammett correlation in the case of aryl substituted tetrathiafulvalene dications (Figure 2).[379]

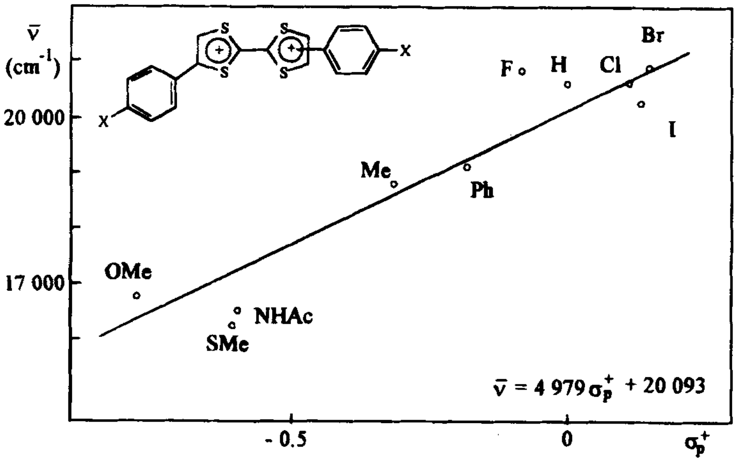


FIGURE 2 Wavenumbers of the longest-wavelength band of 2,6(7)-diaryl substituted TTF dications as a function of substitutent constants.

^{77}Se NMR spectroscopy shows the formation of a mixture of *cis*/*trans* isomers in the synthesis of unsymmetrically substituted TSF. The proton noise-decoupled ^{77}Se NMR spectra of this mixture display four well-resolved lines corresponding to separate signals for *cis* and *trans* isomeric compounds. These signals allow the *cis*/*trans* isomer ratio to be determined. The coupling constants between the selenium atoms in both rings are different in the *cis* and the *trans* structure and allow their assignment to the *cis*- and *trans*-isomer.[420,421]

The mass spectrometric fragmentation of TTF, DSDTF, and TSF shows great similarities within the selenium and the sulfur series. The main difference between the selenium and the sulfur compounds results from the more facile loss of selenium compared with sulfur, and from the first fragmentation of the molecular ion. Whereas the TSF first lose an alkyne molecule, the TTF first lose an (SCR) radical.[422]

6.3. *Charge-Transfer Complexing of Tetrathia-, Tetraselena-, and Tetratellurafulvalenes*

In unpolar solvents TTF form with electron acceptors, like quinones,[423] carbon tetrachloride[377,412] or TCNQ,[424] charge-transfer complexes. In their spectra they show a new absorption band which can be associated neither with the neutral donor or acceptor nor with their radical cations or radical anions. The energy of this charge-transfer absorption of TTF and TCNQ increases linearly with decreasing E_T value of the solvent (Figure 3) and decreasing oxidation potential of the TTF (Figure 4).[424]

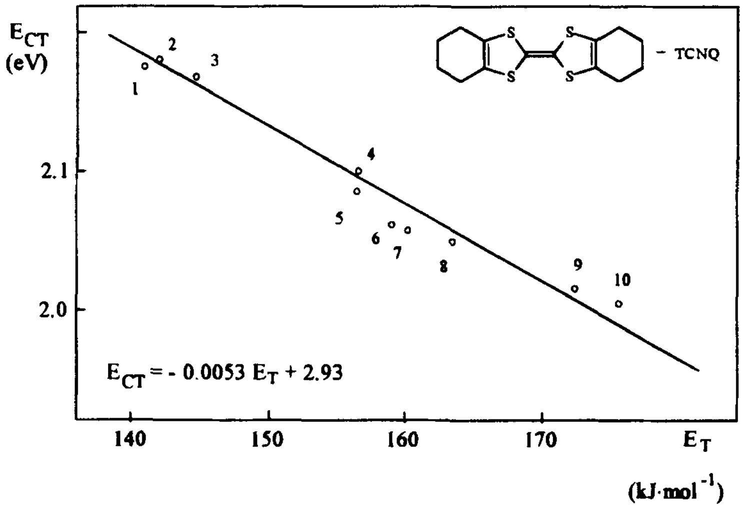


FIGURE 3 Solvent dependence of the charge-transfer absorption of the octamethylenetetrathiafulvalene-TCNQ charge-transfer complex (1 *t*-butylbenzene, 2 toluene, 3 benzene, 4 chlorobenzene, 5 bromobenzene, 6 iodobenzene, 7 1,2-dichlorobenzene, 8 chloroform, 9 dichloromethane, 10 1,2-dichloroethane).

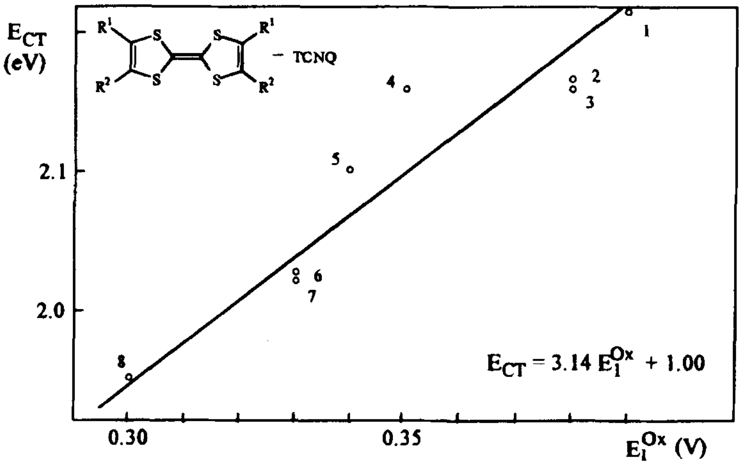


FIGURE 4 Charge-transfer absorption of substituted TTF-TCNQ charge-transfer complexes in chloroform as a function of half-wave oxidation potentials.

Compound	R^1	R^2	Compound	R^1	R^2
1	C_6H_5	H	5	C_6H_5	CH_3
2	p-ClC_6H_4	H	6	p-$CH_3C_6H_4$	CH_3
3	p-BrC_6H_4	H	7	p-$CH_3OC_6H_4$	H
4	p-$CH_3C_6H_4$	H	8	p-$CH_3OC_6H_4$	CH_3

TTF, TSF, and TTeF form with many electron acceptors crystalline charge-transfer salts which possess remarkable electric properties. It was discovered in 1973 that single crystals of the charge-transfer complex tetrathiafulvalene-TCNQ show metallic conductivity.[425] Over the years many charge-transfer salts with high conductivity have been encountered.

In the series of TTF, TSF, and TTeF high conductivity of charge-transfer salts is only observed when the following two conditions are fulfilled (in a simplified fashion):

—the donor and the acceptor or only the donor crystallize in segregated and uniform stacks
—partial charge transfer from donor to acceptor in the range 0.5 to 0.9 electrons per molecule takes place

Within the stack the interplanar separations between molecules along the stacking axis are uniform and in the range of the van der Waals radii. Thereby along the stack an overlap of π-orbitals on neighboring molecules arises and a conducting band is formed. By reason of partial charge transfer from donor to acceptor the conduction band is partially filled resulting in a high electron mobility along the stacking axis. Accordingly, the conductivity is much higher in single crystals along the stack than perpendicular to it. An anisotropy of conductivity is the consequence of this system, which is characterized as a one-dimensional conductor. The ratio of the conductivity along the stacking axis to the one perpendicular to it reaches values from 10^2 to 10^3.[426]

In general the charge-transfer salts of TSF exhibit a higher conductivity than the corresponding salts of TTF. Despite diminished donor ability of the TSF in comparison to the TTF the larger orbitals of selenium cause a better overlap of the π-orbitals within the donor stack and allow a better electron transport. Therefore a further enhancement of the conductivity should be expected in the salts of TTeF. The conductivity values obtained by different laboratories for the same complexes are quite different, so that only further investigations of complex composition and crystal structure will give a more exact explanation.[427]

Cooling of a single crystal of a one-dimensional conductor causes an increase in the conductivity, which decreases dramatically with further decreasing temperature. This metal-insulator transition can essentially have three reasons.[428]

—For energetic reasons there is a variation of distances between molecules within the stacks. These are no longer uniform and in the simplest cases the molecules dimerize. Thereby the band structure with uncompletely filled bands is lost. Dimerization gives rise to a band splitting with completely filled bands and empty ones and consequently an insulating state (Peierls transition).
—The Coulomb repulsion between electrons within a molecule stack is more important than any possible interaction with the lattice. In this way the electron mobility is decreased as a result of a spin orientation in alternating fashion. One observes an insulating magnetic state, called a spin density wave (SDW) state.
—In some charge-transfer salts with inorganic anions (e.g. BF_4^-, ClO_4^-, ReO_4^-, PF_6^-, $SbCl_6^-$, AsF_6^-) there is an ordered array of these anions with a periodicity of 2 a (a = lattice constant). This causes in most cases a gap in the band structure and thus semiconducting behavior.

The charge-transfer salts are three-dimensional crystals and therefore there is not only an intrastack electronic interaction, but also interstack electronic coupling. With increasing two- or three-dimensional character of electronic interaction the metal-insulator transition appears not so pronounced or is suppressed.

Application of pressure on charge-transfer salts squeezes the molecules closer together intrastack as well as interstack. On the one hand the conductivity increases thereby and on the other hand the metal-insulator transition is partially or completely suppressed.

The formation of segregated and uniform stacks with partial charge transfer essentially depends on steric and electronic properties of both complexing agents and crystallization conditions. The investigation of these problems has been the subject of many conferences and reviews, some of which are referred to here.[1-37]

A small selection of charge-transfer complexes with high conductivity and semiconducting properties is shown in Table 8.

TABLE 8 Electric conductivity, temperature of metal-insulator transition and structure of selected charge-transfer complexes

complex	σ_{RT} [S-cm^{-1}]	T_{MI} [K]	structure	reference
TTF-TCNQ	600	53, 38	D, A	429–431
DSDTF-TCNQ	550	45	D, A	106, 432
TSF-TCNQ	800	28	D, A	200, 432, 433
TMTTF-TCNQ	600	34	D, A	434, 435
TMTSF-TCNQ	1 000	57	D, A	436, 437
HMTTF-TCNQ	500	50,43	D, A	438
HMTSF-TCNQ	1 800	no	D, A	439, 440
HMTTeF-TCNQ	550		D, A	427
OMTTF-TCNQ	10^{-5}	—	DA	78, 441
TTF-TNAP	40	185	D, A	442
HMTSF-TNAP	2 400		D, A	443, 444
TMTTF-TCNTP	0.2[a)]			445, 446
DBTTF-TCNQ	10^{-7}	—	DA	79, 93, 447
DBTTF-2,5-TCNQF$_2$	10^{-6}	—	DA	447
DBTTF-2,5-TCNQCl$_2$	40	180	D, A	448, 449
DBTSF-TCNQ	$2.3 \cdot 10^{-6}$	—	DA	221
DBTSF-2,5-TCNQCl$_2$	20		D, A	221
TTF-chloranil	$8 \cdot 10^{-4}$	—	DA	450, 451
TTF-Br	10^{-6}	—	DD	401
TTF-Br$_{0.76}$	500	180	D	401, 452, 453
(TMTTF)$_2$ClO$_4$	30	70	D	454–456
(TMTSF)$_2$ClO$_4$	500	no	D	457–459
(BEDT-TTF)$_2$ReO$_4$	200	81	D	460, 461
(BEDT-TTF)$_2$I$_3$	250	no	D	462–464
(BEDT-TTF)$_2$AuI$_2$		no	D	465

[a)] compressed pellet, D donor stack, A acceptor stack, DA mixed stack, DD donor dimers, DSDTF diselenadithiafulvalene, TTF tetrathiafulvalene, TSF tetraselenafulvalene, TMTTF tetramethyltetrathiafulvalene, HMTTF and HMTSF and HMTTeF resp. hexamethylenetetrathiafulvalene, -tetraselenafulvalene, and -tetratellurafulvalene, resp., OMTTF octamethylenetetrathiafulvalene, DBTTF and DBTSF, resp., dibenzotetrathiafulvalene and -tetraselenafulvalene, resp., TNAP 11,11,12,12-tetracyano-2,6-naphthoquinodimethane, TCNTP 13,13,14,14-tetracyano-4,5,9,10-tetrahydro-2,7-pyrenoquinodimethane, 2,5-TCNQF$_2$ and TCNQCl$_2$, resp., 2,5-difluoro- and 2,5-dichlorotetracyanoquinodimethane, resp.

Doping of an electrically insulating polymer (polyester, polycarbonate, polysulfone, polyacrylate, polyacetal) with such a highly conducting charge-transfer complex provides an electrically conductive polymer. The conductivity increases by more than 15 orders of magnitude at a doping level lower than 1 wt.%. This can be explained by the fact that the conductive charge-transfer complex is not statistically dispersed in the polymer matrix of the system considered, but forms dendrite-like structures. These electrically conductive compositions can be used as antistatic and conductive layers in electrographic, electrophotographic, and photographic elements.[486–489]

UV illumination of a TTF-halocarbon charge-transfer complex on a surface (glass, paper) forms a coloured TTF radical salt with conductive properties. After subsequent electrolytic deposition of metal from a metal salt solution the conductive pattern can be used as a print circuit. During the metal deposition the organic layer is simultaneously removed which results in a high adhesion of the metal image to the surface.[412,413,470]

Conducting charge-transfer salts with TTF as donors and halogens as acceptors can be used as electron beam resist materials. Exposure of the charge-transfer salt to an electron beam causes the reverse of the charge-transfer, forming TTF and halogen. The reaction products can be sublimed (positive resist) or the unexposed areas dissolved in polar solvents (negative resist). The unexposed parts of the resist are conducting and therefore no build-up of charge occurs and no loss of resolution is caused.[471] The TTF-TCNQ complex is used as an electrode material.[472–474] Analogously the hexamethylenetetratellurafulvalene-TCNQ complex is a suitable electrode material for the amperometric detection of biologically important species (e.g. glutathione, cysteine, ascorbic acid, NADH).[475]

6.4. *Organic Superconductors*

The intense effort to find new charge-transfer salts with high conductivity and metallic properties led to the discovery of superconductivity in these radical salts. First of all in the tetramethyltetraselenafulvalenium salt $(TMTSF)_2ClO_4$ high conductivity was observed down to low temperatures and transition to the superconducting state (1.4 K) at ambient pressure.[457] Since then many charge-transfer complexes with tetrachalcogenafulvalenes as donors have been prepared in the hope to find new organic conductors or even superconductors. Superconductivity has been observed in a complex with the unsymmetrically substituted TTF derivative MDT-TTF, in complexes with the DSDTF derivative DMET and in a complex with $Ni(dmit)_2$ as acceptor (Table 9). However, the highest temperature T_c for the transition of an organic conductor to an organic superconductor is observed with BEDT-TTF salts, a selection of which can be found in Table 9. Also in a complex with the oxygen-containing donor BEDO-TTF superconductivity at ambient pressure has been found.

7. SYNTHESIS AND PROPERTIES OF POLYMERIC TETRATHIAFULVALENES

The high electric conductivity of tetrathiafulvalene charge-transfer complexes has also stimulated the synthesis of polymers with tetrathiafulvalene units. Thus, many polymers with tetrathiafulvalene units have been prepared by different synthetic methods.

TABLE 9 Organic superconductors and their transition temperature T_c to superconductivity

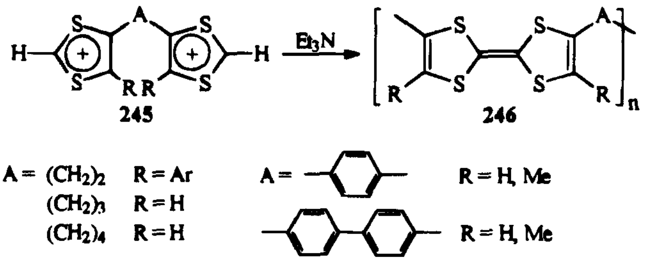
complex	T_c^* [K]		reference
$(TMTSF)_2ClO_4$	1.4		457
$(TMTTF)_2Br$	0.8	(26 kbar)	476
$(MDT\text{-}TTF)_2AuI_2$	3.5		477
$(DMET)_2AuCl_2$	0.83		478
$(DMET)_2AuBr_2$	1.9		479
$(DMET)_2AuI_2$	0.55	(5 kbar)	478
$(DMET)_2Au(CN)_2$	1.1	(2.5 kbar)	480
$(DMET\text{-}TSF)_2AuI_2$	0.58		481
$TTF[Ni(dmit)_2]_2$	1.6	(7 kbar)	482
$(BEDT\text{-}TTF)_2ReO_4$	2	(4 kbar)	483
$\beta\text{-}(BEDT\text{-}TTF)_2I_3$	1.5		462
	8	(1.3 kbar)	484
$\kappa\text{-}(BEDT\text{-}TTF)_2I_3$	3.6		485
$\theta\text{-}(BEDT\text{-}TTF)_2I_3$	3.6		486
$(BEDT\text{-}TTF)_2IBr_2$	2.5		487
$(BEDT\text{-}TTF)_2AuI_2$	3.8–5		465
$(BEDT\text{-}TTF)_2(I_3)_{2.5}$	2.5		488
$\alpha\text{-}(BEDT\text{-}TTF)_2(NH_4)Hg(SCN)_4$	0.8		489
$(BEDT\text{-}TTF)_4Hg_3Cl_8$	1.8	(12 kbar)	490
$(BEDT\text{-}TTF)_4Hg_3Br_8$	4.3		491
$(BEDT\text{-}TTF)_3Cl_2(H_2O)_2$		(16 kbar)	492
$(BEDT\text{-}TTF)_4Pt(CN)_4(H_2O)$	2	(6.5 kbar)	493
$(BEDT\text{-}TTF)_4Pd(CN)_4(H_2O)$	1.2	(7 kbar)	494
$(BEDT\text{-}TTF)_2Ag(CN)_2(H_2O)$	5.0		495
$(BEDT\text{-}TTF)_2Cu(NCS)_2$	10.4		496
$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Br$	11.6		497
$(BEDT\text{-}TTF)_2Cu[N(CN)_2]Cl$	12.5	(0.3 kbar)	498
$(BEDT\text{-}TTF)_2Cu[N(CN)_2]CN$	11.2		499, 500
$(BEDT\text{-}TTF)_2Cu_2(CN)_3$	3.8		498, 500
$(BEDT\text{-}TTF)_2Cu(CF_3)_4(TCE)$	4.0		501
$(BEDT\text{-}TTF)_2Cu(CF_3)_4(TCE)_x$ ($x < 1$)	9.2		502
$(BEDT\text{-}TTF)_2Ag(CF_3)_4(TCE)$	11.1		503
$(BEDO\text{-}TTF)_3Cu_2(NCS)_3$	1.06		504

*unless otherwise noted, T_c is at ambient pressure, MDT-TTF methylenedithiotetrathiafulvalene, DMET dimethyl(ethylenedithio)tetrathiafulvalene, DMET-TSF dimethyl(ethylenedithio)tetraselenafulvalene, TMTTF and TMTSF, resp., tetramethyltetrathiafulvalene, -tetraselenafulvalene, resp., BEDT-TTF and BEDO-TTF, resp., bis(ethylenedithio)- and bis(ethylenedioxo)tetrathiafulvalene, resp., dmit dimercaptoisotrithione or 1,3-dithiole-2-thione-4,5-dithiolate

7.1. *Deprotonation of Bis-1,3-dithiolium Salts*

In a manner analogous to the synthesis of 1,3-dithiolium salts (see Chapter 1.2.) the bis-1,3-dithiolium salts **245** can be synthesized and converted with tertiary aliphatic amines (e.g. triethylamine) in acetonitrile or acetone to the polymeric TTF **246**.[505–508]

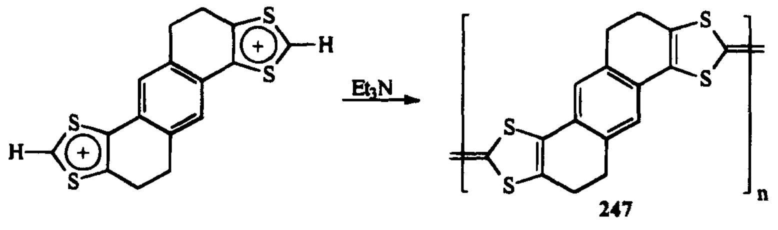
Terminal group analysis showed that the molecular weight (M_n) of the polymeric TTF **246** was about 2000–4000. The polymers **246** are insoluble in all common solvents. Only in sulfuric acid are they soluble by virtue of oxidation to the dications of the TTF unit. With lead(IV) oxide in acetonitrile oxidation to the dication occurs, too. The weaker oxidants bromine and iodine oxidize the polymeric TTF **246** only to the monocations of the TTF unit which can be characterized ESR spectroscopically via a structureless singlet.[506–508] Iodine



SCHEME 141

or TCNQ converts the polymeric TTF **246** to the corresponding charge-transfer complexes with semiconductor properties. Arylene-bridged polymeric tetrathiafulvalene radical salts with their conjugation along the chain possess a higher conductivity, by several orders of magnitude, than the alkylene-bridged derivatives.[508]

Deprotonation of the corresponding bis-1,3-dithiolium salt provides the planar and conjugatively connected polytetrathiafulvalene **247**. This polymer reacts with bromine, iodine or TCNQ and forms radical cation salts with an electrical conductivity of up to $3 \cdot 10^{-4}$ S·cm^{-1}.[509] The radical cation salts of **247** show a higher conductivity than twisted phenylene-bridged polymeric radical cation salts **246**.[509–511] The oxidation of the polymer **247** with bromine or iodine leads to the generation of charge carriers (polarons, bipolarons) which are responsible for conductivity.[512,513]



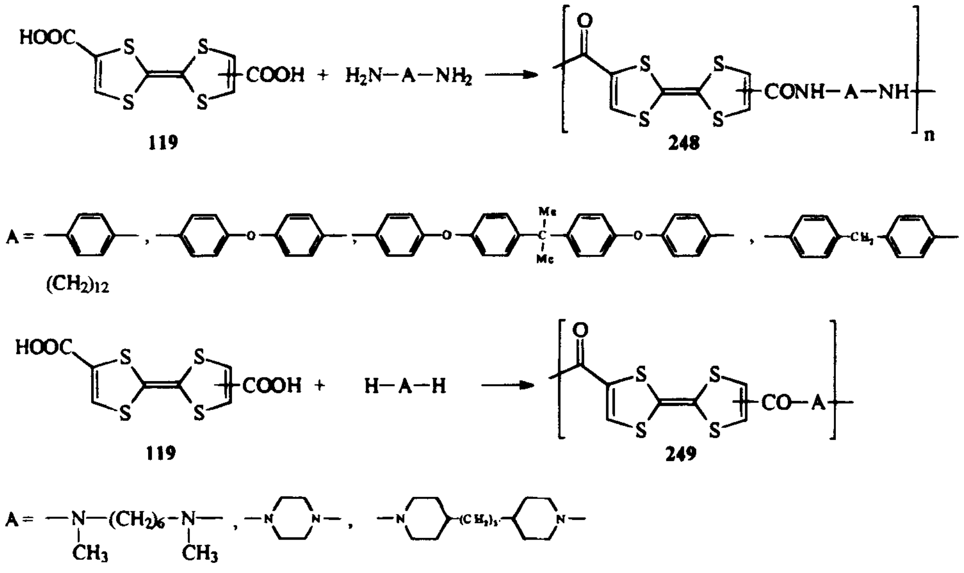
SCHEME 142

Polymeric TTF **246** can be used as a catalyst in electrodes for fuel cells. Addition of tetrathiafulvalene polymer to a carbon electrode decreases the activation energy for cathodic oxygen reduction.[514]

Polymeric TTF **246** are also suitable for ion sensitive electrodes as membrane active substances. Especially, they surpass hitherto known electrodes as an iodide sensitive electrode over a wide range of concentrations with short potential adjusting time and low sensitivity for chloride and bromide.[515]

7.2. *Polycondensation of Tetrathiafulvalene Derivatives*

Bivalent tetrathiafulvalene derivatives condense with bivalent amines, alcohols, carboxylic acids, isocyanates, or sulfonyl chlorides to form polyamides,[516–518] polyesters,[519,520] polyurethanes,[521,522] and polysulfonates,[522] respectively.



SCHEME 143

The polycondensation of the diacid **119** with amines is readily effected in *N*,*N*-dimethylformamide at 70 °C with diphenyl phosphite/pyridine[516] or in hexachloroethane with triphenylphosphine/pyridine[517] as dehydration reagent. The polyamides **248** and **249** are only soluble in trifluoroacetic acid, hexamethylphosphoramide, or dimethyl sulfoxide. With TCNQ they do not form charge-transfer complexes.[516] With bromine the tetrathiafulvalene unit is oxidized to the monocation, causing an increase in the conductivity ($\sigma_{RT} \leq 10^{-5}$ $S{\cdot}cm^{-1}$).[517]

A polypeptide **250** with attached tetrathiafulvalene units has also been prepared.[518]

The polyesters **251** are completely soluble in trifluoroacetic acid and soluble in *N*,*N*-dimethylformamide, dimethyl sulfoxide, and hexamethylphosphoramide. They do not form any charge-transfer complexes with TCNQ, but can be oxidized with bromine to radical salts.[519]

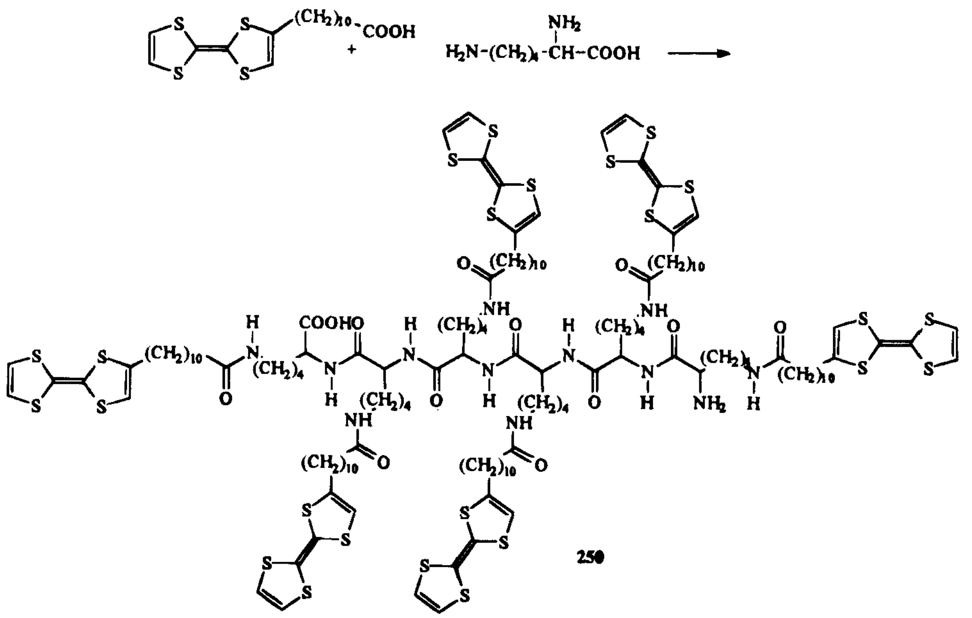
The polyesters **252** (n: 3–5; M_n 2000) are insoluble in organic solvents. Upon grinding of the polyesters **252** with TCNQ their conductivity is enhanced by one to five orders of magnitude ($\sigma_{RT} \leq 10^{-7}$ $S{\cdot}cm^{-1}$).[520]

The brown polyurethane **253** is insoluble, but can be oxidized with iodine, whereby the conductivity increases by two orders of magnitude ($\sigma_{RT} = 2{\cdot}10^{-6}$ $S{\cdot}cm^{-1}$).[281]

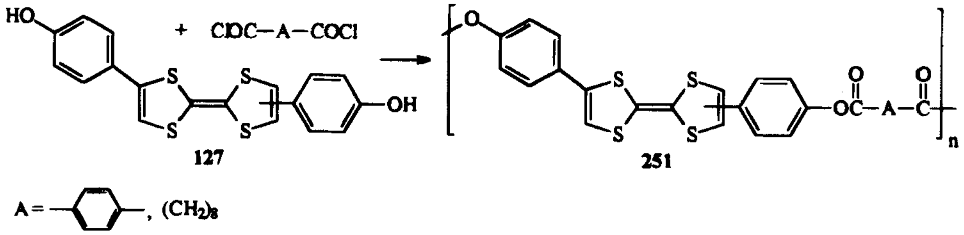
In the same manner a charge-transfer polymer **254** containing covalently bound TCNQ has been prepared. The black polyurethane **254** is amorphous and possesses an electric conductivity of $1{,}7{\cdot}10^{-7}$ $S{\cdot}cm^{-1}$.[281]

The monomers **255** and **256** of similar structure have also been synthesized. Both appear to be zwitterionic.[282]

The polyurethanes **257** as well as the polysulfonates **258** are readily soluble in trifluoroacetic acid at room temperature and in *N*,*N*-dimethylformamide, dimethyl sulfoxide, and hexamethylphosphoramide upon heating. They form no charge-transfer complexes with TCNQ, but can be oxidized with bromine.[521]



SCHEME 144



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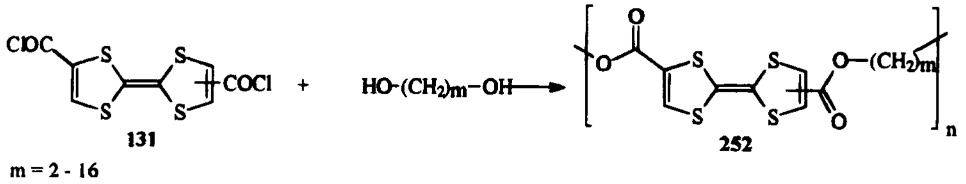
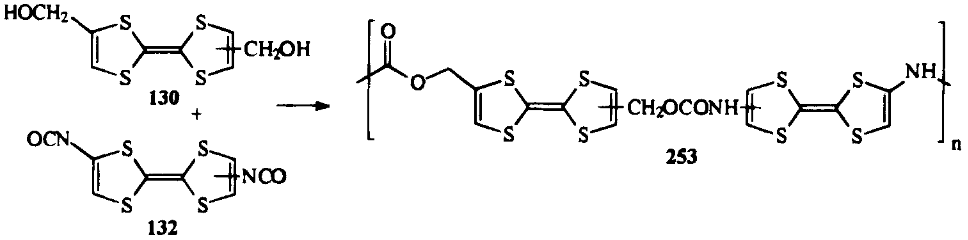


SCHEME 146

7.3. *Polymerization of Tetrathiafulvalene Vinyl Monomers*

Tetrathiafulvalene vinyl monomers can be polymerized under various reaction conditions. The vinyltetrathiafulvalene **259** can be polymerized thermally as well as by UV initiation, azobisisobutyronitrile, or by adding TCNQ, and forms low-molecular weight polymers **260**. This polymer **260** forms charge-transfer complexes with bromine or TCNQ.[305]



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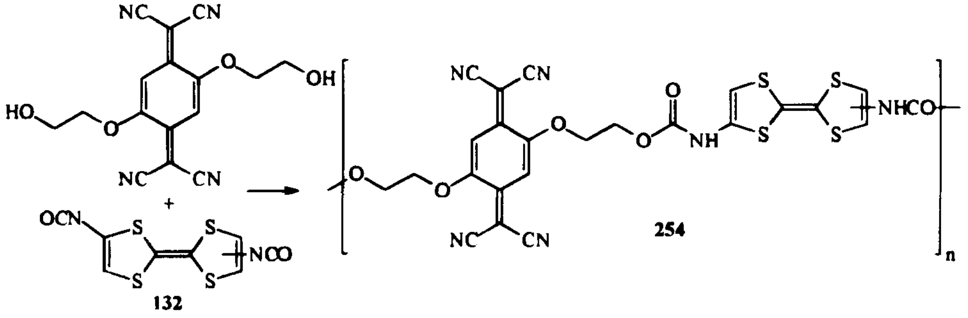
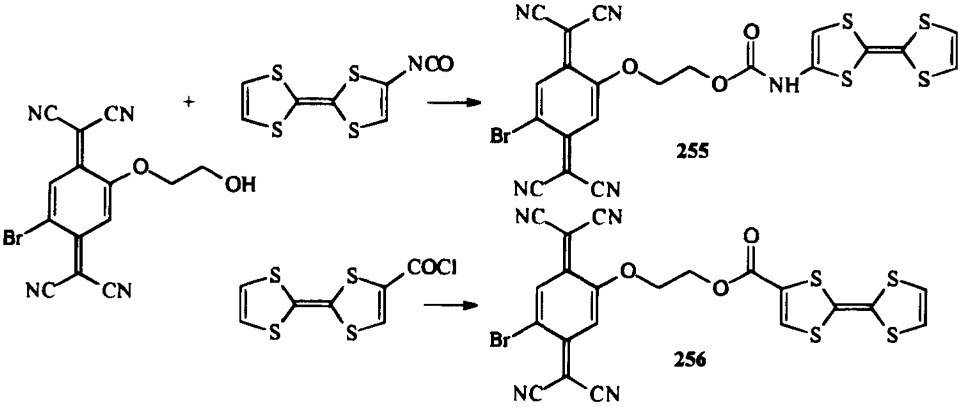


SCHEME 148



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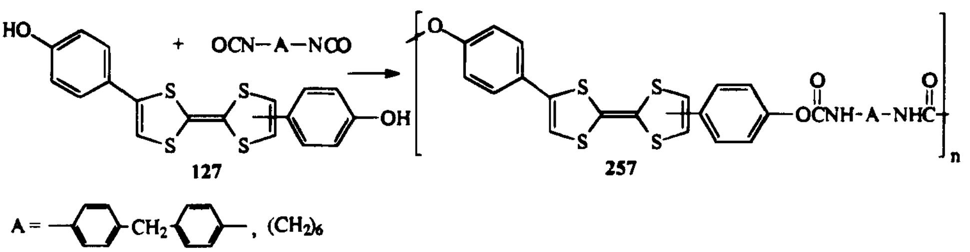
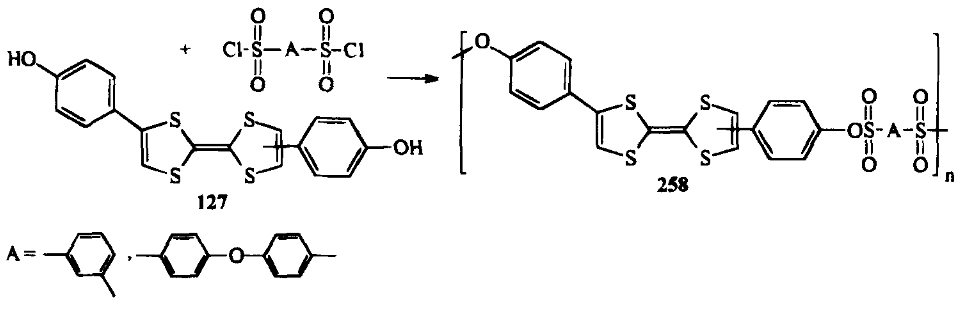


SCHEME 150



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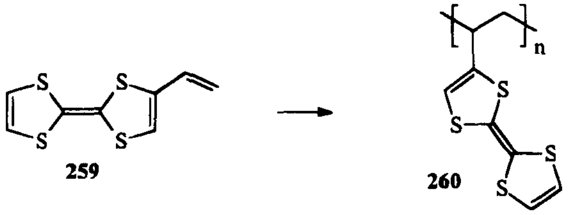
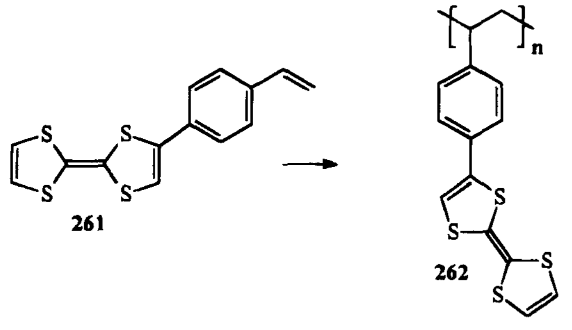


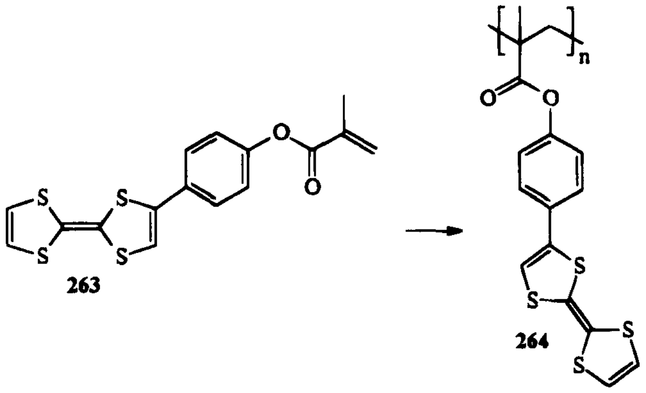
SCHEME 152

The polymerization of (*p*-vinylphenyl)tetrathiafulvalene **261** was carried out in a sealed tube *in vacuo* at 130 °C for 3 days. A polymer **262** with a molecular weight of about 50,000 was isolated. The brown polymer **262** is insoluble in all usual organic solvents. With bromine the polymer **262** is oxidized and its conductivity increases by two orders of magnitude to 10^{-8} S·cm^{-1}. Attempted anionic polymerization or initiation with azobisisobutyronitrile was unsuccessful.[523]



SCHEME 153

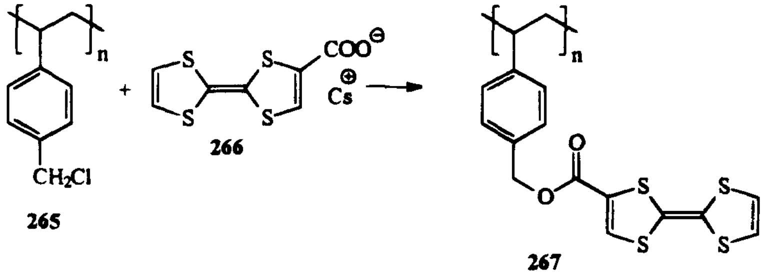
The polymerization of *p*-(2-tetrathiafulvalenyl)phenyl methacrylate **263** is only successful in a sealed tube *in vacuo* upon heating at 120 °C for 2 days. The black polymer **264** is only soluble in strong acids. It is converted with bromine to a bromine complex. The copolymerization with methyl methacrylate gives a soluble copolymer with a molecular weight (M_n) of about 10,000 (93.5% of MMA).[278]



SCHEME 154

7.4. *Reactions of Polymers*

Another polymeric tetrathiafulvalene is formed by reaction of a linear poly(vinylbenzyl chloride) **265** with the caesium salt of carboxytetrathiafulvalene **266**, where 30% of the sites have reacted with the tetrathiafulvalene moiety. The tetrathiafulvalene substituted polystyrene **267** is soluble in tetrahydrofuran and films can be prepared.[522]

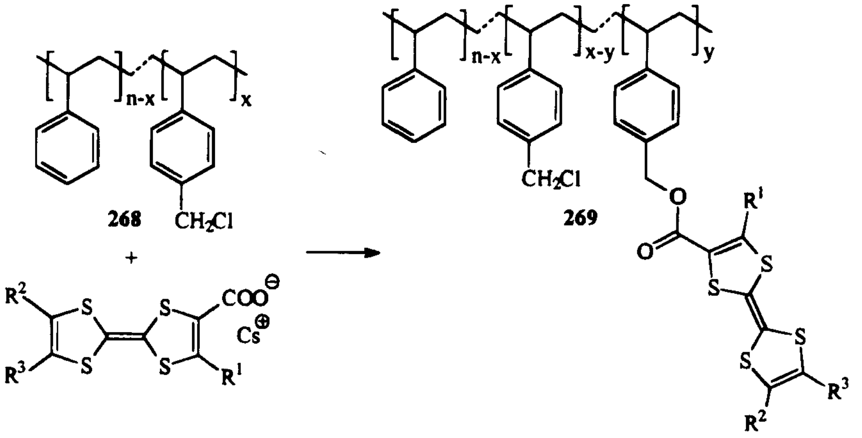


SCHEME 155

Such a polystyrene derivative with attached tetrathiafulvalene units is useful as a negatively working photoresist. Illumination of the polymer in the presence of carbon tetrabromide converts the weakly polar polymer to a more polar polymer, which is insoluble in solvents of low polarity (ether, ketones). After treatment with solvents the insoluble areas do not swell and therefore the resolving power is increased.[524]

Also the copolymer poly[styrene-co-*p*-(chloromethyl)styrene] **268** reacts with caesium tetrathiafulvalenecarboxylate and proceeds new polystyrenes **269** bearing tetrathiafulvalene units in the backbone.[525] The resists show under electron beam irradiation in the presence of dibromotetrachloroethane good properties as negative resists, including high sensitivity and high resolution.[526]

Analogous to the polymer **267** a polymer backbone with attached tetrathiafulvalene units has been prepared by reaction of poly[*p*-(chloromethyl)styrene] **270** or poly[styrene-co-



SCHEME 156

p-(chloromethyl)styrene] **268** with potassium 4-tetrathiafulvalenylphenoxide. The polymers **271** and **272** with a variety of controlled molecular weights and molecular weight distributions differ in their solubilities. Whereas polymers **271** are highly insoluble, the polymers **272** are soluble in dichloromethane.[527] The suitability of the polymers **271** and **272** for microlithographic processes is being studied.

