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Thiophenethiols†

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ABSTRACT. **New data on the chemistry of thiophenethiols have been reviewed systematically. Synthetic routes to these compounds, their reactions with nucleophiles and radicals, and a number of other properties have been examined.**

The bibliography contains 78 references.

Contents

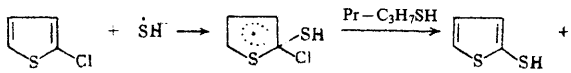
I. Introduction 1309
II. Synthesis of thiophenethiols 1309
III. Physical properties 1310
IV. Reactions of thiophenethiols 1311

I. Introduction

Thiophenethiols are highly reactive compounds which are used widely in organic synthesis. Since the publication of an earlier review in this field,[1] the chemistry of this class of compound has developed and a number of new aspects have been brought to light. These include, in particular, new methods of their synthesis, their reactions with radicals, and their recently discovered autothiylation. The reactivity of certain thiophenethiols has also been noted with much interest as new routes to key compounds have been developed. These include drugs and biologically active compounds, scents, polymers and 'organic metals'.

II. Synthesis of thiophenethiols

The primary route to thiophenethiols, notably for industrial syntheses, involves the reaction of halogenothiophenes with H_2S or lower alkyl mercaptans in the gas phase.[2] The reaction proceeds by a radical mechanism. It has been found that the yield of 2- and 3-thiophenethiols is higher with alkyl mercaptans than with H_2S. This would be expected from energetic considerations, since the C–S bond energy is 25–30 kcal/mol less than that of the S–H bond energy in H_2S. Thus, 2-chlorothiophene reacts with isopropyl mercaptan at 540 °C in a quartz tube to give 2-thiophenethiol in 23.6% yield.[3]



+ HCl + CH_2=$CHCH_3$ + $\dot{S}H$.

† This article received the third prize in a competition of reviews on 'Organic and Elementoorganic Synthesis' (Section No. 4), organised by the Inter-departmental Scientific and Technical Council set up to monitor the direction of chemical research and technology.

Dialkyl disulphides, which also produce thiyl radicals on thermolysis, may be used instead of alkyl mercaptans:

$$C_2H_5SSC_2H_5 \longrightarrow 2C_2H_5\dot{S} \xrightarrow{2(C_2H_5)_2S} 2C_2H_5SH + CH_3\dot{C}HSSCH_2CH_3,$$
$$2C_2H_5SH \longrightarrow 2CH_3\dot{C}H_2 + 2SH^{\cdot}.$$

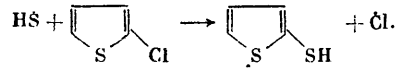
The yield of 2-thiophenethiol by this route is 36.4%.[4]

The low yield of thiophenethiols obtained by the high temperature reaction of halogenothiophenes with H_2S is explained, in part, by their thermal instability and, in part, by the formation of a number of side-products, of which bis(2-thienyl) sulphides predominate. It has been shown that thermolysis of bis(2-thienyl) sulphide, e. g. on hydrothiolysis in the liquid phase at 180 °C in the presence of an excess of sulphur, affords 2- and 3-thiophenethiol in 29.9% and 12.6% yield, respectively.[5,6] A similar yield of thiols is obtained under the same conditions by hydrothiolysis of bis(2-thienyl) disulphides.[7] The added elemental sulphur provides an additional source of thiyl radicals, both by interaction with H_2S and by fission of the S_8 ring.

$$S_8 + H_2S \rightarrow H\dot{S} + H\dot{S}_8 \rightarrow H\dot{S} + S_7.$$

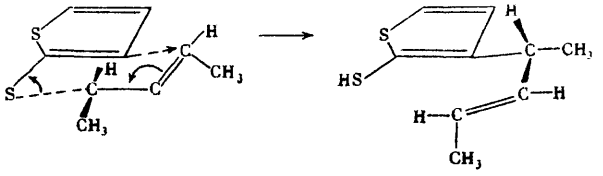
It has been found[8] that the addition of dimethyl selenide, which is readily cleaved into radicals above 300 °C, increases the yield of 2-thiophenethiol on reaction of 2-chlorothiophene with H_2S:

$$(CH_3)_2Se \longrightarrow \dot{C}H_3 + CH_3\dot{S}e \longrightarrow Se + \dot{C}H_3,$$
$$\dot{C}H_3 + H_2S \longrightarrow CH_4 + \dot{S}H,$$



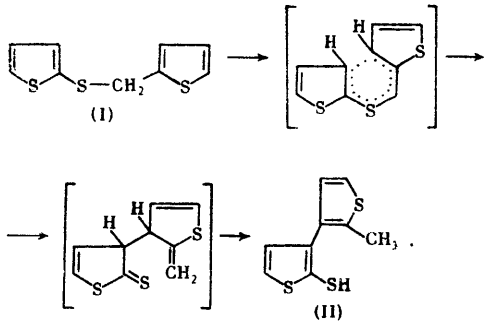
The maximum yield of 2-thiophenethiol is 31.1%.