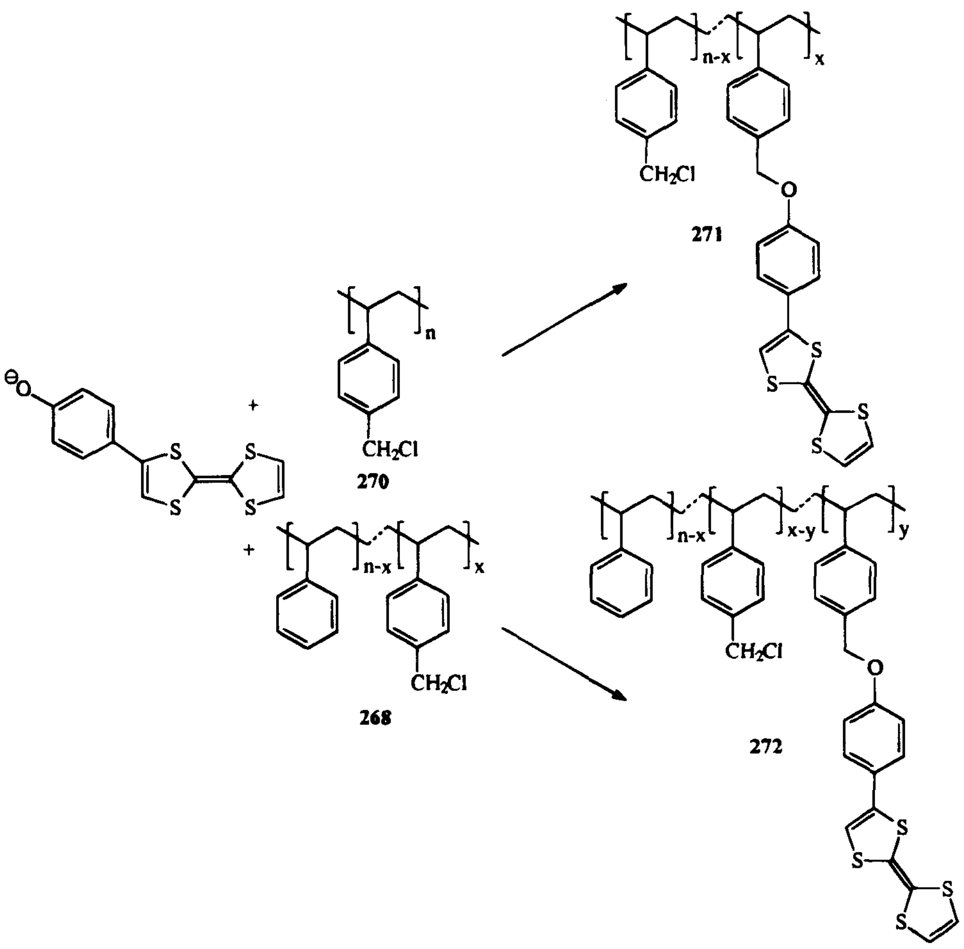
Tetrathiafulvalene substituted polystyrenes **274** and **275** are also obtained by acylation of an atactic polystyrene (M_n 3200) with α-halo acid halides and further conversion to the corresponding dithiolium salts **273** which are further treated with triethylamine.[528] These polymers **274** and **275** are insoluble in organic solvents and concentrated sulfuric acid. With iodine and TCNQ they form charge-transfer complexes whose conductivity differs only little from that the conductivity of the polymer (σ_{RT} about 10^{-10} S·cm^{-1}).[528]

7.5. *Miscellaneous Methods*

The reaction of dithione **276** with triethyl phosphite yields a black insoluble polymer, presumably **277**, which could not be characterized. The same product was also achieved by silylation of **276** with trimethylsilyl chloride, subsequent coupling with triethyl phosphite and hydrolysis with dilute HCl. Pressed pellets of the polymer show an electrical conductivity of about 10^{-2} S·cm^{-1} which increased up to two orders of magnitude upon exposure to iodine vapor. After acylation of **276** with hexanoyl chloride the dithione formed gives with triethyl phosphite a slightly soluble dark reddish-brown polymer, presumably with the structure **278**.[529]

Polymers with 1,3-dithiol-2-ylidene units has also been formed by electrolytic oxidation of the corresponding 1,3-dithiole monomers. After electrical doping the polymer films exhibit electrical conductivity (in the range of 10^{-4} to 50 S·cm^{-1} for polymer **279** and 10^{-3} to 10^{-2} S·cm^{-1} for polymer **280**).[530]

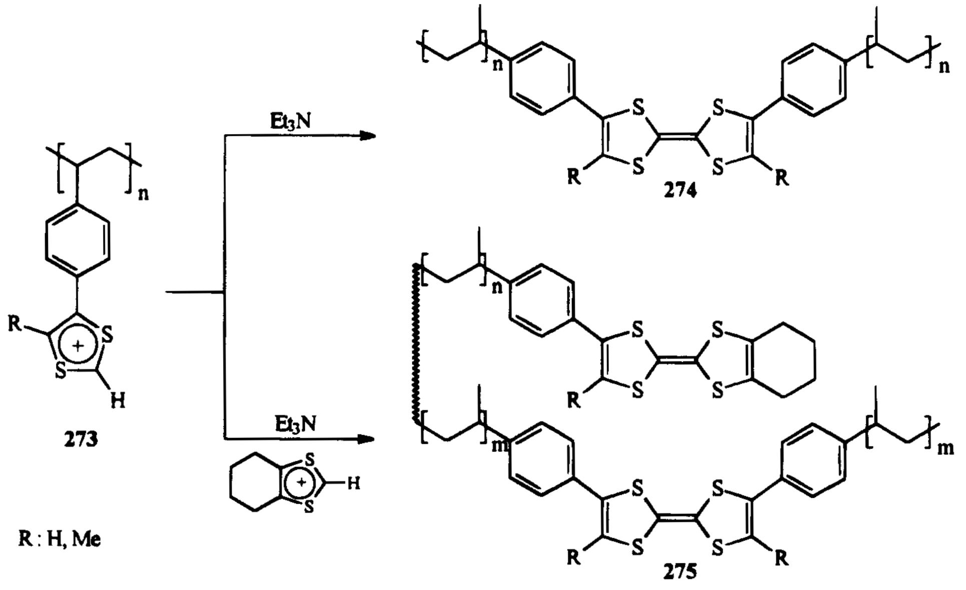
Molecules with more than one 1,4-dithiafulvene unit form polymers upon electrochemical oxidation (in analogy to the electrochemical oxidation of 1,4-dithiafulvenes, but in contrast to 1,2-xylene-α,α′-diylidenebis(1,3-dithioles), see Chapter 5).[236,531]



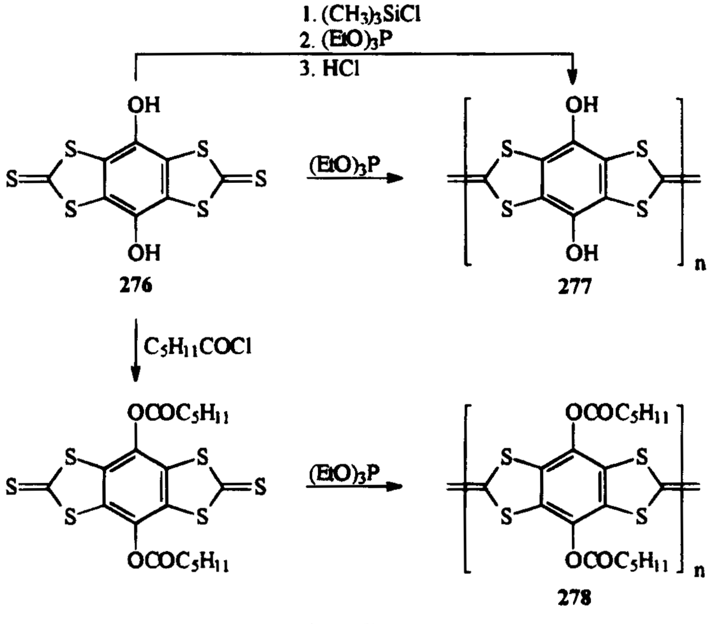
SCHEME 157

A TTF moiety covalently attached to a thiophene ring has been synthesized. Electropolymerization of this thiophene **281** in acetonitrile is unsuccessful, presumably because of the scavenging of the thiophene cation radicals by TTF, but can be achieved by application of repetitive potential scans in nitrobenzene solution. In agreement with UV-VIS absorption spectra the formation of a donor-acceptor complex between nitrobenzene and the TTF moiety decreases its reactivity toward thiophene cation radicals and enables polymerization.[532]

Tetracyanotetrathiafulvalene forms with metal acetylacetonates polymers with phthalocyanine-like structures **282** and with diamines polymers of the hemiporphyrazine type **283**, both containing tetrathiafulvalene units.[533,534] Both types of polymers **282** and **283** are dark coloured, insoluble in all solvents and show semiconductor properties ($\sigma_{RT} < 10^{-5}$ S·cm^{-1}). In the case of the hemiporphyrazine type polymers **283** the two hydrogen atoms inside the macroheterocyclic ring can be substituted by metal atoms. Depending on the metal atom



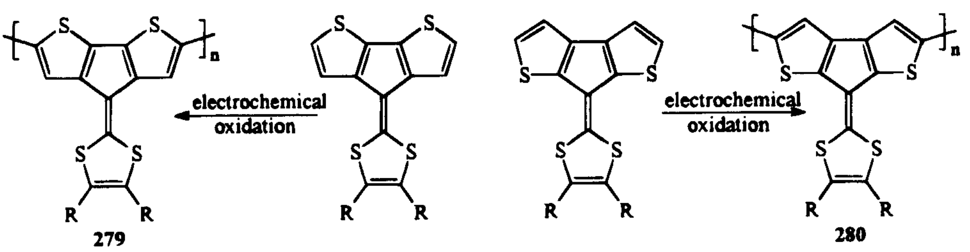
SCHEME 158



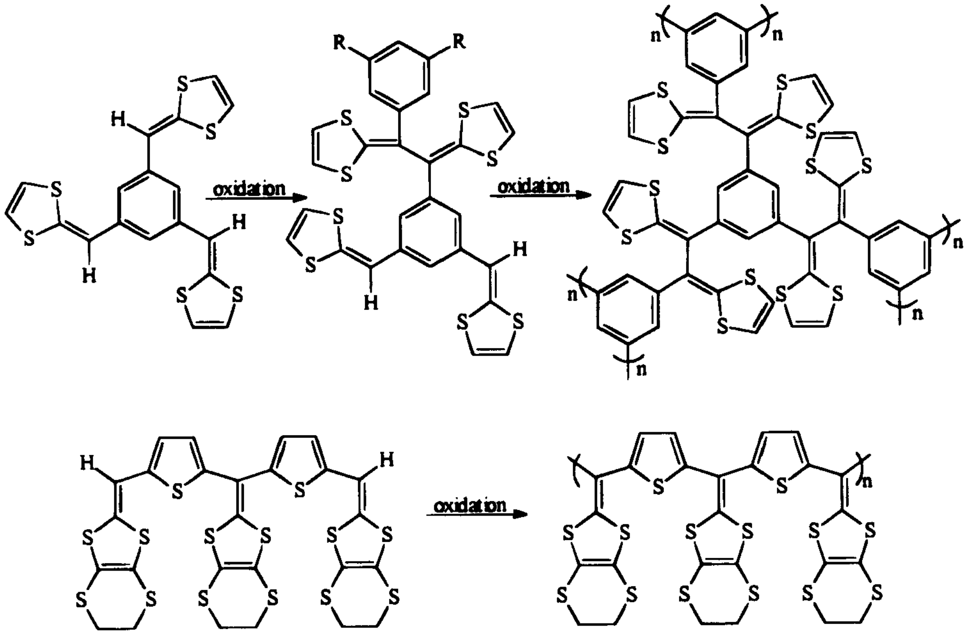
SCHEME 159

and the bridging group A the electric conductivity increases up to two orders of magnitude.[534]

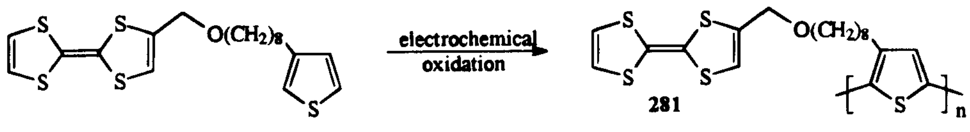
A polymer with bridged tetrathiafulvalene units by S-S linkages is formed by treatment of bis(tributylstannylthio)- TTF **238** with gaseous hydrogen chloride (see Chapter 6.1.).[95]



SCHEME 160



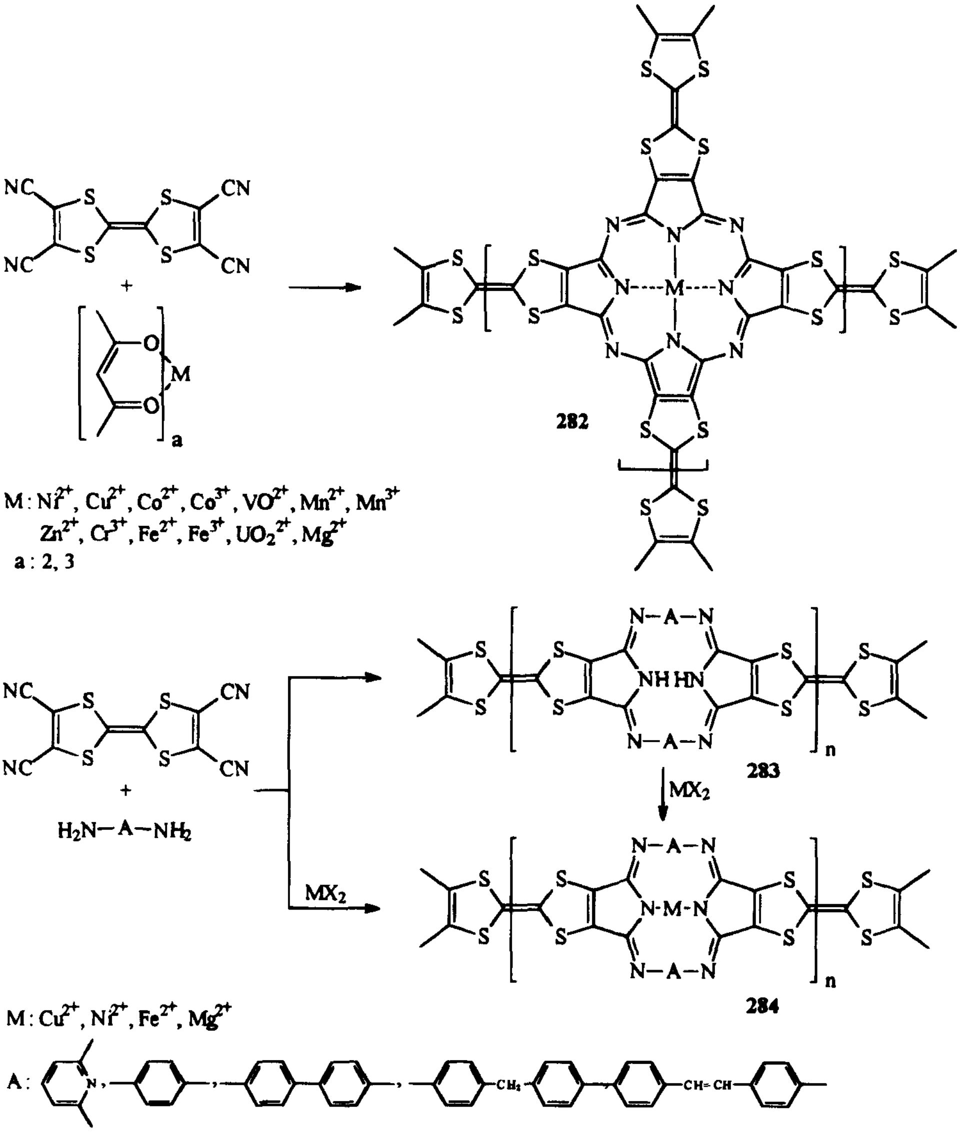
SCHEME 161



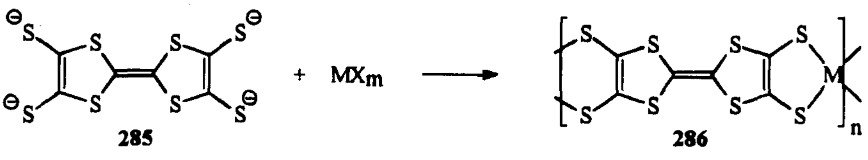
SCHEME 162

7.6. *Polymeric Tetrathiafulvalene Metal Bisdithiolene Complexes*

The reaction of tetrathiafulvalenetetrathiolate **285** with metal salts leads to the precipitation of insoluble, amorphous powders **286** with high electric conductivity (M: Cu $\sigma_{RT} = 10^{-1}$ S·cm^{-1}).[63,292,294] A ribbon structure, but also two-dimensional layers could be formed. Qualitative MO and band structure calculations support this assumption.[535]



SCHEME 163

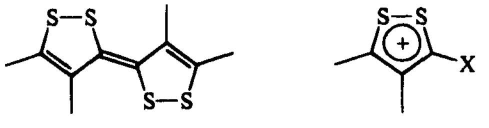


M : Ni, Cu, Fe, Pd, Pt

SCHEME 164

8. SYNTHESIS OF 1,2-TETRACHALCOGENAFULVALENES (1,2-TXF)

While the 1,3-tetrathiafulvalenes have been known for many years, especially since the discovery of the high electric conductivity of their charge-transfer complexes, and a lot of new and efficient syntheses have been described, the chemistry of the analogous 1,2-tetrathiafulvalenes **287** is poorly developed. The first synthesis of 1,2-TTF was reported in 1975.[536] Most pathways to 1,2-TTF start from the 1,2-dithioles **288–292**. In this report we are only reviewing some of the more common possibilities for the synthesis of these compounds (for more detailed information see reviews[83,84,537–539a]).

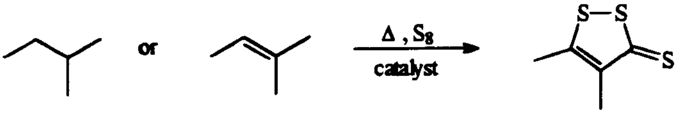

287 **288-292**

X=	S^-	O^-	H	Cl	SR
	288	**289**	**290**	**291**	**292**

SCHEME 165

8.1. *Synthesis of 1,2-Dithiole Derivatives*

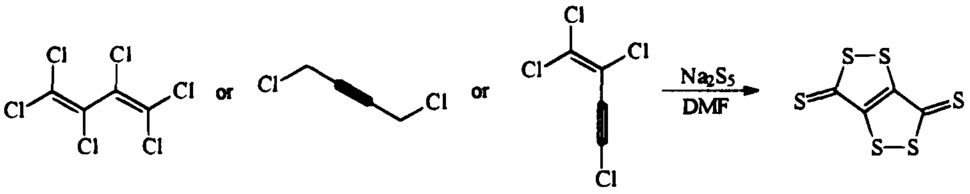
8.1.1. *1,2-Dithiole-3-thiones.* 1,2-Dithiole-3-thiones (trithiones) **288** are available by high-temperature sulfurization (200–500 °C) of the hydrocarbons **293** or **294** and their derivatives.[540] The yields are good in some cases and can be increased by the use of basic catalysts (e.g. diphenylguanidine).[541]



293 **294** **288**

SCHEME 166

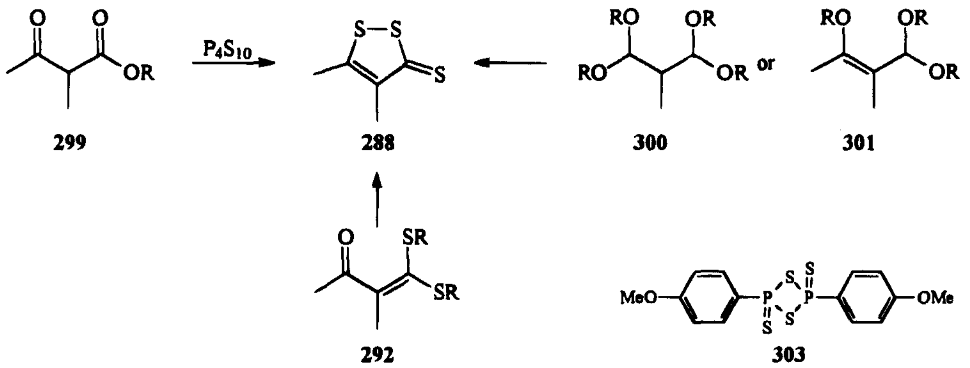
Polyhalogenated hydrocarbons can be thiated at lower temperatures in aprotic solvents (e.g. **295**, **296**, **297** to **298**).[542] A very efficient way to the trithiones **288** is the sulfurization of β-keto esters **299** and their functional derivatives (e.g. **300–302**) with phosphorus pentasulfide,[543,544] preferably in the presence of sulfur.[545]



295 **296** **297** **298**

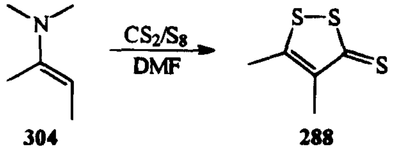
SCHEME 167

In many cases the yields can be considerably increased by use of the 2,4,1,3-diphosphadithietane **303** (Lawessons's reagent) as sulfurization agent.[546]



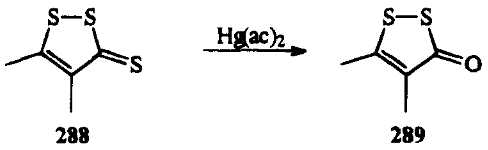
SCHEME 168

Another common way to trithiones **288** starts from the enamines **304**, carbon disulfide, and sulfur.[547]



SCHEME 169

8.1.2. *1,2-Dithiol-3-ones.* The 1,2-dithiol-3-ones **289** can be obtained from the corresponding 1,2-dithiole-3-thiones by desulfurization with mercury (II) acetate.[107]

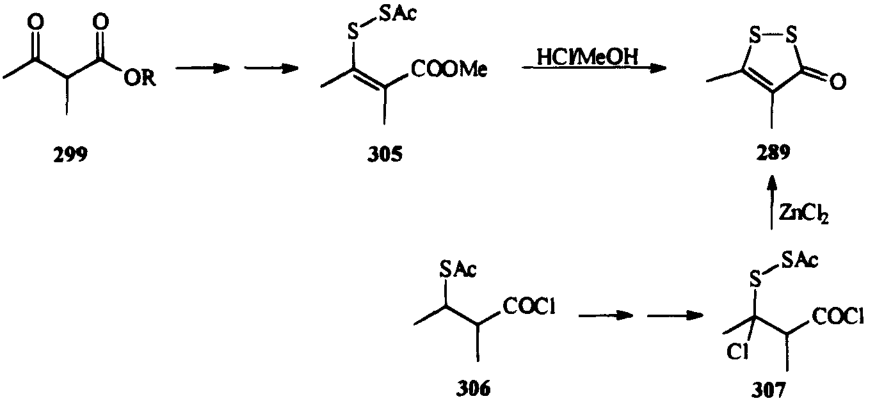


SCHEME 170

Furthermore, 1,2-dithiol-3-ones **289** can be synthesized from acyclic compounds. For instance the sulfurization of cinnamic esters with sulfur at 250 °C gives 5-phenyl-1,2-dithiol-3-one in yields of up to 65%.[548]

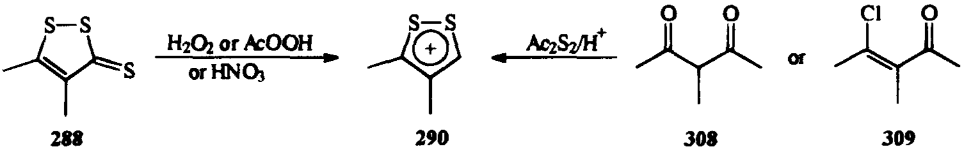
A common method used to obtain 1,2-dithiol-3-ones **289** starts from β-keto esters **299** and leads to the 1,2-dithiol-3-ones via the β-acetyldithioacrylic esters **305**.[549]

In a similar way the β-acetyl thioacid chlorides **306** give the compounds **289** via the β-acetyldithio-β-chloro acid chlorides **307**.[550]



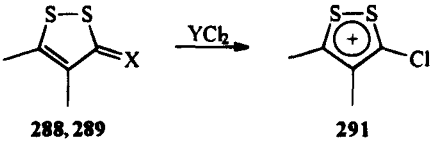
SCHEME 171

8.1.3. *3H-1,2-Dithiolium salts.* 3*H*-1,2-Dithiolium salts **290** can be obtained in a manner similar to that for the 2H-1,3-dithiolium salts **26** by oxidation of the corresponding trithiones with peracetic acid,[551] hydrogen peroxide,[552] or nitric acid.[553] Furthermore they are available from acyclic educts (e.g. **308** or **309**) by reaction with diacetyl disulfide in the presence of strong acids.[554]



SCHEME 172

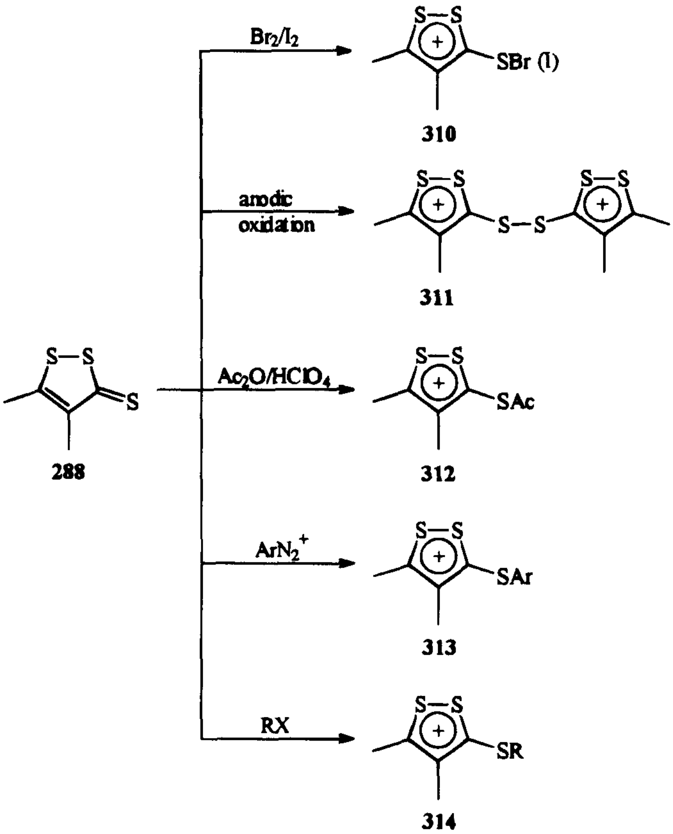
8.1.4. *3-Chloro-1,2-dithiolium salts.* 3-Chloro-1,2-dithiolium salts **291** can be synthesized from the corresponding 1,2-dithiole-3-thiones **288** or 1,2-dithiol-3-ones **289** by reaction with halogen transfer compounds (e.g. phosphorus pentachloride,[555] oxalyl chloride,[555,556] phosphorus oxychloride[555]).



Y : PCl_3, $POCl$, SO, CO
X : O, S

SCHEME 173

8.1.5. *Organylthio-1,2-dithiolium salts.* 1,2-Dithiole-3-thiones **288** are compounds with a strongly polar thione group which is readily attacked by electrophiles. With halogens the sulfenyl halides **310**[557] and by anodic oxidation the disulfides **311**[558] are formed. In the presence of perchloric acid the acylation to the reactive acylthiodithiolium salts **312** is possible.[559] Diazonium salts arylate to give the 3-arylthio-1,2-dithiolium salts **313**.[560] Due to the simple replacement of the arylthio group by nucleophiles and due to their ready availability[561] especially the 3-(alkylthio)- 1,2-dithiolium salts **314** are of high preparative interest.



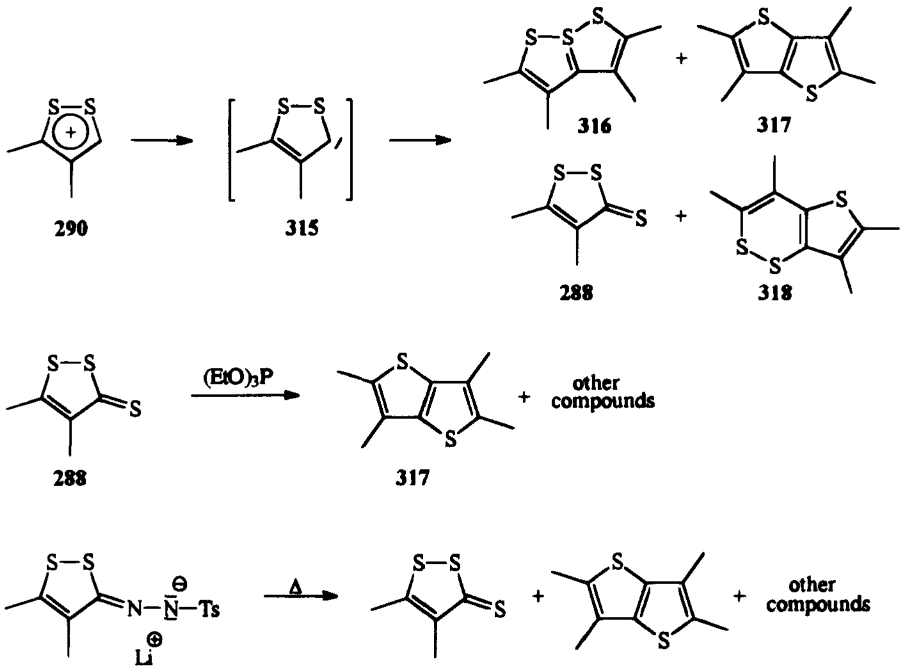
SCHEME 174

8.2. *Unsuccessful Attempts to Synthesize 1,2-TTF*

All attempts to make 1,2-TTF by deprotonation of the 3*H*-1,2-dithiolium salts **290** via the corresponding carbenes **315** failed. However, it has been claimed in a patent that methylthio, alkoxy and acetyloxy substituted 1,2-dithiolylium salts give rise to 1,2-TTF when treated with base in acetic anhydride.[841] The deprotonation of the 3H-1,2-dithiolium salts **290** leads to product mixtures from which 1,2-dithiole-3-thiones **288**,[562,563] thiathiophthenes **316**,[564] thieno[3,2-*b*]thiophenes **317**[565] and a thieno[3,2-*c*]dithiin **318**[563] could be isolated. The formation of these compounds can be explained via the 3-carbena-1,2-dithiole **315** as intermediate. Other reasons for the postulation of the carbene **315** as the primary deprotonation product of the dithiolium salt **290** are the results of the base catalyzed H/D exchange[562,566] and the observation that the yields of trithiones **288** increase markedly if the deprotonation is carried out with additional amounts of sulfur.[562]

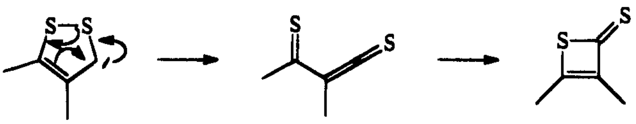
Also thermolysis of alkali salts of 1,2-dithiol-3-one hydrazones **319** leads not to 1,2-TTF, but to compounds of the types **288** and **317**.[563]

Furthermore, the reaction of trithiones **288** with trivalent phosphorus compounds—very successful in the synthesis of 1,3-TTF—leads to thieno[3,2-*b*]thiophenes **317** and **320**.[563,567,567a] Alkylthio substituted trithiones **288** form with triethyl phosphite compounds which have erroneously been ascribed the 1,2-TTF structure.[568] It has, however, been shown that these products are actually the isomeric thioxodesaurines.[567a,568a,573]



SCHEME 175

An explanation of the experimental observation that the postulated carbene **315** as intermediate does not yield 1,2-TTF has been given.[569] These authors carried out quantum chemical calculations on a CNDO/2 basis. They came to the conclusion that the carbenes **315** isomerize very fast to the thiete-2-thiones **321** with S-S bond cleavage. The open-chain thioacyl thioketenes **320** are discussed as intermediates. According to this calculation the possible dimerization of the carbene **315** to the 1,2-TTF is kinetically depressed by competition reactions.

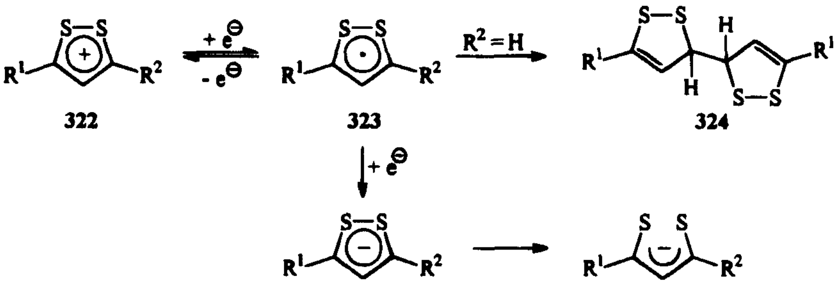


315 **320** **321**

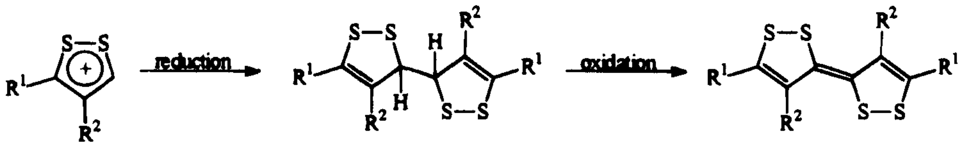
SCHEME 176

8.3. *Synthesis of 1,2-TTF*

8.3.1. *1,2-TTF by reductive dimerization of 1,2-dithiolium salts.* The cathodic reduction of the dithiolium salts **322** leads, with reception of one electron, to the corresponding 1,2-dithiolium radical **323**, which can dimerize to compound **324**.[570] The stability of the radical **323** and the equilibrium **323** ⇌ **324** depend on the nature of the substituents R^1 and R^2 and the temperature. Whereas in the case of the radicals **323** with $R^2 = H$ the equilibrium lies definitely on the side of the dimers the equilibrium in the case of **333** with $R^1 = R^2 = Ph$ lies on the opposite side. Reception of a further electron leads to the dithiolium anions **325**. This reduction step is irreversible, the reason being the formation of the dithiodiketonate anion **326**. The intermediate steps **322**–**326** can also be carried out photochemically.[570]



SCHEME 177



290 **327** **287**

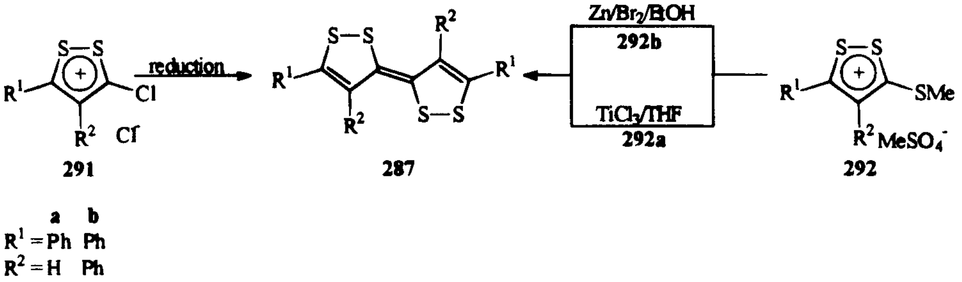
$R^1 = Ph, H$ $R^2 = Ph, Me, H$ $R^1, R^2 = (CH{=}CH)_2$

SCHEME 178

Preferred reagents for preparative reductions for the synthesis of the dimers **327** are zinc and aqueous titanium(III) chloride solutions.[571]

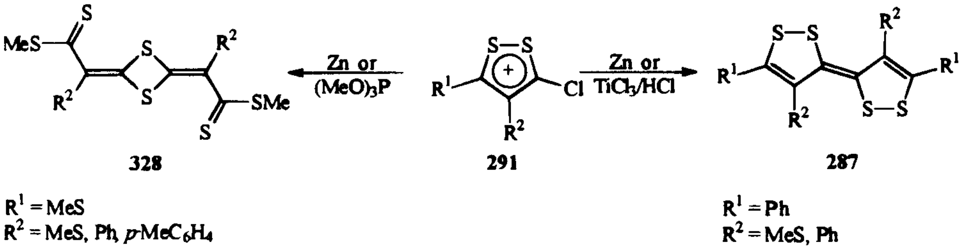
Contrary to earlier claims[572] some dimers **327** could be dehydrogenated by several oxidation agents to the 1,2-TTF **287**.[536,571]

The oxidation of the dimers **327** with iodine or bromine or their electrochemical oxidation leads to the dithiolium salts **290**. Reduction of 3-chloro-1,2-dithiolium salts **291** with zinc, silver, or aqueous titanium(III) chloride solution gives the 1,2-TTF **287** in one step.[571] The yield in the reduction with activated zinc is increased by supersonication.



SCHEME 179

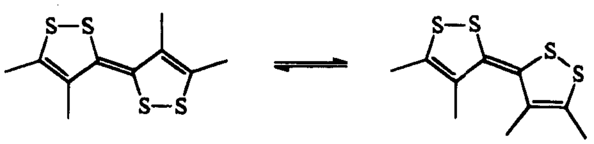
The conversion of the 3-(methylthio)-1,2-dithiolium salt **292b** to **287b** requires zinc as reducing agent and bromine as oxidative "extractor" for the alkylthio groups.



SCHEME 180

Reduction of the compound **292a** with titanium(III) chloride alone gave the 1,2-TTF **287a** in only about 3% yield. The dimerization of the 5-methylthio substituted 1,2-dithiolium salts with activated zinc or trimethyl phosphite gives the 1,3-dithietanediylidenebis(methyldithioacetate) derivatives **328**.[573]

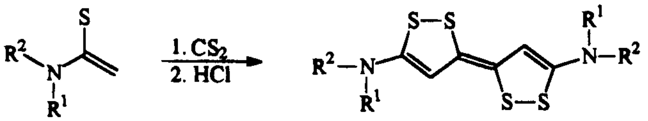
The choice of the reducing agent and the reduction conditions (solvent, temperature) have a great influence on the success of the dimerization reaction.[571]



287 **287'**

SCHEME 181

Some 1,2-TTF **287** could be isolated in two isomeric forms, characterized by their different UV-VIS spectra. The conversion of one form into another—considered as the *cis-trans* isomers **287/287'**—can be carried out thermally or photochemically.

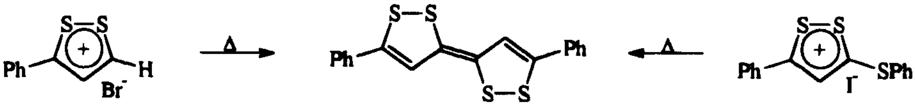


329 **287**

$R^1 = Me$ $R^2 = Ph, Me$ $R^1, R^2 = (CH_2)_2, (CH_2)_2O(CH_2)_2$

SCHEME 182

Besides the 1,2-TTF the reaction mixture also contains the corresponding trithiones **288**, in some cases additionally 1,2-dithiol-3-ones **289** and thieno[3,2-*b*]thiophenes **317**. The intermediate formation of the carbenes **315** has been discussed as one of the reasons for this observation.



290 **292**

SCHEME 183

8.3.2. *Formation of 1,2-TTF in other ways.* A further approach to 1,2-TTF starts from the thiolates **329**. Reaction of **329** with carbon disulfide, followed by treatment with hydrochloric acid, yields the amino substituted 1,2-TTF **287** in 7–30% yield.[574]

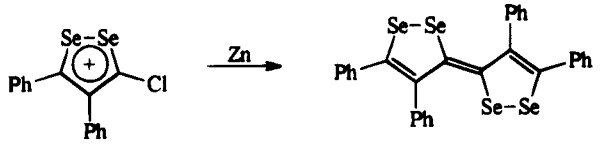
Already before the first announcement of the preparation of 1,2-TTF the possible formation of 1,2-TTF by thermolysis of the salts **290** or **292** in the ion source of a mass spectrometer was reported.[575] This was confirmed in a later publication.[576]

1,2-TTF and their data are summarized in Table 16.

8.4. *Synthesis of 1,2-TSF*

Diphenyl substituted 1,2-tetraselenafulvalenes are obtained in low yield (5%) by reduction of the corresponding 1,2-diselenolium salts with activated zinc.[573]

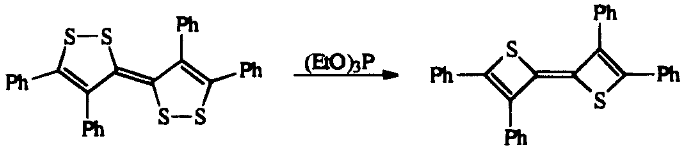
1,2-TSF and their data are summarized in Table 17.



SCHEME 184

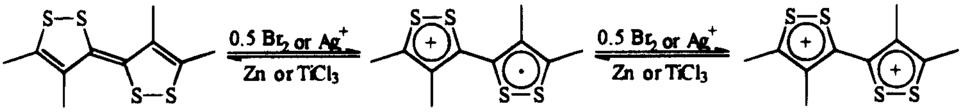
9. PROPERTIES OF 1,2-TETRATHIAFULVALENES

The 1,2-TTF are deep coloured solids, which are only sparingly soluble in most solvents. Of the amino substituted compounds it has only been reported that they are yellow. 1,2-TTF are thermally less stable than 1,3-TTF. In boiling tetraline compound **287b** is converted to the thieno[3,2-*b*]thiophene **317** in about 80% yield.[536,562]



287b **330**

SCHEME 185



287 **331** **332**

SCHEME 186

Other 1,2-TTF react analogously. In some cases addition of sulfur during the thermolysis is a further reaction so that the trithiones **288** and a thienodithiine **318** can be isolated.

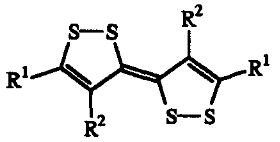
The reaction behaviour of 1,2-TTF with trivalent phosphorus compounds was studied in the case of **287b**. Whereas no reaction took place with triphenylphosphine in boiling benzene, with triethyl phosphite or tributyl phosphite a new compound was formed which contains two sulfur atoms less than **287b**. As a possible structure **330** was postulated.

In their redox properties 1,2-TTF show great similarity to their 1,3-analogues. Depending on the nature and amount of the oxidizing agent, oxidation to the radical cation **331** or the dication **332** takes place. Neutral 1,2-TTF comproportionate with the dications **332** to the radical cations **331**. Zinc or titanium(III) chloride reduces the salts **331** and **332** to the neutral 1,2-TTF. The 1,2-TTF are typical reversible redox systems.[562]

9.1. *Charge-Transfer Complexes of 1,2-TTF*

Like the analogous 1,3-TTF the 1,2-TTF also form charge-transfer complexes with several acceptors (Table 10). Only 1:1 donor-acceptor complexes have been isolated. In the electron spectra of the charge-transfer complexes long-wavelength absorptions of the donor and acceptor radical ions have been identified. Although the first isolation of a charge-transfer complex from a 1,2-TTF and TCNQ was reported in 1975,[536] the experimental data available until now are very limited. General conclusions about the interdependence between structure and redox properties or electric conductivity of the donors **277** and their charge-transfer complexes cannot be drawn.

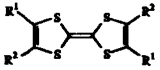
TABLE 10 Charge-transfer complexes of 1,2-TTF with acceptors and their electrical conductivity (compressed powder)



R^1	R^2	acceptor	σ_{RT} [S-cm^{-1}]	reference
Ph	H	DDQ$^{a)}$		571
		TCNQ$^{b)}$	3–5	577
Ph	Ph	DDQ	10^{-5}	571, 578
		TCNE$^{b)}$		571
		TCNQ	10^{-5}	578
		$TCNQF_4$	10^{-3}	578
Ph	SMe	$TCNQF_4$	10^{-5}	578
SMe	SMe	TCNQ	10^{-5}	578
$(CH{=}CH)_2$		DDQ		571
CH=CH-S		TCNQ	10^{-3}	578

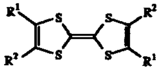
a) both isomeric forms provide the same complex, b) only one of the isomeric forms provides a complex, DDQ 2,3-dichloro-5,6-dicyanobenzoquinone, TCNE tetracyanoethylene, $TCNQF_4$ tetrafluorotetracyanoquinodimethane

TABLE 11 Molecular Properties of Tetrathiafulvalenes (redox potential in acetonitrile vs. sce)

R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H		H	A	119 (hexane/c-C_6H_{12})	91	446 (263), 357 sh, 316 (12 300), 308 (12 200)[a]	0.33 0.1 M TEAP	0.70	53, 87, 90, 306, 376, 377
			B Z = S $(MeO)_3P$		1	450 (230), 368 (2 000), 316 (11 400), 303 (12 800)[b]	0.32 0.1 M TBAP	0.68	39, 121
			B Z = Se $(MeO)_3P$		70	449 (257), 362 (2 000), 304 (13 100)[c]	0.33 0.1 M TBAT	0.70	106, 379
			I Z = S M = Co_2 n = 8		35	450 (270), 368 (1 900), 317 (12 500) 303 (13 300)[d]	0.40 0.05 M TBAT[e]	0.86	149, 376, 579
			M				0.38 0.1 M TBAHP	0.77	147, 580
			R = Me		33				
			R = Bu		25				
							0.31 0.1 M LiCl[f]	0.74	379
							0.35 0.1 M TBAP	0.71	581
							0.47 0.025 M TBAHP[e]	0.94	582
							0.45 0.1 M TEAT[g]	0.82	90
							0.02 0.1 M TEAP[h]	0.40	583
							0.40 0.1 M TBAHP[k]	0.82	308
							0.38 0.1 M TBAP[g]	0.74	343
CH_3		H	A	96–100 (EtOH)	35	440 (1 340), 355 sh, 321 (13 500), 308 (14 800), 298 (15 800)[b]	0.32 0.1 M TEAP	0.68	39, 121, 303, 584
				109–111[i]		453 (180), 298 (7 500)[c]	0.29 0.1 M TBAP	0.71	39, 584

(*table continues*)

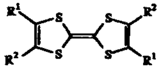
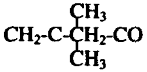
TABLE 11 (*continued*)

R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
							0.24 0.05 M TBAT	0.62	579
n-C_6H_{13}		H	A	oil	25				585
n-$C_{17}H_{35}$		H	A	91–92 (hexane)		480 (480), 360 (2 570), 320 (11 700), 300 (12 900)[b]			586
n-$C_{18}H_{37}$		H	A	33	65				585
CH_3		CH_3	A	245 (MeCN)	61	473 (248), 327 (13 800), 315 (14 000), 297 sh, 287 sh[b]	0.24 0.1 M TEAP	0.60	45, 119, 121 586
							0.25 0.1 M TBAP	0.62	39
							0.29 0.1 M TBAP	0.65	581
			1 Z = S M = Co_2 n = 8	244	25		0.21 0.05 M TBAT	0.59	149, 579
			M R = Bu		77		0.24 TBAA[e]	0.73	146, 225
							0.35 0.2 M TBAT[k]	0.83	226, 587
CD_3		CD_3		241–243 (MeCN)					73
CH_3		C_2H_5	A	102			0.24 0.1 M TBAP	0.61	39
C_2H_5		C_2H_5	A	125			0.23 0.1 M TBAP	0.75	39
n-C_3H_7		n-C_3H_7	A			325 (15 100), 310 (14 200)[a]	–0.06 0.1 M TEAP[h]	0.32	583
n-C_5H_{11}		n-C_5H_{11}	A	48–49 (pentane)					585
n-$C_{16}H_{33}$		n-$C_{17}H_{35}$	A	91–92 (benzene)	80	480 (400), 330 (12 300), 315 (13 800), 300 (12 300)[b]			586
n-$C_{17}H_{35}$		n-$C_{17}H_{35}$	A	93–95 (benzene)		482 (303),[l] 480, 363, 328, 314, 300[b]			586

$(CH_2)_2$			68				146
$(CH_2)_3$	A	244 (MeCN/trichlorobenzene)	71	465 (340), 315 (12 300)[m]	0.33 0.1 M TEAP	0.66	46, 78
				462 (177), 327 (9 280), 307 (10 500)[a]	0.31 0.1 M TBAP	0.74	39
	A	254		464 (374), 305 (22 900)[c]	0.26 0.1 M LiCl[f]	0.67	39, 379
	F		50		0.27 0.1 M TEAP	0.60	119, 139
	G	270–274 (benzene)	42				411
	I Z = S M = Co_2 n = 8	243	20				149
$CH_2\text{-}\underset{CH_3}{CH}\text{-}CH_2$	A	155–156 (hexane)	32	468 (400), 330 sh, 315 (13 800)[m]	0.30 0.1 M TEAP	0.63	46
$\overset{CH_3}{CH}\text{-}CH_2\text{-}CH_2$	A	119–120 ($EtNO_2$)	37	465 (500), 330 sh, 313 (16 600)[m]	0.30 0.1 M TEAP	0.64	46
$CH_2\text{-}CH_2\text{-}\underset{CH_3}{CH}$		137–139 (hexane)					
$CH_2\text{-}O\text{-}CH_2$			92		0.35 0.1 M TBAHP[q]	0.66	350
$CH_2\text{-}S\text{-}CH_2$	B Z = S $(MeO)_3P$	222–230	40	530 (215), 463 (360), 365 (2 040), 308 (12 850)[v]	0.55 0.1 M TBAHP[q]	0.72	588, 589, 590
$CH_2\text{-}SO_2\text{-}CH_2$	B Z = Se $(MeO)_3P$	190 dec.	47		0.65 0.1 M TBAHP[q]	0.80	588, 591
$(CH_2)_4$	A	248 (PhCl)	74	472 (195), 323 (9 550), 297 (10 600)[a]	0.40 0.1 M TEAT[s]	0.80	78, 90
	F		74	470 (290), 323 (14 200), 300 (15 100)[a]	0.25 0.1 M LiCl[f]	0.67	90, 139, 379
	G	255–258 (benzene/MeOH)	57	477 (324), 301 (19 400)[c]			140, 379
	I Z = S M = Co_2 n = 8	245	37				149
	M R = Bu		68				146

(*table continues*)

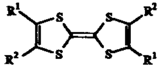
TABLE 11 (*continued*)

R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
	 $CH_2CH_2CH_2CH(CH_3)$		A	202–203	46	478 (298), 303 (16 500)[c]	0.25 0.1 M LiCl[f]	0.68	379
	 $CH_2\text{-}C(CH_3)_2\text{-}CH_2\text{-}CO$		B Z = Se Ph_3P	270–278 dec. (DMF)	50	444 (3 800), 318 (13 800), 305 (14 100)[l]	0.7	1.0	592
	$(CH_2)_5$		A	199–206 (benzene/MeOH)	65	500 (236), 291 (21 600)[c]	0.21 0.1 M LiCl[f]	0.64	379, 411
	$CH_2S(CH_2)_2SCH_2$			250 dec.	26				593
CH_2OH		H		178–180 (EtOH)	13				100
				160					281
					95				283, 286
CH_2OH		CH_2OH		220 dec.	80		0.39 0.1 M TBAP[q]	0.65	174, 198
					39				350
				190 dec.	92				594
CH_2OCH_3		CH_2OCH_3		74	62				594
$CH_2OC_3H_7$		$CH_2OC_3H_7$		47	73	490 (500), 370 (4 100)[k]	0.31 0.1 M TBAP	0.63	594
$CH_2OC_5H_{11}$		$CH_2OC_5H_{11}$		35	68	490 (500), 370 (4 100)[k]	0.31 0.1 M TBAP	0.63	594
$CH_2OC_6H_{13}$		$CH_2OC_6H_{13}$		44	70				594
$CH_2OC_7H_{15}$		$CH_2OC_7H_{15}$		44	77	490 (500), 370 (4 000)[k]	0.31 0.1 M TBAP	0.63	594
$CH_2OC_8H_{17}$		$CH_2OC_8H_{17}$		48	73				594
$CH_2OC_{10}H_{21}$		$CH_2OC_{10}H_{21}$		61	75				594
$CH_2OC_{12}H_{25}$		$CH_2OC_{12}H_{25}$		70	74				594
$CH_2OC_{14}H_{29}$		$CH_2OC_{14}H_{29}$		73	65				594
CH_2OCOCH_3		CH_2OCOCH_3		131	77				594
$CH_2OCOC_6H_{13}$		$CH_2OCOC_6H_{13}$		69	73		0.46 0.1 M TBAP	0.77	594
	$S\text{-}CH_2\text{-}CH_2$		B Z = Se	184–186 ($CHCl_3$)	13	476 (480), 370 sh, 340 sh, 323 (20 000), 310 sh[m]	0.43 0.1 M TEAP	0.69	46

S-CH_2-CH_2 CH_2-CH_2·S		B Z = Se	195–196 (PhCl)	37	483 (645), 370 sh, 323 (12 600), 310[m)]	0.43 0.1 M TEAP	0.69	46
		B Z = S $(MeO)_3P$	191–193 (PhCl)	23	530 (3 980), 345 (400), 290 (12 600)[e)]			589, 590
S-$(CH_2)_5$ $(CH_2)_5$·S			127–129	26				168
$CH_3SCH_2CH_2$	$CH_3SCH_2CH_2$		110 (MeCN)	60		0.22 0.1 M TEAP[h)]	0.47	59
$CH_3COS(CH_2)_3$	$CH_3COS(CH_2)_3$		95 (MeCN)	60		0.19 0.1 M TEAP[h)]	0.42	59
H	$C_{18}H_{37}S$		62	12				595
CH_3	CH_3S		136 (MeCN)	43		0.03 0.1 M TBAHP[h)]	0.35	161, 596
			141 (heptane)	49		0.36 0.1 M TBAHP[k)]	0.72	96
CH_3	$C_{18}H_{37}S$	A	85 (heptane)	40		0.35 0.1 M TBAHP[k)]	0.73	96
CH_3	$(C_2H_5OOC)_2$ $CH(CH_2)_6S$	A	55–57 (EtOH)	79				289
CH_3	$([HOCH_2]_3CHNCO)_2$ $HC(CH_2)_6S$		145–150 dec.	73		0.46	0.78	289
CH_3	$C_6F_5CH_2S$		177 (benzene)	98	269 (10 900)[k)]			162
CH_3	p-$CNC_6H_4CH_2S$		248 (benzene)	98	316 (12 000)[k)]			162
CH_3	p-$NO_2C_6H_4CH_2S$		201 (benzene)	80	265 (31 500)[k)]			162
CH_3	p-$NO_2C_6H_4(CH)_2S$		188 (benzene)	71	263 (73 500)[k)]			162
C_2H_5	CH_3S		86 (MeCN)	69		0.43	0.71	161, 581, 597
$CH_3(CH_2)_{10}$	$HOOC(CH_2)_{10}S$		119 (MeCN)	60				95
$CH_3(CH_2)_{10}$	CH_3OOCCH_2S		58 (MeCN)	78				95
$CH_3(CH_2)_{10}$	$HOOCCH_2S$		90–95 (MeCN)	88				95
C_6H_5	CH_3S		173 (MeCN)	66				161
p-$CH_3C_6H_4$	CH_3S		190 (MeCN)	38				161
p-ClC_6H_4	CH_3S		186 (MeCN)	76				161
p-ClC_6H_4	C_2H_5S		130 (MeCN)	71				161
p-$HOOCC_6H_4$	$CH_3(CH_2)_{17}S$		153 (EtOAc)	90				95
p-ClC_6H_4	$HOOC(CH_2)_{10}S$		139 (MeCN)	66				95
p-$CH_3C_6H_4$	$C_6F_5CH_2S$		164 (benzene)	63				162
p-$CH_3C_6H_4$	p-$CNC_6H_4CH_2S$		>260 (benzene)	77	254 (30 100)[k)]			162
p-$CH_3C_6H_4$	2,6-$(CN)_2C_6H_3CH_2S$		>260 (benzene)	96				162
p-$CH_3C_6H_4$	p-$NO_2C_6H_4CH_2S$		>260 (benzene)	51	268 (40 000)[k)]			162

(*table continues*)

TABLE 11 (*continued*)

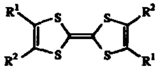


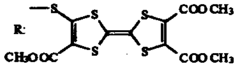
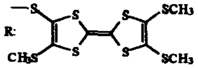
R^1	R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
p-$CH_3C_6H_4$	$3,5$-$(NO_2)_2C_6H_4CH_2S$		188 (MeCN)					598
p-$CH_3C_6H_4$	p-$NO_2C_6H_4(CH_2)_2S$		120 (benzene)	80	265 (50 000)$^{k)}$			162
p-ClC_6H_4	$C_6H_5CH_2S$		170	30				161
p-ClC_6H_4	$C_6F_5CH_2S$		190 (benzene)	74	267$^{k)}$			161
p-ClC_6H_4	p-$CNC_6H_4CH_2S$		>260 (benzene)	91				162
p-ClC_6H_4	p-$NO_2C_6H_4CH_2S$		266 (benzene)	63	264 (54 800)$^{k)}$			162
p-ClC_6H_4	p-$NO_2C_6H_4(CH_2)_2S$		160 (benzene)	85	264 (51 800)$^{k)}$			162
p-ClC_6H_4	$(C_6H_5)_2CHS$		218 (EtOH)	98				95
p-$CH_3C_6H_4$	$(C_6H_5)_2CHS$		222 (EtOH)	92				95
$CH_3(CH_2)_{10}$	$(C_6H_5)_2CHS$		86 (EtOH)	93				95
$CH_3(CH_2)_8$	$Br(CH_2)_3S$			91				95
$CH_3(CH_2)_{10}$	$Br(CH_2)_3S$			99				95
p-ClC_6H_4	$Br(CH_2)_3S$			90				95
p-$CH_3C_6H_4$	$Br(CH_2)_3S$			86				95
$CH_3(CH_2)_{10}$	$Br(CH_2)_4S$			97				95
$CH_3(CH_2)_{10}$	$Br(CH_2)_6S$		58 (EtOAc)	95				95
p-ClC_6H_4	$Br(CH_2)_6S$			87				95
p-$CH_3C_6H_4$	$Br(CH_2)_6S$			89				95
p-ClC_6H_4	$N_3(CH_2)_3S$			87				95
$CH_3(CH_2)_{10}$	$N_3(CH_2)_3S$			85				95
p-ClC_6H_4	$N_3(CH_2)_6S$			86				95
p-$CH_3C_6H_4$	$N_3(CH_2)_6S$			87				95
p-ClC_6H_4	$(CH_3O)_2OPNH(CH_2)_3S$							95
p-$CH_3C_6H_4$	$(CH_3O)_2OPNH(CH_2)_3S$							95
$CH_3(CH_2)_{10}$	$(CH_3O)_2OPNH(CH_2)_6S$							95
p-ClC_6H_4	$(CH_3O)_2OPNH(CH_2)_6S$							95
p-ClC_6H_4	$H_2N(CH_2)_3S$							95
p-$CH_3C_6H_4$	$H_2N(CH_2)_3S$							95
$CH_3(CH_2)_{10}$	$H_2N(CH_2)_6S$							95
p-ClC_6H_4	$H_2N(CH_2)_6S$							95
p-ClC_6H_4	C_4H_9SnS							95
p-$CH_3C_6H_4$	C_4H_9SnS							95
$CH_3(CH_2)_{10}$	C_4H_9SnS							95

$COOCH_3$	CH_3S					0.63 0.1 M TBAP	0.93	581, 597
$COOCH_3$	CH_3OCOCH_2S	B Z = O $(EtO)_3P$	230 dec. (diglyme)	36	440 (3 980), 330 sh (15 135), 307 sh (22 910), 295 (24 000)$^{t)}$	0.79 0.1 M TBAP$^{c,g)}$	1.09	288
CH_3O	CH_3O	B Z = S $(EtO)_3P$	71–73	30		0.44 0.1 M TBAP$^{e)}$	0.68	599
CH_3S	CH_3S	B		56	490(400), 380 (3 715), 332 (13 500), 311 (14 450)$^{t)}$	0.49 0.05 M TEAP	0.67	102, 295
		K	94, 5–96 (MeCN)	75	370 (1 700), 329 (7 750), 309 (8 700), 260 (7 750)$^{a)}$	0.47 0.05 M TEAP	0.71	58, 119
			96.5			0.55		600
			95–96 (MeCN)			0.64 0.1 M TBAT$^{n)}$	0.93	346, 601
						0.40 0.1 M TBAP	0.73	581
						0.50	0.81$^{h,k)}$	602
		95–96	95			0.60 0.1 M TBAHP$^{k)}$	0.94	355
RCH_2S R: $(CH_3)_3Si(CH_2)_2O$	RCH_2S	B Z = O,S $(EtO)_3P$	64–65		470, 380, 328, 305, 278, 254, 225$^{a)}$			109
RCH_2S R: CH_3OOC	RCH_2S		108	18	390, 326, 307, 264, 210$^{e)}$			603
		B Z = O $(EtO)_3P$	117 (MeOH)			0.69 0.1 M TBAHP$^{m)}$	1.03	604
$C_6H_5CH_2S$	$C_6H_5CH_2S$	B Z = O $(EtO)_3P$	166,5–168,5 ($CHCl_3$/hexane 2:1)	70	395, (3 390), 332 (15 800)$^{w)}$	0.67* 0.1 M TBAHP$^{h,k)}$	1.03*	605
RCH_2S R: *p*-$CH_3COOC_6H_4$	RCH_2S	B Z = O Z = S $(EtO)_3P$	170–173 dec. (toluene/hexane 5:1, v:v)	71 27				76, 353, 354
CH_3S	$NCCH_2CH_2S$	B Z = O $(EtO)_3P$	*cis*/*trans* mixture	80				173
C_2H_5S	C_2H_5S		72–73			0.64 0.1 M TBAT$^{n)}$	0.94	601

(*table continues*)

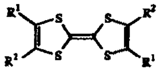
TABLE 11 (*continued*)

R^1 	R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2	References
						0.52 0.1 M TBAT	0.77	301
$ClCH_2CH_2S$	$ClCH_2CH_2S$	B	158–160 (MeCN)		386, 330, 310[i]	0.61 0.1 M TBAT	0.87	301, 330, 332
		Z = O		89				
		Z = S		76				
		$(EtO)_3P$						
$BrCH_2CH_2S$	$BrCH_2CH_2S$	B Z = O $(EtO)_3P$	169–171 (CCl_4/benzene)	90	338, 330, 310[i]	0.62 0.1 M TBAT	0.88	301
ICH_2CH_2S	ICH_2CH_2S		166–167 (benzene)	78	389, 332, 308[i]	0.60 0.1 M TBAT	0.87	301
$NCCH_2CH_2S$	$NCCH_2CH_2S$		184.5–185.5	56				607
		B Z = O $(EtO)_3P$	209–210	72		0.75 0.1 M TBAHP[k]	1.06	355
$HOCH_2CH_2S$	$HOCH_2CH_2S$	137–138 (acetone)		95	379, 330, 310[i]	0.53 0.1 M TBAT	0.74	301
			140–141	75		0.57 0.1 M TBAHP	0.80	304
			139–140	95		0.59 0.1 M TBAHP[k]	0.80	355
RCH_2CH_2S R: $t\text{-}C_4H_9(C_6H_5)_2SiO$	RCH_2CH_2S	B Z = O $(EtO)_3P$	oil	62				304
RCH_2CH_2S R: TTFCOO	RCH_2CH_2S		191–195	57	435 (52 000), 318 (310 000), 305 (310 000),271 (260 000), 228 (310 000)[k]	0.63 0.75 0.1 M TBAHP[c]	0.95	304
$CH_3SCH_2CH_2S$	$CH_3SCH_2CH_2S$	B Z = O $(EtO)_3P$	65–67 (ether/acetone 5:1)	73		0.60	0.84	355
RCH_2CH_2S R: C_6H_5COO	RCH_2CH_2S	B Z = O $(EtO)_3P$	107–109 (*i*-PrOH)	96	388, 330, 308[i]	0.59 0.1 M TBAT	0.91	301

RCH_2CH_2S R: *p*-$ClC_6H_4SO_2O$	RCH_2CH_2S		146–148 (benzene)	94	385, 330, 308[c]	0.60 0.1 M TBAT	0.87	301
$CH_2{=}CHS$	$CH_2{=}CHS$		45–46	89	475, 388, 325 sh, 309, 299 sh, 229[a]	0.59 0.1 M TBAHP[a,k]	0.92	109, 330, 332
C_3H_7S	C_3H_7S		96					600
			30–31			0.63 0.1 M TBAT[a]	0.94	601
$ClCH_2CH_2CH_2S$	$ClCH_2CH_2CH_2S$		62–63 (*i*-PrOH)	52		0.60 0.1 M TBAHP[k]	0.92	365
$ICH_2CH_2CH_2S$	$ICH_2CH_2CH_2S$		68–70	53		0.61 0.1 M TBAHP[k]	0.94	365
$RCH_2CH_2CH_2S$	$RCH_2CH_2CH_2S$		>91 (darkening) (acetone)	69		0.57 0.72 0.1 M TBAHP[k]	0.77 1.06	365
R: 								
$RCH_2CH_2CH_2S$	$RCH_2CH_2CH_2S$		47–50	84		0.52 0.1 M TBAHP[k]	0.82	365
R: 								
C_4H_9S	C_4H_9S		24.6	87				600
			26–26.5			0.64 0.1 M TBAT[a]	0.94	601
$C_5H_{11}S$	$C_5H_{11}S$		31.5–32.5			0.64 0.1 M TBAT[a]	0.93	601
$C_6H_{13}S$	$C_6H_{13}S$		29.5–30.5			0.64 0.1 M TBAT[a]	0.93	601
$C_7H_{15}S$	$C_7H_{15}S$		44–46			0.64 0.1 M TBAT[a]	0.94	601
		B Z = O	44–46	17		0.55 0.1 M TBAT	0.79	608
$C_8H_{17}S$	$C_8H_{17}S$		48.5–49.5			0.64 0.1 M TBAT[a]	0.94	601
$C_9H_{19}S$	$C_9H_{19}S$		57.5–59			0.64 0.1 M TBAT[a]	0.94	601
$C_{10}H_{21}S$	$C_{10}H_{21}S$		59–60			0.64 0.1 M TBAT[a]	0.94	601
$C_{11}H_{23}S$	$C_{11}H_{23}S$		64–65.5			0.64 0.1 M TBAT[a]	0.94	601

(*table continues*)

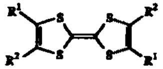
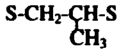
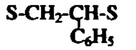
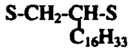
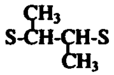
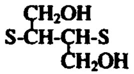
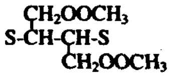
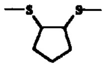
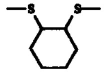
TABLE 11 (*continued*)

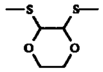
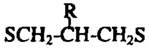
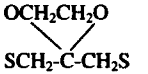
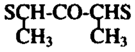
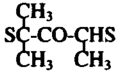
R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2 [V]	References
$C_{12}H_{25}S$		$C_{12}H_{25}S$		68–69			0.64 0.1 M TBAT[a]	0.95	601
$C_{13}H_{27}S$		$C_{13}H_{27}S$		74.5–75.5			0.64 0.1 M TBAT[a]	0.95	601
$C_{14}H_{29}S$		$C_{14}H_{29}S$		78–79			0.64 0.1 M TBAT[a]	0.94	601
$C_{15}H_{31}S$		$C_{15}H_{31}S$		80–81.5			0.64 0.1 M TBAT[a]	0.94	601
$C_{16}H_{33}S$		$C_{16}H_{33}S$		81.5–83			0.65 0.1 M TBAT[a]	0.94	601
			B Z = O $(EtO)_3P$	83			0.58 0.1 M TBAHP[a]	0.95	604
			B Z = S $(BuO)_3P$	85–86	61	384, 318, 304, 268[v]			609
$C_{17}H_{35}S$		$C_{17}H_{35}S$		84–85.5			0.64 0.1 M TBAT[a]	0.94	601
$C_{18}H_{37}S$		$C_{18}H_{37}S$		92			0.64 0.1 M TBAT[a]	0.94	601
				87.7					600
$C_{20}H_{41}\underset{COOH}{CH}S$		$C_{20}H_{41}\underset{COOH}{CH}S$							610
$C_{20}H_{41}\underset{COOC_2H_5}{CH}S$		$C_{20}H_{41}\underset{COOC_2H_5}{CH}S$							610
$HOOCC_{21}H_{42}S$		$HOOCC_{21}H_{42}S$							610
$C_2H_5OOCC_{21}H_{42}S$		$C_2H_5OOCC_{21}H_{42}S$							610
C_6H_5S		C_6H_5S		167–169 (EtOAc/hexane)	64		0.55 0.1 M TBAHP[k]	0.90	351
	S-CH_2-S		A	154–156		542 (4 080), 354 (4 900), 282 (7 600), 264 (8 100)[k]	0.57 0.1 M TBAP[v]	0.77	611, 612
			B Z = O $(EtO)_3P$	191–192 (1,2-dichlorobenzene)	48		0.60 0.025 M TBAHP[a]	0.98	103, 296, 582, 613

$\overset{COOCH_3}{\text{S-CH-S}}$	B Z = O $(EtO)_3P$	198	17	346, 306, 220[a]			614
O-$(CH_2)_2$-O	B Z = Se $(MeO)_3P$	178	63	517 (148), 337 (9 690), 315 (11 400), 200 (13 300)[a]	0.44 0.1 M TBAHP[g]	0.70	615, 616
O-$(CH_2)_2$-S	B Z = O $(MeO)_3P$		26		0.39 0.1 M TBAHP[k]	0.81	617
S-$(CH_2)_2$-S	B Z = O $(EtO)_3P$	242,5	82	467 (276), 349 (10 960), 324 (14 800)[v]	0.49 0.1 M TEAP	0.74	74, 103, 119, 178, 296, 618, 619, 620
	Z = S $(EtO)_3P$	>250	45	490 (400), 347 (10 470), 321 (15 150)[t]	0.53 0.05 M TEAP	0.77	102, 295
	Z = S $(EtO)_3P$	258–260 (PhCl)	23		0.55 0.1 M TBAP[v]	0.85	104, 296, 612
	B Z = O $(MeO)_3P$ $(EtO)_3P$	235–237 $(CHCl_3)$	69 85				605
				480 (290), 345 sh (10 000), 320 (17 000), 197 (36 000)[a]	0.57 0.1 M TBAHP[g]	0.83	615, 616
					0.56 0.1 M TBAP	0.80	581
					0.60 0.025 M TBAHP[e]	0.98	582
					0.54 0.1 M TBAHP[m]	0.96	580
					0.49 0.1 M TBAHP[k]	0.64	621
					0.69 0.1 M TBAP[c]	0.82	71
S-$(CD_2)_2$-S	B Z = O $(EtO)_3P$ Z = S $(EtO)_3P$	244 dec.	74	481 (269)[c]			622

(*table continues*)

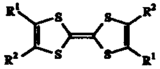
TABLE 11 (*continued*)

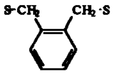
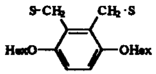
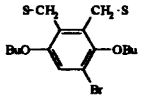
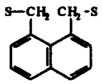
R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1	E^{0x}_2 [V]	References
	 $S\text{-}CH_2\text{-}\underset{CH_3}{CH}\text{-}S$		B Z = S $(EtO)_3P$	175	35	345, 321, 260$^{k)}$			623
			B Z = S $(EtO)_3P$	175	21	458, 322, 263$^{k)}$	0.51 0.1 M TBAHP$^{k)}$	0.93	624
	 $S\text{-}CH_2\text{-}\underset{C_6H_5}{CH}\text{-}S$		B Z = S $(EtO)_3P$	207–210 dec.	50	344 (39 800), 308 (56 300), 218 (170 000)$^{k)}$	0.47 0.1 M TBAHP$^{k)}$	0.62	621
	 $S\text{-}CH_2\text{-}\underset{C_{16}H_{33}}{CH}\text{-}S$		B Z = S $(EtO)_3P$	109–110 (hexane)	10				625
	 $S\text{-}\overset{CH_3}{CH}\text{-}\underset{CH_3}{CH}\text{-}S$		B Z = S $(EtO)_3P$	204					626, 627
			B Z = O $(EtO)_3P$	200–203 ($CHCl_3$)			0.44 0.1 M TBAP$^{g,k)}$	0.86	628
	 $S\text{-}\overset{CH_2OH}{CH}\text{-}\underset{CH_2OH}{CH}\text{-}S$			165–167 (MeCN)	80	500 (450), 345 (10 000), 323 (18 000)$^{p)}$	0.62	0.87$^{c,g)}$	629
	 $S\text{-}\overset{CH_2OOCH_3}{CH}\text{-}\underset{CH_2OOCH_3}{CH}\text{-}S$		B Z = O $(EtO)_3P$	138–140 (MeCN)	59	333 (12 200), 319 (16 600)$^{i)}$	0.65	0.80$^{c,g)}$	629
			B Z = O $(EtO)_3P$	190–205 dec. (pyridine)	71	470, 343 (17 000), 314 (14 500)$^{i)}$	0.68 0.1 M TBAP$^{c,g)}$	0.97	64
							0.62 0.1 M TBAP	0.90	581
			B Z = O $(EtO)_3P$	205 dec. (pyridine)	75	460 (398), 351 (12 600), 326 (14 800)$^{i)}$	0.66 0.1 M TBAP$^{c,g)}$	0.92	64

		B			346 (15 000), 316	0.58	0.82	65
		Z = O		32	(13 000)[w]	0.1 M TBAHP		
		Z = S		20				
		Z = O, S		42				
		$(EtO)_3P$						
	$S-(CH_2)_3-S$	B	>250	45		0.58	0.86	102
		Z = S				0.05 M TEAP		
		$(EtO)_3P$						
		Z = O	265	89		0.50	0.78	119, 296, 630
		Z = S	(trichlorobenzene)	40		0.1 M TEAP		
						0.54	0.81	581
						0.1 M TBAP		
	 $SCH_2-\overset{R}{C}H-CH_2S$							
R: OH		B	>230	5		0.66	1.03	170
		Z = S				0.1 M TBAHP[s,k]		
		$(EtO)_3P$						
R: $OSi(C_6H_5)_2$*t*-C_4H_9		B	201–202					170
		Z = O	$(MeOH(CH_2Cl_2)$	50				
		Z = S		33				
		$(EtO)_3P$						
R: $OCOCH_3$			>230	69				170
R: $OCO(CH_2)_3Br$			224–225	43				170
			(toluene)					
R: $O(CH_2CH_2O)_4C_6H_4O$		B		58				375
$(CH_2CH_2O)_3C_6H_4C(C_6H_5)_3$		Z = O						
		$(EtO)_3P$						
	 OCH_2CH_2O / SCH_2-C-CH_2S	B	>230	66				170, 631
		Z = O						
		$(EtO)_3P$						
	$SCH_2-CO-CH_2S$	G	235–236	51				141
			(pyridine)					
			>340	87		0.68	1.04	170, 631
						0.1 M TBAHP[s,v]		
	 $S\underset{CH_3}{C}H-CO-\underset{CH_3}{C}HS$	G	210–216	25				142
			(pyridine)					
	 $S\overset{CH_3}{\underset{CH_3}{C}}-CO-\underset{CH_3}{C}HS$	G						142

(*table continues*)

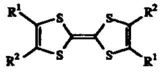
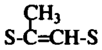
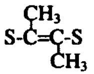
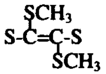
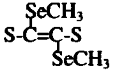
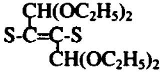
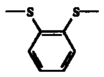
TABLE 11 (*continued*)

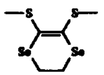
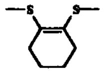
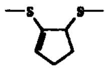
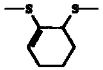
R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E$^{Ox}_1$ [V]	E$^{Ox}_2$	References
	SCH-CO-CHS		G	203–206	60				142
	C_2H_5 C_2H_5			(pyridine)					
	SCH_2-O-CH_2S		B	260–262		500 (400), 396 (5 130),	0.67	0.90	581, 597,
			Z = O	(pyridine)	5	335 (13 200)$^{i)}$	0.1 M TBAP		632, 633
			Z = S		18				
			$(EtO)_3P$						
			K		65				141
			B	247–248	78	394 (240), 336 (945),	0.75*	1.10*	634
			Z = O	($CHCL_3$)		300 (420), 258 (810)$^{m)}$	0.1 M TBAHP$^{e,k)}$		
			$(EtO)_3P$						
	SCH_2S-CH_2-S		B	235–240					103
			Z = O						
			$(EtO)_3P$						
			B	>300					634
			Z = O	(>240 color change)	81				
			Z = S		27				
			$(EtO)_3P$						
			A	275–278	90				141
	S-$(CH_2)_4$-S		B	151	37	310, 227$^{k)}$	0.53	0.95	635, 636
			Z = S	(CH_2Cl_2/MeOH)			0.1 M TBAHP$^{e,k)}$		
			$(EtO)_3P$						
							0.67	1.01	581
							0.1 M TBAP		
	S-$(CH_2)_5$-S		B	240 dec.	9	401, 325, 310, 232$^{k)}$	0.57	1.03	624
			Z = S				0.1 M TBAHP$^{k)}$		
			$(EtO)_3P$						
	S-$(CH_2)_6$-S		B	228 dec.	12	365, 338, 268, 225$^{k)}$	(0.96)		624
			Z = S				0.1 M TBAHP$^{k)}$		
			$(EtO)_3P$						
	$S(CH_2CH_2O)CH_2CH_2S$		B	230–233	27		0.48	0.64	637
			Z = S	($CHCl_3$)			0.1 M TBAHP		
			$(EtO)_3P$						
	$S(CH_2CH_2O)_2CH_2CH_2S$		B	230–233	34		0.48	0.64	637, 638
			Z = S	($CHCl_3$)			0.1 M TBAHP		
			$(EtO)_3P$						

$S(CH_2CH_2O)_3CH_2CH_2S$	B Z = S $(EtO)_3P$	211–212 (MeCN)	44		0.52	0.75[h,k]	602, 638
	B Z = S $(EtO)_3P$	181 ($CHCl_3$)	28		0.48 0.1 M TBAHP	0.64	637
$S(CH_2CH_2O)_4CH_2CH_2S$	B Z = S $(EtO)_3P$	98–99 (EtOAc)	40		0.48 0.1 M TBAHP	0.64	637, 639
$S(CH_2CH_2O)_5CH_2CH_2S$	B Z = S $(EtO)_3P$	59 (EtOAc)	38		0.48 0.1 M TBAHP	0.64	637
$SCH_2CH_2S(CH_2)_3SCH_2CH_2S$	B Z = S $(EtO)_3P$	236–238	33		0.53 0.1 M TBAHP[k]	0.89	640
SCH_2-CH=CH-CH_2S	B Z = Se $(MeO)_3P$	165–167 dec.	43	408 (955), 333 (8 700), 301 (12 300)[q]			641
	B Z = S $(MeO)_3P$	270 dec. (pyridine)	50	408 (1 290), 380 (5 000), 340 (12 300), 275 (10 960)[v]			642
	B Z = O Z = S $(EtO)_3P$	265–267 dec. ($CHCl_3$)	18 61	340, 286, 243, 231[k]	0.52 0.1 M TBAHP[g,k]	0.98	636
	B Z = S $(EtO)_3P$	184	33	417 (2 239), 333 (15 813), 314 (16 143), 227 (27 855)[k]	0.33 TBAHP[k]	0.82	388, 643
	B Z = S $(EtO)_3P$	206 dec.	50	303 (22 730)[k]	0.39 TBAHP[k]	0.84	643, 644
	B Z = O Z = S $(EtO)_3P$	243–244 dec. (CH_2Cl_2)	26 11	334, 294, 237, 220, 204[k]	0.53 0.1 M TBAHP[g,k]	1.06	635, 636
S-CH=CH CH=CH-S	A	214–215 (hexane)	30		0.47 0.1 M TEAP	0.74	46

(*table continues*)

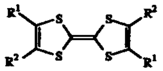
TABLE 11 *(continued)*

R¹		R²	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2	References
			B Z = S $(MeO)_3P$	213–214	7	490 (200), 379 (2 090), 329 (10 000), 297(12 600)$^{a)}$			589, 590
	S-CH=CH-S		B Z = O $(EtO)_3P$	>220 (pyridine)	43	480 (355), 340 (14 450), 313 (14 120)	0.68 0.1 M TBAP$^{a)}$	0.94	581, 645, 646
							0.70 0.1 M TBAP		581
			Z = O $(EtO)_3P$	227–230 ($CHCl_3$)	72	340 (6 910), 316 (6 750)$^{c)}$	0.83 0.1 M TBAP$^{c)}$		103, 647, 648
			Z = S $(EtO)_3P$						649
	 $S\text{-}\overset{CH_3}{C}\text{=}CH\text{-}S$		B Z = O $(EtO)_3P$	204 dec. ($CHCl_3$)	92	342 (4 900), 316 (4 900)$^{c)}$	0.80 0.1 M TBAP$^{c)}$		647, 648
	 $S\text{-}\overset{CH_3}{C}\text{=}\underset{CH_3}{C}\text{-}S$		B Z = O $(EtO)_3P$	249–250 ($CHCl_3$)	97	344 (3 310), 314 (3 160)$^{c)}$	0.80 0.1 M TBAP$^{c)}$		72, 103, 647
			B Z = O $(EtO)_3P$	>230 dec. (pyridine)	75	490 (426), 344 (14 120), 312 (13 200)	0.65 0.1 M TBAP$^{c)}$	0.91	581, 645, 646
	 $S\text{-}\overset{SCH_3}{C}\text{=}\underset{SCH_3}{C}\text{-}S$		B Z = O $(EtO)_3P$	200 dec. (CCl_4)	68	337 (20 000), 308 (21 400)$^{c)}$	0.83 0.1 M TBAP$^{c)}$		650
	 $S\text{-}\overset{SeCH_3}{C}\text{=}\underset{SeCH_3}{C}\text{-}S$		B Z = O $(EtO)_3P$	185 dec. (CCl_4)	75	338 (20 000), 306 (23 430)$^{c)}$	0.86 0.1 M TBAP$^{c)}$		71
	 $S\text{-}\overset{CH(OC_2H_5)_2}{C}\text{=}\underset{CH(OC_2H_5)_2}{C}\text{-}S$		B Z = O $(i\text{-}PrO)_3P$		60				66
			B Z = O $(EtO)_3P$	287 dec. (CCl_4)	75	495, 347, 303$^{E)}$	0.75* 0.1 M TBAHP$^{k)}$	1.13*	642

			B Z = S $(EtO)_3P$	>320	13	340 sh, 312, 274k)			651
			B Z = O $(EtO)_3P$	238–239 dec. (pyridine)	64	490 (645), 348 (27 300), 313 (33 100)i)	0.76 0.1 M TBAPe)	1.02	581, 646
			B Z = O $(EtO)_3P$	270 dec. (pyridine)	57	490 (708), 345 (29 350), 316 (44 600)i)	0.69 0.1 M TBAPe)	0.97	581, 646
			B Z = O $(EtO)_3P$	228–229 dec. (pyridine)	40	488 (416), 348 (14 100), 320 (13 800)i)	0.67 0.1 M TBAPe)	0.96	581, 646
			B Z = O $(EtO)_3P$	250 dec. (pyridine)	47	480 (416), 347 (15 100), 321 (19 100)i)	0.61 0.1 M TBAPe)	0.91	581, 646
C_6H_5COS		C_6H_5COS	G	163–169	1		0.66 0.1 M TBAP	0.89	63, 581, 597
			I Z = S M = Co_2 n = 8	195	25		0.66 0.1 M TBAHPk)	0.87	352
			C	195–197	82				652
CH_3Se		CH_3Se		93.5–93.7 (hexane/benzene)	52		0.58 0.1 M TBATe)	0.93	359
				109.5–109.7 (hexane)					359
				88–89 (EtOAc/hexane)	80		0.54	0.93	351
					45				134
C_2H_5Se		C_2H_5Se		62.7–63.5 (hexane/benzene)	70				359
				57–58 (ether/hexane)	45				356
C_3H_7Se		C_3H_7Se		37.4–38.3 (hexane/benzene)	63				359
					85		0.44 0.1 M TBAHPk)	0.86	351
C_4H_9Se		C_4H_9Se		38.8–39.3 (hexane)	55				359

(*table continues*)

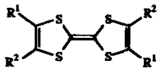
TABLE 11 *(continued)*

R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
					84		0.44 0.1 M TBAHP[k]	0.86	351
$C_5H_{11}Se$		$C_5H_{11}Se$		29.9–30.7 (hexane)	56				359
					90		0.51 0.1 M TBAHP[k]	0.92	351
$C_6H_{13}Se$		$C_6H_{13}Se$		32.5–32.9 (hexane)	56				359
$C_7H_{15}Se$		$C_7H_{15}Se$		39.0–39.7 (hexane)	62				359
$C_8H_{17}Se$		$C_8H_{17}Se$		48.7–49.2 (hexane/benzene)	69				359
$C_9H_{19}Se$		$C_9H_{19}Se$		56.0–56.8 (hexane/benzene)	65				359
$C_{10}H_{21}Se$		$C_{10}H_{21}Se$		62.2–62.8 (hexane/benzene)	84				359
$C_{11}H_{23}Se$		$C_{11}H_{23}Se$		66.2–67.5 (hexane)	65				359
$C_{12}H_{25}Se$		$C_{12}H_{25}Se$		74.3–75.2 (hexane)	70				359
$C_{13}H_{27}Se$		$C_{13}H_{27}Se$		79.7–80.3 (hexane)	78				359
$C_{14}H_{29}Se$		$C_{14}H_{29}Se$		83.3–84.1 (hexane)	73				359
$C_{15}H_{31}Se$		$C_{15}H_{31}Se$		86.0–86.6 (hexane)	78				359
$C_{16}H_{33}Se$		$C_{16}H_{33}Se$		87.7–89.2 (hexane)	75				359
$C_{17}H_{35}Se$		$C_{17}H_{35}Se$		90.4–97.3 (hexane)	71				359
$C_{18}H_{37}Se$		$C_{18}H_{37}Se$		94.2–94.6 (hexane)	77				359
	Se-CH_2-Se			192–195 (CS_2)	31		0.11 0.1 M TBAT[c,h]	0.36	356, 357

			B Z = O (EtO)$_3$P	257 dec. (dichlorobenzene)	69	326 (12 200)$^{A)}$	0.40 0.1 M TBAA$^{e)}$	0.67	67
	Se-(CH$_2$)$_2$-Se			220–223 (CS$_2$)	35		0.15 0.1 M TBAT$^{e,h)}$	0.55	356, 357
			B Z = O (EtO)$_3$P	241 (CS$_2$)	60	345 (15 800)$^{m)}$	0.44 0.1 M TBAA$^{e)}$	0. 77	67, 68
	Se-(CH$_2$)$_3$-Se		B Z = O (EtO)$_3$P	272–277 (dichlorobenzene)	55	400 (6 000), 340 (16 000)	0.45 0.1 M TBAA$^{e)}$	0.77	67, 69
					65				357
C_6H_5Se		H		114					327
C_6H_5Se		C_6H_5Se		154–156 (THF/hexane)	75		0.50 0.1 M TBAHP$^{k)}$	0.89	351
					60				134
				116					327
C_2H_5Te		H		85	20				347
Ch_3Te		CH_3Te		175–176	66		0.52 0.1 M TBAT$^{n)}$	0.91	347, 358
C_2H_5Te		C_2H_5Te		90,2–91,2	67		0.51 0.1 M TBAT$^{n)}$	0.91	358
				88–90 (CHCl$_3$)	35				347
				86–87 (ether/hexane)	32				356
C_3H_7Te		C_3H_7Te		62.2–62.7	60		0.51 0.1 M TBAT$^{n)}$	0.91	358
C_4H_9Te		C_4H_9Te		85.7–86.5	38		0.51 0.1 M TBAT$^{n)}$	0.91	358
$C_5H_{11}Te$		$C_5H_{11}Te$		56.9–57.4	62		0.51 0.1 M TBAT$^{n)}$	0.93	358
$C_6H_{13}Te$		$C_6H_{13}Te$		46.0	68		0.52 0.1 M TBAT$^{n)}$	0.92	358
$C_7H_{15}Te$		$C_7H_{15}Te$		49.9–50.2	67		0.51 0.1 M TBAT$^{n)}$	0.92	358
$C_8H_{17}Te$		$C_8H_{17}Te$		57.7	48		0.51 0.1 M TBAT$^{n)}$	0.92	358
$C_9H_{19}Te$		$C_9H_{19}Te$		65.2–65.7	63		0.51 0.1 M TBAT$^{n)}$	0.92	358

(table continues)

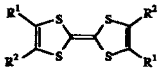
TABLE 11 (*continued*)

R¹	 R²	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2 [V]	References
$C_{10}H_{21}Te$	$C_{10}H_{21}Te$		72.7	52		0.52 0.1 M TBAT[a]	0.93	358
$C_{11}H_{23}Te$	$C_{11}H_{23}Te$		77.5–78.5	58		0.52 0.1 M TBAT[a]	0.93	358
$C_{12}H_{25}Te$	$C_{12}H_{25}Te$		82.2–82.7	58		0.52 0.1 M TBAT[a]	0.92	358
$C_{13}H_{27}Te$	$C_{13}H_{27}Te$		87.2–87.4	33		0.50 0.1 M TBAT[a]	0.91	358
$C_{14}H_{29}Te$	$C_{14}H_{29}Te$		89.2–90.3	45		0.50 0.1 M TBAT[a]	0.90	358
$C_{15}H_{31}Te$	$C_{15}H_{31}Te$		92.2–93.0	52		0.50 0.1 M TBAT[a]	0.90	358
$C_{16}H_{33}Te$	$C_{16}H_{33}Te$		94.2–94.6	77		0.51 0.1 M TBAT[a]	0.91	358
$C_{17}H_{35}Te$	$C_{17}H_{35}Te$		96.2–97.2	69		0.51 0.1 M TBAT[a]	0.91	358
$C_{18}H_{37}Te$	$C_{18}H_{37}Te$		97.5–98.2	76		0.51 0.1 M TBAT[a]	0.91	358
C_6H_5Te	H	*cis*	152.0–153.7	12		0.54 0.1 M TBAT[c,g]	0.84	653
		trans	179.6			0.54 0.1 M TBAT[c,g]	0.84	653
C_6H_5Te	C_6H_5Te		170.1–170.6 (CS_2/petroleum ether, 3:1)	84		0.46 0.1 M TBAT[c,g]	0.87	653
$COOCH_3$	H	B Z = S $(EtO)_3P$ *trans*	244–246 (glyme)	20				100
		cis	198–200 (benzene)	18				100
		trans	238–242	45	444 (2 455), 312 (10 960), 302 (10 720), 290 (10 000)[e]			171

			cis	198–200 (MeOH)	85				172
		D		244–245 (glyme)	21	444 (2 470), 315 (11 000), 302 (10 600), 290 (10 000)[k]			53
		E		242–244 (glyme)	96	450 (2 000), 312 (9 154), 301 (8 846), 290 (8 000), 235 (10 500)[k]			132, 654
				226–228	53				276
				232	74				655
$COOCH_2CH\text{-}CH_2$		H		103–105	20	430 (2 950), 313 (13 180), 300 (13 490),286 (13 180)[a]			171
$COOCH_2C_6H_5$	H			123–125	20	440 (3 160), 312 (15 490), 299 (15 140), 286 (15 490)[a]			171
$COOC_2H_5$	H	D			26				129
		E		169–172 (MeOH)	2				98
				140–160			0.60 0.1 M TEAP	0.94	306
$COOC_4H_9$	H		*trans*	114–115 (hexane)	18	434 (3 980), 312 (18 200), 301 (16 980), 288 (16 220)[a]	0.65 0.1 M TBAP	0.98	171, 581, 597
			cis	69–70 (hexane)	4	415 (3 020), 312 (15 150), 300 (14 130), 288 (13 180)[a]	0.65 0.1 M TBAP	0.98	171, 581, 597
$COOC_6H_4NO_2\text{-}p$	H	B Z = S $(EtO)_3P$		280 (toluene)	9				100
$COOCOCH_3$	H			350 (MeCN)	47				100
COOH	H			>350 (pyridine)		407 (2 570), 313 (13 000), 302 (12 600), 285 sh[c]			53, 100, 101
		E		360	69				132, 654
			trans			418 (3 550), 313 (19 055), 302 (18 200), 287 (15 850)[o]			171
			cis			404 (2 570), 315 (14 125), 304 (13 490), 288 (12 020)[o]			171
$CONHC^6H^5$	H			224-226 (MeCN)	72				100

(*table continues*)

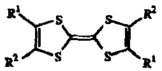
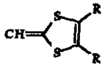
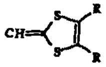
TABLE 11 *(continued)*

R¹ 	R²	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
$CONHC_6H_4N(C_{18}H_{37})_2$-*P*	H		156					656
$CON(CH_3)_2$	H		231-232 (CH_2Cl_2/ether)	22				276
$CON(C_{18}H_{37})_2$	H		75					656
COCl	H		240-245					281
NCO	H		156 (benzene/heptane)					281
$COOCH_3$	CH_3		238-240					655
$COOCH_3$	$COOCH_3$	B Z = S $(Et0)_3P$		63	445 (1 930), 315 (13 100),284 (14 300), 245 (15 500)[p]	0.80 0.1 M TEAP	1.08	98,101,412 657
		Z = S $(Et0)_3P$		52	446[q]	0.83 0.1 M TBAP	1.10	100,101,581
		Z = S Ph_3P		90	445[k]			101
		D		28	461[h]			101,129,130
		E	169-170 (MeOH)	10	462[i]			98,101
		E	169-170 (benzene/hexane)	36				135
		E	163-167 (ether/hexane)	92				132,654
		I Z = S M = Co_2 n = 8	168	20				149
		M R = Bu		73				146
$COOC_4H_9$	$COCH_3$		98–99 (hexane)		446 (2 040), 318 (12 900), 291 (17 400)[a]	0.86 0.1 M TBAP	1.17	370, 581