A V Anisimov et al. have studied the thio-Claisen rearrangement of various thienyl sulphides to give the corresponding substituted thiophenethiols on heating in different solvents.[9,10] (–)-*S*-2-(*trans*-Pent-3-en-2-ylthio)thiophene has been shown to rearrange with retention of optical activity, confirming that reaction proceeds by a concerted [3.3]-sigmatropic shift.[11]



Quantum chemical calculations on the rearrangement of allyl 2-thienyl sulphide have shown that the sulphide and thiol exist in the stable 'open' conformation with a clear separation of the reactive centres.[12] It is thus considered that the rate of the reaction will be determined predominantly by the energetics of the conformational interconversion and by entropy factors.

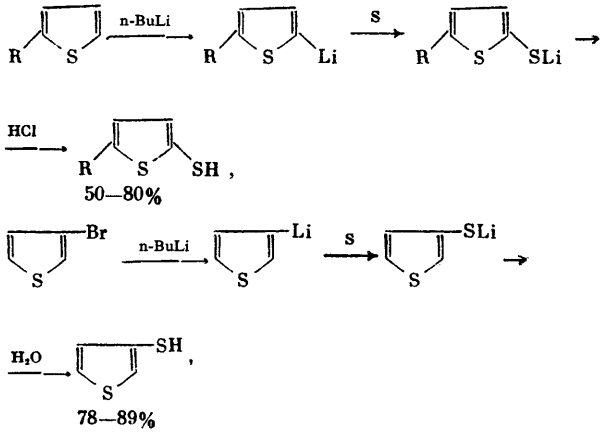
Thio-Claisen rearrangement also provides a convenient route to binuclear heterocyclic thiols.[13] Thus, 2-thienyl 2-thienylmethyl sulphide (I) and 2-benzofurylmethyl 2-thienyl sulphide afford 3-(2-methyl-3-thienyl)-2-thiophenethiol (II) and 3-(2-methyl-3-benzofuryl)-2-thiophenethiol, respectively, in 31% and 30% yield.



(I)

(II)

The commonest laboratory preparations of thiophenethiols are based on the intermediate formation of lithiothiophenes:[10]



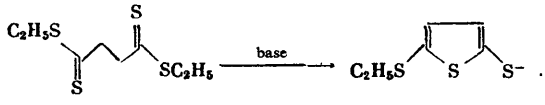
50—80%

78—89%

where R = H, Me, Et, Bu^t, Pr^i, etc. 3,4,5-Trichloro-2-thiophenethiol has also been prepared by this route in 86% yield.[14]

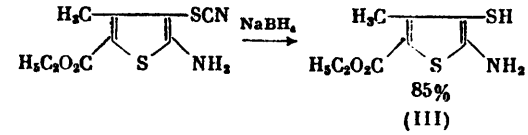
3,4-Thiophenedithiol has recently been obtained in one step by treatment of 3,4-dibromothiophene with two equivalents of butyllithium, followed by addition of flowers of sulphur and water.[15] This contrasts with the former two-step route.[16,17]

Among alternative methods of preparing thiophenethiols, it is worth noting the cyclisation of diethyl tetrathiosuccinate.[18] Cooling is necessary during the reaction, for which a number of tertiary amines or dimethylformamide acetals may serve as catalyst.



60%

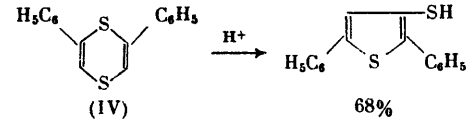
The 2-amino-3-thiophenethiol (III) may be obtained by reduction of the corresponding 3-thienyl thiocyanate with $NaBH_4$ or sodium dithionite:[19]



85%

(III)

A substituted 3-thiophenethiol was also isolated by an unusual acid-catalysed rearrangement of the 1,4-dithiin (IV).[20] The reaction proceeds on heating at 180 °C in σ-dichlorobenzene in a sealed tube.



(IV)

68%

2-Thiophenethiol may also be obtained by reduction of 2-thiophenesulphonic acid with PPh_3.[21]

III. Physical properties

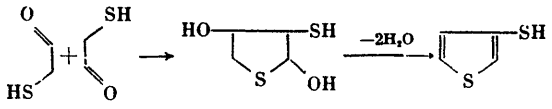
The physical properties of a number of thiophenethiols are given in Table 1. These data were reported after the publication of the previous review.

Table 1. Physical properties of thiophenethiols.

R^2	R^3	R^4	R^5	m.p. °C	b.p. °C/mmHg	^{1}H NMR (ppm)*	Reference
SH	Cl	Cl	Cl	39.0–41.0	56/0.1	3.55 (s) [$CDCl_3$]	[14]
SH	2-Methyl-3-benzofuryl	H	H	–	–	7.5–6.8 (m, 6H, arom.,thiophene); 3.35 (s, 1H, SH); 2.3 (s, 3H, CH_3) [CCl_4]	[13]
SH	2-Methyl-3-thienyl	H	H	–	–	6.5–7.3 (m, 4H, thiophene); 3.15 (s, 1H, SH); 2.27 (s, 3H, CH_3) [CCl_4]	[13]
SH	H	H	SC_2H_5	–	125/0.3	1.26 (t, 3H, CH_3); 2.78 (t, 2H, CH_2); 3.57 (s, 1H, SH); 6.90 (s, 2H, 3-, 4-H) [$CDCl_3$]	[18]
OMe	SH	H	SCH_3	–	123–127/3	2.34, (s, 3H, SCH_3); 3.0 (s, 1H, 4-H); 3.89 (s, OCH_3); 6.68 (s, 1H, 4-H) [CCl_4]	[37]
NH_2	SH	CH_3	CO_2Et	125–128	–	–	[19]

*In parenthesis are given the multiplicity of the signal, its integral intensity (the number of protons) and the position of the protons in the molecule.

Recently much attention has been paid to thermolytically prepared thiophenethiols for their aromatic properties and as starting materials for the synthesis of novel aromatic compounds.[22–34] Chromato-mass spectrometric and gas chromatographic analyses have indicated that 2-thiophenethiol, 3-thiophenethiol and 2-methyl-3-thiophenethiol are responsible for the fleshy odours which have been detected in certain pyrolytic experiments, supporting the view that thiophenethiols play a key role in the formation of sulphur-containing compounds in meat. It is thought that thiophenethiols are formed by condensation of the products derived by thermolysis of cysteine or by reaction with hydrocarbons and hydrogen sulphide. Thus, 3-thiophenethiol may be formed from two molecules of mercaptoacetaldehyde:



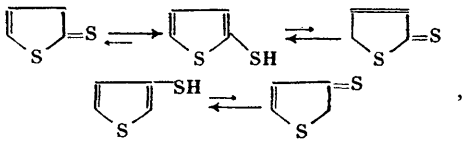
To assist identification and separation of aromatic thiophenethiols in mixtures, mass and IR spectroscopic studies of simple thiophenethiols and the corresponding alkyl thienyl sulphides have been undertaken and retention times on capillary gas chromatography columns determined.[35]

The dipole moments of a number of 3-thiophenethiols and 3-(alkylthio)thiophenes in benzene have also been determined in order to provide conformational information on the structures of the compounds.[36]

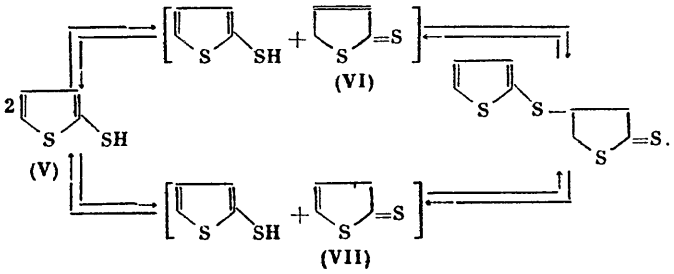
IV. Reactions of thiophenethiols

1. Nucleophilic addition

In the tautomeric system:



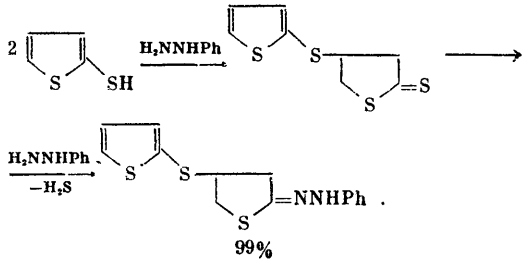
the equilibrium is clearly shifted in favour of the thiol form. This has been established both by quantum chemical calculations[38] and by the fact that proton signals from the thione form are absent in the ^{1}H NMR spectra of thiophenethiols. However, there is evidence that certain reactions of thiophenethiols involve the thione form, such as the recently discovered autothiylation of 2-thiophenethiol to give 4-(2-thienylthio)tetrahydrothiophene-2-thione.[39–42] Reaction takes place on exposing the thiol to air or in the presence of an amine as catalyst. It is most likely that the conversion takes place by a radical mechanism or by the ionic addition of 2-thiophenethiol (V) to the tautomeric thione form (VI) or (VII):