						0.91	1.22	370
						0.1 M TBAP[e]		
$COOC_4H_9$	I		141–143		439 (2 550), 331			370
					(15 850), 270 (17 800)[a]			
$CONH_2$	$CONH_2$		>360	85	449 (2 040), 319 (15 150)[q]			101, 654
COOH	COOH		>300		477 (2 140), 311 (14 800)[q]			100, 101
			(H_2O or acetone)					
COOCO								101
CF_3	CF_3	B	90–91	89	412 (1 520), 310	1.05	1.28	98
		Z = S			(13 600), 222 (12 600)[r]	0.1 M TBAP		
		Ph_3P						
		E	90–91	100		1.08		98, 412
			sublim.			0.1 M TEAP		
			89,5–90,5	40		1.23	1.45	177
			sublim.			0.1 M TBAHP[k]		
CN	CN	B	265–267	95	502 (2 000), 328	1.12	1.22	101, 412, 658
		Z = O	(CH_2Cl_2)		(15 100)[l]	0.1 M TEAP		659
		$(MeO)_3P$						
		Z = S		20	500 (2 300), 325			99, 101, 658
		$(MeO)_3P$			(15 800), 262 (25 700)[m]			
		Z = S		5	492 (2 000), 323			99, 101, 658
		Ph_3P			(14 500), 261 (21 400)[n]			
		Z = S		75	480 (2 450), 320			101, 658
		$(PhO)_3P$			(11 000), 261 (15 800)[s]			
		I		10				99
		Z = S						
		$M = Fe_3$ n = 12						
CHO	H			45		0.74*	1.08*	286
						0.1 M TBAHP[g]		
CHO	CHO	I	280	60		1.20*	1.48*	87, 174
		$M = Co_2$ n = 8				0.1 M TBAP[m]		
CHO	$CH(OC_2H_5)_2$	I *trans*	172		526 (4 800)[k]			55, 660
		$M = Co_2$ n = 8		70		0.84*	1.24*	
		cis	130–132		504 (4 250)[k]	0.1 M TBAP[v]		
						0.95*	1.30*	174
						0.1 M TBAP[m]		
$CH(OC_2H_5)_2$	$CH(OC_2H_5)_2$	B	136.5–137.5	42		0.62*	1.05*	174
		Z = S				0.1 M TBAP[m]		
		$(MeO)_3P$						
CHBr	CHBr	B		13				

(*table continues*)

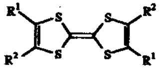
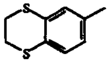
TABLE 11 (*continued*)

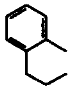
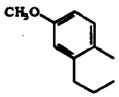
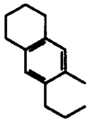
R¹	R²	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
 661								
CHBr	CBr_2	Z = O $(EtO)_3P$ B Z = O $(EtO)_3P$		43				661
$(CH_3)_2C=CH$	$(CH_3)_2C=CH$		114–115 (ether/EtOH)	60		0.33* 0.1 M TBAP	0.65*	174
$CH_3COCH=CH$	$CH_3COCH=CH$		>260	91		0.78* 0.1 M TBAP	0.95*	174
$C_2H_5OOCCH=CH$	$C_2H_5OOCCH=CH$		250	92		0.86* 0.1 M TBAP	1.14*	174
$C_6H_5CH=CH$	$C_6H_5CH=CH$		152–154 (THF)	73		0.42* 0.1 M TBAP	0.67*	174
								
R: H				70		0.19* 0.34* 0.1 M TBAP[q)]	(0.50)*	343, 662
R: $(CH_2)_4$						0.08* 0.17* 0.1 M TBAP[r)]	0.51*	662, 663
						0.13* 0.29* 0.1 M TBAP[q)]	0.48*	662
R: $COOCH_3$				65		0.34* 0.65* 0.1 M TBAP[r)]	0.95*	343, 662
						0.32* 0.65* 0.1 M TBAP[q)]	(0.81)*	662
R: $(CH=CH)_2$				73				343
Cl	H	*cis/trans* mixture	118–119 (hexane)	10				323
Br	H	*cis/trans* mixture	128.5–129.5 (hexane)	5		0.59 0.1 M TEAP		323, 412
I	H		91–92	16	478 (398), 372 sh (2 570), 314 (12 900), 288 (12 900)[s)]	0.58 0.1 M TBAP	0.96	370, 581

						0.63 0.1 M TBAP[g]	1.01	370
			122.4–122.7 (needles) *trans* 100, 130 dec. (plates)	72		0.69 0.1 M TBAT[c,g]	1.05	324
Cl	Cl		221–223	15		0.83 0.1 M TBAHP[h]	1.12	664
				30				321
Br	Br		227–228	15		0.79 0.1 M TBAHP[h]	1.13	664
I	I		175 dec.	68		0.71 0.1 M TBAHP[c,g]	1.03	665
C_6H_5	H	A	202–206 (benzene)	35	403 (2 700), 329 (9 000), 259 (12 100)[c]			584
		F		73	416 (5 950), 331 (22 900), 258 (36 640)[c]	0.39 0.1 M LiCl[f]	0.78	139, 379
		G	207–213 (benzene/MeOH)	64				411
					412 (6 300), 328 (22 200), 280 (27 400), 260 (30 700)[d]	0.43 0.1 M TBAP	0.78	581, 666
		I Z = S M = Co_2 n = 8		25				149
		M		5				145
		A	204–206			0.38 0.05 M TEAP	0.75	667
p-HOC_6H_4	H		207–208 (MeOH)					100
m-HOC_6H_4	H		224–225 (MeOH)					100
p-$CH_3OC_6H_4$	H	A	254–256 (benzene/ether)	35	392 (5 100), 332 (12 100), 270 (22 700)[c]			584
		A	252–254			0.34 0.05 M TEAP	0.71	667
		G	256–261 (benzene/MeOH)		404 (6 080), 334 (18 090), 271 (38 500)[c]	0.33 0.1 M LiCl[f]	0.73	379
p-$CH_3SC_6H_4$	H	A	249–253 (DMF)	88	417 (7 650), 296 (59 600)[c]	0.36 0.1 M LiCl[f]	0.75	379

(table continues)

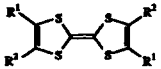
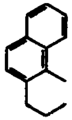
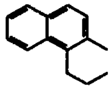
TABLE 11 (*continued*)

R¹ 	R²	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2	References
p-$CH_3CONHC_6H_4$	H	A	329–333 (DMF/MeOH)	73	413 (7 080), 331 (11 080), 295 (23 500)$^{c)}$	0.35 0.1 M LiCl$^{f)}$	0.70	379
p-$CH_3C_6H_4$	H	G	250–256 (benzene/MeOH)	60	411 (5 750), 332 (20 300), 262 (35 900)$^{c)}$	0.35 0.1 M LiCl$^{f)}$	0.76	140, 379
p-$C_6H_5C_6H_4$	H	A	321–323 (DMF)	84	418 (8 540), 331 (28 500), 299 (53 800)$^{c)}$	0.40 0.1 M LiCl$^{f)}$	0.70	379
						0.36 0.1 M TBAHP$^{k)}$	0.86	668
p-FC_6H_4	H	A	231–232 (benzene)	50	408 (4 480), 331 (14 900), 258 (36 640)$^{c)}$	0.40 0.1 M LiCl$^{f)}$	0.81	379
p-ClC_6H_4	H	G	245–251 (benzene/MeOH)	50	424 (6 390), 330 (17 860), 269 (32 940)$^{c)}$	0.43 0.1 M LiCl$^{f)}$	0.79	140, 379
						0.50 0.1 M TBAP	0.84	581
p-BrC_6H_4	H	G	249–255 (benzene/MeOH)	33	400 (6 070), 331 (19 620), 272 (35 540)$^{c)}$	0.43 0.1 M LiCl$^{f)}$	0.79	140, 379
p-IC_6H_4	H	A	290–295 (DMF)	71	410 (6 740), 328 (21 000)$^{c)}$	0.42 0.1 M LiCl$^{f)}$	0.75	379
p-$CH_3COOC_6H_4$	H	A	228–230 (benzene)	15				100, 669
m-$CH_3COOC_6H_4$	H	A	185–186 (EtOH)					100
2,3,4,5-$(CH_3)_4C_6H$		B Z = S $(MeO)_3P$		20		0.28 0.1 M TBAHP$^{k)}$	0.51	668
	H	B Z = S $(EtO)_3P$		20		0.40 0.1 M TBAHP$^{k,l)}$	0.83	668
1-naphthyl	H	A	79–81	87	395 sh (2 420), 292 (32 000)$^{w)}$	0.32 0.1 M LiCl$^{f)}$	0.67	175
2-naphthyl	H	A	243–247	92	416 (6 640), 323 (33 500)$^{w)}$	0.32 0.1 M LiCl$^{f)}$	0.67	175

							0.38 0.1 M TBAHP[k]	0.86	668
C_6H_5	CH_3	G		145–154 (benzene/MeOH)	50	484 (448), 381 (3 970), 290 (23 050)[c]	0.34 0.1 M LiCl[f]	0.73	379, 411
		A		160			0.32 0.05 M TEAP	0.69	667
p-$CH_3OC_6H_4$	CH_3	A		187–191 (MeCN)	67	485 (422), 370 (5 360), 307 (22 650)[c]	0.30 0.1 M LiCl[f]	0.71	379, 670
p-$CH_3C_6H_4$	CH_3	A		192–196 (MeCN)	82	482 (453), 370 (4 830)[c]	0.33 0.1 M LiCl[f]	0.75	379, 670
p-ClC_6H_4	CH_3	A		223–226 (benzene)	62	480 (463), 381 (4 220), 287 (26 870)[c]	0.38 0.1 M LiCl[f]	0.79	379, 670
p-BrC_6H_4	CH_3	A		212–215 (benzene)	73	480 (479) 385 (4 170), 289 (23 700)[c]	0.38 0.1 M LiCl[f]	0.78	379, 670
p-$CH_3COC_6H_4$	CH_3	A		136	12				671
p-$C_6H_5C_6H_4$	CH_3	A		276–278 (DMF)	66	488 (840), 396 (5 780), 274 (41 400)[c]	0.37 0.1 M LiCl[f]	0.71	379
1-naphthyl	CH_3	A	*cis*	110–111	43	482 sh (360), 395 (2 170), 296 (35 000)[w]	0.30 0.1 M LiCl[f]	0.66	175
1-naphthyl	CH_3	A	*trans*	206–209	43	482 sh (415), 395 (2 560), 296 (38 500)[w]	0.30 0.1 M LiCl[f]	0.66	175
2-naphthyl	CH_3	A		216–218	90	585 sh (420), 391 (5 500), 330 (20 500)[w]	0.30 0.1 M LiCl[f]	0.66	175
		A		209–211 (benzene)	91	485 (546), 406 (6 410), 279 (20 500)[c]	0.36 0.1 M LiCl[f]	0.72	93, 163, 379
		F			90				139
		A		211–213 (benzene)	75	472 (597), 396 (8 210), 284 (35 450)[c]	0.30 0.1 M LiCl[f]	0.68	93, 379
		L							163
		A		309–310	86	395 (8 270), 348 (15 790)[h]	0.33 0.05 M TEAP[h]	0.75	176

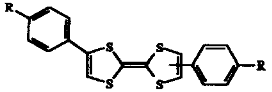
(*table continues*)

TABLE 11 (*continued*)

R^1		R^2	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E$^{ox}_1$ [V]	E$^{ox}_2$	References
		*cis*	A	204–208	20	436 (5 840), 328 (22 000)$^{A)}$ 423 (5 790), 328 (22 000)$^{B)}$	0.32 0.05 M TEAP$^{A)}$	0.69	176
		trans	A	224–228	63	446 (8 570), 328 (31 500)^{A} 433 (8 240), 328 (30 000)$^{B)}$	0.31 0.05 M TEAP$^{A)}$	0.70	176
			A	>360 (1,2-dichlorobenzene)	80	430 (7 260), 365 sh, 334 (80 910)$^{A)}$	0.37 0.05 M TEAP$^{A)}$	0.76	176
C_6H_5		C_8H_5	A	263 (CHCl$_3$/acetone)	80	405 (3 600), 296 (20 500)$^{F)}$	0.41 0.1 M LiCl$^{f)}$	0.77	44, 379
			B Z = S (MeO)$_3$P	263	21	407 (4 470), 302 (29 800)$^{c)}$			101, 379
			G	268–270 (benzene/MeOH)	50				140
			I Z = S M = Co$_2$ n = 8	262	25				149
							0.45 0.1 M TBAP	0.77	581
			M R = Bu		50				146
			M		11				145
.p-$C_4H_9OC_6H_4$		$pC_4H_9OC_6H_4$	A	172					280
p-$C_8H_{17}OC_6H_4$		p-$C_8H_{17}OC_6H_4$	A	121					280
p-$C_{10}H_{21}OC_6H_4$		p-$C_{10}H_{21}OC_6H_4$	A	127					280

2-pyridyl	2-pyridyl	E	287–290 (THF)	80	425 (2 000), 385 (9 150), 355 (8 000)			133
$C_5H_4FeC_5H_5$	H	B Z = Se $(MeO)_3P$	>300 ($CHCl_3$)	31				672
$(CH_3)_3Si$	H	E	131–133	25				134
$(CH_3)_3Si$	$(CH_3)_3Si$	E	223–224 (hexane)	65				134
$(C_6H_5)_2P$	CH_3		241–246 (toluene)	32		0.31 0.1 M TBAHP[*]	0.76	673
$(C_6H_5)_2P$	$(C_6H_5)_2P$		251–252 (toluene)	44		0.33 0.1 M TBAHP[*]	0.73	673

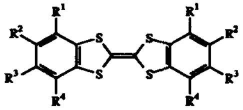
(*table continues*)



liquid crystals

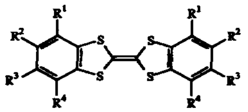
R	preparative method	K	S_G	S_C	N	I	E_1^{ox}	E_2^{ox}	References
				[°C]			[V]		
C_4H_9	A	136	145	—	191		0.43	0.88[k]	280,674
	B								674
	Z = O								
	$(EtO)_3P$								
C_8H_{17}	A	164	184	—	189				280
$C_{10}H_{21}$	B	102	—	—	180		0.43	0.89[k]	674
	Z = O								
	$(EtO)_3P$								
C_4H_9O	A	171	186	—	210				280
$C_8H_{17}O$	A	146	156	172	—				280
$C_{10}H_{21}O$	A	157	—	207	—				280
$C_5H_{11}COO$		186	195	—	—				280
$C_6H_{13}COO$		175	187	192	—				280
$C_7H_{15}COO$		174	182	214	—				280
$C_8H_{17}COO$		171	174	207	—				280
$C_9H_{19}COO$		165	165	205	—				280
$C_{10}H_{21}COO$		162	—	198	—				280
$C_{11}H_{23}COO$		160.5	—	200	—				280
$C_{12}H_{25}COO$		157	—	194	—				280

TABLE 11 (*continued*)



R1	R2	R3	R4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	E_2^{Ox} [V]	References
H	H	H	H	A (benzene)	243	46	428 (297), 340 sh, 308 (20 700), 286 (18 400), 253 (20 600)[a)]	0.72 0.1 M TEAT[g)]	1.06	90
				A		85	436 (309), 310 (19 900), 289 (19 130)[e)]	0.59 0.1 M LiCl[f)]	0.89	89, 93, 379
				B Z = S (EtO)$_3$P	243	45	432 (400), 315, 296[v)]	0.61 0.1 M TEAP	0.93	90, 105, 178, 248
								0.62 0.1 M TBAP	0.93	581
				G	242–243 (benzene/MeOH)	30		0.71 0.2 M TBAT[k)]	1.14	140, 226
				H		10				143, 144
				K		95				150
				M		25				145
CH$_3$	H	H	H	A	248–249 (pyridine)	94				92
H	CH$_3$	H	H	A	260–262 (pyridine)	67	432 (297), 343 sh, 311 (19 700), 287 (18 200), 256 (17 800)[a)]	0.68 0.1 M TEAT[g)]	1.02	90, 92
				A	253	44				91
				B Z = S (EtO)$_3$P	262 (benzene)	25	436 (417), 318, 294[v)]			90, 178
				H	260–262 (pyridine)	33				79
				K		86				150
H	CH$_3$	CH$_3$	H	B Z = S (MeO)$_3$P	>360 (DMF)	45	427 (525), 316 (13 200), 293 (12 000)[v)]			178
H	Cl	H	H	A	315–318 (pyridine)	88				92
H	I	H	H	A	303–304	95				92

TABLE 11 (*continued*)

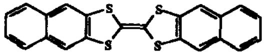
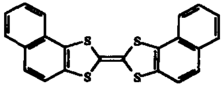
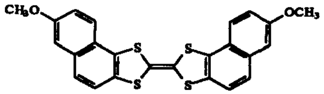
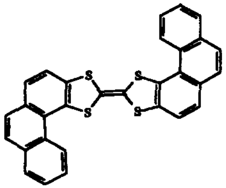
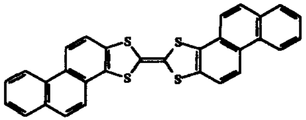
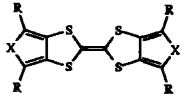


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
H	NO_2	H	H	B	(pyridine) 360–366	60				92
F	F	F	F	I	(pyridine) 175–177	62		1.14 0.1 M TBAHP$^{k)}$	1.45	675
H	CH_3S	CH_3S	H	B Z = O $(EtO)_3P$	281.5–282.5 (xylene)	45		0.43 0.1 M TBAHP$^{k)}$	1.04	676
CH_3S	CH_3S	CH_3S	CH_3S	B Z = S $(EtO)_3P$	302–304	90	409 (10 500)$^{w)}$	0.74 0.1 M TBAP$^{m)}$	1.09	298, 677
CH_3S	C_2H_5S	C_2H_5S	CH_3S		267–269	85	409 (12 600), 315 (38 000)$^{w)}$	0.71 0.1 M TBAP$^{m)}$	1.06	299
CH_3S	$C_5H_{11}S$	$C_5H_{11}S$	CH_3S		232–233	78	409 (12 300), 316 (38 000)$^{w)}$	0.74 0.1 M TBAP$^{m)}$	1.09	299
CH_3S	i-$C_5H_{11}S$	i-$C_5H_{11}S$	CH_3S		222	75	408, 315, 302, 267$^{w)}$			299
CH_3S	$C_{12}H_{25}S$	$C_{12}H_{25}S$	CH_3S		185–187	89	409 (10 700), 316 (34 700)$^{w)}$	0.74 0.1 M TBAP$^{m)}$	1.12	299
CH_3S		$S(CH_2)_2S$	CH_3S		>320 dec.	89	418 (9 330), 271 (37 150)$^{w)}$	0.62 0.1 M TBAP$^{A)}$	0.97	299
C_2H_5S	C_2H_5S	C_2H_5S	C_2H_5S	B Z = S $(EtO)_3P$	161–163	62	409 (11 250)$^{w)}$	0.71 0.1 M TBAP$^{m)}$	1.09	677
i-$C_5H_{11}S$	CH_3S	CH_3S	i-$C_5H_{11}S$		162–164	93	410 (8 320), 315 (27 550), 302 (26 900), 266 (25 100)$^{w)}$			299
$C_5H_{11}S$	$C_5H_{11}S$	$C_5H_{11}S$	$C_5H_{11}S$	B Z = S $(EtO)_3P$	155–157	40	410 (12 050)$^{w)}$	0.74 0.1 M TBAP$^{m)}$	1.11	677
$S(CH_2)_2S$		$S(CH_2)_2S$		B Z = S $(EtO)_3P$	>360	35	381 (6 170)$^{A)}$	0.90 0.05 M TBAP$^{A)}$		677

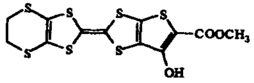
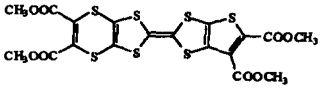
H	S-CO-S	H	B Z = O $(EtO)_3P$	>320 (1,2,4-trichlorobenzene)	96				676
CH_3S	S-CO-S	CH_3S	B Z = O $(EtO)_3P$	>360	96	413, 303, 263[w)]			298
3,5-di-*t*-butylbenzyl-S	S-CO-S	3,5-di-*t*-butylbenzyl-S	B Z = O $(EtO)_3P$	287–293	70	434 (13 800), 311 (47 000), 264 (60 250)[w)]			299
C_2H_5S	S-CO-S	C_2H_5S	B Z = O $(EtO)_3P$	>360	60	415, 303, 263[w)]			299
$C_5H_{11}S$	S-CO-S	$C_5H_{11}S$	B Z = O $(EtO)_3P$	319–320	82				299
i-$C_5H_{11}S$	S-CO-S	iso-$C_5H_{11}S$	B Z = O $(EtO)_3P$	343–345	86	413 (12 600), 303 (39 800), 264 (56 250)[w)]	0.90 0.1 M TBAP[w)]	1.20	298
$C_{12}H_{25}S$	S-CO-S	$C_{12}H_{25}S$	B Z = O $(EtO)_3P$	225–227	70	413 (12 900), 303 (42 660), 263 (54 950)[u)]	0.86 0.1 M TBAP[u)]	1.17	299
$C_5H_{11}S$	S-S-S	$C_5H_{11}S$		266–268	73	422 (14 450), 314 (46 800)[u)]			678
H	S-CS-S	H	B Z = S $(EtO)_3P$		42				679
$C_6H_{13}O$	S-CS-S	$C_6H_{13}O$	K	>245 ($CHCl_3$/MeOH)	73		0.66 0.1 M TBAHP[v)]	1.03	384
i-$C_5H_{11}S$	S-CS-S	iso-$C_5H_{11}S$		>360	76	445, 352[A)]	0.88 0.1 M TBAP[A)]	1.14	299
$C_{12}H_{25}S$	S-CS-S	$C_{12}H_{25}S$		266–270	30	444, 350, 308, 268[w)]			299
H	$S(CH_2)_2S$	H	B Z = O $(EtO)_3P$	>320 (1,2-dichlorobenzene)	73		0.45 0.1 M TBAHP[x)]		676
S-CO-S	S-CO-S		B Z = O $(EtO)_3P$	>360	89	368 sh[A)]			677
$C_6H_{13}O$	SCH_2S	$C_6H_{13}O$		134	41		0.41 0.1 M TBAHP[v)]	0.82	384
CH_3S	SCH_2S	CH_3S		>360	73	416[w)]			298

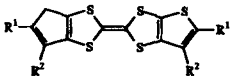
(*table continues*)

TABLE 11 (*continued*)

	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
	A	>400	88				92
	A	302–303 (benzene)	61	375 (2 870), 321 (21 650), 294 (41 250)$^{c)}$	0.52 0.1 M LiCl$^{f)}$		93, 379
	A	279–284 (benzene)	27	397 (303), 323 (22 550), 292 (45 850)$^{c)}$	0.49 0.1 M LiCl$^{f)}$	0.85	93, 379
	B Z = S $(EtO)_3P$	359–361 (1,2-dichlorobenzene)	63	396 (11 860), 335 (24 858)$^{A)}$	0.53 0.05 M TEAP$^{A)}$	0.95	176
	B Z = S $(EtO)_3P$	>360	61	387 (6 210), 328 (36 000)$^{A)}$	0.52 0.05 M TEAP$^{A)}$	0.98	176
							

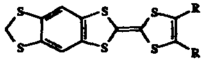
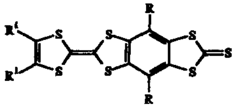
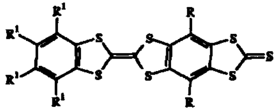
R	X

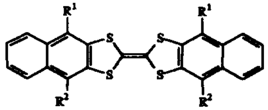
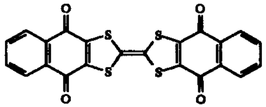
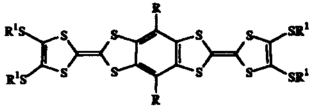
H	S	A	259–260 (CH_2Cl_2/hexane)	31		0.70		80, 680
		B Z = Se $(MeO)_3P$		58				80
		B Z = O,S $(RO)_3P$		0				80
		B Z = S $(MeO)_3P$	263–265	31	449 (126), 313 (20 000)[a]			589, 590
CH_3	S	B Z = S Ph_3P	295–297 (CCl_4)	60				81, 82
CH_3S	S	B Z = Se $(MeO)_3P$	183–184 ($CHCl_3$)	45		0.87 0.1 M TBAP	1.16	681
H	Se	B Z = S $(MeO)_3P$	260–263	54				682
CH_3	NH	M Z = S R = *n*-Bu	>300			0.31 0.1 M TBAHP	0.70	148
CH_3	NC_6H_5	M Z = S R = *n*-Bu				0.39 0.1 M TBAHP	0.74	148
			194–195 dec. ($CHCl_3$/EtOH)	42	404 (3 890), 335 sh (13 180), 307 (23 990)[w]	0.71 0.1 M TBAP[c,g]	1.03	288
				48		0.81 0.1 M TBAHP[b]	1.18	683



(*table continues*)

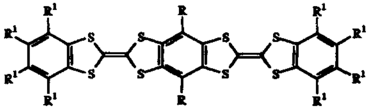
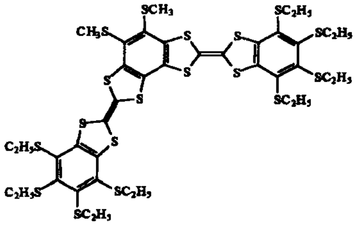
TABLE 11 (*continued*)

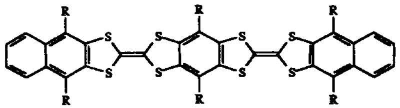
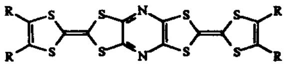
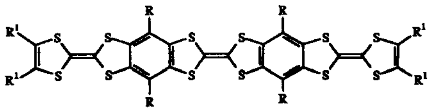
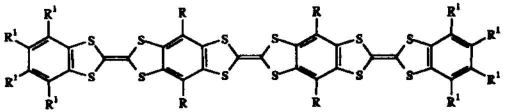
		preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
R^1	R^2							
$COOCH_3$	H		234 dec. (dioxan)	40	402 (8 320), 322 sh (14 125), 307 (39 800)[v]	0.59 0.1 M TBAP[c,g]	0.87	288
$COOCH_3$	$OCOC_6H_5$		223 dec. (benzene/hexane)	45	410 (7 940), 325 sh (17 780), 290 (32 360)[w]	0.85 0.1 M TBAP[c,g]	1.12	288
								
R:CH_3			>300	13		0.40 0.1 M TBAHP[e]	0.83	684
R: $(CH_2)_3$			>300	11		0.41 0.1 M TBAHP[e]	0.83	684
R: $S(CH_2)_2S$			>300	15		0.51 0.1 M TBAHP[e]	0.88	684
								
R	R^1							
S-*i*-C_5H_{11}	SC_6H_{13}	K	158–160 (CH_2Cl_2/MeOH)	58				384
OC_6H_{13}	SC_4H_9	K	41 (acetone)	20				384
OC_6H_{13}	$S(CH_2)_2)S$	K	140 (CH_2Cl_2/petroleum ether)	77				384
								
R	R^1							
OC_2H_5	H	K	273	82				155, 685
OC_6H_{13}	H	K		87				155
O-3,5-di-*t*-butylbenzyl	H	K						686

S-iso-C_5H_{11}	SC_2H_5	K	251–252 (CH_2Cl_2/MeOH)	89			384
							
R^1	R^2						
OH	OH	B	216–218	94			529
		Z = S $(EtO)_3P$		95			687
OCH_3	OCH_3	B	315–316	90			687
		Z = S $(MeO)_3P$					
$OP(O)(OC_2H_5)_2$ 529	OC_2H_5	B	*cis* 129	95			
		Z = S $(EtO)_3P$					
$OCOCH_3$	$OCOCH_3$	B	333–334	90			687
		Z = S $(MeO)_3P$					
$OCOC_5H_{11}$	$OCOC_5H_{11}$		200–204	86			529
			>300	87			529, 687
							
R	R^1						
OC_2H_5	$(CH_2)_2$	K			0.39 (0.98) 0.1 M TBAHP*)	0.57	688
OC_6H_{13}	$(CH_2)_2$	K		86	0.38 0.95 0.1 M TBAHP*)	0.59 (1.16)	156
		B		2.5			

(*table continues*)

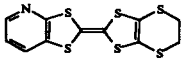
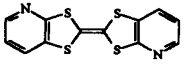
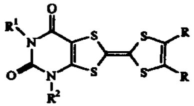
TABLE 11 (*continued*)

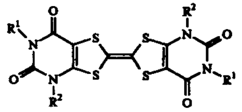
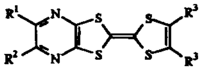
		preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
		Z = S $(EtO)_3P$						
OC_6H_{13}	C_6H_{13}	K		87		0.36 0.86 0.1 M TBAHP[k)]	0.53 0.96	381
OCH_2CH_2Cl	C_6H_{13}	K		97		0.41 0.90 0.1 M TBAHP[k)]	0.56 1.00	381
								
R	R^1							
OC_2H_5	H		280 dec.					154
OC_6H_{13}	H	K	198–199 ($CHCl_3$)	82	313 (60 000), 276 (51 500),247 (36 000)[k)]			154, 685
3,5-di-*t*-butylbenzyl	H	K	242 dec.	68	314 (56 100), 277 (46 000),247 (41 300)[k)]	0.46 1.13 0.1 M TBAHP[k)]	0.68 1.29	154, 157, 685, 686
S-*i*-C_5H_{11}	H	K		66		0.44 1.10 0.1 M TBAHP[k)]	0.68 1.29	157
S-*i*-C_5H_{11}	SCH_3	K						157
S-*i*-C_5H_{11}	SC_2H_5	K				0.41 1.06 0.1 M TBAHP[k)]	0.63 1.22	157
		K				0.54 1.06 0.1 M TBAHP[k)]	0.69 1.22	157

							
R: OH							529
R: $OCOC_5H_{11}$			oil				529
							
R: CH_3				17			689
R: C_6H_{13}			239–240 (c-C_6H_{12})	25		0.49 0.71 1.24 1.50 0.1 M TBAHP$^{k)}$	689
							
R	R^1						
OC_6H_{13}	$S(CH_2)_2S$	B Z = S $(EtO)_3P$	>300	45		0.28 0.42 0.68 (0.87) (1.08) 0.1 M TBAHP$^{v)}$	384, 688
OC_6H_{13}	SC_4H_9	B Z = S $(EtO)_3P$	125	15		0.34 0.45 0.78 1.07 1.31 0.1 M TBAHP$^{v)}$	384
S-i-C_5H_{11}	SC_6H_{13}	B Z = S $(EtO)_3P$	320	58	442, 322, 266$^{k)}$	0.34 0.44 0.69 0.78 1.10 1.33 0.1 M TBAHP$^{G)}$	384
							
R	R^1						
OC_2H_5	H	B Z = S		16			155, 685

(*table continues*)

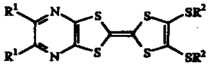
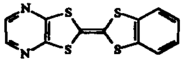
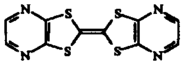
TABLE 11 *(continued)*

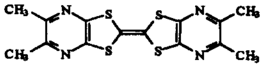
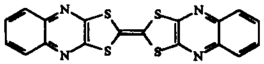
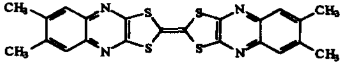
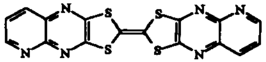
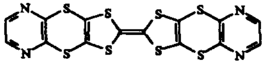
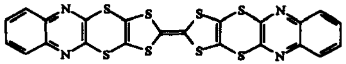
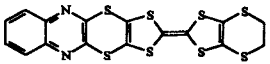
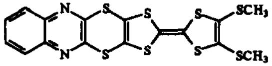
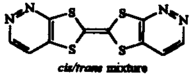
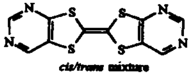
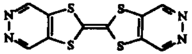
			preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2 [V]	References
			$(EtO)_3P$						
OC_6H_{13}		H	B Z = S $(EtO)_3P$		28		0.31 0.61 1.01 1.28 0.1 M TBAHP[v]	0.81	155, 384
S-*i*-C_5H_{11}		SC_2H_5	B Z = S $(EtO)_3P$	>360	91	431 (34 670), 318 (112 200), 265 (93 000)[k]	0.44 0.61 1.01 1.28 0.1 M TBAHP[F]	0.81	384
			B Z = O,S $(EtO)_3P$	206	5.6				690
			B Z = S $(EtO)_3P$	278 ($CHCl_3$)	25	358 (2 290), 302 (3 420), 280 (3 420), 211 (8 700)			691
									
R	R^1	R^2							
CH_3	H	H	B Z = Se Ph_3P	>250 dec. (DMF)	30		0.62*	0.80*[g,q]	111
							0.7*[g,k]		
							0.72	0.94	692
$S(CH_2)_2S$	H	H	B Z = Se Ph_3P	>260 dec. (DMF)	20		0.75*	0.8*[g,q]	111
CH_3	H	CH_3	B Z = Se Ph_3P	256–260 (DMF)	65		0.79*	1.0*[g,q]	111
$S(CH_2)_2S$	H	CH_3		278–279	80		0.92*	1.04*[g,q]	111
CH_3	H	C_8H_{17}		236–239 (MeCN)	70		0.7*	0.99*[g]	111
							0.72*	0.96*[g,q]	111
							0.86*[g,k]		111

CH_3	H	$C_{16}H_{33}$		206–210 (DMF)	65		0.73*	0.99*[g]	111
							0.77*	0.95*[g,q]	111
CH_3	CH_3	CH_3		241–243 (acetone)	92		0.65*	0.97*[g]	111
							0.7*	0.88*[g,q]	111
$S(CH_2)_2S$	CH_3	CH_3		244–245 (acetone)	85		0.83*	0.97*[g,q]	111
CH_3	C_8H_{17}	C_8H_{17}		114–116	52				111
									
R^1		R^2							
H		H	B Z = Se Ph_3P	>350	25		0.8 [g,k]		111
H		CH_3		>300 dec.	90		0.75 [g,k]		111
H		C_8H_{17}		>300 (DMF)	93				111
CH_3		CH_3		>300	95				111
									
R^1	R^2	R^3							
H	H	H	B $(EtO)_3P$	184	420[a]				693
H	H	CH_3	B Z = O,S $(EtO)_3P$	249	420[a]		0.63 0.1 M TEAP	1.02	693
H	H	$O(CH_2)_2O$	B Z = O $(EtO_3P$	209	6	430, 294 · 311 sh, 240[a]	0.70 0.025 M TBAHP[e]	1.19	300, 694
H	H	$COOCH_3$	B Z = O,S $(EtO)_3P$						693

(*table continues*)

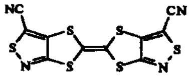
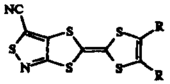
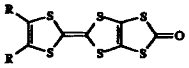
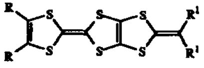
TABLE 11 (*continued*)

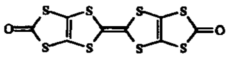
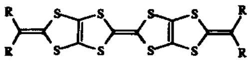
			preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{0x}_1 [V]	E^{0x}_2	References
CH_3	CH_3	CH_3	B $(EtO)_3P$	290	414[a]				693
CH_3	CH_3	$O(CH_2)_2O$	B Z = O $(EtO)_3P$	>280	4	402, 290 · 311 sh, 238 sh, 218 sh[a]			300
Cl	Cl	CH_3		100 dec. ($c-C_6H_{12}$)	35		0.66 0.1 M TBAHP[k]	1.12	689
Cl	SCH_3	CH_3		242–243 ($c-C_6H_{12}$)	20				689
			B Z = O $(EtO)_3P$						
R^1		R^2							
H		CH_3		169	7				695
H		CH_2		218	6	404[a]	0.68	1.07	693, 695
H		$(CH_2)_2$		209	5	414, 326 sh, 306[a]	0.68 0.1 M TEAP	1.02	695, 696
							0.79 0.025 M TBAHP[e]	1.22	694
H		$(CH_2)_3$		222	9				695
H		$\underset{CH_3}{CH}\text{-}CH_2$		145	6				695
CH_3		CH_3		195	9				695
CH_3		CH_2		208	5				695
CH_3		$(CH_2)_3$		261	6				695
CH_3		$\underset{CH_3}{CH}\text{-}CH_2$		187	5				695
$(CH\text{-}CH)_2$		$(CH_2)_2$		272	7				695
			B $(EtO)_3P$	242		408[a]			693
			B Z = S $(EtO)_3P$	303	35	395, 300 sh, 277[a]	0.89 0.1 M TEAP	1.17	695, 697, 698
							(1.05) 0.1 M TBAP[g]		174

	B Z = S (EtO)$_3$P	>310	22		0.99 0.1 M TEAP	1.28	695, 698
	B Z = S (EtO)$_3$P	>310	32	445, 300 sh, 265[k]			695, 697
	B Z = S (EtO)$_3$P	>310	27				695
	B Z = S (EtO)$_3$P	>310	19				695
		295	65				695
	B Z = S (EtO)$_3$P	350–351 dec. (1,2,4-trichlorobenzene)	69				699
	B Z = S (EtO)$_3$P	252–255		307[p]	0.82 0.1 M TBAT[v]	1.06	699
	B Z = S (EtO)$_3$P	179–181		445 (2 240), 337 (12 300), 307 (12 900), 247 (21 900)[p]	0.78 0.1 M TBAT[v]	1.27	699
	B Z = S (EtO)$_3$P						697
	B Z = S (EtO)$_3$P						697
	B Z = S (EtO)$_3$P	300 dec.	80				55, 697

(table continues)

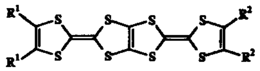
TABLE 11 (*continued*)

	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
	B Z = S $(EtO)_3P$	>300	52	400			700
							
R: SCH_3	B Z = O,S $(EtO)_3P$	136	3	430			700
R: SCH_2S	B Z = O,S $(EtO)_3P$	234	6	430			700
R: $S(CH_2)_2S$	B Z = O,S $(EtO)_3P$	247	10	434			700
							
R: H			50				701
R: $COOCH_3$	B Z = O,S $(EtO)_3P$	215–220	18	430 (1 410), 295 (20 000), 260 (12 900), 238 (17 800)*)	0.89 TEAP	0.98	702
R: CN	B Z = O $(MeO)_3P$	196 (MeCN)					291
R: CF_3	B Z = O,S $(MeO)_3P$	146–147 (hexane)					291
R: CH_3S		178	54				76, 293
	B Z = O,S $(EtO)_3P$						
R R^1							

H	CH_3		167	67	0.45 0.76 (1.41)* 0.1 M TBAP[e]	703
$COOCH_3$	CH_3		212 dec.	67	0.62 0.91 (1.51)* 0.1 M TBAP[e]	703
CH_3S	CH_3		164–166 dec.	47	0.50 0.75 (1.39)* 0.1 M TBAP[e]	703
SCH_2S	CH_3		195 dec.	36	0.51 0.83 0.1 M TBAP[e]	703
$S(CH_2)_2S$	CH_3		201 dec.	37	0.51 0.79 (1.39)* 0.1 M TBAP[e]	703
$S(CH_2)_3S$	CH_3		200–202 dec.	23	0.50 0.78 (1.45)* 0.1 M TBAP[e]	703
H	$(CH_2)_4$		161–162 dec.	89	0.43 0.74 (1.43)* 0.1 M TBAP[e]	704
$COOCH_3$	$(CH_2)_4$		214.5 dec.	64	0.60 0.89 (1.47)* 0.1 M TBAP[e]	704
CH_3S	$(CH_2)_4$		172–173 dec.	57	0.50 0.75 (1.37)* 0.1 M TBAP[e]	704
SCH_2S	$(CH_2)_4$		215–215.5 dec.	55	0.51 0.74 (1.37)* 0.1 M TBAP[e]	704
$S(CH_2)_2S$	$(CH_2)_4$		213–214 dec.	58	0.51 0.77 (1.38)* 0.1 M TBAP[e]	703
$COOCH_3$	$(CH_2)_2\text{-S-}(CH_2)_2$			73		705
CH_3S	$(CH_2)_2\text{-S=}(CH_2)_2$			44		705
H	CH=CH-S-CH-CH		200–201 dec.	45	0.37 0.60 0.94 (1.11)* 0.1 M TBAP[e]	705
$COOCH_3$	CH=CH-S-CH=CH		218 dec.	83		705
CH_3S	CH=CH-S-CH=CH		183–184 dec.	79	0.38 0.63 0.93 (1.10)* 0.1 M TBAP[e]	705
		B	>360	76		63, 293, 294
		B Z = O $(EtO)_3P$				
R: CH_3			222.5–223 dec.	47		706

(*table continues*)

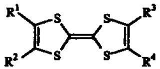
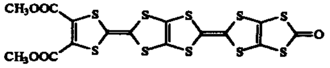
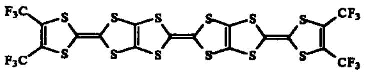
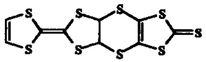
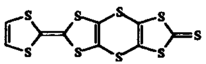
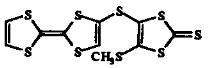
TABLE 11 (*continued*)

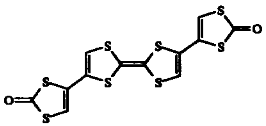
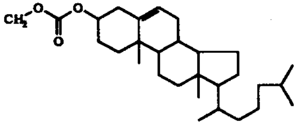
		preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
R: C_2H_5			195–196.5	51	370 sh (6 450), 336 (14 450), 282 sh (21 880), 254 sh (24 000), 249 (24 550)$^{k)}$	0.52 0.1 M TBAP$^{e)}$	0.73 (1.27)	706
R: n-C_3H_7			167–168	58	370 sh (6 170), 337 (13 800), 278 sh (21 880), 256 sh (22 900), 249 (23 450)$^{k)}$	0.51 0.1 M TBAP$^{e)}$	0.73 (1.31)	706
R: $(CH_2)_4$			200–200.5 dec.	65				706
								
R¹	R²							
H	H	B Z = O,S $(EtO)_3P$	179–180 dec. (CS_2/toluene)			0.44 (1.05)* 0.1 M TBAP$^{e)}$	0.62 (1.13)*	701, 707
H	CH_3		179–180 dec.	72				380
H	$O(CH_2)_2O$			55				708
H	CH_3S		163–164.5 dec.	54		0.49 0.99 0.1 M TBAP$^{e)}$	0.71 (1.13)	709
H	SCH_2S	B Z = O,S $(MeO)_3P$	215–215.5 dec.	61				701
H	$S(CH_2)_2S$	B Z = O,S $(MeO)_3P$	210–210.5 dec.	66				701
H	$S(CH_2)_3S$	B Z = O,S $(MeO)_3P$	215–215.5 dec.	33				701
H	$COOCH_3$	B Z = O,S $(MeO)_3P$	191–192 dec.	82				701, 707

CH_3	$O(CH_2)_2O$	B Z = O,S $(MeO)_3P$	193–194 dec.	28				380
CH_3	CH_3S	B Z = O,S $(MeO)_3P$	182–183 dec.	58		0.43 0.98 0.1 M TBAP[e)]	0.68 1.17	380
CH_3	SCH_2S	B Z = O,S $(MeO)_3P$	189–190 dec.	53				380
CH_3	$S(CH_2)_2S$	B Z = O,S $(MeO)_3P$	202–203 dec.	59				380
CH_3	$S(CH_2)_3S$	B Z = O,S $(MeO)_3P$	214–215 dec.	34				380
CH_3	$COOCH_3$	B Z = O,S $(MeO)_3P$	190–191 dec.	79				380
CN	CN	B Z = O $(EtO)_3P$	290 dec. ($CHCl_3$)					291
CF_3	CF_3	B Z = O,S $(EtO)_3P$	233–235 (benzene)					291
CH_3S	CH_3S	B Z = O,S $(MeO)_3P$	192–193	35	385 sh (6 610), 325 (32 360), 288 sh (26 300)[w)]	0.53 0.99 0.1 M TBAP[e)]	0.72 1.11	76
$O(CH_2)_2O$	$O(CH_2)_2O$	B Z = O,S $(EtO)_3P$				0.47 0.99* 0.1 M TBAP[e)]	0.62 1.14*	708
$O(CH_2)_2O$	$S(CH_2)_2S$	B Z = O,S $(EtO)_3P$				0.54 1.12* 0.1 M TBAP[e)]	0.79 1.13*	708
$O(CH_2)_2O$	$S(CH_2)_2S$	B Z = O,S $(EtO)_3P$						708
$O(CH_2)_2O$	$COOCH_3$	B Z = O,S $(EtO)_3P$						708
CH_3S	SCH_2S	B Z = O,S	193–194 dec.	58		0.51 0.1 M TBAP[e)]	0.70	709

(table continues)

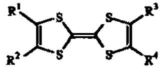
TABLE 11 (*continued*)

 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
CH_3S		$O(CH_2)_2O$		$(MeO)_3P$ B Z = O,S $(MeO)_3P$	187.5–189 dec.	17		0.51 0.97 0.1 M TBAP$^{e)}$	0.72 1.12	709
CH_3S		$S(CH_2)_2S$		B Z = O,S $(MeO)_3P$	200.5–202 dec.	26		0.53 1.00 0.1 M TBAP$^{e)}$	0.74 (1.18)	709
CH_3S		$S(CH_2)_3S$		B Z = O,S $(MeO)_3P$	212–213 dec.	25		0.51 1.02 0.1 M TBAP$^{e)}$	0.73 (1.20)	709
CH_3S		$COOCH_3$		B Z = O,S $(MeO)_3P$	202.5–203.5 dec.	78		0.58 0.99 0.1 M TBAP$^{e)}$	0.81 (1.13)	709
				B	265–270 dec.	3	440 (1 380), 310 (19 500), 280 (15 850), 239 (16 600)$^{w)}$	0.78 TEAP	0.92	702
				B Z = O $(EtO)_3P$	252 dec. (PhCl)	10				291
					165 dec. (benzene)	50		0.37* 0.1 M TBAP$^{q)}$	0.72*	710
					231–232 dec. (toluene)	80		0.59* 0.1 M TBAP$^{q)}$	0.71*	710
					oil	92		0.48* 0.1 M TBAP$^{q)}$	0.67*	710

					275–277		410, 325, 295[w]	0.73 0.1 M TEAP[q]	0.90	711
H	H	CH_3	CH_3	A	117–119 (MeCN)	78	445 (640), 360 sh, 322 sh, 308 sh, 299 (14 300), 290 sh, 278 sh	0.28 0.05 M TBAT	0.65	121, 579
					96–98	74				712
								0.28 0.1 M TBAP	0.67	581
H	H		$(CH_2)_3$	A	141 subl.			0.20 $NaClO_4$	0.54	713, 714
								0.35 TEAP[k]	0.75	715
H	H		$(CH_2)_4$	C	105–106	41	460 (260), 364 (1 500), 322 (9 550), 310 (9 800), 299 (10 500)			113
				122				0.42 0.1 M TBAP[v]	0.84	714
H	CH_2OH	H								656
CH_3	CH_3	$(CH_2)_3$		A	232			0.26 0.1 M TBAT	0.61	122, 714, 716
CH_3	CH_3	$(CH_2)_4$		C	174–175		478 (214), 323 (9 550), 314 (10 500), 298 (11 200), 286 (10 200)			113
				C	194	53		0.32 0.1 M TBAP[v]		127, 714, 717
CH_3	C_2H_5	$(CH_2)_3$		A	132 subl.			0.16 $NaClO_4$	0.52	713
$(CH_2)_3$		$(CH_2)_4$				217		0.34	0.83	714 0.1 M TBAP[v]
$CH_2CHCH_2CH_2$ $CH_2OCOC_{17}H_{35}$		$(CH_2)_4$		B Z = O,S $(EtO)_3P$	86-88	33		0.7	0.87[p]	718

(table continues)

TABLE 11 (*continued*)

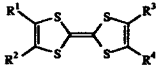


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
CH_3	CH_3	H	CH_3		110	50				719
CH_3	CH_3	H	$C_{17}H_{35}$	A	82–83 (MeCN)		480 (447), 320 (11 700), 310 (12 300), 300 (12 900)^{b}			586
CH_3	CH_3	H	$C_8H_{16}COOCH_3$	C						720
CH_3	CH_3	H	$C_8H_{16}COOH$							720
CH_3	CH_3	H	$C_8H_{16}CONH$ $C_{10}H_{20}COOH$							720
CH_3	CH_3	H	$C_8H_{16}CONH$ $C_{10}H_{20}CONH$ $C_{10}H_{20}COOH$							720
CH_3	CH_3	H	$CH{=}CHC_9H_{18}$ COOH							720
CH_3	CH_3	H	p-$CH_3COOC_6H_4$	A C	130	17 9				671
CH_3	CH_3	H	p-HOC_6H_4		191	87				671
CH_3	CH_3	CH_3	p-$CH_3COOC_6H_4$	A C	232	15 11				671
CH_3	CH_3	CH_3	p-HOC_6H_4		165	82				671
CH_3	CH_3	$C_{16}H_{33}$	$C_{17}H_{35}$	A	82–83	43	490 (159), 313 (14 800), 300 (14 500)^{b}			586
CH_3	CH_3	$C_{17}H_{35}$	$C_{17}H_{35}$	A	80–81	33	485 (240), 360 sh, 323 sh, 312 (11 000), 300 (11 000)$^{b)}$			586
CH_3	CH_3	CH_3	CH_3S		119 (hexane)	51		0.28 0.1 M TBAHP$^{a)}$	0.73	96
CH_3	CH_3	CH_3	$C_{18}H_{37}S$		68 (hexane)	31		0.27 0.1 M TBAHP$^{a)}$	0.78	96
CH_3	CH_3	CH_3	CH_3OOC $C_{10}H_{20}S$	C						720
CH_3	CH_3	CH_3	CH_3OOC $C_{15}H_{30}S$	C						720
CH_3	CH_3	CH_3	HOOC $C_{10}H_{20}S$							720
CH_3	CH_3	CH_3	HOOC $C_{15}H_{30}S$							720

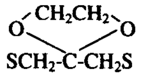
CH_3	CH_3	CH_3	HOOC $C_{10}H_{20}NH$ $COC_{10}H_{20}S$							720
CH_3	CH_3	CH_3	HOOC $C_{10}H_{20}NH$ $COC_{15}H_{30}S$							720
CH_3	CH_3	CH_3	$C_6H_5CH_2S$		127 (hexane)	57		0.27 0.1 M TBAHP[k]	0.81	96
CH_3	CH_3	CH_3	C_2H_5OOC $(CH_2)_{10}S$		59 (hexane)	41		0.27 0.1 M TBAHP[k]	0.77	96
CH_3	CH_3	CH_3	C_2H_5OOC $(CH_2)_{15}S$		71 (hexane)	32		0.27 0.1 M TBAHP[k]	0.78	96
CH_3	CH_3	CH_3	$(C_6H_5)_2P$		158–160 (MeCN)	62		0.23 0.1 M TBAHP[k]	0.74	673
CH_3	CH_3	p-$CH_3C_6H_4$	$C_{18}H_{37}S$		65–70			0.47 0.1 M TBAP[g]	0.82	275
H	H		$O(CH_2)_2O$	B Z = O $(EtO)_3P$	92	2.5	483, 296–308 sh, 246[a]			300
				B Z = O,S $(EtO)_3P$	91.0–91.5	3		0.39 0.1 M TBAHP[c,g]	0.83	721
					162	18				722
H	H	CH_3S	CH_3S	B $(EtO)_3P$	60		450, 370, 324, 292[a]			723
					63–65	64		0.40 0.1 M TBAHP	0.73	724
H R: p-$CH_3COOC_6H_4$	H	RCH_2S	RCH_2S	B Z = O,S $(EtO)_3P$		47				701
H R: OH	H	RCH_2CH_2S	RCH_2CH_2S		98–101	31		0.49 0.1 M TBAHP	0.80	304, 332
H R: t-$C_4H_9(C_6H_5)_2SiO$	H	RCH_2CH_2S	RCH_2CH_2S							304
H R: TTFCOO	H	RCH_2CH_2S	RCH_2CH_2S		66–67	77	436 (4 000), 317 (33 000), 305 (32 000), 276 (27 000), 230 (23 000)[k]	0.69 0.1 M TBAHP[c]	0.96	304

(*table continues*)

TABLE 11 (*continued*)

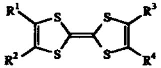


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	[V]	E_2^{Ox}	References
H	H	RCH_2CH_2S	RCH_2CH_2S			60		0.45	0.62	0.82	333
R: $C_5H_5Fe\ C_5H_4COO$										0.2 M TBAHP g)	
H	H	n-C_3H_7S	n-C_3H_7S		oil	85					652
H	H	C_6H_5COS	C_6H_5COS	C	180 (dichlorobenzene)	35					652
H	H	SCH_2		B $(EtO)_3P$	154	29	448, 370, 326, 307, 294 u)	0.54		0.92	582, 723
								0.025 M TBAHP m)			
H	H	$S(CH_2)_2S$		B $(EtO)_3P$	200	36	444, 363, 327, 306, 293 u)	0.55		0.97	582, 723
								0.025 M TBAHP m)			
					206–207 (MeCN/toluene 1:2)	35		0.42		0.74	724
								0.1 M TBAHP			
					210–212 (MeCN)	25					712
					199	20					332
				A				0.39		0.69	119
								0.1 M TEAP			
								0.44		0.75	581
H	H	$S\underset{CH_3}{CH}\text{-}CH_2S$		B $(EtO)_3P$	79	12	444, 365, 328, 306, 292	0.1 M TBAP			723
H	H	$S\overset{CH_3}{CH}\text{-}\underset{CH_3}{CH}S$		B 146 $(EtO)_3P$			442, 362, 321, 306, 290 u)				723
H	H	$S(CH_2)_3S$		B $(EtO)_3P$	151	33	450, 386, 322, 300 u)				723
					145–146 (hexane)	52	400 (398), 329 (16 600), 281 (14 450) w)	0.48		0.83 g)	712
				A				0.41		0.73	119
								0.1 M TEAP			
H	H	$S\overset{R}{C}H_2CHCH_2S$									170
R: OH					190–192	70		0.50		0.89	
								0.1 M TBAHP g,k)			
R: OCON $HC_{18}H_{37}$					129–132	10					

R: OSi$(C_6H_5)_2$-C_4H_9-*t*				B Z = O,S $(EtO)_3P$	95–96	32				
H	H	 SCH$_2$-C-CH$_2$S		B Z = O,S $(EtO)_3P$	200–203	37				170
H	H	SCH_2COCH_2S			179–182 dec. (cubes) 186–187 dec. (needles)	94		0.60 0.1 M TBAHP[e,v]	0.91	170, 631
H	H	SCH_2OCH_2S		B Z = O,S $(EtO)_3P$	193–194			0.14 0.1 M TBAP[b]	0.44	725
H	H	$SeCH_2Se$			173	21	450, 325, 306, 225[e]	0.49 0.025 M TBAHP[e]	0.87	582, 726
H	H	$Se(CH_2)_2Se$			195	20	440, 368, 318, 290, 230[e]			726
					194–197	18				332
H	H	$Se(CH_2)_3Se$			148	18	389, 323, 280, 225[e]			726
H	CH_3	CH_3S	CH_3S		72–73 (*c*-C_6H_{12})	38		0.39 0.1 M TBAHP	0.71	724
H	CH_3	$S(CH_2)_2S$						0.41 0.1 M TBAP	0.75	581
					134–135.5 (MeCN)	26		0.40 0.1 M TBAHP	0.73	724
H	CH_2OH	$S(CH_2)_2S$			121–123 ($CHCl_3$/petroleum ether)	45		0.52* 0.1 M TBAP[q]	0.70*	285, 727
H	$(CH_2)_3C{\equiv}CH$	$S(CH_2)_2S$				75				728
H	$(CH_2)_8C{\equiv}CH$	$S(CH_2)_2S$				75				728
H	$(CH_2)_3C{\equiv}C$- $C{\equiv}C(CH_2)_{11}CH_3$	$S(CH_2)_2S$			43–45			0.48	0.68[s,q]	728
H	$(CH_2)_8C{\equiv}C$- $C{\equiv}C(CH_2)_4CH_3$	$S(CH_2)_2S$			72–74			0.48	0.68[s,q]	728
H	$(CH_2)_8C{\equiv}C$- $C{\equiv}C(CH_2)_{11}CH_3$	$S(CH_2)_2S$			52–54			0.48	0.68[s,q]	728
CH_3	CH_3	$P(C_6H_5)_2$	$P(C_6H_5)_2$		162–163	75	420 (2 050), 324 (16 200)[k]	0.34 0.1 M TBAHP	0.71	312
								0.27 0.1 M TBAHP[k]	0.81	673
CH_3	CH_3	CH_3S	CH_3S					0.40	0.70	729

(*table continues*)

TABLE 11 (*continued*)

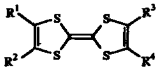


R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
								0.2 M TEAB		
								0.40 0.1 M TBAP	0.73	581
CH_3	CH_3	SCH_2S			139–141					109
CH_3	CH_3	$O(CH_2)_2O$		B Z = O $(EtO)_3P$	162	2				694
				C	132	15		0.33 0.1 M TBAP	0.63	722
CH_3	CH_3	$S(CH_2)_2S$		A	169–170 (benzene)		459 (660), 340 (10 000), 312 (11 750)[v]	0.37 0.1 M TEAP	0.67	119, 178
					169–170 (toluene)	15				724
				B		9		0.40 0.2 M TEAB	0.71	729
								0.45 0.1 M TBAP	0.79	581
								0.44	0.83	730
CH_2OH	CH_2OH	$S(CH_2)_2S$			170–175 (EtOAc)	93		0.53* 0.1 M TBAP[q]	0.72*	285, 727
CH_3	CH_3	$S(CH_2)_3S$		C		62		0.36 0.1 M TEAP	0.69	119
CH_3	CH_3	SCH_2CHCH_2S OH			207–210	90				170, 631
CH_3	CH_3	$O(CH_2)_2O$ SCH_2-C-CH_2S		Z = O,S $(EtO)_3P$	B	218–220	20			170
CH_3	CH_3	SCH_2COCH_2S			220–222	93		0.51 0.1 M TBAHP[s,v]	0.96	170, 631
$(CH_2)_3$		$S(CH_2)_2S$		A				0.37 0.1 M TEAP	0.66	119
								0.43 TEAP[s]	0.83	715
CH_3	C_2H_5OCO-	$C_{10}H_{21}S$	$C_{10}H_{21}S$	C	38–40	22				731

	$(CH_2)_3S$									
CH_3	$NC(CH_2)_3S$	$C_{10}H_{21}S$	$C_{10}H_{21}S$	C	66–69	21				731
CH_3	$H_2N(CH_2)_4S$	$C_{10}H_{21}S$	$C_{10}H_{21}S$		110–111	59				731
CH_3	HOOC-$(CH_2)_3S$	$C_{10}H_{21}S$	$C_{10}H_{21}S$		74–75	49				731
$O(CH_2)_2O$		CH_3S	CH_3S	B Z = O,S $(EtO)_3P$	73.7–74.1	12		0.43 0.1 M TBAHP[e,g]	0.80	721
$O(CH_2)_2O$		SCH_2S		B Z = O,S $(EtO)_3P$	171	3.9				690
				B Z = O,S $(EtO)_3P$	161–163 dec.	9		0.46 0.1 M TBAHP[e,g]	0.79	721
$O(CH_2)_2O$		=CH-S-CH=		B Z = O $(EtO)_3P$	206	8				694
$O(CH_2)_2O$		$S(CH_2)_2S$		B $(MeO)_3P$	184–185			0.01 0.1 M TBAHP[h,k]	0.43	732
				B Z = O $(EtO)_3P$	192	14	490, 445 sh, 324, 310 sh, 220 sh[a]			300
				B Z = O,S $(EtO)_3P$	184–185 dec.	54		0.46 0.1 M TBAHP[e,g]	0.86	721
$O(CH_2)_2O$		$S(CH_2)_3S$		B Z = O $(EtO)_3P$	194	6				694
				B Z = O,S $(EtO)_3P$	184–185 dec.	59		0.46 0.1 M TBAHP[e,g]	0.91	721
$O(CH_2)_2O$		SCH_2OCH_2S		B Z = O,S $(EtO)_3P$	188–189 dec.	58		0.47 0.1 M TBAHP[e,g]	0.88	721
$O(CH_2)_2O$		SCH = CHS		B Z = O,S $(EtO)_3P$	194	4.5		0.62 0.025 M TBAHP[e]	1.00	690, 694
$O(CH_2)_2O$		$SeCH_2Se$		B Z = O,S $(EtO)_3P$	218	<1				690

(table continues)

TABLE 11 (*continued*)

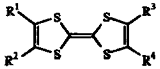


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
$O(CH_2)_2O$		$Se(CH_2)_2Se$		B Z = O $(EtO)_3P$	187	4,5	486, 442 sh, 324, 310 sh, 230 sh[a)]			300
$O(CH_2)_2O$		$Se(CH_2)_3Se$		B Z = O $(EtO)_3P$	172	2				694
$O(CH_2)_2O$		$(CH{=}CH)_2$		 Z = O,S $(EtO)_3P$	B	175	7			690
$S(CD_2)_2S$		$S(CH_2)_2S$			237–238					109
RCH_2S R: p-CH_3 $COOC_6H_4$	RCH_2S	CH_3S	CH_3S	B $(EtO)_3P$	80–85 Z = O	30				353, 354
				B Z = O,S $(EtO)_3P$	80–85	62				76
RCH_2S R: p-C_6H_5 $COOC_6H_4$	RCH_2S	CH_3S	CH_3S		120–122	66				354
CH_3S	CH_3S	CH_3S	$NCCH_2$ CH_2S	102–104 (toluene/c-C_6H_{12})		94		0.57 0.1 M TBAHP[k)]	0.92	365
CH_3S	CH_3S	$NCCH_2$ CH_2S	$NCCH_2$ CH_2S	B Z = O,S $(EtO)_3P$	113–114	68		0.59 0.1 M TBAHP[k)]	0.92	355
CH_3S	CH_3S	C_4H_9S	C_4H_9S			54				353, 354
RCH_2S R: p-CH_3 $COOC_6H_4$	RCH_2S	C_4H_9S	C_4H_9S	B $(EtO)_3P$	90–92 Z = S	17				353, 354
RCH_2S R: HOOC	RCH_2S	$C_{10}H_{21}S$	$C_{10}H_{21}S$		126–129 dec.	56				731
RCH_2S R: CH_3OOC	RCH_2S	$C_{10}H_{21}S$	$C_{10}H_{21}S$	B Z = O $(EtO)_3P$	67–69	21				731
CH_3S	CH_3S	$C_{12}H_{25}S$	$C_{12}H_{25}S$	65–66						109

CH_3S	CH_3S	$C_{16}H_{33}S$	$C_{16}H_{33}S$	B Z = O $(EtO)_3P$						733
RCH_2S R: HOOC	RCH_2S	$C_{16}H_{33}S$	$C_{16}H_{33}S$		131 (toluene/hexane)	80		0.61 0.1 M TBAHP$^{h)}$	0.97	604
RCH_2S R: CH_3OOC	RCH_2S	$C_{16}H_{33}S$	$C_{16}H_{33}S$		87 (hexane)	25		0.65 0.1 M TBAHP$^{a)}$	1.02	604
RCH_2S R: CH_3OOC	RCH_2S	C_8F_{13} $(CH_2)_{11}S$	C_6F_{13} $(CH_2)_{11}S$	B Z = O,S $(EtO)_3P$	70,5–71,0					734
CH_3S	CH_3S	$C_{18}H_{37}S$	$C_{18}H_{37}S$		65–66					109
RCH_2S R: CH_3OOC	RCH_2S	$C_{18}H_{37}S$	$C_{18}H_{37}S$	B Z = S $(EtO)_3P$	64–66	12	380 sh, 330, 310, 265$^{k)}$	0.7	0.96	735
CH_3S	CH_3S	C_6H_5COS	C_6H_5COS		165	22				354
CH_3S	CH_3S	SCH_2S			83	11	480, 384, 342, 313$^{a)}$			603
RCH_2S R: CH_3OOC	RCH_2S	SCH_2S		B	87	20	480, 418, 340, 312, 298, 274$^{a)}$			603
CH_3S	$HO(CH_2)_8S$	SCH_2S			oil					109
CH_3S	$C_{18}H_{37}S$	SCH_2S			46–49					109
$C_{18}H_{37}S$	$C_{18}H_{37}S$	SCH_2S			65–67					109
$S(CH_2)_2S$		SCH_2S		B Z = O $(MeO)_3P$	183,5–184 $(CHCl_3)$			0.07 0.1 M TBAHP$^{h,k)}$	0.45	736
					184	19	495, 342, 319, 280, 226$^{a)}$			614
$S(CH_2)_2S$		$S\underset{COOCH_3}{CH}S$		B $(EtO)_3P$	167	29	478, 348, 319, 306, 230, 212$^{a)}$			614
$S\underset{CH_3}{CH}\text{-}CH_2S$		SCH_2S			166	16				614
$S\underset{CH_3}{CH}\text{-}CH_2S$		$S\underset{COOCH_3}{CH}S$		B $(EtO)_3P$	131	16				614
$S\underset{CH_3}{CH}\text{-}\overset{CH_3}{CH}S$		SCH_2S			167	8				614
$S\underset{CH_3}{CH}\text{-}\overset{CH_3}{CH}S$		$S\underset{COOCH_3}{CH}S$		B $(EtO)_3P$	141	21				614
$S(CH_2)_3S$		SCH_2S		B Z = O $(MeO)_3P$	192–193 dec.			0.08 0.1 M TBAHP$^{h,k)}$	0.46	737

(*table continues*)

TABLE 11 (*continued*)

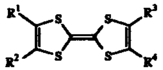


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	E_2^{Ox} [V]	References
					189	12				614
$S(CH_2)_3S$		$SCHS$ $COOCH_3$		B $(EtO)_3P$	178	18				614
CH_3S	CH_3S	$S(CH_2)_2S$		A	64	6		0.48	0.72	119, 603
					75	8		0.1 M TEAP		
					50					354
RCH_2S R: p-$CH_3COOC_6H_4$	RCH_2	$S(CH_2)_2S$		99–102	29					354
						47				353
CH_3S	CH_3S	$SCH\text{-}CH_2S$ CH_3			61	4				603
CH_3S	CH_3S	$SCH\text{-}CH_2S$ 4-pyridyl		B Z = O,S $(MeO)_3P$	158–159 (MeCN)	22				738
CH_3S	CH_3S	$SCH\text{-}CH_2\text{-}S$ 4-methylpyridinium			160–170	96				738
CH_3S	CH_3S	CH_3 $SCH\text{-}CHS$ CH_3		93	6				603	
RCH_2S R: CH_3OOC	RCH_2S	$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	96	16				603
RCH_2S R: CH_3OOC	RCH_2S	$SCH\text{-}CH_2S$ CH_3		B $(EtO)_3P$	65	14				603
RCH_2S R: CH_3OOC	RCH_2S	CH_3 $SCH\text{-}CHS$ CH_3		B $(EtO)_3P$	>82	33				603
RCH^2S R: $(CH_3)_3$ $Si(CH_2)_2O$	RCH_2S	CH_3 $SCH\text{-}CHS$ CH_3		B Z = O,S $(EtO)_3P$	oil		470, 375 sh, 312, 299 sh, 250 sh, $229^{a)}$			109
RCH_2S R: $(CH_3)_3$ $Si(CH_2)_2O$	$BrCH_2CH_2S$	CH_3 $SCH\text{-}CHS$ CH_3		B Z = O,S $(EtO)_3P$	76–78		470, 378 sh, 337 sh, 312, 300 sh, 250 sh, $228^{a)}$			109

$C_6H_5CH_2$ CH_2S	$C_6H_5CH_2$ CH_2S	$NCCH_2$ CH_2S	$NCCH_2$ CH_2S	B Z = O,S $(EtO)_3P$						173
C_2H_5S	C_2H_5S	$S(CH_2)_2S$		B Z = O $(EtO)_3P$						739
$ClCH_2$ CH_2S	$ClCH_2CH_2S$	$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	65–66	27				330, 332
$NCCH_2$ CH_2S	$NCCH_2CH_2S$	$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	141–142	48		0.60 0.1 M TBAHP[k]	0.98	355
$CH_2{=}CHS$	$CH_2{=}CHS$	$S(CH_2)_2S$			50–51	91	475, 380, 338 sh, 313, 300 sh, 227[a]	0.54 0.1 M TBAHP[e,k]	0.91	109, 330, 332
$C_7H_{15}S$	$C_7H_{15}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$	11.4–13.4	22	480 sh (290), 385 sh (3 020), 343 (12 590), 316 (13 490), 302 sh (12 300)[w]	0.54 0.1 M TBAT	0.78	608
$C_{10}H_{21}S$	$C_{10}H_{21}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$						739
$C_{12}H_{25}S$	$C_{12}H_{25}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$						606
$C_{13}H_{27}S$	$C_{13}H_{27}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$						739
$C_{16}H_{33}S$	$C_{16}H_{33}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$						733, 739
$C_{18}H_{37}S$	$C_{18}H_{37}S$	$S(CH_2)_2S$		B Z = O $(EtO)_3P$				0.63	0.94	606, 733, 735, 739
C_6H_5COS	C_6H_5COS	$S(CH_2)_2S$			122	26				354
$S(CH_2)_2S$		$S\underset{CH_3}{CH}\text{-}CH_2S$			193–194					109
$S(CH_2)_2S$		$S\underset{C_{16}H_{33}}{CH}\text{-}CH_2S$		B Z = O $(EtO)_3P$	92–93 (hexane)	13		0.63 0.59	0.90[e] 0.89[e]	625 625
$S(CH_2)_2S$		$S\underset{C_{18}H_{37}}{CH}\text{-}CH_2S$		B Z = 0 $(EtO)_3P$	95.5–96	16		0.42 0.1 M TBAHP[e,k]	0.90	740

(*table continues*)

TABLE 11 *(continued)*

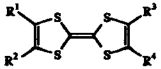
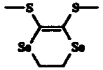
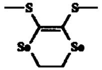
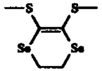


R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
$S(CH_2)_2S$		$SCH\text{-}CH_2S$ $CH_2COOC_{17}H_{35}$		B Z = O,S $(EtO)_3P$	86–88	33		0.7	$0.87^{g)}$	718
$S(CH_2)_2S$		CH_3 $SCH\text{-}CHS$ CH_3			212 ($CHCl_3$)	30	343 sh, 321, $230^{e)}$			109
				B Z = O $(EtO)_3P$	196–200 dec. ($CHCl_3$)			0.39	(0.87)*	628
CH_3S	CH_3S	$S(CH_2)_3S$		A	124	17		0.48 0.1 M TEAP	0.73	119, 603
RCH_2S R: CH_3OOC	RCH_2S	$S(CH_2)_3S$		B $(EtO)_3P$	119	20				603
CH_3S	CH_3S	SCH_2CHCH_2S R								170
R: OH					143–144 (CH_2Cl_2/hexane)	67		0.56 0.1 M $TBAHP^{g,k)}$	0.92	
R: $OCOCH\text{-}CH_2$					156–157 (CH_2Cl_2/hexane)	31		0.57 0.1 M $TBAHP^{g,k)}$	0.93	
R: $OCONH(CH_2)_2Cl$					219–221 (CH_2Cl_2/hexane)	80		0.56 0.1 M $TBAHP^{g,k)}$	0.92	
R: $OSi(C_6H_5)_2C_4H_9$-*t*					Bnot analytical pure	30				
				Z = O $(EtO)_3P$						
CH_3S	CH_3S	$O(CH_2)_2O$ ╲ ╱ $SCH_2\text{-}C\text{-}CH_2S$		B Z = O $(EtO)_3P$	152–154	18				170
CH_3S	CH_3S	SCH_2COCH_2S			175–178	92		0.64 0.1 M $TBAHP^{g,v)}$	0.98	170, 631
CH_3S	CH_3S	$SCH_2CH_2SCH_2CH_2S$			oil	11			354	
CH_3S	CH_3S	$S(CH_2CH_2O)_3CH_2CH_2S$		B Z = S $(EtO)_3P$	125–126 (EtOH/$CHCl_3$)	21		0.51	$0.76^{h,k)}$	602

$S(CH_2)_2S$		$S(CH_2)_3S$		C		22		0.49 0.1 M TEAP	0.77	119
					235–236 dec.			0.11 0.1 M TBAHP$^{h,k)}$	0.52	737
				B Z = O,S $(EtO)_3P$	255 dec.			0.19 0.1 M TBAP$^{h)}$	0.47	725
$S(CH_2)_2S$		$O(CH_2)_2O$		B Z = O $(EtO)_3P$	226–228	25				170
$S(CH_2)_2S$		$SCH_2\text{-}C\text{-}CH_2S$ SCH_2COCH_2S			218–220	95		0.64 0.1 M TBAHP$^{g,v)}$	1.02	170, 631
$S(CH_2)_2S$		SCH_2OCH_2S		B Z = O,S $(EtO)_3P$	258–260 dec. (benzene)	61	343 (10 700), 318 (13 200)$^{c)}$	0.54 0.1 M TBAP	0.79	741
$S(CH_2)_2S$		SCH_2SCH_2S		B Z = O,S $(EtO)_3P$	233–235 dec.	58	342 (12 020), 316 (15 850)$^{c)}$	0.73 0.1 M TBAP$^{c)}$		742
SCH_2CHCH_2S R R: $OSi(C_6H_5)_2C_4H_9\text{-}t$		$O(CH_2)_2O$ $SCH_2\text{-}C\text{-}CH_2S$		B Z = O $(EtO)_3P$	>230	37				170
$S(CH_2)_3S$		SCH_2OCH_2S		B Z = O,S $(EtO)_3P$	257–258			0.25 0.1 M TBAP$^{h)}$	0.50	725
$C_{18}H_{37}S$	$C_{18}H_{37}S$	$Se(CH_2)_2Se$		B Z = O $(EtO)_3P$	84	20				743
$C_{18}H_{37}S$	$C_{18}H_{37}S$	$Se(CH_2)_3Se$		B Z = O $(EtO)_3P$	80	17				743
SCH_2S		CH_3Se	CH_3Se		oil					109
SCH_2S		$Se(CH_2)_2Se$		B Z = O $(EtO)_3P$	285	11				743
SCH_2S		$Se(CH_2)_3Se$		B Z = O $(EtO)_3P$	212	14				743
$S(CH_2)_2S$		CH_3Se	CH_3Se							739
$S(CH_2)_2S$		$C_{18}H_{37}Se$	$C_{18}H_{37}Se$	B Z = O $(EtO)_3P$	84	14				743

(*table continues*)

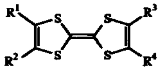
TABLE 11 *(continued)*

 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ$_{max}$ (ε) [nm]	E$_1^{Ox}$ [V]	E$_2^{Ox}$	References
$S(CH_2)_2S$		CH_3Te	CH_3Te							739
$S(CH_2)_2S$		$SeCH_2Se$		B Z = O $(EtO)_3P$	230	4				743
$S(CH_2)_2S$		$Se(CH_2)_2Se$			294	12				743
$S(CH_2)_3S$		$C_{18}H_{37}Se$	$C_{18}H_{37}Se$	B Z = O $(EtO)_3P$	86	17				743
$S(CH_2)_3S$		$\vert SeCH_2Se\vert$		B Z = O $(EtO)_3P$	230	8				743
C_6H_5Se	C_6H_5Se	C_6H_5Se	H			84				327
SCH=CHS		H	H	B $(EtO)_3P$	181	18	440, 380, 323, 300$^{a)}$			723
		H	H		220					651
		H	$COOCH_3$	B Z = S $(EtO)_3P$	141					651
SCH=CHS		CH_3S	CH_3S		117	7				109, 603
SCH=CHS		RCH_2S	RCH_2S	B $(EtO)_3P$	107	12				603
R: CH_3OOC										
SCH=CHS		SCH_2S			189	8				614
SCH=CHS		SCHS $COOCH_3$		B	171–173	23	$(EtO)_3P$			614
		$O(CH_2)_2O$		B Z = O,S $(EtO)_3P$	290	19	349 sh, 320, 274$^{b)}$			651
SCH=CHS		$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	218 dec. (benzene)	44	344 (17 380), 318 (21 380)$^{c)}$	0.75 0.1 M TBAP$^{c)}$		744,745

CH_3 SCH=CHS		$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	210 dec.	61	346 (10 470), 318 (13 180)$^{c)}$	0.76 0.1 M TBAP$^{c)}$		742, 745
CH_3 SC=CS CH_3		$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	217 dec.	49	347 (11 480), 318 (14 125)$^{c)}$	0.76 0.1 M TBAP$^{c)}$		742, 744, 745
$CH(OC_2H_5)_2$ SC=CS $CH(OC_2H_5)_2$		$COOCH_3$	$COOCH_3$	B Z = O,S $(i\text{-}PrO)_3P$		45				66
CH0 SC=CS CHO			$COOCH_3$	$COOCH_3$		100				66
SCH=CHS		$S(CH_2)_3S$		B Z = O,S $(EtO)_3P$	231–232 dec. (benzene)	58	339 (14 125), 308 (13 490)$^{c)}$	0.78 0.1 M TBAP$^{c)}$		742
SCH=CHS		SCH_2SCH_2S		B Z = O,S $(EtO)_3P$	225–227 dec.	45	338 (13 800), 307 (13 800)$^{c)}$	0.80 0.1 M TBAP$^{c)}$		742
SCH=CHS		$Se(CH_2)_2Se$		B Z = O,S $(EtO)_3P$	284	13				743
SCH=CHS		$Se(CH_2)_3Se$		B Z = O,S $(EtO)_3P$	231	8				743
CH_3S	CH_3S	CH=CH-CH=CH						0.55 0.2 M TEAB	0.83	729
$S(CH_2)_2S$		CH=CH-CH=CH		B Z = S $(MeO)_3P$	225–226 (benzene)	14	420 (390), 331 (12 300), 306 (14 500)$^{v)}$	0.57 0.2 M TEAB	0.87	178, 729
$S(CH_2)_2S$		CH=C-CH=CH CH_3		B Z = S $(MeO)_3P$	195–197 (benzene)	2	439 (515), 331 (9 350), 306 (11 500)$^{v)}$			178
$S(CH_2)_2S$		CH_3 CH-C-C-CH CH_3		B Z = S $MeO)_3P$	243 (pyridine)	5	442 (600), 334 (12 900), 310 (14 500)$^{v)}$			178
CH_3S	CH_3S	H	$COOCH_3$	B $(EtO)_3P$			480, 418, 320, 302$^{a)}$			723
SCH_2S		H	$COOCH_3$	B $(EtO)_3P$	193	3	480, 408, 326, 303$^{a)}$			723

(*table continues*)

TABLE 11 *(continued)*

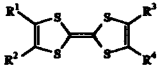


R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
$S(CH_2)_2S$		H	$COOCH_3$	B Z = O,S $(MeO)_3P$		95				285, 727
$S(CH_2)_2S$		H	$CH{=}CHCH_2OH$			92				287
$S(CH_2)_2S$		H	$CH{=}CHCH_2OCOC_5H_4FeC_5H_5$			15		0.51 0.73 0.1 M TBAHP[k]	0.96	287
$S(CH_2)_2S$		H	$CH{=}CHCOOCH_3$			70				287
$S(CH_2)_2S$		CH_2OH	$COOCH_3$	B $(MeO)_3P$		70				287, 727
$S(CH_2)_3S$		H	$COOCH_3$	B $(EtO)_3P$			482, 406, 321, 308[a]			723
CH_3S	$COOCH_3$	CH_3	CH_3		145 (MeOH)	70		0.50 TBAHP[e]	0.86	385
$NCCH_2CH_2S$	$COOCH_3$	$COOCH_3$	$COOCH_3$	B Z = O,S $(EtO)_3P$	168–169 (MeOH)	52		0.76 0.1 M TBAHP	1.08	365
CH_3S	CH_3S		COOCO		140	30	570 (832), 480 (631), 322 (11 490), 297 (12 300)[f]			712
CH_3S	CH_3S	$COOCH_3$	$COOCH_3$		88–90 (MeCN)					291
				B Z = O,S $(EtO)_3P$	83	35	434, 322, 294, 257[a]			723
$NCCH_2CH_2S$	$NCCH_2CH_2S$	$COOCH_3$	$COOCH_3$	B Z = O,S $(EtO)_3P$						173
$C_{10}H_{21}S$	$C_{10}H_{21}S$	COOH	COOH		175–177	75				731
$C_{10}H_{21}S$	$C_{10}H_{21}S$	$COOCH_3$	$COOCH_3$	B Z = O $(EtO)_3P$	oil	15				731
$C_{18}H_{37}S$	$C_{18}H_{37}S$	$COOCH_3$	$COOCH_3$	B Z = S $(EtO)_3P$	75	18	440 sh, 370 sh, 325, 295, 260[k]	0.76	1.02	735

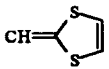
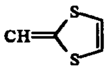
SCH_2S	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	143	39	440, 332, 306, 270[a]			723
$S(CH_2)_2S$	H	CHO		153	69				287, 383
$S(CH_2)_2S$	H	CH=CHCHO			85				287
$S(CH_2)_2S$	CHO	$CH(OC_2H_5)_2$	I M = Co_2 n = 8		13				285, 727
$S(CH_2)_2S$	CHO	CHO		210–211 dec.	68				285, 727
$O(CH_2)_2O$	$COOCH_3$	$COOCH_3$	B Z = O,S $(EtO)_3P$	122–123	37		0.58 0.1 M TBAHP[c,d]	1.03	721
			B Z = O,S $(MeO)_3P$		60				727
			B Z = S $(EtO)_3P$	94	30				722
$S(CH_2)_2S$	COOH	COOH		>150 dec.	75				712
$S(CH_2)_2S$	COOCO			210–213	95				712
$S(CH_2)_2S$	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	119	30	430, 326, 305, 298[a]			723
CH_3 SCH-CH_2S	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	110	12	435, 327, 305, 292[a]			723
CH_3 SCH-CHS CH_3	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$		9	432, 328, 305, 295[a]			723
$S(CH_2)_2S$	$COOCH_3$	CH_3OCOCH_2S	B Z = O $(EtO)_3P$	173–174 (MeCN)	20	440 (2 190), 343 sh (11 480), 315 (14 450)[a]	0.68 0.1 M TBAP[c,d]	0.98	742
$S(CH_2)_3S$	COOH	COOH		>120 dec.	70				712
$S(CH_2)_3$	COOCO			193–195	97				712
$S(CH_2)_3S$	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	165	22	430, 325, 288, 270[a]			723
SCH-CHS	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	146	8	430, 322, 296, 227[a]			723
$SeCH_2Se$	H	$COOCH_3$	B $(EtO)_3P$	210	9	417, 326, 304, 294, 220[a]			726
$SeCH_2Se$	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	152	35	436, 330, 306, 264, 233[a]			726
$Se(CH_2)_2Se$	$COOCH_3$	$COOCH_3$	B $(EtO)_3P$	113	20	433, 326, 312, 296, 245, 229[a]			726

(table continues)

TABLE 11 (*continued*)

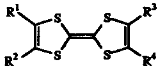


R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
Se(CH$_2$)$_3$Se		COOCH$_3$	COOCH$_3$	B (EtO)$_3$P	159	18	428, 325, 295, 232$^{a)}$			726
CH$_3$	CH$_3$	H	COOH		190–192	70	435 (2 190), 312 (13 800), 290 (13 180)$^{a)}$			746
CH$_3$	CH$_3$	H	COOCH$_3$	C	132–134 (MeCN)	21	438 (1 860), 313 (11 500), 292 (11 200)			115
H	CH$_3$	H	COOH		164					655
H	CH$_3$	H	COOCH$_3$		92–94					655
H	CH$_3$	H	COOC$_2$H$_5$		67–70 (isooctane)	35	432 (1 500), 313 (12 000), 303 (11 400), 294 (11 000)$^{a)}$	0.45 0.1 M TEAP	0.81	306
H	CH$_3$	CH$_3$	COOH		184					655
H	CH$_3$	CH$_3$	COOCH$_3$		88—94					655
H	CH$_3$	CH$_3$	COOC$_2$H$_5$		62–64 (hexane)		415 (1 300), 319 (9 600), 310 (9 200), 289 (8 900)$^{a)}$	0.43 0.1 M TEAP	0.79	306
H	CH$_3$	CH$_3$	COOCH$_2$C$_6$H$_5$		127	65				525
CH$_3$	CH$_3$	CH$_3$	COOH		172	46				655
CH$_3$	CH$_3$	CH$_3$	COOCH$_3$		142	56				655
CH$_3$	CH$_3$	CH$_3$	COOCH$_2$C$_6$H$_5$		165	65				525
H	H	Cl	Cl		103–106	4		0.70 0.01 M TBAP$^{a,k)}$	0.97	321
H	H	Br	Br		129–130	5		0.65 0.01 M TBAP$^{a,k)}$	0.95	321
					128.5–130.4					322
					126–128 (c-C$_6$H$_{12}$)	9				323
S(CH$_2$)$_2$S		H	Cl		174–176	17		0.57 0.1 M TBAP$^{a)}$	0.88	326
S(CH$_2$)$_2$S		H	Br		113–114			0.56 0.1 M TBAP$^{a)}$	0.88	326
S(CH$_2$)$_2$S		H	I		119–120	94		0.53 0.1 M TBAP$^{a)}$	0.85	326
S(CH$_2$)$_2$S		Cl	Cl		119–120	82		0.68	0.94	326

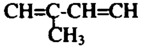
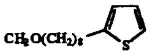
								0.1 M TBAP[e]		
$S(CH_2)_2S$		Br	Br		167–168	92		0.66 0.1 M TBAP[e]	0.93	326
$S(CH_2)_2S$		I	I		146–147	3		0.60 0.1 M TBAP[e]	0.89	326
H	Br	Br	Br		146.5–148 (hexane)	3				323
H	H	$COOC_2H_5$	$COOC_2H_5$		63 (isooctane)	33	460 (1 060), 314 (19 500), 302 (18 750), 292 (17 700)[a]	0.57 0.1 M TEAP	0.91	306
		H	H			82		0.22 0.1 M TBAP[g]	0.30	343, 662
H	CH_3	$COOCH_3$	$COOCH_3$		82–85					655
H	CH_3	$COOC_2H_5$	$COOC_2H_5$		33–34 (hexane)		444 (1 450), 316 (13 800), 293 (13 700)[a]	0.54 0.1 M TEAP	0.87	306
CH_3	CH_3	COOH	COOH		140 dec.	90				712
CH_3	CH_3	COOCO			138–140	98				712
CH_3	CH_3	$COOCH_3$	$COOCH_3$					0.55 0.1 M TBAP	0.88	581
H	COOH	$COOCH_3$	H		224–225 dec.	5	435 (2 570), 313 (13 800), 302 (12 590), 288 (11 220)[a]			171
H	COOH	$COOCH_2CH{=}CH_2$	H		196–198 dec.	21	428 (3 160), 313 (12 880), 300 (13 180), 286 (12 590)[a]			171
H	COOH	$COOCH_2C_6H_5$	H		196–198 dec.	23	442 (3 310), 312 (15 850), 299 (16 220), 286 (16 220)[a]			171
H	COOH	$COOC_4H_9$	H	*trans*	184–186 dec.	21	439 (4 070), 313 (19 950), 302 (18 200), 289 (16 600)[a]			171
H	COOH	$COOC_4H_9$	H	*cis*	192–194 dec.	9	432 (3 890), 313 (17 780), 301 (16 600), 287 (15 140)[a]			171
H	COOH	$CON(C_{18}H_{37})_2$	H		70					656
H	$COOC_4H_9$	$COOC_4H_9$	I		87–89	37	438(2 515), 311 (14 450), 298 (13 200), 278 (13 200)[a]	0.71 0.1 M TEAP	1.01	370, 581

(*table continues*)

TABLE 11 *(continued)*

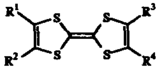


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E$_1^{Ox}$ [V]	E$_2^{Ox}$ [V]	References
								0.76 0.1 M TBAP[e]	1.06	370
H	COOC$_4$H$_9$	COOC$_2$H$_5$	COOC$_4$H$_9$			66		0.75 0.1 M TBAP[e]	1.05	370, 597
H	COOC$_4$H$_9$	COOC$_4$H$_9$	COCH$_3$		75–77	53	442 (3 550), 312 (21 400), 301 (23 450), 285 (23 450)[a]	0.76 0.1 M TBAP	1.14	370, 581
								0.81 0.1 M TBAP[e]	1.13	370
CH$_3$	CH$_3$	CN	CN		230	20				297
CH$_3$	CH$_3$	C$_6$H$_5$	C$_6$H$_5$					0.39 0.1 M TBAP	0.73	581
H	H	(CH=CH)$_2$			138–140	68	444 (100), 313 (16 200), 294 (15 800)[d]	0.40	0.75	123
				C	141	40		0.60 0.1 M TBAP[v]	0.94	113, 714
						29				159
CH$_3$	CH$_3$	(CH=CH)$_2$		C	200	30	454 (316), 319 (12 900), 292 (14 100), 260 (10 000), 253 (8 700), 247	0.50 0.1 M TBAP[v]	0.96	113, 714
								0.47 0.1 M TBAP	0.89	581
				K		89				150
CH$_3$	CH$_3$	CH=C-CH=CH CH$_3$		K		81				150
(CH$_2$)$_3$		CH=C-CH=CH CH$_3$		K		40				150
(CH$_2$)$_3$		(CH=CH)$_2$		C	197			0.53 0.1 M TBAP[v]	0.93	714
(CH$_2$)$_4$		(CH=CH)$_2$		C	208	31	456 (190), 319 (9 750), 307 (10 000), 293 (11 000)	0.53 0.1 M TBAP[v]	1.00	113, 467, 507, 517, 535,714
C$_6$H$_5$COS	C$_6$H$_5$COS	(CH=CH)$_2$		C	187 (benzene)	66				652

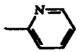
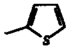
$COOCH_3$	$COOCH_3$	$(CH{=}CH)_2$		B	173–174		416 (100), 313 (17 400), 255 (19 100)$^{a)}$			123, 143
				Z = S		3				
				Z = Se		31				
 $CH{=}\underset{CH_3}{C}{-}CH{=}CH$		$(CH{=}CH)_2$		H	207–210 (pyridine/H_2O)	15				79
				K		75				150
 $CH{=}\underset{Cl}{C}{-}CH{=}CH$		$(CH{=}CH)_2$		K		98				150
 $CH{=}\underset{Cl}{C}{-}CH{=}CH$		 $CH{=}\underset{CH_3}{C}{-}CH{=}CH$		K		82				150
H	H	H	CH_3					0.33 0.1 M TEAP	0.70	306
H	H	H	CH_2OH		70–71 (isooctane/toluene)	34	452 (244), 353 (2 300), 316 (13 400), 309 (13 200)$^{a)}$	0.41 0.1 M TEAP	0.79	306
						93				532
						75				283
					70–72	92				334
H	H	H	$CHOHC_{17}H_{35}$		31–33	70				595
H	H	H	CH_2OCH_3		oil	79				334
H	H	H	$CH_2OC_{18}H_{37}$		41	71				334
H	H	H	CH_2OCOCH_3		94–95	92				334
H	H	H	CH_2OCOCF_3							283
H	H	H	$CH_2OCOC_{17}H_{35}$		56	81				334
H	H	H			oil	35		0.37* 0.1 M TBAHP	0.74*	532
H	H	H	CH_2NH_2		oil	15		0.29 0.1 M TBAHP	0.67	342
H	H	H	$CH_2NHC_4H_9$		oil	20		0.29 0.1 M TBAHP	0.68	342
H	H	H	$CH_2NHC_{18}H_{37}$		53–55	15		0.29 0.1 M TBAHP	0.67	342
H	H	H	$CH_2N(CH_3)_2$		78–80	44		0.36 0.1 M TBAHP$^{b)}$	0.87	308
								0.30 0.1 M TBAHP	0.69	342
H	H	H	$CH_2N(CH_3)_5$		110–112	38		0.34	0.68	308

(*table continues*)

TABLE 11 (*continued*)

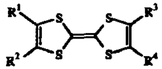


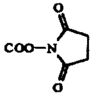
R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
								0.1 M TBAHP$^{k)}$		
								0.30	0.68	342
								0.1 M TBAHP		
H	H	H	$CH_2NHCH_2C_6H_5$		62–64	35		0.29	0.67	342
								0.1 M TBAHP		
H	H	H	$CH_2N(CH_3)CH_2C_6H_5$		68–70	35		0.34	0.87	308
								0.1 M TBAHP$^{k)}$		
								0.29	0.67	342
								0.1 M TBAHP		
H	H	H	$CH_2NHC_6H_4CH_3$ -*p*		85–97	30		0.32	0.70	342
								0.1 M TBAHP		
H	H	H	C_2H_5					0.33	0.70	306
								0.1 M TEAP		
H	H	H	$CH=CH_2$		32			0.42	0.77	305, 747
					(hexane)			0.1 M TEAP		
							467 sh (562), 391	0.42	0.65	325
							(3 470), 318 (28 200)$^{k)}$	0.1 M TBAP$^{e,i)}$		
H	H	H	$CH=CH(CH_2)_9COOH$							335
H	H	H	$CH=CHCH_2OH$			85				287
H	H	H	$CH=CHCH_2OCOC_5H_4FeC_5H_5$			20		0.46 0.73	0.88	287
								0.1 M TBAHP$^{k)}$		
H	H	H	$CH=CHCHO$		121–122	86				287, 334
H	H	H	$CH=CHCOOCH_3$		137–140	89				287, 334
H	H	H	$CH=CHCOOC_{16}H_{33}$		79–80	76				334
H	H	H	$CH=C(COOCH_3)_2$		102	10				334
H	H	H	$CH=CHC_5H_4FeC_5H_5$					0.26 0.44	0.77	333
								0.2 M TBAHP$^{e)}$		
H	H	H	$C(CH_3)=CHCOC_5H_4FeC_5H_5$		175	36				372
H	H	H	$C(CH_3)=CHCOC_6H_5Cr(CO)_3$		174	30				372
H	H	H	$C\equiv CH$		44–45	72	484 sh (339), 399	0.51	0.71	325
							(2 150), 320 (11 750)$^{k)}$	0.1 M TBAP$^{e,i)}$		
H	H	H	$C\equiv CSi(CH_3)_3$		65–66	70				325
H	H	H	C_6H_5	A	78–81		400 (2 820), 322	0.35	0.70	523, 579

					(hexane)		(12 500), 302 (14 100), 277 (13 900), 228	0.05 M TBAT		
					79.5–81	67		0.38 0.1 M TBAP[c,e]	0.80	316
H	H	H	*p*-CNC_6H_4		210.5–213 dec.	98		0.47 0.1 M TBAP[c,e]	0.87	316
H	H	H	*p*-$CH_3OC_6H_4$		160–162 dec.	33		0.38 0.1 M TBAP[c,e]	0.80	316
H	H	H	*p*-$BrCH_2CH_2C_6H_4$	A	128–133 (*c*-C_6H_{12})					523
H	H	H	*p*-CH_2=CH-C_6H_4	A	100–104 (*c*-C_6H_{12})			0.33 0.05 M TBAT	0.70	523, 579
H	H	H	*p*-HOC_6H_4		122–124 ($EtOH/H_2O$)	96				278
				A	125	94				527
H	H	H	*p*-CH_3COO-C_6H_4	A	125–127 (MeCN)	70				278
				A	134–135	21				527
H	H	H	*p*-$CH_3C(=CH_2)$-$COOC_6H_4$		85–86	70				278
H	H	H			173–175	82		0.39 0.1 M TBAP[c,e]	0.85	316
H	H	H			79–80	62		0.41 0.1 M TBAP[c,e]	0.80	316
H	H	H	COOH		182–184 (benzene)	60	420 (100), 313 (7 100), 301 (6 900)[e]	0.47 0.1 M TEAP	0.83	306
					181–185 (toluene)	90				334
					183–184 dec. (benzene/hexane)	66	431 (1 995), 313 (14 125), 302 (13 800), 288 (12 020)[a]			171
H	H	H	COCl		130–135 (hexane)	90				282
					122–125 dec.	50				595
H	H	H	$COOCH_3$		114 (hexane)	67				655
					112–113 (MeOH)	93	430 (1 995), 313 (13 800), 302 (12 880), 292 (12 020)[a]			171, 172
H	H	H	$COOCH_2CH=CH_2$		77–78	73	432 (1 950), 313 (13 180), 300 (13 180), 288 (12 880)[a]			171

(*table continues*)

TABLE 11 (*continued*)

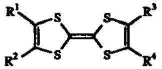


R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E$_1^{Ox}$ [V]	E$_2^{Ox}$ [V]	References
H	H	H	$COOCH_2C_6H_5$		77–79	70	430 (2 820), 313 (14 125), 299 (13 490), 290 (12 880)[a]			171
H	H	H	$COOC_2H_5$		79.5–80.5 (MeOH)	50	424 (1 920), 314 (13 600),303 (12 900), 292 sh[a]	0.47 0.1 M TEAP	0.83	306
					80–82	64				595
H	H	H			92–93	50				748
H	H	H			84–85	79				748
H	H	H	$COOC_4H_9$		74–76 (hexane)	78	435 (2 040), 315 (13 490), 304 (12 880), 289(10 970)[a]	0.51 0.1 M TBAP	0.86	171, 581, 597
H	H	H	$COOC_6H_{13}$		61–64	18				595
H	H	H	$COOC_8H_{17}$		60–62	15				595
H	H	H	$COOC_8H_{16}SH$		wax					749
H	H	H	$COOC_{11}H_{22}SH$		oil					749
H	H	H	$COOC_{12}H_{24}SH$		35–44 (hexane/EtOH)					749
H	H	H	$COOC_{16}H_{33}$		71–72 (ether/MeOH)	29		0.51	0.87[g]	307, 595, 750
H	H	H	$COOC_{16}H_{32}SH$		37–40	26		0.59 0.1 M TBAT[c,g]		749
H	H	H	$COOC_{16}H_{32}Br$		57–58	61				749
H	H	H	$COOCH_2C_6H_5$		82	65				525
H	H	H			172.5–173.5 (benzene)					751

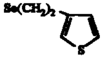
H	H	H	$CONHC_{18}H_{37}$	96–99	30					307
H	H	H	$CONH(CH_2)_2C_6H_5$	114.5–115.5 (benzene)						751
H	H	H	$CONHC_6H_5$	147–155	50					307
H	H	H	$CON(CH_3)_2$	162–163	18					276
H	H	H	$CON(CH_2)_5$	149.5–151 (benzene)						751
H	H	H	$CSOC_4H_8Cl$	95–97	58					307
H	H	H	$CSOC_{16}H_{33}$	78–80	38	539, 315, 309[k)]	0.49		0.86[g,k)]	595, 752
H	H	H	$CSNHCH_3$	188–190 (CH_2Cl_2/hexane)	58		0.43 0.01 M TBAP[g,k)]		0.98	307, 309
H	H	H	$CSNHC_{18}H_{37}$	107–111	42		0.43		1.00[g)]	307, 750
H	H	H	$CSNHC_6H_5$	169–172 (CH_2Cl_2)	30					307
H	H	H	NCO	75–78 (benzene/hexane)	80					282
H	H	H	$NHCOOC_6H_4NO_2$-*p*	154–155 (benzene/hexane)	86	371 (4 700), 350 (5 500), 310 (14 800), 272 (20 400)[e)]				282
H	H	H	CHO	110–111 (isooctane)	44	462 (2 260), 312 (12 900), 298 (13 300), 285 (13 300)[e)]	0.53 0.1 M TEAP		0.89	306
				109–110	82					334
H	H	H	$COCH_3$	152–153 (isooctane)	67	455 (1 940), 312 (10 200), 300 (9 800), 285 (9 600)[e)]	0.47 0.1 M TEAP		0.83	306
H	H	H	$COC_5H_{10}Br$							307
H	H	H	$COC_{13}H_{27}$	80–81	25					307
H	H	H	$COC_{15}H_{31}$	81 (ether/MeOH)	20		0.52		0.90[g)]	595, 753
H	H	H	$COC_{17}H_{35}$	85	20					595, 754
H	H	H	$CO(CH_2)_3Br$	205 dec.	30					595
H	H	H	$CO(CH_2)_5Br$	114	27					595
H	H	H	$CO(CF_2)_6CF_3$	155–158	10					595
H	H	H	$COC_5H_4FeC_5H_5$		12		0.38 0.2 M TBAHP[g)]	0.73	0.88	333
H	H	H	$COCH{=}C(CH_3)C_5H_4FeC_5H_5$	75	7					372
H	H	H	$COCH{=}C(CH_3)C_6H_5Cr(CO)_3$	204	18					372
H	H	H	$COCH{=}C(CH_3)C_6H_5$	150	6					372
H	H	H	$CSC_{17}H_{35}$ 71–76	50						595
H	H	H	SCH_3 oil	96			0.42		0.80	331

(*table continues*)

TABLE 11 (*continued*)

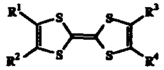
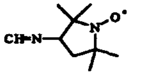
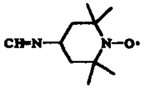
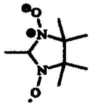
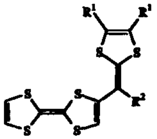


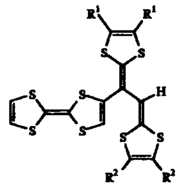
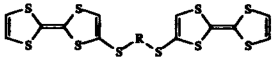
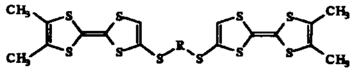
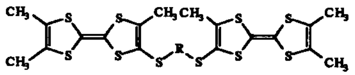
R^1	R^2	R^3	R^4	preparative method	m. p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
					$(CH_2Cl_2/c\text{-}C_6H_{12})$			0.1 M TBAHP$^{g,k)}$		
H	H	H	$SCH_2O(CH_2)_2Si(CH_3)_3$		oil	48				332, 363
H	H	H	$SCH_2CH_2N_3$		oil	92				332
H	H	H	$SCH_2CH_2NH_2$		semi-solid	76				332
H	H	H	$SCH_2CH_2N\ HCOCH_3$		54–55	81				332
H	H	H	SCH_2CH_2OH		101–102	75		0.41	0.81	331,332
					$(CH_2Cl_2/c\text{-}C_6H_{12})$			0.1 M TBAHP$^{g,k)}$		
H	H	H	$SCH_2CH_2OC_{18}H_{37}$		oil	79		0.49	0.87$^{g)}$	332, 750
H	H	H	$SCH_2CH_2OCOCH{=}CH_2$		oil	82		0.43	0.83	330, 332
								0.1 M TBAHP$^{g,k)}$		
H	H	H	$SCH_2CH_2OCO\underset{CH_3}{C}{=}CH_2$		oil	83		0.43	0.89	330, 332
									0.1 M TBAHP$^{g,k)}$	
H	H	H	$SCH_2CH_2OCOC_{15}H_{31}$		wax	88		0.48	0.90$^{g)}$	332, 750
H	H	H	$S(CH_2)_2OCOC_5H_4FeC_5H_5$			80		0.35 0.61	0.75	333
								0.2 M TBAHP$^{g)}$		
H	H	H	$SCH_2CH_2\ OCONHC\ H_2CH_2Cl$		74–77	63				332
H	H	H	$SCH_2CH_2O\ CONHC_6H_5$		86–88	56				332
H	H	H	$SCH_2CH_2OSO_2CH_3$		oil	95				332
H	H	H	SCH_2CH_2Cl		oil	86				332
H	H	H	$SCH_2CH_2SCH_3$		oil	81				332
H	H	H	$SCH_2CH_2SC_6H_5$		71–73	76				332
H	H	H	$S(CH_2)_3I$		oil	50		0.46	0.86	331
								0.1 M TBAHP$^{g,k)}$		
H	H	H	SCH_2CHCH_2 \ / 'O'		oil	38				332
H	H	H	$S(CH_2)_4I$		oil	43		0.44	0.81	331
								0.1 M TBAHP$^{g,k)}$		
H	H	H	$SC_{18}H_{37}$		52	15		0.50	0.88$^{g)}$	332, 595, 755
H	H	H	$SCH{=}CH_2$		oil	87		0.45	(0.92)	330, 332
								0.1 M TBAHP$^{g,k)}$		
H	H	H	$SCOC_{15}H_{31}$		79–83	36		0.52	0.90$^{g)}$	750

H	H	H	$SCOC_6H_5$	126–127 (CH_2Cl_2/c-C_6H_{12})	78		0.45 0.1 M TBAHP[g,k]	0.81	331
H	H	H	$SCOC_5H_4FeC_5H_5$		27		0.45 0.2 M TBAHP[g]	0.73	333
H	H	H	$SeCH_3$	oil	84				332
H	H	H	$SeCH_2O(CH_2)_2Si(CH_3)_3$	oil	57				332, 363
H	H	H	$SeCH_2CH_2OH$	oil	53				332
H	H	H			10				748
H	H	H	$SeC_{18}H_{37}$	52	15				595, 755
H	H	H	SeC_6H_5	98					327
				78–79 (MeCN)	67				332
H	H	H	$TeC_{18}H_{37}$	50	10				595, 755
H	H	H	TeC_6H_5	100.6–101.1 (CS_2/petroleum ether)	38		0.42 0.1 M TBAT[c,g]	0.84	653
H	H	H	$Si(CH_3)_3$	oil	26				595
H	H	H	$Si(CH_3)_2C_{18}H_{37}$	39–42	27				595
H	H	H	$P(C_6H_5)_2$	41–43	51	388 (2 140), 312 (12 600), 254 (14 500)[k]	0.38 0.1 M TBAHP	0.74	312
H	H	H	Cl	57–58	48		0.56 0.01 M TBAP[g,k]	0.78	321
				65 (hexane)	11				323
H	H	H	Br	40–45 dec.	38		0.55 0.01 M TBAP[g,k]	0.76	321, 322
				52 (hexane)	34				323
H	H	H	I	66–68 (hexane)	9	480 (302), 368 sh (1 590), 314 (10 700), 292 (9 330)[e]	0.45 0.1 M TBAP	0.83	370, 581
				110–112	34		0.55 0.01 M TBAP[g,k]	0.80	321
							0.50 0.1 M TBAP[g]	0.88	370
				68.8–69.3 (light petroleum)	70		0.60 0.1 M TBAT[c,g]	1.00	324
				65–66	78				325

(*table continues*)

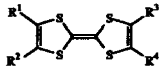
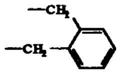
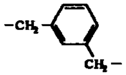
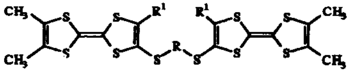
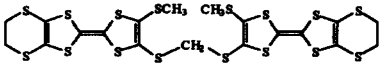
TABLE 11 (*continued*)

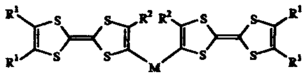
 R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{ox}	[V]	E_2^{ox}	References
H	H	H			75		g = 2,0067	0.56 0.1 M TBAT$^{s)}$	0.90	0.92	341
H	H	H			66		g = 2,0071	0.57 0.1 M TBAT$^{s)}$	0.88	0.92	340, 341
H	H	H					g = 2,0063	0.32		0.77$^{g,h)}$	339
											
R¹		R²									
H		H									336
CH_3		H									336
CH_3S		H									336
$S(CH_2)_2S$		H			174–176	83					336
H		CH_3									336
CH_3		CH_3									336
CH_3S		CH_3									336
$S(CH_2)_2S$		CH_3									336
CH_3S		CHO									336
$S(CH_2)_2S$		CHO			234–237	30					336

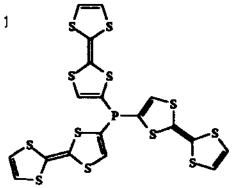
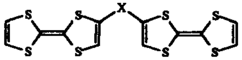
							
R¹	R²						
S(CH$_2$)$_2$S	|S(CH$_2$)$_2$S|						336
CH$_3$S	|S(CH$_2$)$_2$S|			0.18 0.1 M TBAHP[k]	0.40	(1.18)	336
S(CH$_2$)$_2$S	CH$_3$S	102–105	60	0.19 0.1 M TBAHP[k]	0.41	(1.22)	336
							
R: (CH$_2$)$_3$		oil (hexane)	26	0.44 0.1 M TBAHP[e,k]		0.78	331
R: (CH$_2$)$_4$		124–126 (hexane)	11	0.44 0.1 M TBAHP[e,k]		0.81	331
							
R: (CH$_2$)$_2$		116 (benzene/hexane)	78	0.53 TBAHP[e]		0.87	385
R: (CH$_2$)$_3$		112–113 (benzene/hexane)	81	0.46 TBAHP[e]		0.78	385
R: (CH$_2$)$_4$		115–116 (benzene/hexane)	81	0.44 TBAHP[e]		0.78	385
							
R: CH$_2$		250 (hexane)	24	0.22 0.1 M TBAHP[k]	0.36	0.80	96
R: (CH$_2$)$_2$		239 (hexane)	15	0.23 0.1 M TBAHP[k]	0.36	0.74	96
R: (CH$_2$)$_3$		196	33	0.28		0.75	96

(*table continues*)

TABLE 11 *(continued)*

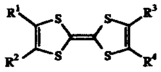
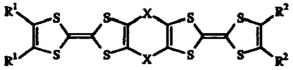
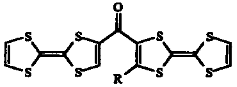
 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
					(hexane)			0.1 M TBAHP[k]		
R: $(CH_2)_{10}$					110 (hexane)	50		0.27 0.1 M TBAHP[k]	0.77	96
R: 					229 (hexane)	23		0.29 0.1 M TBAHP[k]	0.79	96
R: 					181 (hexane)	31		0.26 0.1 M TBAHP[k]	0.75	96
R: 					253 (hexane)	35		0.28 0.1 M TBAHP[k]	0.76	96
										
R		R^1								
$(CH_2)_2$		$COOCH_3$			183 (pyridine/hexane)	61	434 (3 980), 333 sh (21 900), 312 (28 840), 286 sh (25 100)[l]	0.60 TBAHP[e]	0.94	385
$(CH_2)_3$		$COOCH_3$			180–181 (pyridine/hexane)	68	434 (3 980), 333 sh (22 400), 309 (28 840), 280 sh (25 100)[l]	0.40 0.56 TBAHP[e]	0.94	385
$(CH_2)_4$		$COOCH_3$			183–184 (pyridine/hexane)	75	431 (4 680), 333 sh (25 700), 309 (35 500), 280 sh (28 840)[l]	0.56 TBAHP[e]	0.91	385
					170–173 dec. (benzene/MeOH 3:1, v:v)			0.47 0.55 0.1 M TBAP[e]	(0.81)	756

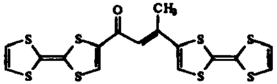
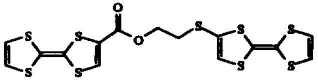
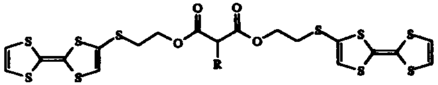
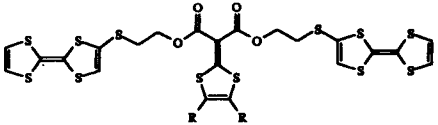
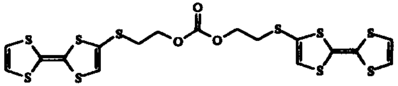
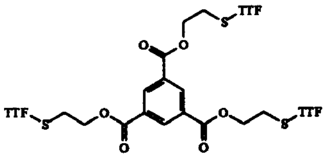


R¹	R²	M					
H	H	Hg	214	80		0.35 0.68 0.05 M TBAHP[q]	311
CH_3	H	Hg	210–213 (DMF)	85		0.27 0.66 0.05 M TBAHP[q]	311
CH_3	CH_3	Hg	236 (DMF)			0.28 0.63 0.05 M TBAHP[q]	311
H	H	$Si(CH_3)_2$	142–144 (c-C_6H_{12})	71		0.29 0.38 0.77 0.05 M TBAHP[k]	311
CH_3	H	$Si(CH_3)_2$	210–212 (MeCN)	25		0.22 0.32 0.82 0.05 M TBAHP[k]	311
CH_3	CH_3	$Si(CH_3)_2$	204–210 (MeCN)			0.24 0.34 0.88 0.05 M TBAHP[k]	311
H	H	$P(C_6H_5)$	70–73	34	390 (3 150), 316 (19 700), 250 sh (15 730)[k]	0.37 0.47 0.84 0.1 M TBAHP	312
CH_3	H	$P(C_6H_5)$	197–199 (toluene)	60		0.28 0.38 0.88 0.05 M TBAHP[k]	311
CH_3	CH_3	$P(C_6H_5)$	216–220 (toluene)	68		0.23 0.36 0.76 0.87 0.05 M TBAHP[k]	311
1 			178–180 (toluene)	40	402 (5 750), 316 (35 800)[k]	0.47 0.86 0.1 M TBAHP 0.35 0.47 0.54 0.86 0.1 M TBAHP[k] 0.28 0.52 TBAHP[q]	312 312 310
							
X: S			144–147 (CS_2/hexane)	14		0.49 0.61 (0.86) 0.1 M TEAHP[g,k]	329
X: Se			161–164	9		0.49 0.61 0.1 M TEAHP[g,k]	329
X: Te			176	20		0.31 0.65 (0.8) 0.1 M TBAP[a/c,g]	314

(*table continues*)

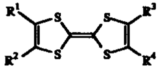
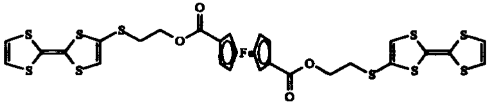
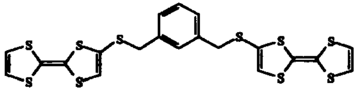
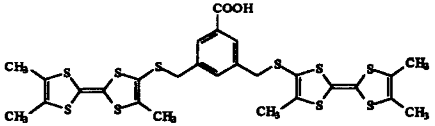
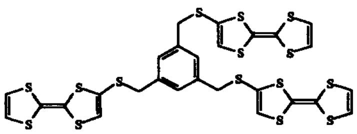
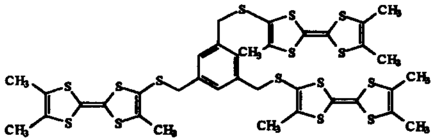
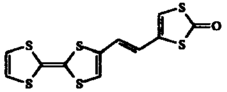
TABLE 11 (*continued*)

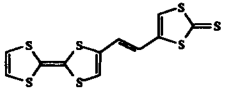
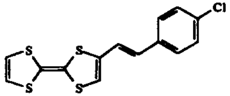
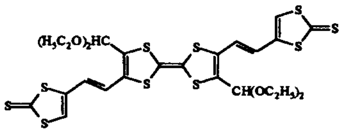
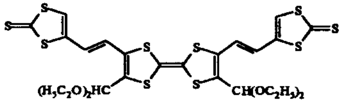
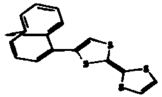
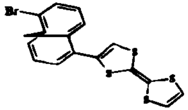
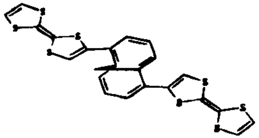
 R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} E_2^{Ox} [V]	References
								0.38 0.72 (0.80) 0.1 M TBAP$^{a/c,g)}$	757
X: (Te)$_2$					28				349
									
R¹	R²	X							
H	H	S		B Z = O,S (MeO)$_3$P	245–246	51		0.47 0.67 0.89 0.1 M TBAP$^{c,g)}$	757
H	CH$_3$	S		B Z = O,S (MeO)$_3$P	247–248	55		0.43 0.64 0.88 0.1 M TBAP$^{c,g)}$	757
H	CH$_3$S	S		B Z = O,S (MeO)$_3$P	240–241	54		0.49 0.75 0.89 0.1 M TBAP$^{c,g)}$	757
CH$_3$	CH$_3$S	S		B Z = O,S (MeO)$_3$P	233–234	50		0.47 0.76 0.87 0.1 M TBAP$^{c,g)}$	757
CH$_3$	CH$_3$	Te			blacken at 250 °C (acetophenone)	63		0.48 0.83 0.1 M TBAP$^{c,g)}$	319, 320
CH$_3$S	CH$_3$S	Te			blacken at 270 °C	26		0.57 0.84 0.1 M TBAP$^{c,g)}$	320
									
R: H						9		0.58 0.67 1.01 1.05$^{e)}$	758
R: COOCH$_3$					220–223	23		0.63 0.71 1.02 1.02$^{e)}$	758

R: $COOC_2H_5$	159–160		0.52 0.60 0.92 0.1 M TEAP	306
R: $CON(CH_3)_2$	214–218	7	0.62 0.70 0.90 0.98$^{e)}$	758
	130	12		372
	91–93 (ether/MeOH)	34	0.45 0.56 0.79 0.1 M TBAHP^{g,k}	331
				
R: H	oil (ether/MeOH)	84	0.47 0.78 0.1 M TBAHP$^{g,k)}$	331
R: $C_{18}H_{37}$	oil (ether/MeOH)	62	0.47 0.79 0.1 M TBAHP$^{g,k)}$	331
				
R: CH_3	138–139 (CH_2Cl_2/c-C_6H_{12})	91	0.44 0.81 (1.45) 0.1 M TBAH$^{g,k)}$	331
R: $S(CH_2)_2S$	67–68 (CH_2Cl_2/c-C_6H_{12})	88	0.43 0.81 (1.26) 0.1 M TBAHP$^{g,k)}$	331
	oil	78	0.46 0.81 0.1 M TBAHP$^{g,k)}$	331
	60–61 (ether/MeOH)	72	0.45 0.78 0.1 M TBAHP$^{g,k)}$	331

(*table continues*)

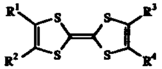
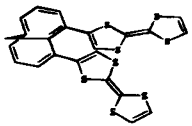
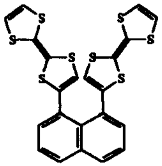
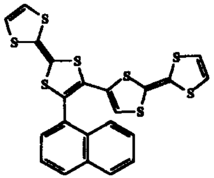
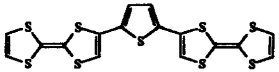
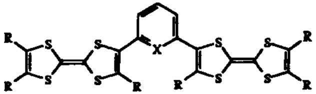
TABLE 11 (*continued*)

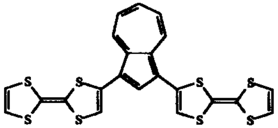
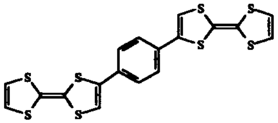
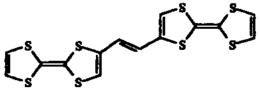
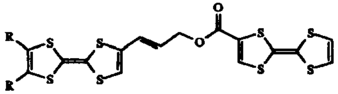
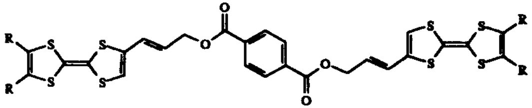
 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	[V]	E_2^{Ox}	References
						20		0.37 0.1 M TBAHP[g,k]	0.71	0.85	333
					73–75 (ether/MeOH)	10		0.45 0.1 M TBAHP[g,k]		0.79	331
											759
					49–52 (ether/MeOH)	17		0.47 0.1 M TBAHP[g,k]		0.81	331
					228–229	23		0.40 0.1 M TBAHP[k]		0.83	236
						65					760

	*trans*	210 dec.	83	0.67 0.1 M TBAP[c)]	0.96	283, 760
						283
			95			760
			95			760
			33	0.39 0.1 M TBAP[c)]	0.78	318
			11	0.40 0.1 M TBAP[c)]	0.73	318
			19	0.40 0.1 M TBAP[c)]	0.79	318

(*table continues*)

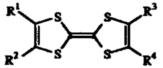
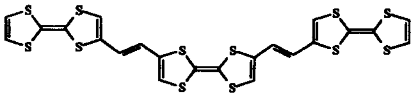
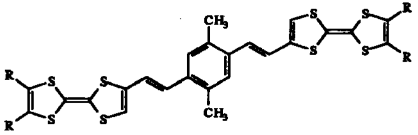
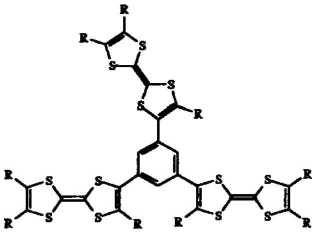
TABLE 11 (*continued*)

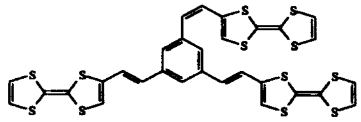
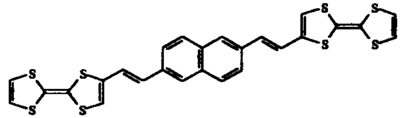
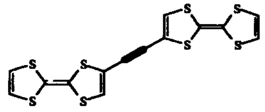
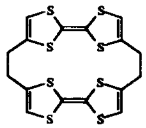
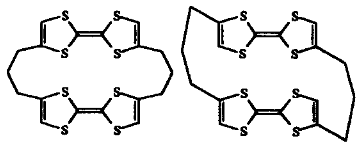
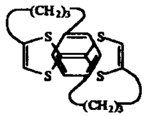
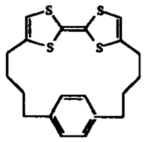
 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox}	References
						21		0.29 0.1 M TBAP[e]	0.89	318
						14		0.30 0.1 M TBAP[e]	0.91	318
						5				318
					212–214 dec.	52		0.38 0.1 M TBAP[e,g]	0.81	316
										
R: H	X: CH				196–197	77		0.40 0.1 M TBAP[e]	0.81	317
R: H	X: N				214–215	72		0.29	0.87	317

					0.1 M TBAP[e]		
R: CH_3S X: CH		141–142	54		0.53 0.1 M TBAP[e]	0.79	317
R: C_2H_5S X: CH		oil	55		0.54 0.1 M TBAP[e]	0.82	317
R: C_4H_9S X: CH		oil	63		0.54 0.1 M TBAP[e]	0.79	317
R: C_2H_5S X: N		120–121	48		0.54 0.1 M TBAP[e]	0.82	317
		245 dec.	74		0.37 0.1 M TBAP[e]	0.75	317
	A	242	21	440(4 020), 320(21 600), 310 (19 800)[i]	0.35 0.1 M TBAP	0.76	579, 761
		237–239 dec.	61		0.35 0.1 M TBAP[e,g]	0.82	316
		135 dec.	56	450(8 700), 310 (28 200)[k]	0.41 0.1 M TBAP[g,q]	0.71	325
							
R: H			25		0.43 0.53 0.69 0.89 0.1 M TBAHP[k]		287
R: $S(CH_2)_2S$			50		0.51 0.1 M TBAHP[k]	0.93	287
							
R: H			20				287
R: $S(CH_2)_2S$			46		0.49 0.1 M TBAHP[k]	0.90	287

(*table continues*)

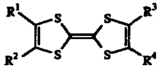
TABLE 11 (*continued*)

 R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} [V]	E_2^{Ox} [V]	References
										283
										
R: H					257 dec. (DMF)			0.51 0.05 M TBAT$^{z,q)}$	0.80	337, 383
								(0.44) (0.69) 0.05 M TBAT$^{c,e)}$	(0.92)	337, 383
R: $S(CH_2)_2S$					207 dec. (DMF)		440(24 000), 359(43 000)$^{q)}$	0.61 0.05 M TBAT$^{z,q)}$	0.86	383
								(0.52) (0.66) 0.05 M TBAT$^{c,e)}$	(0.99)	383
										
R: H					235–240 dec.	72		0.29 0.1 M TBAP$^{e)}$	0.77	317
R: CH_3S						49				317
R: C_2H_5S					128–129	53		0.54 0.1 M TBAP$^{e)}$	0.81	317

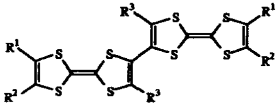
							236
			25				762
		92 dec.	22	445 (7 250), 310 (33 900)[k]	0.56 0.1 M TBAP[g,q]	0.78	325
	A	>210	15				763
	A		9				763
	A	180–182 (c-C_6H_{12})					764
	A	157–158	63				764

(*table continues*)

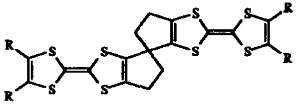
TABLE 11 (*continued*)



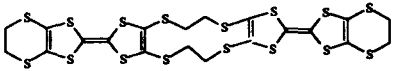
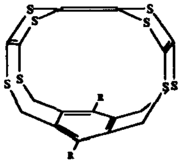
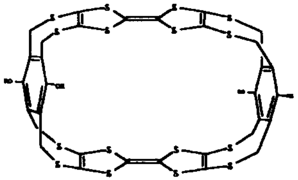
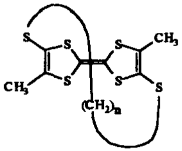
R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	[V]	E_2^{Ox}	References



R^1		R^2	R^3								
H		H	H		158–160 dec.		410(7 250), 320(22 400), 308(21 400)a)	0.45 0.1 M TBAPg)	0.65	0.97	370
					225–226 dec.	62		0.43 0.1 M TBAPe,g)		0.84	316
H		COOH	COOH		>250 dec.						370
H		$COOCH_3$	$COOCH_3$		227–228 (benzene)		442(3 710), 313 (19 050), 302(18 200), 291(16 200)a)	0.85 0.1 M TBAPg)		1.13	370
H		$COOC_4H_9$	$COOC_4H_9$			25	432 (6 025), 315 (38 000), 303 (36 300), 289 (33 900)a)	0.83 0.1 M TBAPg)		1.12	370
CH_3S		CH_3S	H	B Z = O,S $(EtO)_3P$	172–173 $(CHCl_3)$	15		0.50 TEAPk)	0.59	0.85	765

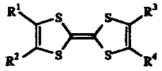
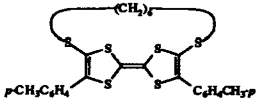
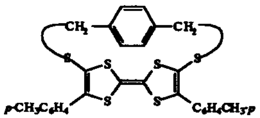
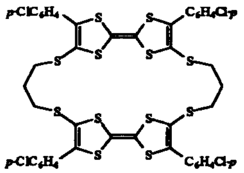
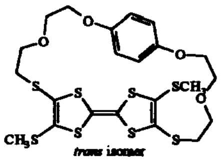


R: H				A	>200 dec. $(CHCl_3)$	18		0.41 0.66 TEAPk)		0.50 0.78	715
R: $S(CH_2)_2S$				A	>225 dec.	22		0.46		0.55	715

			(PhCl)			0.78 TEAP[k]		
		B Z = O (EtO)$_3$P	239–242 dec. (CCl$_4$/CS$_2$)			0.59 TBAP[e]		(0.84)	387
	R: OC$_3$H$_7$	B Z = S (EtO)$_3$P	227	38	393 sh (3 200), 311 (8 600), 228 (39 700)[k]	(0.93) TBAHP[k]			643, 644, 685
	R: OC$_4$H$_9$	B Z = S (EtO)$_3$P	236	34					643
	R: OC$_6$H$_{13}$	B Z = S (EtO)$_3$P	205 dec. (EtOAc)	37	333 (3 473), 312 (2 297)[k]				643, 644, 685
		A		61					643
	R: OCOC$_5$H$_{11}$	B Z = O (EtO)$_3$P	186 dec.	59					643
	R: OC$_3$H$_7$			53		0.36 0.1 M TBAHP[k]	1.01	1.06	388
	R: OC$_4$H$_9$			44					388
	R: OC$_6$H$_{11}$			47	422 (2 632), 330 (22 578), 307 (17 807), 227 (47 129)[k]				388
									

(table continues)

TABLE 11 (*continued*)

 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox} E_2^{Ox} [V]	References
n: 10				*cis*				0.40 0.1 M TBAHP[k)]	166
n: 10				*trans*	158–159	55		0.47 0.1 M TBAHP[k)]	166
n: 12				*trans*	115–116	70			166
						72		(0.46) 0.1 M TBAT[g,k)]	165
						91		(0.46) 0.1 M TBAT[g,k)]	165
						81		0.18 0.45 0.1 M TBAT[g,k)]	165
					99–101			0.46 0.74 1.01 0.1 M TBAHP[e)]	173

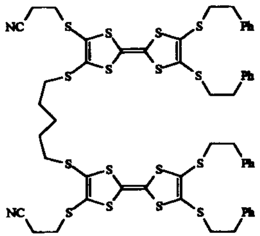
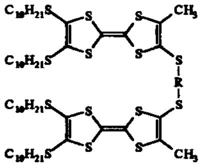
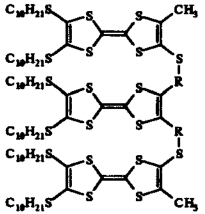
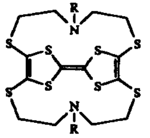
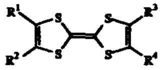
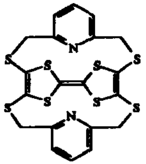
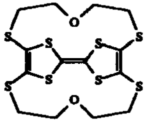
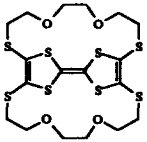
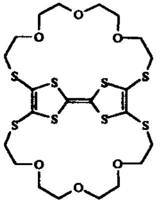
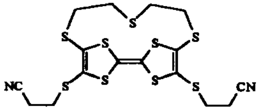
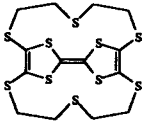
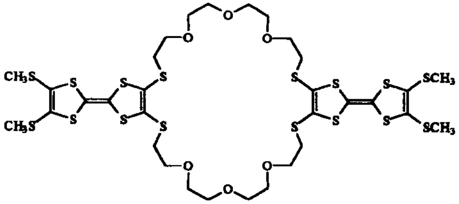
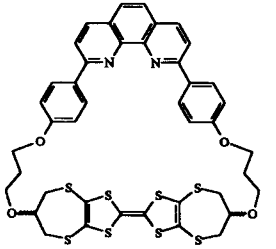
							173
							
R: $(CH_2)_3CONH(CH_2)_4$			62–64 (MeCN)	40			731
R: $(CH_2)_3CONH(CH_2)_3NHCO(CH_2)_3$			95–97	92			731
							
R: $CONH(CH_2)_4$			129–132	13			731
R: $SCH_2CONH(CH_2)_4$			64–66	59			731
	R: CH_3	B Z = S $(EtO)_3P$	248–249 ($CHCl_3$/MeOH)	31	344 (11 750), 265 (18 200), 241 (14 800)[m]	(0.95) TBAHP[k]	766

TABLE 11 (*continued*)

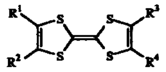
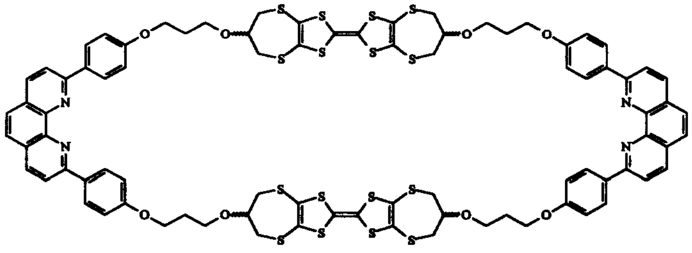
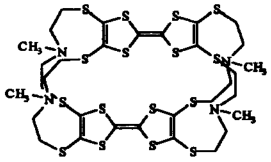
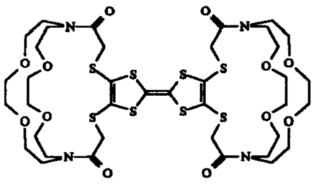


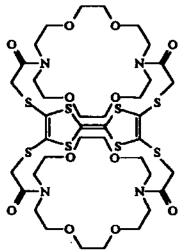
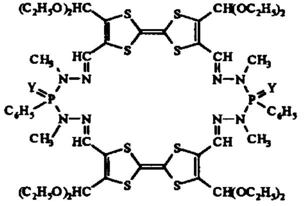
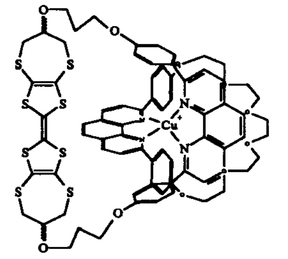
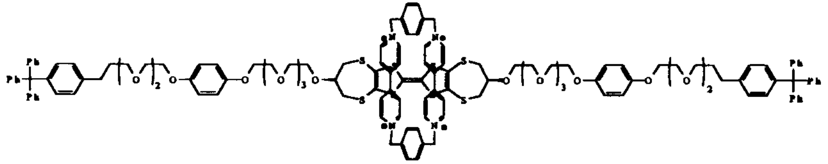
R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{ox} [V]	E_2^{ox}	References
				B Z = S $(EtO)_3P$	237–238 dec. ($CHCl_3$/MeOH)	22		0.95 TBAHP		767
		R: C_2H_5		B Z = S $(EtO)_3P$	203–204 (ether/$CHCl_3$)	23				767
				B Z = S $(MeO)_3P$	290–292 dec. (CH_2Cl_2)	22	340 (7 080), 262 (16 600), 240 (16 200)[w]	0.27 0.1 M TBAP[t,v]	0.57	768
				B Z = S $(EtO)_3P$	290–292	20		0.27 0.1 M TBAHP		767
				B Z = S $(EtO)_3P$	>270 dec. (CH_2Cl_2/MeOH)	25		(1.26) 0.1 M TBAHP		767, 769
				B Z = S $(EtO)_3P$	247 (EtOH/$CHCl_3$)	22	338(1 445), 304 (537), 259 (1 410)[w]	0.41 0.1 M TBAP[t,v]	0.73	638
								0.41 0.1 M TBAHP		767, 769

	B Z = S $(EtO)_3P$		1	770
	B Z = O $(EtO)_3P$	172–176	30	364
	B Z = S $(EtO)_3P$	130–132 dec.	20	640
	B Z = S $(EtO)_3P$	109–111 (MeCN)	8	602
			13	771

(*table continues*)

TABLE 11 (*continued*)

 R¹	R²	R³	R⁴	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{ox} [V]	E_2^{ox}	References
						29				771
				B Z = S $(EtO)_3P$		1				769
				B Z = S $(EtO)_3P$	230–232 dec.	19		0.50* 0.1 M TBAHP[c)]	0.81*	769, 772

	B Z = S $(EtO)_3P$	210 dec.	22	0.52* 0.1 M TBAHP[c]	0.76*	769, 772
	Y = O Y = S		80 60	0.61* 0.1 M TBAP[v]	1.05*	660
			14			771
			8			375

(*table continues*)

TABLE 11 (*continued*)

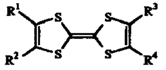
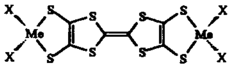
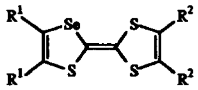
 R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield (%)	λ_{max} (ε) [nm]	E_1^{Ox}	E_2^{Ox} [V]	References
										
Me	X									
Ti	C_5H_5				129–132	30	760 (7 500), 454 (4 100), 319 (22 300), 225 (53 800)[c]	0.50 0.2 M TBAHP[N]	0.85	362
							788, 470[l]			362
Ti	$C_5(CH_3)_5$					20	920, 470, 319, 225[l]	0.35 0.2 M TBAHP[N]	0.70	362
Pt	$[P(C_6H_5)_3]_2$						720, 450, 340, 280, 250[c]			362

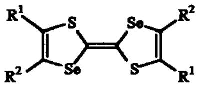
TABLE 12 Molecular properties of Monoselenatrithiafulvalenes (redox potential in acetonitrile vs. sce)



R^1	R^2	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2 [V]	References
$S(CH_2)_2S$	H	O,S $(EtO)_3P$	195	4.5	360 sh, 330 sh, 298, 218			773
$S(CH_2)_2S$	$CH_2O(CH_2)_2Si(CH_3)_3$	O,S $(EtO)_3P$						774
$S(CH_2)_2S$	$O(CH_2)_2O$	O $(EtO)_3P$	196	4.5	480, 313, 260 sh			773
$S(CH_2)_2S$	$S(CH_2)_2S$		250					774
$COOCH_3$	SCOS	O,S $(EtO)_3P$	204 dec.					291

(*table continues*)

TABLE 12 Molecular properties of Diselenadithiafulvalenes (vedox potential in acetonituile vs. sce)

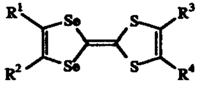


R^1	R^2	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H		119	84	470 (200), 325 (5 900), 297 (9 800), 282 (9 100), 235 sh (3 700), 235 (8 900)[b)]	0.40 0.1 M TEAP	0.72	106, 184, 185, 191, 377
		Se	117 (hexane)	70				106, 184
CH_3	H	Se	123–125 (hexane)	60	490 (200), 370 (2 500), 295 (21 000), 240 (6 200)[b)]			108
CH_3	CH_3	Se			495 (172), 327 (953), 298 (13 980)	0.33 TBAT	0.68	186
$(CH_2)_3$		Se $(EtO)_3P$	244 dec.	37	472 (257), 433 (204), 347 (3 100), 334 (5 900), 305 (10 700)[a)]			187
CH_2-$\underset{CH_3}{CH}$-CH_2		S $(MeO)_3P$	160–163 (hexane)	5	475 (159), 340 sh, 310 (14 100)[a)]	0.39 0.1 M TEAP	0.70	46
		Se $(MeO)_3P$						46
$(CH_2)_4$		S $(EtO)_3P$	sublim.	11		0.28 TEAP	0.63	190
C_6H_5	H	S	202–208 (MeCN)	10	505 sh (350), 400 (3 470), 323 sh (13 800), 294 (22 400), 269 (24 550), 241 (19 950)[c)]			145
				40				145
$(CH{=}CH)_2$		S $(EtO)_3P$	247–248	4	448 (230), 315 (9 800), 284 (15 100), 254 (19 000)[a)]			187
		Se $(EtO)_3P$		39				150
$S(CH_2)_2S$		O $(EtO)_3P$	250	5	440, 336 sh, 310, 217[a)]			773

H	$COOCH_3$		254–255 (benzene/MeOH)	85		654
H	COOH			70		654
$COOCH_3$	$COOCH_3$	Se Ph_3P	138 (MeOH)	50	434 (2 750), 325 (10 700), 290 (26 900), 250 (24 500)	185, 191
				89		654
$CONH_2$	$CONH_2$			80		654
COOH	COOH		>280			185, 191, 215

(*table continues*)

TABLE 12 (*continued*)

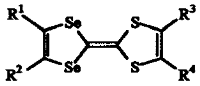
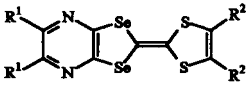


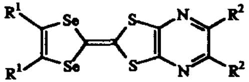
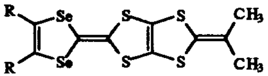
R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
CH_3	CH_3	CH_3	CH_3			25				180, 182
CH_3	CH_3	$(CH_2)_4$				10				182
$(CH_2)_4$		CH_3	CH_3			20				182
$(CH_2)_4$		$(CH_2)_4$				8				180
H	H	SCH_2S			158					775
H	H	$O(CH_2)_2O$		B Z = O $(EtO)_3P$	>90	<1	492, 302, 248$^{a)}$			300
H	H	$S(CH_2)_2S$			207					775
H	H	$Se(CH_2)_2Se$		B Z = O $(EtO)_3P$	210	20	486, 360 sh, 297–320 sh, 240 sh$^{a)}$			300
CH_3	CH_3	SCH_2			176–177					109
CH_3	CH_3	$O(CH_2)_2O$		B Z = O $(EtO)_3P$	168	<1				694
				B Z = Se $(EtO)_3P$	240	25		0.42 0.1 M TBAP	0.70	722
CH_2OH	CH_2OH	H	H		172	80				776
CH_2OH	CH_2OH	$O(CH_2)_2O$			150	80				776
$CH(OC_2H_5)_2$	$CH(OC_2H_5)_2$	H	H	B Z = Se $(EtO)_3P$		35				776
$CH(OC_2H_5)_2$	$CH(OC_2H_5)_2$	$O(CH_2)_2O$		B Z = Se $(EtO)_3P$		15				776
CHO	CHO	H	H		203	15				776
CHO	CHO	$O(CH_2)_2O$			149	65				776
CH_3	CH_3	SCH_2Br	$SCH_2O(CH_2)_2Si(CH_3)_3$							109

CH_3		CH_3	$SCH_2O(CH_2)_2Si(CH_3)_3$	$SCH_2O(CH_2)_2Si(CH_3)_3$							109
CH_3		CH_3	$S(CH_2)_2S$		B Z = O,Se $(MeO)_3P$	198		490 (645), 340 sh (6 020), 313 (15 130), 208 (19 500)[a]	0.52 0.1 M TBAP	0.78	179
					B Z = S,Se $(MeO)_3P$	204–205 (pyridine)	5	485 (575), 397 (2 190), 346 (6 910), 307 (12 000)[v]			178
							43				183
	$(CH_2)_3$		$S(CH_2)_2S$		B Z = O,Se $(EtO)_3P$	214	27	480 (302), 355 sh (6 310), 315 (14 120), 209 (19 500)[a]	0.48 0.1 M TBAP	0.73	179, 777
							37				184
CH_3		CH_3	$S(CH_2)_3S$		B Z = O,Se $(MeO)_3P$	232		380 (1 480), 336 (7 580), 300 (14 800), 212 (19 950)[a]	0.48 0.1 M TBAP	0.75	179
		$(CH_2)_3$	$S(CH_2)_3S$		B Z = O,Se $(MeO)_3P$	234		378 (1 480), 332 (7 750), 303 (15 850), 208 (21 400)[a]	0.44 0.1 M TBAP	0.74	179
	$Se(CH_2)_2Se$		H	$COOCH_3$	B Z = O,S $(EtO)_3P$						300
	$Se(CH_2)_2Se$		$COOCH_3$	$COOCH_3$	B Z = O,S $(EtO)_3P$						300
H			$COOCH_3$	$O(CH_2)_2O$	B Z = O,Se $(EtO)_3P$						300
$COOCH_3$			$COOCH_3$	$O(CH_2)_2O$	B Z = O,Se $(EtO)_3P$						300
	$O(CH_2)_2O$			$S(CH_2)_2S$	B Z = O $(EtO)_3P$	186	3				694
	$O(CH_2)_2O$			$Se(CH_2)_2Se$	B Z = O $(EtO)_3$	183	<1				694

(*table continues*)

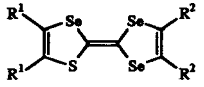
TABLE 12 (*continued*)

R^1	R^2	R^3	R^4	preparative method	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
										
$S(CH_2)_2S$		SCH_2S		B Z = O $(EtO)_3P$		6.8		0.18 0.1 M $TBAP^{a,b)}$	0.41	778
$S(CH_2)_2S$		$S(CH_2)_2S$								779
$S(CH_2)_2S$		SCH_2OCH_2S								779
										
R^1		R^2								
H		H		B $(EtO)_3P$	174		$418^{a)}$			693
H		CH_3		B $(EtO)_3P$	180		$418^{a)}$			693
H		$COOCH_3$		B Z = O,S $(EtO)_3P$						693
H		$O(CH_2)_2O$		B Z = O $(EtO)_3P$	210	5.5	410, 298–314 sh, 244 sh, $218^{a)}$			300
H		SCH_2S		B Z = O,S $(EtO)_3P$	218	7	$400^{a)}$	0.69 0.1 M TEAP	0.96	693, 698
H		$S(CH_2)_2S$		B Z = O,S $(EtO)_3P$	229	25	$405^{a)}$	0.74 0.1 M TEAP	1.03	693, 698
CH_3		SCH_2S		B Z = O,S $(EtO)_3P$	218	10	$380^{a)}$	0.66 0.1 M TEAP	0.92	693, 698

CH_3	$S(CH_2)_2S$	B Z = O,S $(EtO)_3P$	238	18	386[a]	0.59 0.1 M TEAP		0.99	693, 698
									
R^1	R^2								
CH_3	H	B Z = O,S or Se $(EtO)_3P$	282	10	415[a]	0.69 0.1 M TEAP		1.02	693, 698
$(CH_2)_3$	H	B $(EtO)_3P$	220		414[a]				693
CH_3	CH_3	B Z = O,S $(EtO)_3P$	302	1	405[a]	0.63 0.1 M TEAP		0.93	693, 698
									
R: CH_3		B Z = O $(EtO)_3P$		0.4		0.07 0.1 M TBAP[c,h]	0.36	1.00*	778
R: $S(CH_2)_2S$		B Z = O $(EtO)_3P$		4.4		0.17 0.1 M TBAP[c,h]	0.41	0.95*	778, 780

(*table continues*)

TABLE 12 Molecular properties of Triselanamonothiafulvalenes (redox potential in acetonitrile vs. sce)



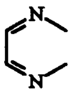
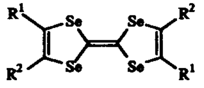
R¹	R²	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
CH_3	CH_3	B Z = S $(MeO)_3P$	253–254 (isooctane)					194
CF_3	CF_3	B Z = S $(MeO)_3P$	103.5–104 (hexane)					194
$COOCH_3$	$COOCH_3$	B Z = S $(MeO)_3P$	140–141 (benzene/ MeOH)					194
$\|S(CH_2)_2S\|$		B Z = O $(EtO)_3P$	233	8	384, 292, 224 sh, 213			773

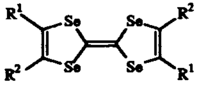
TABLE 13 Molecular properties of Tetraselenafulvalenes (redox potential in acetonitrile vs. sce)

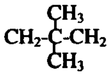
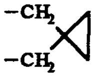


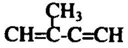
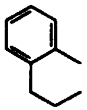
R¹	R²	Z		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H	Se (MeO)$_3$P or Ph$_3$P		133 (hexane)	70	495 (130), 365 (1 400), 300 (12 300), 287 (14 200)[b)]	0.48	0.76	106, 196, 200, 377, 412
H	CH$_3$	Se (MeO)$_3$P	*cis/trans* mixture						421
H	C$_6$H$_5$	S		189–195 (MeCN)	5	483 (355), 388 (5 010), 350 sh (2 400), 295 (22 900), 266 (24 000), 247 sh (22 900)[c)]			145
		Se Ph$_3$P			13				145
		Se (MeO)$_3$P	*cis/trans* mixture						421
H	COOH				62				654
H	COOCH$_3$			161–162	85	420 (2 950), 326 (7 950)[d)]			654, 781
H	COOC$_2$H$_5$	Se (MeO)$_3$P	*cis/trans* mixture						421
H	SeCH$_2$O(CH$_2$)$_2$ Si(CH$_3$)$_3$								363
CH$_3$	CH$_3$	S (EtO)$_3$P		250 dec.	30	508 (200), 299 (12 600)[k)]	0.44 0.1 M TEAP	0.72	46, 204, 782
		Se (EtO)$_3$P				500 (152), 365 (1 600), 304 (16 200)	0.54 0.2 M TBAT[k)]	0.93	46, 209, 210, 587
		Se (MeO)$_3$P			87	534 (176)[a)]	0.42 TBAA[e)]	0.81	225, 783
							0.43 0.1 M TBAT[k)]	0.73	215
CD$_3$	CD$_3$	Se (MeO)$_3$P			90	508, 299[k)]	0.43 0.1 M TBAT[k)]	0.73	215
CH$_3$	C$_2$H$_5$	Se (MeO)$_3$P	*cis/trans* mixture						421

(*table continues*)

TABLE 13 (*continued*)

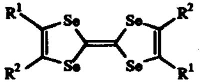


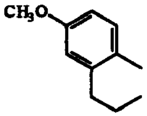
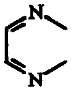
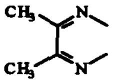
R^1	R^2	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
$(CH_2)_3$		Se $(EtO)_3P$	207–209 (PhCl)	45	498 (166), 370 (1 250), 301 (13 800)	0.55 0.2 M TBAT[k)]	0.92	193, 201, 209, 210, 587
					493 (230), 350 sh, 310 sh, 303 (16 600)[m)]	0.18 0.1 M TBAT[h,y)]	0.45	46, 784
$CH_2\text{-}CH\text{-}CH_2$ CH_3		Se $(MeO)_3P$	181–184 (hexane)	60	498 (210), 350 sh, 303 (16 200)[m)]	0.48 0.1 M TEAP	0.75	46
				5		0.15 0.1 M TBAT[h,y)]	0.39	784
 $CH_2\text{-}C(CH_3)_2\text{-}CH_2$				9		0.16 0.1 M TBAT[h,y)]	0.39	785
				10		0.17 0.1 M TBAT[h,y)]	0.41	784
$CH_2\text{-}S\text{-}CH_2$		Se $(EtO)_3P$	228–230	10	495 (525), 438 (500), 317 (8 300)[e)]			219
CH_2OH	CH_2OH		247 dec.	53		0.48 0.1 M TBAP[q)]	0.70	198
CHO	$CH(OC_2H_5)_2$	I *cis* Z = Se	153–157	35	466 (3 100), 286 (22 500)[k)]			198
		M = Co_2 n = 8 *trans*	173–176		472 (3 670), 286 (30 100)[k)]			
CHO	CHO		315 dec.	85				198
COOH	COOH		>300	28	360 (3 890), 322 (6 450), 277 (8 320), 205 (12 900)[p)]			786
$COOCH_3$	$COOCH_3$	Se Ph_3P	144–145 (MeOH/benzene)	75	422 (3 630), 328 (5 000), 285 (26 900), 260 (24 600), 212 (14 500)[d)]			277, 654
			145 dec.	40				786
$CONH_2$	$CONH_2$			87				654
CF_3	CF_3		111–112					194

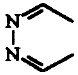
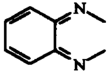
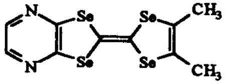
CH=C(CH$_3$)$_2$		CH=C(CH$_3$)$_2$		124.5–126			0.71 0.1 M TBAP$^{c)}$	0.89	198
	(CH=CH)$_2$		Se Ph$_3$P	291–293	66	470 (158), 320 (5 000), 286 (16 200) 221 (28 200)$^{a/c)}$	0.78 0.2 M TBAT$^{k)}$	1.17	220, 226
			Se Ph$_3$p	288–291 (CCl$_4$)	65				221
	 $CH{=}\overset{CH_3}{C}{-}C{=}CH$		Se Ph$_3$P						220
CH$_3$S		CH$_3$S		119–119.7 (benzene/hexane)	66	492, 360, 305$^{a)}$	0.64 0.1 M TBAT	0.84	367
							0.78 0.1 M TBAT$^{n)}$	1.03	360
	S(CH$_2$)$_2$S		O (EtO)$_3$P	220	49				70, 103
			O (EtO)$_3$P				0.27 0.1 M TBAP$^{c,h)}$	0.49	780
			Se (EtO)$_3$P		0				103
	S(CH$_2$)$_3$S		O (EtO)$_3$P				0.27 0.1 M TBAP$^{c,h)}$	0.51	780
C$_6$H$_5$S		C$_6$H$_5$S		140 dec.	44	386 (795), 277 (5 250), 241 (7 950), 216 (8 320)$^{p)}$			786
CH$_3$Se		CH$_3$Se		112–113	10	430, 308, 280$^{a)}$	0.58 0.1 M TEAP	0.81	195
				115.2–116.2	43		0.62 0.1 M TBAT	0.86	367
							0.75 0.1 M TBAT$^{n)}$	1.04	360
(CH$_3$)$_3$Si(CH$_2$)$_2$OCH$_2$Se		(CH$_3$)$_3$Si(CH$_2$)$_2$OCH$_2$Se							363
	Se(CH$_2$)$_2$Se		O (EtO)$_3$P	235–240	10		0.94 0.1 M TBAP$^{v)}$		195, 612, 787
			Se (RO)$_3$P		0		0.30 0.1 M TBAT$^{c,h)}$	0.55	195, 356, 612
C$_6$H$_5$Se		C$_6$H$_5$Se		155 dec.	52	388 (1 620), 297 (6 610), 219 (13 820)$^{p)}$			786
		*cis*	Se (EtO)$_3$P	228–230 (DMF)	15	505 (243), 365 (8 100), 318 (18 200), 281 (25 800)$^{c)}$	0.50 0.1 M LiCl$^{q)}$		192

(*table continues*)

TABLE 13 (*continued*)

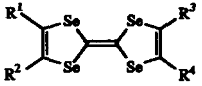


R¹	R²	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
	*trans*	Se $(EtO)_3P$	242–245 (DMF)	22	505(255), 365(8 350), 317 (20 350), 281 (26 800)[c)]	0.49 0.1 M LiCl[q)]	192
	cis	Se $(EtO)_3P$	235–236 (DMF)	34	510(238), 362 (10 200), 321 (19 100), 283 (30 700)[c)]	0.48 0.1 M LiCl[q)]	192
	trans $(EtO)_3P$	Se (DMF)	240–243	27 325	510(243), 362 (10 800), (19 500), 283 (32 400)[c)]	0.47 0.1 M LiCl[q)]	192
$(CH_3)_3Si$	$(CH_3)_3Si$		104–105 (hexane)	20			134
		Se $(RO)_3P$					80
		Se Ph_3P	313–315 (CCl_4)	40			81, 82
		S $(EtO)_3P$	>310		384[a)]		697, 698
		O $(EtO)_3P$	>310		378[a)]		698

		296 dec.	71		198
	S (EtO)$_3$P				697
	O (EtO)$_3$P	258		407[a)]	693

(*table continues*)

TABLE 13 (*continued*)

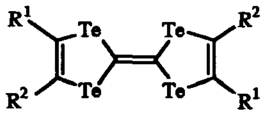


R^1	R^2	R^3	R^4	Z	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H	H	COOH		165			0.63 0.1 M TEAP	0.90	657
H	H	H	$COCH_3$		147–149 (c-C_6H_{12}/ CS_2 1:1)	20		0.62 0.01 M TBAHP[g,k]	1.00	369
H	H	H	COC_3H_7		115–118 (CS_2/hexane 1:1)	18		0.62 0.01 M TBAHP[g,k]	1.00	369
H	H	H	$COC_{13}H_{27}$		wax	17		0.62 0.01 M TBAHP[g,k]	1.00	369
H	H	H	$COC_{15}H_{31}$		83–85 (MeOH/ CH_2Cl_2 1:1)	14		0.62 0.01 M TBAHP[g,k]	1.00	369
H	H	H	$SC_{18}H_{37}$							363
H	H	CH_3	CH_3	Se $(MeO)_3P$	137–138			0.52 0.1 M TBAP[v]	0.89	788
H	H	$(CH_2)_3$		Se $(MeO)_3P$	153			0.52 0.1 M TBAP[v]	0.88	716, 788
H	H	$(CH_2)_4$			130			0.58 0.1 M TBAP[v]	0.96	714
H	CH_3	CH_3	CH_3	Se $(EtO)_3P$						789
H	CH_2OH	CH_3	CH_3		203	77		0.45	0.72	776, 790
H	$CH(OC_2H_5)_2$	CH_3	CH_3	Se $(EtO)_3P$	41	50		0.47 0.1 M TBAP	0.74	776, 790
H	CHO	CH_3	CH_3		199	50		0.65 0.1 M TBAP	0.96	776, 790
CH_3	CH_3	$(CH_2)_3$		Se $(MeO)_3P$	257			0.48 0.1 M TBAP[v]	0.87	791, 792
CH_3	CH_3	$(CH_2)_4$		Se $(MeO)_3P$	212			0.5 0.1 M TBAP[v]		714, 717

$(CH_2)_4$		$(CH_2)_3$			223		0.50	0.90	714
							0.1 M TBAP[c]		
CH_2OH	CH_2OH	CH_3	CH_3		243	70	0.42	0.71	776, 790
							0.1 M TBAP		
CH_2OH	CH_2OH	$(CH_2)_4$			206	85			776
$CH(OC_2H_5)_2$	$CH(OC_2H_5)_2$	CH_3	CH_3	Se	79	88	0.47	0.74	776, 790
				$(EtO)_3P$			0.1 M TBAP		
$CH(OC_2H_5)_2$	$CH(OC_2H_5)_2$	$(CH_2)_4$		Se	103	42			776
				$(EtO)_3P$					
CHO	CHO	CH_3	CH_3		259	90	0.70	0.97	776, 790
							0.1 M TBAP		
CHO	CHO	$(CH_2)_4$			251	97			776
CH_3	CH_3	$S(CH_2)_2S$		O,Se			0.15	0.44	780
				$(EtO)_3P$			0.1 M TBAP[e,h]		

(*table continues*)

TABLE 14 Molecular Properties of Tetratellurafulvalenes (redox potential in acetonitrile vs. sce)



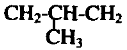
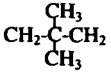
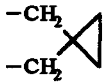
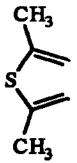
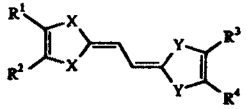
R^1	R^2	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H			324	0.59 0.2 M TBAT[k)]	0.84	229, 793
$(CH_2)_3$		260 (PhC)	32	610 (189), 410 (9 115), 335 (19 350), 320 (15 560)[v)]	0.40 TBAA[e)]	0.69	225, 794
					0.12 0.1 M TBAT[h,y)]	0.34	784
 $CH_2\text{-}CH(CH_3)\text{-}CH_2$			20		0.08 0.1 M TBAT[h,y)]	0.34	784
 $CH_2\text{-}C(CH_3)_2\text{-}CH_2$			35		0.10 0.1 M TBAT[h,y)]	0.31	784, 785
					0.11 0.1 M TBAT[h,y)]	0.34	785
$(CH{=}CH)_2$			10		0.71 0.2 M TBAT[k)]	1.05	226
					0.29 0.1 M TBAT[h,y)]	0.72	784
		295–298 ($ClCH_2CHCl_2$)	75		0.78 0.1 M TBAT[h,y)]	1.20	227
					0.40 0.1 M TBAT[h,y)]	0.80	784

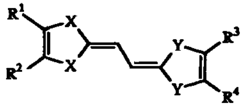
TABLE 15 Molecular Properties of Vinylogous, Cumulenic, Acetylenic, and Quinoid Tetrathia- and Tetraselenafulvalenes (redox potential in acetonitrile vs. sce)



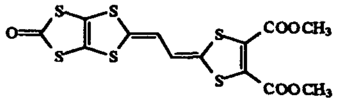
R^1	R^2	R^3	R^4	X	Y	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H	H	H	S	S	164–165 dec. (benzene)	83	404 (26 500), 384 (24 700)[w]	0.20 0.1 M TEAP[g]	0.36	117, 240
H	H	CH_3	CH_3	S	S	151–152 dec. (benzene)	86		0.23 0.1 M TEAP[g]	0.39	240
CH_3	CH_3	CH_3	CH_3	S	S	220–222 dec. (benzene)	78	415 (29 500), 394 (28 200)[w]	0.19 0.1 M TEAP[g]	0.34	117, 240
$(CH_2)_4$		CH_2OH	CH_2OH	S	S	161–162 (EtOAc/light petroleum)	82		0.37 0.1 M TBAP	0.49	284
$(CH_2)_4$		H	CH_2OH	S	S *cis/ trans* mixture	128–136 (CH_2Cl_2)	72		0.38 0.1 M TBAP	0.51	284
$(CH_2)_4$		H	$COOCH_3$	S	S		75				284
CH_3	CH_3	$COOCH_3$	$COOCH_3$	S	S	147–148 (CH_2Cl_2/hexane)	71		0.38 0.1 M TEAP[g]	0.59	240
$(CH_2)_4$		$COOCH_3$	$COOCH_3$	S	S	84					284
$COOCH_3$	$COOCH_3$	$COOCH_3$	$COOCH_3$	S	S	184–185 (CH_2Cl_2/hexane)	73	389 (26 300), 374 (28 200)[w]	0.60 0.1 M TEAP[g]	0.74	117, 240
C_6H_5	C_6H_5	C_6H_5	C_6H_5	S	S	180 dec. (benzene)	80		0.28 0.1 M TEAP[g]	0.46	240
H	H	$(CH{=}CH)_2$		S	S	126–127 (benzene/hexane)	37	400 (13 800), 380 (15 500)[w]	0.37 0.1 M TEAP[g]	0.53	117, 270
CH_3	CH_3	$(CH{=}CH)_2$		S	S	202–203 (benzene)	69		0.33 0.1 M TEAP[g]	0.50	240
$COOCH_3$	$COOCH_3$	$(CH{=}CH)_2$		S	S	134–135 (benzene/hexane)	46	393 (33 900), 374 (36 300)[w]	0.49 0.1 M TEAP[g]	0.64	117, 240
$(CH{=}CH)_2$		$(CH{=}CH)_2$		S	S	220 dec. ($CHCl_3$)	48	394 (33 900), 376 (35 500)[w]	0.47 0.1 M TEAP[g]	0.64	232, 240, 248

(*table continues*)

TABLE 15 (*continued*)

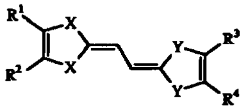


R^1	R^2	R^3	R^4	X	Y	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
						225	80	392 (87 100), 374 (64 600)[a]	0.26	0.41[e]	233, 261
H	H	CH_3S	CH_3S	S	S	oil			0.40 0.1 M TEAHP[g,k]	0.63	795
H	H	$C_{18}H_{37}S$	$C_{18}H_{37}S$	S	S	68–69			0.40 0.1 M TEAHP[g,k]	0.63	795
H	H	$S(CH_2)_2S$		S	S	158	57	417 (20 900)[a]	0.40 TBAHP	0.57	796
						161–162 (CH_2Cl_2/ c-C_6H_{12} 1:3)	80	415, 401, 237, 197[k]	0.43 0.1 M TEAHP[g,k]	0.69	797
H	H	$S(CH_2)_3S$		S	S	157–158	70		0.41 0.1 M TBAHP[g,k]	0.64	795
CH_3	CH_3	CH_3S	CH_3S	S	S	106–107			0.33 0.1 M TEAHP[g,k]	0.57	795
CH_3	CH_3	$S(CH_2)_2S$		S	S	201–202 (CH_2Cl_2/ c-C_6H_{12} 1:3)	82	423, 401, 233, 197[k]	0.36 0.1 M TEAHP[g,k]	0.62	797
CH_3	CH_3	$S(CH_2)_3S$		S	S	229–230	70		0.34 0.1 M TEAHP[g,k]	0.61	795
CH_3	CH_3	$Se(CH_2)_2Se$		S	S	198–200	62		0.29 0.1 M TEAHP[g,k]	0.54	798
$COOCH_3$	$COOCH_3$	$S(CH_2)_2S$		S	S	142 (toluene)	55		0.58 TBAHP	0.76	796
CH_3S	CH_3S	CH_3S	CH_3S	S	S	99			0.43 0.1 M TEAHP[g,k]	0.59	795
CH_3S	CH_3S	$S(CH_2)_2S$		S	S	137–139			0.42 0.1 M TEAHP[g,k]	0.62	795
CH_3S	CH_3S	$S(CH_2)_3$		S	S	151–152	70		0.43 0.1 M TEAHP[g,k]	0.63	795

SCH_2S		SCH_2S		S	S	246–247 dec. (DMSO)	16		0.41 0.1 M TBAHP[k]	0.60	799
$S(CH_2)_2S$		$S(CH_2)_2S$		S	S	>250 dec. (PhCN)	82	430 (14 120), 408 (13 500)[t]	0.36	0.59	800
						245 dec. (DMSO)	80	452 (37 200), 428 (36 300), 389 (33 100)[u]	0.40 0.1 M TBAHP[k]	0.57	796, 799
						239–240 (CS_2/hexane 2:1)	85	429, 403, 261, 230, 197[k]	0.48 0.1 M TBAHP[g,k]	0.71	797
$S(CH_2)_2S$		$S(CH_2)_3S$		S	S	219–220	70		0.48 0.1 M TBAHP[g,k]	(0.67)	795
$S(CH_2)_3S$		$S(CH_2)_3S$		S	S	247–249	70		0.47 0.1 M TBAHP[g,k]	(0.68)	795
CH_3S	CH_3S	CH_3Se	CH_3Se	S	S	120–122	76		0.39 0.1 M TEAHP[g,k]	0.57	798
CH_3Se	CH_3Se	CH_3Se	CH_3Se	S	S	132–134	58		0.39 0.1 M TEAHP[g,k]	0.59	798
CH_3Se	CH_3Se	$Se(CH_2)_2Se$		S	S	123–125	65		0.35 0.1 M TEAHP[g,k]	0.56	798
$Se(CH_2)_2Se$		$Se(CH_2)_2Se$		S	S	>340 (CS_2/MeOH)	55		0.41 0.1 M TEAHP[g,k]	0.63	798
H	H	H	H	S	Se	162 dec. (benzene)	61	398 (12 900), 372 (14 500)[w]	0.26 0.1 M TEAP[g]	0.40	231, 240
						165 (c-C_6H_{12})	81				801
$COOCH_3$	$COOCH_3$	H	H	S	Se	120 (CH_2Cl_2/hexane)	63	403 sh (13 942),383 (18 810), 231 (11 250)[k]			801
$COOCH_3$	$COOCH_3$	$COOCH_3$	$COOCH_3$	S	Se	175–176 (CH_2Cl_2/hexane)	94		0.62 0.1 M TEAP[g]	0.77	231, 240
H	H	H	H	Se	Se	183–184 dec. (benzene)	68	390 (11 500), 365 (11 500)[w]	0.33 0.1 M TEAP[g]	0.47	231, 240
$COOCH_3$	$COOCH_3$	$COOCH_3$	$COOCH_3$	Se	Se	156–157 (CH_2Cl_2/hexane)	60		0.64 0.1 M TEAP[g]	0.81	231, 240
							57				382

(*table continues*)

TABLE 15 (*continued*)



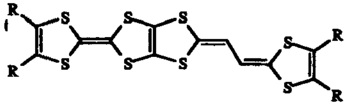
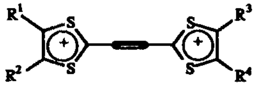
R^1	R^2	R^3	R^4	X	Y	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
										
R: H						197 dec.	88	402 (34 700), 345 (28 850), 283 (17 000)[w)]	0.37 0.50 0.81 1.05[e)]	382
R: $COOCH_3$							76		0.56 0.70 1.07 1.30[e)]	382

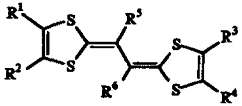
TABLE 15 (*continued*)

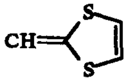
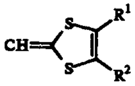
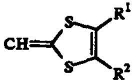
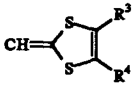
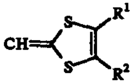
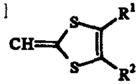


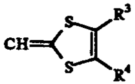
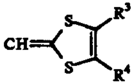
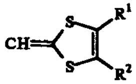
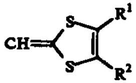
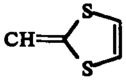
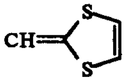
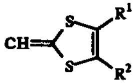
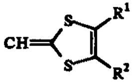
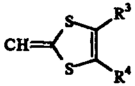
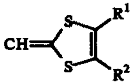
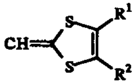
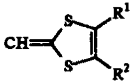
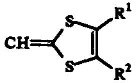
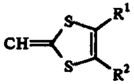
R^1	R^2	R^3	R^4	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
CH_3	CH_3	CH_3	CH_3		89	454 (19 500), 424 sh (14 800)[y]	(0.07) 0.1 M TEAP[g]	(0.31)	254
C_6H_5	C_6H_5	C_6H_5	C_6H_5		73				254

(*table continues*)

TABLE 15 (*continued*)

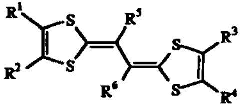


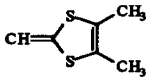
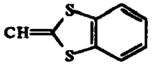
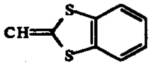
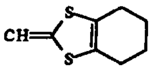
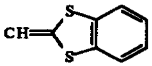
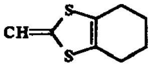
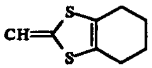
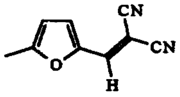
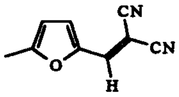
$R^1 = R^2$	$R^3 = R^4$	R^5	R^6	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
CH_3S	CH_3S	CH_3S	CH_3S	76		334(20 420)[a]	0.55 0.1 M TEAP	243
$COOCH_3$	$COOCH_3$	CHO	H	75				247
$(CH{=}CH)_2$	$(CH{=}CH)_2$	CHO	H	74				247
H	H		H	80			0.26 0.40 1.32 TBAP[e]	247
CH_3	CH_3		H	>230	62–68		0.08 0.33 1.25 0.01 M TBAHP[g,k]	802
CH_3S	CH_3S		H	oil	62–68		0.36 0.49 1.20 0.01 M TBAHP[g,k]	802
CH_3	$S(CH_2)_2S$		H	232	62–68		0.25 0.47 1.24 0.01 M TBAHP[g,k]	802
$COOCH_3$	$COOCH_3$		H		83		0.70 0.79 1.75 TBAP[e]	247
$(CH{=}CH)_2$	$(CH{=}CH)_2$		H	92	399 [k]		0.50 0.62 1.53 TBAP[e]	247

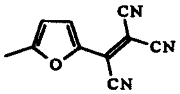
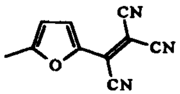
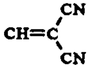
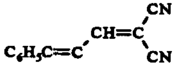
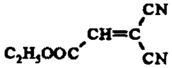
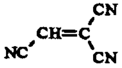
CH_3	CH_3S		CHO		53–65				802
CH_3	$S(CH_2)_2S$		CHO		53–65				802
$COOCH_3$	$COOCH_3$		CHO		86				247
$(CH{=}CH)_2$	$(CH{=}CH)_2$		CHO		85				247
H	H			87–92	80	0.25 TBAP[e]	0.37	0.72	247
CH_3S	CH_3S				57–63	0.26 0.01 M TBAHP[e,k]	0.37	0.69	802
CH_3	$S(CH_2)_2S$			192–195	57–63	0.23 0.01 TBAHP[e,k]	0.39	0.81	802
$COOCH_3$	$COOCH_3$				89	0.73 TBAP[e]		1.14	247
$(CH{=}CH)_2$	$(CH{=}CH)_2$				73	0.54 TBAP[e]		0.97	247

(*table continues*)

TABLE 15 (*continued*)

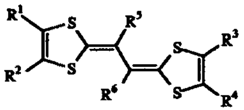


$R^1 = R^2$	$R^3 = R^4$	R^5	R^6	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
	$(CH=CH)_2$	C_6H_5	H		61		0.16* 0.33* 1.08* 1.42* 0.1 M TBAHP$^{k)}$	249
	$(CH=CH)_2$	C_6H_5	H		80		0.23* 0.43* 1.12* 1.45* 0.1 M TBAHP$^{k)}$	249
	$S(CH_2)_2S$	CH_3	CH_3		73		0.34* 0.50* 0.96* 1.40* 0.1 M TBAHP$^{k)}$	249
	$(CH_2)_4$	p-$CH_3C_6H_4$	H		30		0.04* 0.22* 0.86* 1.29* 0.1 M TBAHP$^{k)}$	249
	$S(CH_2)_2S$	p-$CH_3C_6H_4$	H		90		0.22* 0.39* 1.03* 1.37* 0.1 M TBAHP$^{k)}$	249
	$S(CH_2)_2S$	p-$CH_3C_6H_4$	H		89		0.06* 0.23* 0.85* 1.26* 0.1 M TBAHP$^{k)}$	249
	H	p-$CH_3C_6H_4$	H		70		0.04* 0.21* 0.88* 1.27* 0.1 M TBAHP$^{k)}$	249
$(CH=CH)_2$	$(CH=CH)_2$						0.49 0.1 M TBAHP$^{l)}$	235

(CH=CH)$_2$	(CH=CH)$_2$					0.56 0.1 M TBAHP[b)]	235
H	H	C$_6$H$_5$	 $CH{=}C(CN)_2$	33		0.76 0.1 M TBAP[e)]	373, 803
H	H	C$_6$H$_5$	 $C_6H_5C{\equiv}C{-}CH{=}C(CN)_2$	77			373
H	H	C$_6$H$_5$	 $C_2H_5OOC{-}CH{=}C(CN)_2$	47			373
H	H	C$_6$H$_5$	 $NC{-}CH{=}C(CN)_2$	77	511 (27 450), 364 (22 400), 247 (25 700), 199 (51 300)[a)]	0.85 0.1 M TBAP[e)]	373, 803

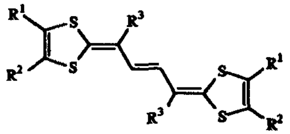
(*table continues*)

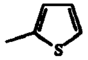
TABLE 15 (*continued*)



$R^1 = R^4$	$R^2 = R^3$	R^5	R^6	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
C_6H_5	H	C_6H_5	C_6H_5						239
CH_3S	CH_3	*p*-$NO_2C_6H_4$	*p*-$NO_2C_6H_4$	>270 (CH_2Cl_2/MeOH)	68		0.48 0.1 M TBAHP		237
CH_3S	CH_3	*p*-CNC_6H_4	*p*-CNC_6H_4	267 (CH_2Cl/MeOH)	78		0.44 0.1 M TBAHP		237
CH_3S	CH_3	C_6H_5	C_6H_5	163 (CH_2Cl_2/MeOH)	70		0.34 0.1 M TBAHP		237
CH_2S	CH_3	*p*-$CH_3OC_6H_4$	*p*-$CH_3OC_6H_4$	164 (CH_2Cl_2/MeOH)	71		0.32 0.1 M TBAHP		237
CH_3S	CH_3	*p*-$(CH_3)_2NC_6H_4$	*p*-$(CH_3)_2NC_6H_4$	168–170 (CH_2Cl_2/MeOH)	73		0.23 0.1 M TBAHP		237
CH_3S	CH_3	thienyl	thienyl	177–180 (CH_2Cl_2/MeOH)	66		0.35 0.1 M TBAHP		237
CH_3S	CH_3	C_3H_7	C_3H_7	oil	65		0.32 0.1 M TBAHP		237
CH_3S	CH_3	*p*-$CH_3OC_6H_4$	*p*-$NO_2C_6H_4$	139 (CH_2Cl_2/MeOH)	18		0.38 0.1 M TBAHP		237
CH_3S	CH_3	*p*-$CH_3OC_6H_4$	*p*-CNC_6H_4	128–130 (CH_2Cl_2/MeOH)	20		0.35 0.1 M TBAHP		237

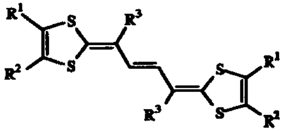
TABLE 15 (*continued*)

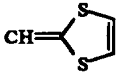
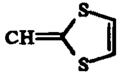
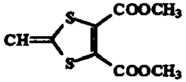
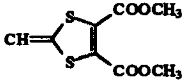
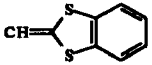
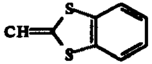
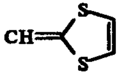
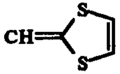
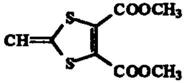
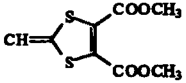
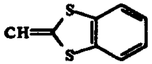
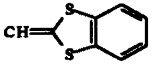
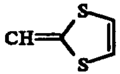
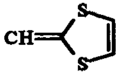


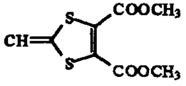
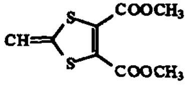
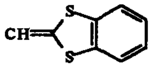
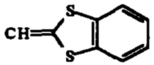
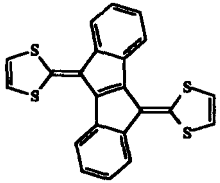
R^1	R^2	R^3	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
H	H	H	118–120	55	432 (53 100),409 (43 400)[w]	0.22 0.1 M TEAP[e]	240, 248
H	H	CH_2CH_2	175–176 dec. (hexane)	68		0.23 0.1 M TEAP[e]	240
H	H	C_6H_5	171–172 (benzene)	45		0.28 0.1 M TEAP[e]	240, 248
H	H		179–180 (toluene)	73	442 (46 670),420 (40 490), 228 (23 930)[e]	0.02 0.1 M TEAP[e]	804
CH_3	CH_3	H	176–178 dec. (benzene)	52		0.21 0.1 M TEAP[e]	240
C_6H_5	C_6H_5	H	193–194 dec. (CH_2Cl_2/hexane)	100		0.32 0.1 M TEAP[e]	240
	$(CH{=}CH)_2$	H	225–227 dec. (benzene)	69		0.47 0.1 M TEAP[e]	240, 248
CHO	CHO	H		30	413 (10 000), 391 (7 940)		805
$COOCH_3$	$COOCH_3$	H	195–197 ($MeNO_2$)	42	418 (14 130),396 (4 790)	0.55 0.1 M TEAP[e]	240, 248, 805, 806
COC_6H_5	COC_6H_5	H	203–205 ($MeNO_2$)	60			248
$COOCH_3$	$COOCH_3$	CH_2CH_2	202–204 (benzene/c-C_6H_{12})	94		0.57 0.1 M TEAP[e]	118, 240
$COOCH_3$	$COOCH_3$	C_6H_5	212–216 ($MeNO_2$)	80		0.60 0.1 M TEAP[e]	240, 248
CHO	CHO	C_6H_5		78			805
COC_6H_5	COC_6H_5	C_6H_5	244–246	51			248
CHO	CHO	-p$CH_3C_6H_4$	272 (CH_2Cl_2)	77			805

(*table continues*)

TABLE 15 (*continued*)

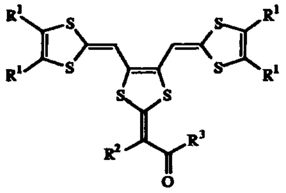


R¹	R²	R³	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2		References
CHO	$CH(OC_2H_5)_2$	p-$CH_3C_6H_4$	83–85						805
		H		50		0.12* 0.1 M TBAP[e]	0.47*		345, 805
		H		50		0.22* 0.1 M TBAHP[k]	0.40*	0.89*	805
		H		51					805
		C_6H_5		51		0.25* 0.1 M TBAP[e]	0.56*		345, 805
		C_6H_5		80		0.35* 0.1 M TBAP[e]	0.55*	0.81*	345, 805
		C_6H_5		55					805
		p-$CH_3C_6H_4$	180–183 dec. (DMF/EtOH)	63		0.20* 0.1 M TBAP[e]	0.42*		345, 805

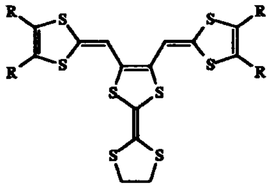
	*p*-$CH_3C_6H_4$ 		79		0.25 0.35 0.84 0.1 M TBAHP[k)]	805
					0.30 0.56 0.81 0.1 M TBAP[q)]	345, 805
			73		0.20 0.55 0.1 M TBAP[q)]	345, 805
	*p*-$CH_3C_6H_4$	260–262		518[a)]	0.42 0.1 M TEAT	770

(*table continues*)

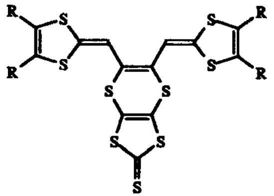
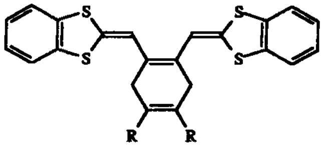
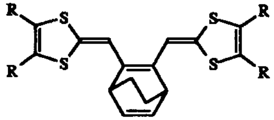
TABLE 15 (*continued*)



R^1	R^2	R^3	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H	C_6H_5		86				249
$(CH=CH)_2$	CH_3	CH_3		91		0.48 0.1 M TBAHP[k)]	0.66	249
$S(CH_2)_2S$	CH_3	CH_3		83		0.38 0.1 M TBAHP[k)]	0.50	249
CH_3	C_6H_5	H		86		0.28 0.1 M TBAHP[k)]	0.43	249
$S(CH_2)_2S$	C_6H_5	H		88		0.45 0.1 M TBAHP[k)]	0.54	249
$(CH_2)_4$	p-$CH_3C_6H_4$	H		91		0.28 0.1 M TBAHP[k)]	0.42	249
$(CH=CH)_2$	p-$CH_3C_6H_4$	H		93		0.53 0.1 M TBAHP[k)]	0.67	249

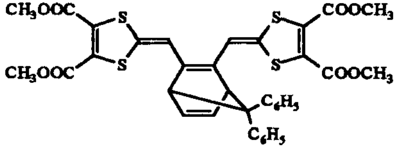
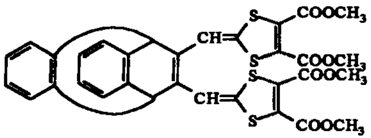
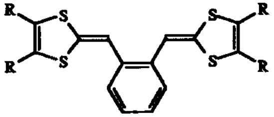


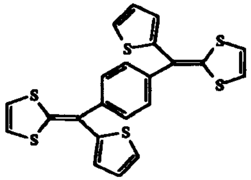
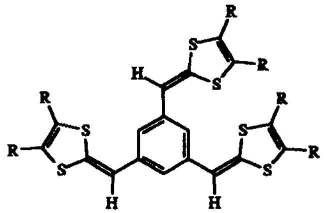
R^1	R^2	R^3	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
R: H						0.26 0.1 M TBAP[v)]	0.39	662

R: COOCH$_3$		70	0.29 0.1 M TBAP[q]		343, 662
			0.51 0.1 M TBAP[v]	0.74	662
			0.51 0.1 M TBAP[q]	0.58	662
					
R: CH$_3$	174	80	0.39 0.1 M TBAHP[x]		66
R: CH$_3$S		80	0.54 0.1 M TBAHP[x]		66
R: COOCH$_3$	239 dec.	80	0.81 0.1 M TBAHP[x]		66
					
R: H	152–154 (MeCN)	31			807
R: CH$_3$	210–212 (THF)	33			807
					
R: (CH$_2$)$_4$	174 dec. (MeOH)	56	0.21 0.1 M TBAHP[x]		807

(*table continues*)

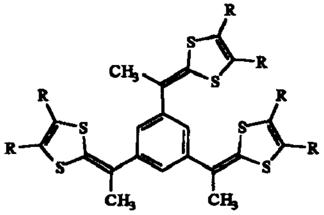
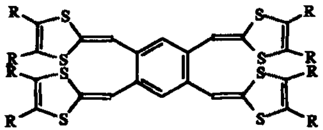
TABLE 15 (*continued*)

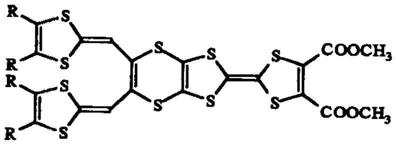
	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
R: $COOCH_3$	133 dec. ($CHCl_3$/MeOH)	70				807
R: $(CH{=}CH)_2$	160 dec. (MeOH)	47				807
	210 (toluene/pentane)	73				807
	110 (CH_2Cl_2/MeOH)	75				807
						
R: CH_3		75				807
R: $(CH_2)_4$	164	59		(0.69)* 0.1 M TBAHP[c]		807

R: COOCH$_3$			90		(1.04)* 0.1 M TBAP[q]		255, 807
					(1.20)* 0.1 M TBAP[v]		255
R: (CH=CH)$_2$		175 (CHCl$_3$/hexane)	75		(0.85)* 0.1 M TBAP[q]	(0.95)*	255, 807
					(0.93)* 0.1 M TBAP[v]	(1.05)*	255
							
R: COOCH$_3$					(0.88)* 0.1 M TBAP[q]	(1.08)*	255
					(1.03)* 0.1 M TBAP[v]		255
R: (CH=CH)$_2$		292–294 (DMF)		409 (50 100), 390 (56 200)	0.78 0.1 M TBAP[s]		241
		291 dec.	40		(0.72)* 0.1 M TBAP[q]	(0.86)*	255
		173–174 (DMF)	86	351 (21 250), 240 (14 740)[a]	0.04 0.1 M TEAP[e,q]	(0.23)	804
	R: H	160–165 (toluene)	12				236
	R: CH$_3$	232–234 (toluene)	21				236

(*table continues*)

TABLE 15 *(continued)*

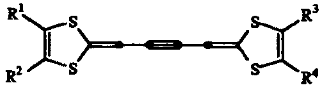
		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
	R: H	157–158 (MeCN)	50				236
	R: CH_3	205–209 (toluene)	25				236
							
R: H		159 dec. (PhCl)	59		(0.46)* 0.1 M TBAP$^{q)}$		255
					0.54 0.1 M TBAP$^{v)}$	0.66	255
R: $COOCH_3$		221–226	50		(0.85)* 0.1 M TBAP$^{q)}$	(0.99)*	255
					(0.96)* 0.1 M TBAP$^{v)}$	(1.15)*	255

R: $(CH{=}CH)_2$	>260 (PhCl)	90	(0.68)* 0.1 M TBAP[q]		255
			(0.68)* 0.1 M TBAP[v]	(0.96)*	255
					
R: CH_3					66
R: $COOCH_3$					66

(*table continues*)

TABLE 15 (*continued*)

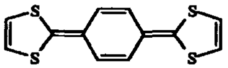
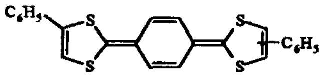
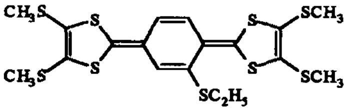
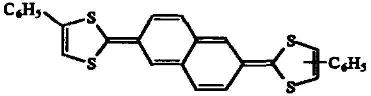
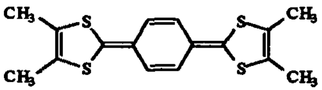
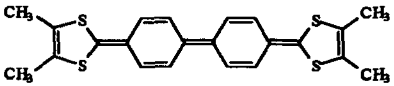
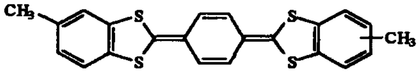
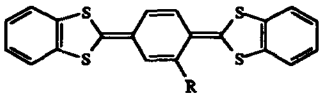
R^1	R^2	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
CH_3	CH_3		87	465 (33 100), 431 (28 200), 388 sh (19 500)[j]	(–0.12) 0.1 M TEAP[g]	(0.25)	254
C_6H_5	C_6H_5		74				254

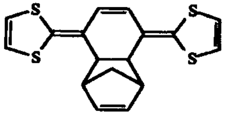
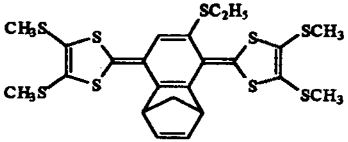
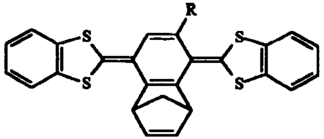


R^1	R^2	R^3	R^4	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
H	H	H	H	98	23		(0.48) 0.1 M TBAP		251, 253
H	H	CH_3	CH_3	136–138	54		(0.47) 0.1 M TBAP		251, 253
CH_3	CH_3	CH_3	CH_3		20		(0.42) 0.1 M TBAP		251, 253
$(CH_2)_3$		$(CH_2)_3$					(0.41) 0.1 M TBAP		251, 253
$(CH_2)_4$		$(CH_2)_4$		216 dec.			(0.42) 0.1 M TBAP		251, 253
H	H	$COOCH_3$	$COOCH_3$		59		0.61 0.1 M TBAP	1.18	251, 253
CH_3	CH_3	$COOCH_3$	$COOCH_3$		54		0.54 0.1 M TBAP	1.18	251, 253
$(CH_2)_3$		$COOCH_3$	$COOCH_3$				0.54 0.1 M TBAP	1.14	251, 253
$(CH_2)_4$		$COOCH_3$	$COOCH_3$				0.54 0.1 M TBAP	1.13	251, 253
CH_3	CH_3	$(CH=CH)_2$		102					251, 253
$COOCH_3$	$COOCH_3$	$COOCH_3$	$COOCH_3$	180	60		0.80 0.1 M TBAP	1.18	251, 253
$COOCH_3$	$COOCH_3$	$(CH=CH)_2$			74		0.72 0.1 M TBAP	1.18	251, 253
$(CH=CH)_2$		$(CH=CH)_2$			35		0.67 0.1 M TBAP	1.02	251, 253

(*table continues*)

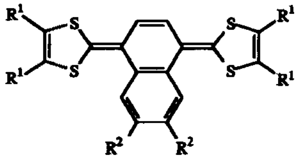
TABLE 15 (*continued*)

	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
	230–245 dec.	54	495 (60 200), 469 (31 600), 257 (11 750) [k)]	–0.11 0.1 M TEAP	–0.04	269
				0.23		808
						259
	211 dec.			0.20 0.1 M TEAT[i)]	0.34	809
						261
						258
				0.13 0.1 M TBAP[e)]		270
						264
						

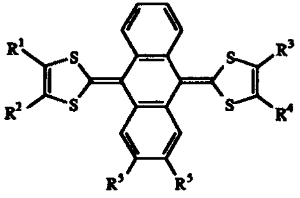
R: H	280	38	503 (91 100), 478 (64 500) [u]	0.27 0.1 M TBAHP[g,x]		241, 262, 263, 269, 810
R: SC$_2$H$_5$	190–200			0.26 0.1 M TEAT[x]	0.47	809
R: SC$_5$H$_{11}$	144–147			0.25 0.1 M TEAT[x]	0.48	809
R: SC$_7$H$_{15}$	123–130			0.27 0.1 M TEAT[x]	0.49	809
R: SC$_{10}$H$_{21}$	90–92			0.26 0.1 M TEAT[x]	0.50	809
R: Cl	290 dec.			0.73 0.1 M TEAT[x]	1.15	809
	168–170 dec.	27	440 (44 650), 416 (38 000), 395 (20 400)[k]	0.17 0.1 M TEAP		269
	117 dec.	56				809
						
R: H	260–265 dec.	73		0.37 0.1 M TEAP		269
R: SC$_2$H$_5$	150–154	61				809
R: SC$_5$H$_{11}$	131–132					809
R: SC$_7$H$_{15}$	131–132					809
R: SC$_{10}$H$_{21}$	oil					809
R: Cl	158 dec.					809

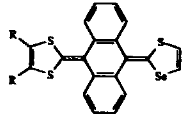
(*table continues*)

TABLE 15 (*continued*)



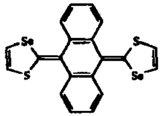
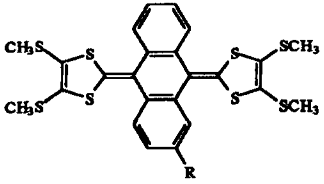
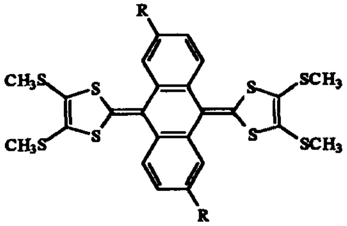
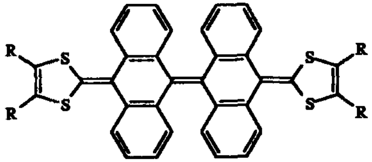
R^1	R^2	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
H	H	160–170 dec.			0.00 0.1 M TEAP	269
H	CH_3	190–194 dec.			0.04 0.1 M TEAP	269
CH_3	H				0.03 0.1 M TBAP[g,k)]	811, 812
					0.06 0.1 M TBAP[g)]	270
$(CH=CH)_2$	H	260–270		468 (55 000)[k)]	0.18 0.1 M TEAP	269
					0.27 0.1 M TBAHP[g,x)]	241, 265, 810
					0.40 0.1 M TBAP[k)]	267
$(CH=CH)_2$	CH_3	243–248 dec.			0.15 0.1 M TEAP	269

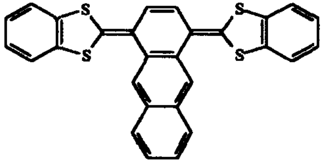
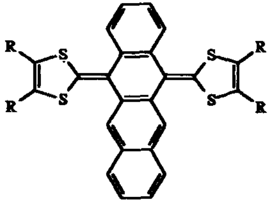
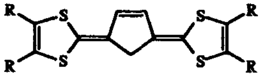
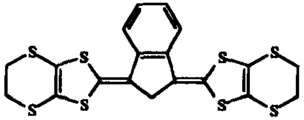


R^1	R^2	R^3	R^4	R^5					
H	H	H	H	H	320–325 dec.	70	415, 323, 273, 253, 206[a)]	0.40 0.1 M TBAP[g)] 0.25 0.1 M TEAP 0.52* 0.1 M TBAT[c)]	270, 811 269 813
H	H	CH_3	CH_3	H	>260	74	430, 360, 235, 200[a)]	0.36 0.1 M TBAP[g)]	270, 275
CH_3	CH_3	CH_3	CH_3	H	>230	75	433, 369, 235, 206[a)]	0.43 0.1 M TBAP[g)]	270, 811, 814
CH_3	CH_3	CH_3	CH_3	C_5H_{11}	199–202 (CH_2Cl_2/MeCN)	28		0.28	815
CH_3	CH_3S	CH_3	CH_3S	H		80			816
H	H	$C_{18}H_{37}S$	p-$CH_3C_6H_4$	H	59	55		0.56 0.1 M TBAP[g)]	275
CH_3	CH_3	$C_{18}H_{37}S$	p-$CH_3C_6H_4$	H	45	55		0.54 0.1 M TBAP[g)]	275
CH_3S	CH_3S	CH_3S	CH_3S	H	310–311 dec.			0.52 0.1 M TEAT[h)]	809
$S(CH_2)_2S$		$S(CH_2)_2S$		H	196–197		440, 269, 238[a)]	0.48 0.1 M TBAP[g)]	271
$(CH{=}CH)_2$		$(CH{=}CH)_2$		H	262–263 (EtOH)	73	415 (14 100), 398 (26 300), 348 (19 500), 237 (70 800)[k)]	0.39 0.1 M TBAP[g)] 0.78* 0.1 M TBAT[c)]	114, 241, 272 813
									
R: H					>260	75	419, 365, 240[a)]	0.47 0.1 M TBAP[g)]	270, 275
R: CH_3					>260	75	427, 363, 242[a)]	0.45 0.1 M TBAP[g)]	270, 275

(*table continues*)

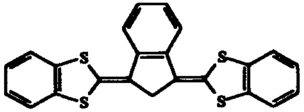
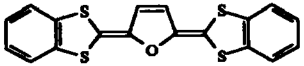
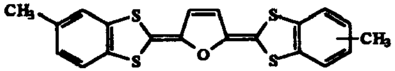
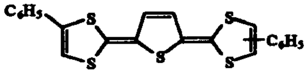
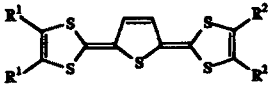
TABLE 15 (*continued*)

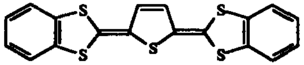
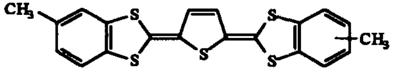
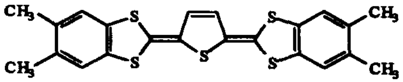
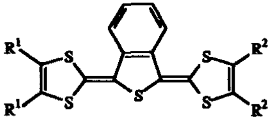
		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
		>240	63	409, 377, 265, 236a)	0.51 0.1 M TBAPe)		270
	R: $CH_2OSi(C_6H_6)_2Bu$-*t*		50				817
	R: CH_2OH	110	91				817
	R: $CH_2OCOCH{=}CH_2$		84				817
	R: $CH_2OCONH(CH_2)_2Cl$		70				817
	R: $CH_2OCOC_5H_4FeC_5H_5$		60		0.59 0.1 M TEAHPe,k)		817
	R: H				0.60 0.1 M TBAHPe,k)		818
	R: $OSi(C_6H_5)_2Bu$-*t*	242–244	59				818
	R: $OCOC_5H_4FeC_5H_5$	> 200 (CH_2Cl_2/hexane)	55		0.61 0.1 M TBAHPe,k)	0.78	818
	R: OCO-TTF	178–180	30		0.50 0.1 M TBAHPe,k)	0.94	818
							
R: H		>240	41	438, 378, 235, 200a)	0.48 0.1 M TBAPe)		270
R: CH_3		>240	38	447, 381, 240, 200a)	0.45 0.1 M TBAPe)		270

		314–317 dec.			0.24 0.1 M TEAP	269
	R: H	>240	59	417, 385, 235, 200[a]	0.44 0.1 M TBAP[e]	270
	R: CH_3	>240	55	428, 327, 230, 205[a]	0.41 0.1 M TBAP[e]	270
	R: $(CH{=}CH)_2$	285–290	48		0.39 0.1 M TBAP[e]	241
						
R: CH_3		233–235	8		0.23* 0.1 M TBAT[c]	819
R: CH_3S		152–153	46		0.40* 0.1 M TBAT[c]	819
R: $S(CH_2)_2S$		243–245	36		0.38* 0.1 M TBAT[c]	819
R: $(CH{=}CH)_2$		272–277	31		0.46* 0.1 M TBAT[c]	819
		249–252	7		0.62* 0.1 M TBAT[c]	819

(*table continues*)

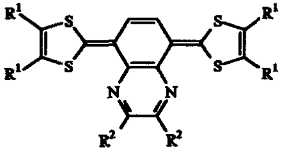
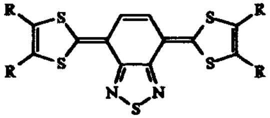
TABLE 15 (*continued*)

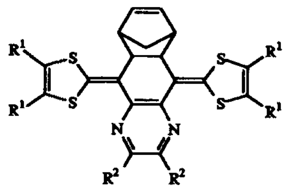
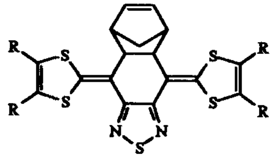
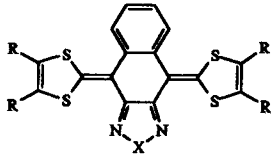
		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
		290–295	8		0.67* 0.1 M TBAT$^{e)}$		819
		218		478 (35 500)	–0.21 0.1 M TBAHP$^{C,D)}$	0.07	235
		214		477 (39 800)	–0.26 0.1 M TBAHP$^{C,D)}$	0.03	235
					0.09 0.1 M TBAP$^{k)}$	0.40	812
							
R^1	R^2						
H	H	149–150 dec.	91	489 (38 000), 462 (32 360)$^{c)}$	0.11 0.1 M TBAP$^{e)}$	0.34	669
H	CH_3S	102–105	60	492 (30 200), 466 (28 200)$^{c)}$	0.19 0.1 M TBAP$^{e)}$	0.37	669
H	$S(CH_2)_2S$	188–190	63	496 (30 200), 470 (26 900)$^{c)}$	0.18 0.1 M TBAP$^{e)}$	0.38	669
CH_3	CH_3				0.04 0.1 M TBAP$^{k)}$	0.31	812
CH_3S	$COOCH_3$	150–151	65		0.34 0.1 M TBAP^{e}	0.55	669

S(CH$_2$)$_2$S	COOCH$_3$	179–181	53		0.35 0.1 M TBAP$^{e)}$	0.55	669
S(CH$_2$)$_2$S	S(CH$_2$)$_2$S	170–175 dec.	64	504 (22 400), 473 (19 050), 279 (13 500)$^{c)}$	0.26 0.1 M TBAP$^{e)}$	0.43	820
S(CH$_2$)$_2$S	S(CH$_2$)$_3$S	139–140 dec.	60	501 (31 600), 474 (28 200), 274 (15 100)$^{c)}$	0.24 0.1 M TBAP$^{e)}$	0.42	820
S(CH$_2$)$_3$S	S(CH$_2$)$_3$S	240–241 dec.	73	496 (39 800), 474 (38 000), 271 (17 800)$^{c)}$	0.26 0.1 M TBAP$^{e)}$	0.44	820
COOCH$_3$	COOCH$_3$	227–229	98		0.47 0.1 M TBAP$^{e)}$	0.67	669
		>300	70	476 (95 500), 453 (81 300)$^{c)}$	0.34 0.1 M TEAP$^{e)}$	0.58	822
					0.32 0.1 M TBAP$^{e)}$	0.57	241
		266–268		480 (48 900), 454 (42 600)$^{c)}$	0.30 0.1 M TEAP$^{e)}$	0.54	821, 822
		295–298		483 (22 400), 456 (19 500)$^{c)}$	0.28 0.1 M TEAP$^{e)}$	0.52	822
							
R^1	R^2						
H	SCH$_2$S	162–164			0.36 0.1 M TBAP$^{e)}$	0.55	823
H	S(CH$_2$)$_2$S	71–72			0.37 0.1 M TBAP$^{e)}$	0.58	823
H	S(CH$_2$)$_3$S	112–114			0.37 0.1 M TBAP$^{e)}$	0.58	823
COOCH$_3$	SCH$_2$S		10				823
COOCH$_3$	S(CH$_2$)$_2$S		29				823
COOCH$_3$	S(CH$_2$)$_3$S		15				823

(*table continues*)

TABLE 15 (*continued*)

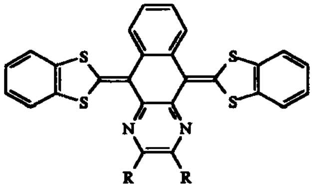
		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
							
R^1	R^2						
H	H	279–280		504 (63 100), 475 (44 700)k)	0.23 0.1 M TBAPe)	0.43	268
CH_3	H	320 dec.		518 (64 600), 489 (44 700)k)	0.17 0.1 M TBAPe)	0.35	268
$S(CH_2)_2S$	H	265–267		522 (51 300), 490 (34 700)k)	0.33 0.1 M TBAPe)	0.49	268
$(CH{=}CH)_2$	H	340–345	90	492 (33 100), 462 (24 000)k)	0.37 0.1 M TBAPe)	0.61	267
					0.44 0.1 M TBAPe)	0.60	268
$(CH{=}CH)_2$	CH_3	315–318	90	489 (79 500), 462 (53 700)k)	0.31 0.1 M TBAPe)	0.56	267
$(CH{=}CH)_2$	C_6H_5	322–324	90	582 (5 880), 500 (69 200)k)	0.44 0.1 M TBAPe)	0.67	267
							
R: H		297–298	95	494 (56 200), 464 (44 600)k)	0.36 0.1 M TBAPe)	0.53	824
R: CH_3		330 dec.	95	507 (56 200), 478 (44 600)k)	0.30 0.1 M TBAPe)	0.48	824
R: $S(CH_2)_2S$		>440	95	511 (50 100), 480 (37 100)k)	0.45 0.1 M TBAPe)	0.60	824
R: $(CH{=}CH)_2$		409–410	95	482, 454k)	0.54 0.1 M TBAPe)	0.70	824

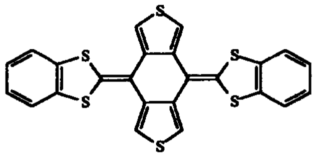
						
R¹	R²					
H	H	8				268
CH_3	H	41				268
$S(CH_2)_2S$	H	50				268
$(CH{=}CH)_2$	H	58	394 (51 300)[k)]			267
$(CH{=}CH)_2$	CH_3	80	389 (45 700)[k)]			267
$(CH{=}CH)_2$	C_6H_5	54	490 (15 130), 428 (32 350)[k)]	0.82 0.1 M TBAP[k)]	0.94	267
						
R: H		22				824
R: CH_3		72				824
R: $S(CH_2)_2S$		75				824
R: $(CH\text{-}CH)_2$		92				824
						
R	X					
H	S			0.66* 0.1 M TBAT[c)]		813
CH_3	S			0.61* 0.1 M TBAT[c)]		813

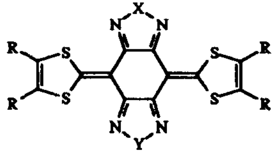
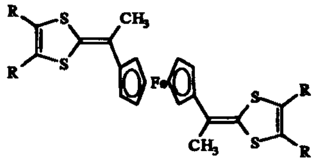
(*table continues*)

TABLE 15 (*continued*)

		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
$S(CH_2)_2S$	S				0.76* 0.1 M TBAT[e]	813
$(CH{=}CH)_2$	S	367–370	90	422 (43 600), 360 (16 200), 235 (38 000)[k]	0.65	272 0.1 M TEAP
					0.83* 0.1 M TBAT[e]	813
H	Se				0.69* 0.1 M TBAT[e]	813
CH_3	Se				0.65* 0.1 M TBAT[e]	813
$S(CH_2)_2S$	Se				0.81* 0.1 M TBAT[e]	813
$(CH{=}CH)_2$	Se	345–347	69	414(39 800), 305 (15 500), 233 (37 200)[k]	0.70 0.1 M TEAP	272
					0.87* 0.1 M TBAT[e]	813

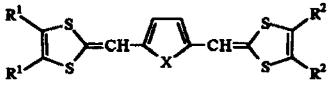


		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
R: H		273–274	4	438 (41 700), 370 (15 130)[k]	0.66 0.1 M TBAP[k]	267
R: CH_3		270–272	8	439 (45 700), 365 (15 130)[k]	0.61 0.1 M TBAP[k]	267
		376–378	38	374 (38 900), 335 (22 900), 248 (39 800)[k]	0.78[k]	272

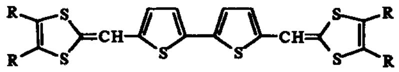
							
R	X	Y					
H	S	S	>400	48	542 sh, 470[g]		273,825
CH_3	S	S	>400	16			273,825
SCH_3	S	S	377–380	43	485 sh (10 970), 456 (20 900), 419 sh (15 400), 396 (10 970), 281 (10 000)[k]	0.81* 0.1 M TBAT[k]	825
SC_2H_5	S	S	>400	82		0.86* 0.1 M TBAT[k]	825
$S(CH_2)_2S$	S	S	>400	61			825
$(CH=CH)_2$	S	S	>400	70	420, 400, 380, 302, 257, 240[k]		272,825
H	S	Se	>400	34	555 sh, 485, 405[g]		825
H	Se	Se	>400	11	620 sh, 520 sh, 400[g]		825
							
R: H				65		0.10 0.56 (1,15) 0.2 M TBAHP[e]	333
R: CH_3				65		0.06 0.51 (1,05) 0.2 M TBAHP[e]	333

(*table continues*)

TABLE 15 (*continued*)



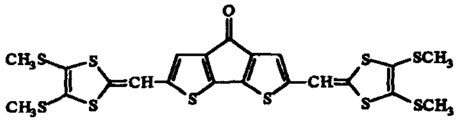
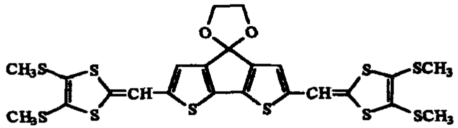
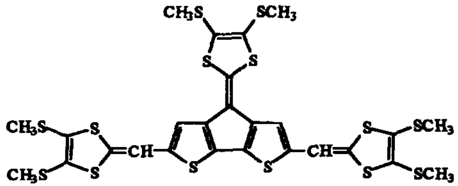
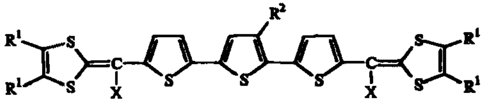
X	R^1		R^2	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V]	E^{ox}_2	References
NCH_3	H		H	148–149	8		0.20 0.1 M TBAHP	0.38	256
					24		0.19 0.1 M TBAHP[k)]	0.40	257
		$(CH_2)_4$			80		0.13 0.1 M TBAHP[k)]	0.41	257
		$S(CH_2)_2S$		199–200 (CH_2Cl_2)	87		0.31 0.1 M TBAHP[k)]	0.58	256
					71		0.29 0.1 M TBAHP[k)]	0.54	257
	$COOCH_3$		$COOCH_3$	188	88		0.48 0.1 M TBAHP	0.71	256
		$(CH{=}CH)_2$		242	60		0.32 0.1 M TBAHP[k)]	0.56	257
O	H		H	122–124	23		0.32 0.1 M TBAHP		256
				137	80		0.23 0.1 M TBAHP[k)]	0.31	257
				136–137 (EtOH)	67	436 (19 500),412 (18 620), 367 (14 130), 318 (12 300)[k)]	0.23 0.1 M TEAP	0.30	826
		$(CH_2)_4$			30		0.22 0.1 M TBAHP[k)]	0.36	257
	H		$COOCH_3$		63				257
	$(CH_2)_4$		$|S(CH_2)_2S|$	207	77		0.34 0.1 M TBAHP[k)]	0.54	257
		$S(CH_2)_2S$		213–214	66		0.38 0.1 M TBAHP[k)]	0.56	256
					45				257

	$COOCH_3$		$COOCH_3$		89	99		0.58 0.1 M TBAHP	0.71	256
		$(CH\text{-}CH)_2$			174		400 (40 750)	0.01 0.1 M TBAHP$^{C.D)}$	0.14	235
						42		0.41 0.1 M TBAHP$^{k)}$	0.61	257
		$CH{=}C(CH_3){-}CH{=}CH$			168		404 (35 500)	–0.04 0.1 M TBAHP$^{C.D)}$	0.10	235
S		H		H	133–134	43		0.39 0.1 M TBAHP	0.49	256
						75		0.35 0.1 M TBAHP$^{k)}$	0.45	257
					147–149 dec.	71	448 (28 200), 424 (27 550), 319 (8 700)$^{k)}$	0.40 0.1 M TEAP		826
		$(CH_2)_4$			205	60		0.28 0.1 M TBAHP$^{k)}$	0.41	257
	$(CH_2)_4$		$S(CH_2)_2S$			70		0.38 0.1 M TBAHP$^{k)}$	0.58	257
	CH_3S		CH_3S		122	90	454, 434$^{k)}$	0.48* 0.1 M TBAHP$^{k)}$	0.64*	827
		$S(CH_2)_2S$			230–231 (toluene)	51		0.46 0.1 M TBAHP$^{k)}$	0.65	256
						83		0.41 0.1 M TBAHP$^{k)}$	0.68	257
	$COOCH_3$		$COOCH_3$		149	91		0.69 0.1 M TBAHP	0.79	256
					164–165 (benzene/MeOH)	73	432 (30 900), 414 (30 900), 310 (9 970)$^{k)}$	0.62 0.1 M TEAP	0.74	826
							432, 412$^{k)}$	0.68* 0.1 M TBAHP$^{k)}$	0.87*	827
		$(CH\text{-}CH)_2$				40		0.48 0.1 M TBAHP$^{k)}$	0.68	257
							438, 416$^{k)}$	0.48* 0.1 M TBAHP$^{k)}$	0.68*	827
 R: CH_3S 827					152–155	53	462$^{k)}$	0.55* 0.64*	0.74*	

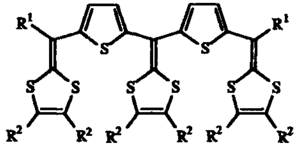
(*table continues*)

0.1 M TBAHP$^{k)}$

TABLE 15 (*continued*)

			m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1	[V]	E^{ox}_2	References
R: $COOCH_3$			224	56	447$^{k)}$	0.74* 0.1 M TBAHP$^{k)}$		0.88*	827
R: $(CH{=}CH)_2$			325 dec.	70	451$^{k)}$	0.58* 0.1 M TBAHP$^{k)}$	0.71*	0.92*	827
						0.38* 0.65* 0.1 M TBAHP$^{k)}$		0.56* 0.90*	828
						0.38* 0.68* 0.1 M TBAHP$^{k)}$		0.46* 0.96*	828
						0.18* 0.61* 0.1 M TBAHP$^{k)}$		0.50* 0.87*	828
									
X	R^1	R^2							
H	CH_3S	CH_3	108–110	40	475$^{k)}$	0.57* 0.16* 0.1 M TBAHP$^{k)}$		0.68* 1.27*	827
H	$COOCH_3$	CH_3	202–203	75	454$^{k)}$	0.70*	0.87*	1.20*	827

				0.1 M TBAHP[k)]	
H	(CH=CH)$_2$	H	417[k)]	0.50 0.69 0.92*	829
				0.1 M TBAP[k)]	
H	(CH=CH)$_2$	CH$_3$	444[k)]	0.46 0.64 0.82*	829
				0.1 M TBAP[k)]	
			454[k)]	0.52* 0.80*	827
				1.12* 1.50*	
				0.1 M TBAHP[k)]	
H	(CH=CH)$_2$	C$_8$H$_{17}$	450[k)]	0.50 0.62 0.82*	829
				0.1 M TBAP[k)]	
H	(CH=CH)$_2$	(CH$_2$CH$_2$O)$_3$CH$_3$	438[k)]	0.52 0.66 0.82*	829
	(CH=CH)$_2$	CH$_3$	440[k)]	0.1 M TBAP[k)]	
				0.45 0.61 1.00*	829
				0.1 M TBAP[k)]	

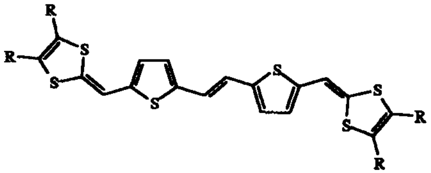
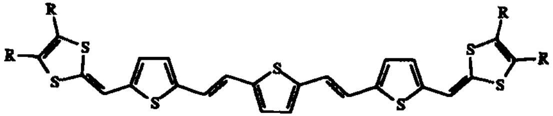
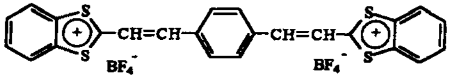


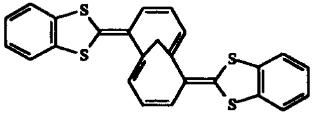
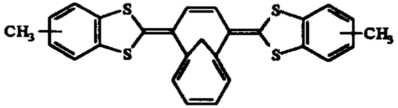
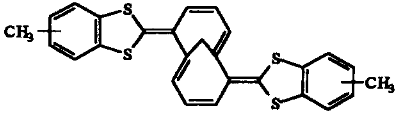
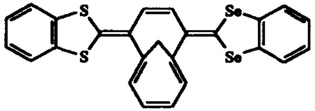
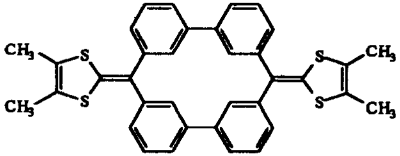
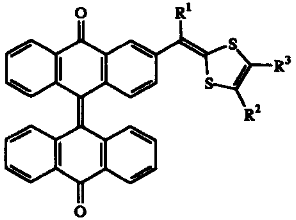
R^1	R^2					
H	H		27		0.48 0.82*	531
					0.1 M TBAT[e)]	
H	SCH$_3$	145–147 (CHCl$_3$/hexane)	69	455 (32 360), 432 (36 300)[k)]	0.58 0.92*	531
					0.1 M TBAT[e)]	
H	S(CH$_2$)$_2$S		54		0.58 0.91*	531
					0.1 M TBAT[e)]	
H	(CH=CH)$_2$		72		0.66 1.02*	531
					0.1 M TBAT[e)]	
CH$_3$	H		26		0.50 0.88 1.22*	531
					0.1 M TBAT[e)]	
CH$_3$	SCH$_3$		54		0.59 0.90 1.18*	531
					0.1 M TBAT[e)]	
CH$_3$	S(CH$_2$)$_2$S		52		0.59 0.92 1.22*	531
					0.1 M TBAT[e)]	
CH$_3$	(CH=CH)$_2$		47		0.68 1.04 0.30*	531

(*table continues*)

0.1 M TBAT[c]

TABLE 15 *(continued)*

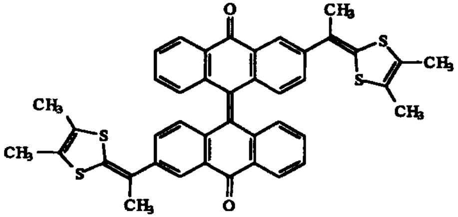
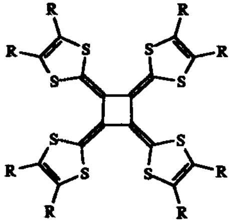
	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
					
R: *n*-C_3H_7	135 (ether)	85	493[k]	0.29* 1.43* 0.1 M TBAT[k]	830
R: CH_3S	192 dec. ($CHCl_3$)	80	484[k]	0.43* 1.40* 0.1 M TBAHP[k]	830
R: $COOCH_3$	245 dec. (ether)	52	468[k]	0.59* 0.68* 0.1 M TBAHP[k]	830
					
R: *n*-C_3H_7	132 dec. ($CHCL_3$)	80	521[k]	0.30* 1.00* 0.37* 0.1 M TBAHP[k]	830
R: CH_3S	175 dec.	75	510[k]	0.46* 1.05* 1.40* 0.1 M TBAHP[k]	830
	>340	64		0.40 0.1 M TBAP[g]	241
	> 220 dec.	89	490 (9 550), 310 (2 000), 262 (5 130), 239 (13 800)[k]	0.44 0.1 M TBAHP[e] 0.57	831 831

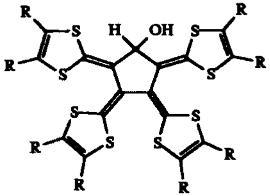
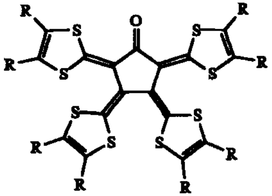
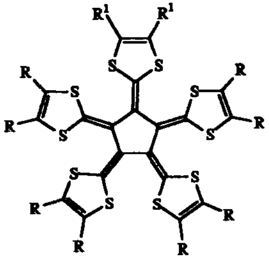
				0.1 M TBAHP${}^{s,k)}$			
	152–154	66	460 (11 500), 384 (12 900)${}^{k)}$				832
	166–168	95	494 (17 720), 316 sh, 271 sh	0.32 0.1 M TBAHP${}^{s,z)}$			832
	275–276	99	451 (38 000), 338 (9 330), 317 (8 320), 242 (38 000)${}^{k)}$				274
	271	61		0.91 0.1 M TBAP${}^{k,z)}$		1.33	833
							
R^1 R^2 R^3 CH_3 CH_3 CH_3	225 dec.	83		0.62	0.44	−0.36	834

(*table continues*)

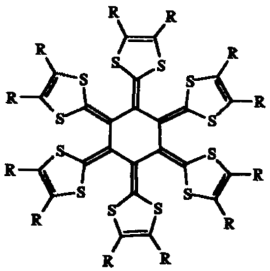
						–0.57 (–1.47) 0.1 M TBAHP$^{a)}$	
$C_{17}H_{35}$	CH_3	CH_3	138–140	32		0.57 0.38 –0.44 –0.61 (–1.37) 0.1 M TBAHP$^{a)}$	834

TABLE 15 (*continued*)

			m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
CH_3	CH_3	$SC_{18}H_{37}$	oil	26		0.64 0.48 –0.42 –0.63 (–1.39) 0.1 M TBAHP$^{a)}$	834
			321–322	42		0.62 0.33 –0.40 –0.56 (–1.37) 0.1 M TBAP$^{a)}$	834
		R: H	170 dec. (benzene)			0.19 (0.98) (1.10)	808, 835
		R: $COOCH_3$	235–238			0.65 0.70	835
		R: $(CH{=}CH)_2$				0.43 0.72	808
		R: $(CH{=}CH)_2$	220	93			836

									
			250 dec.	88		0.17	1.14[c,g]		246
CH$_3$			292	77		0.64	1.35[c,g]		246
=CH)$_2$			298	84		0.38	1.22[c,g]		246
									
R	R^1								
(CH=CH)$_2$	CH$_3$		240 dec.	50		0.28 0.1 M TEAP[e]	0.36		836
(CH=CH)$_2$	CH$_3$S		240 dec.	55		0.366 0.1 M TEAP[e]			836
(CH=CH)$_2$	(CH=CH)$_2$		270	64	463 (8 320), 356 (57 550)[i]	0.39 0.1 M TEAP[e]			836
						0.36[c,g]			244
R: COOCH$_3$		a	>300	35	386 (31 600), 331 (61 600)[k]	0.73 0.1 M TBAP[g,k]	(0.89)	(1.13)	245
		b	>300		354 (44 600), 286 (36 300)[k]	1.14 0.1 M TBAP[g,k]			245
R: (CH=CH)$_2$		a	>300	72	393 (17 000), 338 (35 500)[l]	0.43 0.1 M TBAP[c,g]	(0.65)	(0.81)	245
		b	>300		354 (46 700)[l]	0.82			245

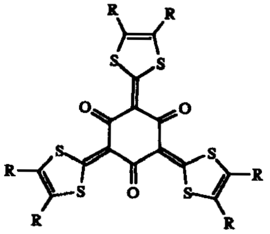
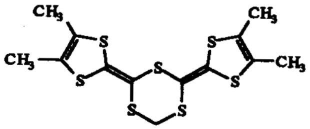
(*table continues*)

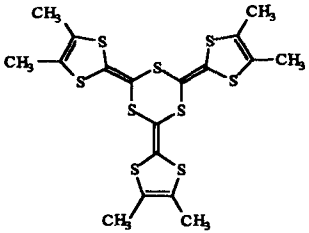
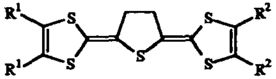


0.1 M TBAP c,g)

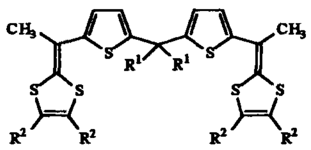
[a] boat conformation
[b] chair conformation

TABLE 15 (*continued*)

		m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
	R: H	>400 (DMSO)	62			837
	R: C_3H_7	312–315 (EtOH)	30		0.65 0.80 (1.25) 0.1 M TEAHP s,x)	838
		203 (acetone/hexane)	30	363 (14 130), 334 (24 550), 322 (21 850), 264 (9 130) w)	0.60 0.1 M TBAHP m)	580
		216 (CS_2)	25	367 (12 900), 334 (18 200), 256 (7 420) w)	0.59 0.68 0.82 0.1 M TBAHP m)	580

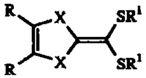
					
					
R^1	R^2				
CH_3S	$COOCH_3$	B Z = O,S $(MeO)_3P$	118–120	53	669
$S(CH_2)_2S$	$COOCH_3$	B Z = O,S $(MeO)_3P$	191–193	55	669
$COOCH_3$	$COOCH_3$	B Z = O,S $(MeO)_3P$		57	669

(*table continues*)



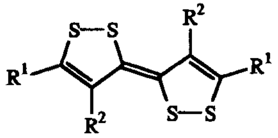
R¹	R²				
H	H	74–75	47	0.82* 0.1 M TBATe)	839
H	SCH_3	90–91	70	0.82* 0.1 M TBATe)	839
H	$S(CH_2)_2S$	167–168	93	0.82* 0.1 M TBATe)	839
H	$(CH{=}CH)_2$	159–160	86	0.82* 0.1 M TBATe)	839
CH_3	$(CH{=}CH)_2$	140–142	33	0.93* 0.1 M TBATe)	839

TABLE 15 *(continued)*



R¹	R²	X	m.p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1 [V] E^{ox}_2	References
CH_3	$(CH_2)_2$	S	140 (EtOAc/hexane)	57	367 (1 950), 328 (11 750), 318 (10 700), 268 (3 540)w)	0.75 1.13 0.1 M TBAHPw)	580
CH_3	CH_2SCH_2	S	168 (hexane)	68	355 (6 025), 330 (13 500), 318 (10 000), 260 (4 070)w)	0.75 1.13 0.1 M TBAHPw)	580
$S(CH_2)_2S$	$(CH_2)_2$	S		57			183
$S(CH_2)_2S$	$(CH_2)_3$	S		61			183
SCH=CHS	$(CH_2)_2$	S		36			183
SCH=CHS	$(CH_2)_3$	S		22			183
=CHSCH=	$(CH_2)_2$	S		67			183
CH_3	$(CH_2)_2$	Se		83			183
$(CH_2)_3$	$(CH_2)_2$	Se		33			183

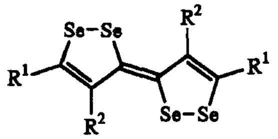
TABLE 16 Properties of 1,2-Tetrahiafulvalenes (redox potential in acetonitrille vs. sce)



R^1		R^2		m. p. [°C] (solvent)	yield [%]	λ_{max} (ε) [nm]	E^{ox}_1	E^{ox}_2	References
							[V]		
CH_3		CH_3							841
$(CH_3)_2N$		H		266 (DMF)	17				574
$(CH_2)_4N$		H		263 (DMF)	30				574
$O(CH_2)_4N$		H		268–269 (DMA)	7				574
$C_6H_5NCH_3$		H		265–266 $(CHCl_3/CCl_4)$	7				574
CH_3S		CH_3S	*cis*-isomer	198 (CH_2Cl_2)	30	481, 440 sh, 350$^{k)}$			568, 623
			trans-isomer	238 (CH_2Cl_2)	45	473, 454 sh, 375$^{k)}$			568
	SCH_2S			181$^{i)}$ (DMF/MeCN)	35	580, 540 sh, 390 sh, 370$^{a)}$			568
	$S(CH_2)_2S$			177$^{i)}$	38	580, 550 sh, 420 sh, 400$^{k)}$			568
CH_3COO		CH_3S							841
C_6H_5		H		179–180$^{L)}$ 180–181$^{L)}$	3–67				536, 571 536, 571
C_6H_5		CH_3		143–144 $(CHCl_3)$	59				571
C_6H_5		CH_3S		208	16	550 (7 950)$^{w)}$	0.30	0.52	573
C^6H^5		C_6H_5		228.5 $(CH_2Cl_2/MeOH)$	13–49	526$^{u)}$ 502$^{4)}$ 502$^{E)}$ 497$^{w)}$			536, 571
				290	52	500 (12 600)$^{w)}$	0.21	0.43	573
	$(CH{=}CH)_2$			147–149$^{L)}$ $(CH_2Cl_2/MeOH)$	2–41				571
				212–213$^{L)}$ $(CH_2Cl_2/MeOH)$					5711

(*table continues*)

TABLE 17 Molecular Properties of 1,2-Tetraselenafulvalenes (redox potential in acetonitrile vs. sce)



R^1	R^2	m.p. [°C]	yield	λ_{max} (ε)	E^{ox}_1		E^{ox}_2	
		(solvent)	[%]	[nm]		[V]		References
C_6H_5	C_6H_5	304	5	497 (15 800), 338 (63 000)$^{w)}$				573

a) in acetonitrile, b) in hexane, c) in tetrahydrofuran, d) in cyclohexane, e) in benzonitrile, f) in methanol/benzene (4:1, v:v), g) vs. Ag/AgCl electrode, h) vs Ag/AgNO$_3$ (0.01 M) electrode, i) probably *trans*-isomer, j) 70 % perchloric acid, k) in methylene chloride, l) in benzene, m) in chlorobenzene, n) in 1,2-dichloroethane, o) in 0.1 N resp. 0.01 N sodium hydroxide, p) in ethanol, q) in *N,N*-dimethylformamide, r) in isooctane, s) in methanol, t) in dioxan, u) in carbon disulfide, v) in 1,1,2-trichloroethane, w) in chloroforme, x) in propionitrile, y) in butyronitrile, z) vs. normal hydrogen electrode, A) in 1,2-dichlorobenzene, B) in KBr, C) in methylene chloride/acetonitrile, D) vs. ferrocene, E) in carbon tetrachloride, F) in 1,1,2-trichloroethane at 90 °C, G) in 1,1,2-trichloroethane at 70 °C, H) in 1,2-dichloroethane at 50 °C, I) in dimethyl sulfoxide, K) in hexamethylphosphoric triamide, L) one isomer, M) in benzonitrile at 60 °C, N) vs. Ag wire

TBAA: tetrabutylammonium hexafluoroarsenate, TBAHP: tetrabutylammonium hexafluorophosphate, TBAP: tetrabutylammonium perchlorate, TBAT: tetrabutylammonium tetrafluoroborate, TEAP: tetraethylammonium perchlorate, TEAT: tetraethylammonium tetrafluoroborate, TEAB: tetraethylammonium bromide

* peak potential, potential in parentheses indicate an irreversible redox reaction

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