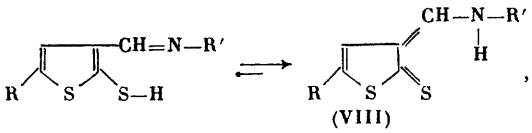
The reaction is reversible, although the equilibrium lies very much to the right. The dimer reverts to the monomer on distillation *in vacuo*. A side-reaction accompanies the autothiylation: the formation of bis(2-thienyl) sulphide with evolution of H_2S, as follows:



If autothiylation takes place in the presence of phenylhydrazine, the hydrazone derivative of the dimer is isolated:[43]

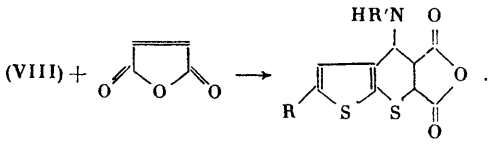


In-depth studies by Ya L Gold'farb et al. of the chemistry of mercaptoaldimines (e.g. Refs. 44 and 45), confirmed the existence of thiol-thione tautomerism in thiophenethiols. In solution, it has been found that mercaptoaldimines (VIII) exist as an equilibriated mixture of tautomers, shifted almost exclusively to the side of the thione form:



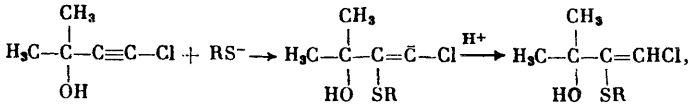
R = Et, OMe; R′ = *cyclo*-C_6H_{11}.

These compounds participate in the Diels–Alder reaction as heterodienes:[46]



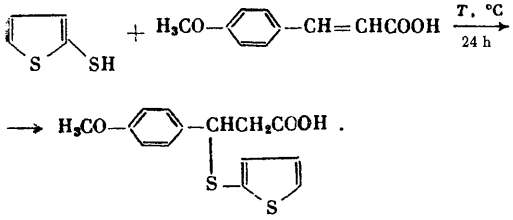
However, from the data available at present, it is clear that thione-type reactions of thiophenethiols are the exception rather than the rule. In fact, all of the reactions outlined below proceed from the thiol form.

The most characteristic and studied reaction of thiophenethiols is their addition to multiple bonds. Thus, treatment of dimethyl(chloroethynyl)carbinol with thienylmercaptide anion leads to the formation of the corresponding chlorovinylcarbinol which may be used subsequently for the preparation of ethynyl sulphides:[47]

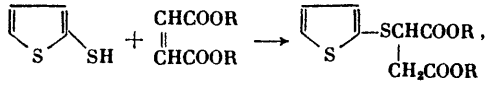


R = thienyl.

There are references to the addition of thiophenethiols to various unsaturated carbonyl compounds.[37,48] Thus, the reaction of 2-thiophenethiol with 4-methoxycinnamic acid yields an adduct which has been used for the preparation of antiglaucoma agents:[49]

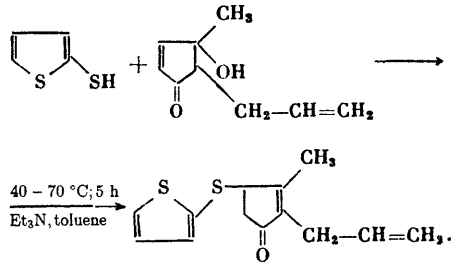


It is interesting that the addition of 2-thiophenethiol to diallyl maleate proceeds exclusively across the double bond of the maleic residue:[50]



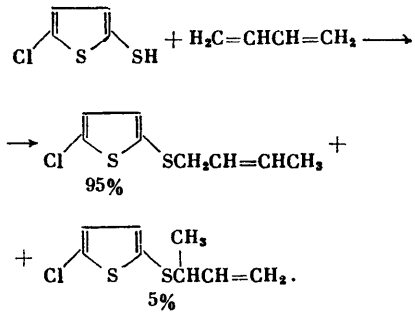
R = $CH_2CH{=}CH_2$.

A series of cyclopentenones, including 2-allyl-3-methyl-4-(2-thienylthio)-2-cyclopentenone, has been prepared in high yield as follows:[51]

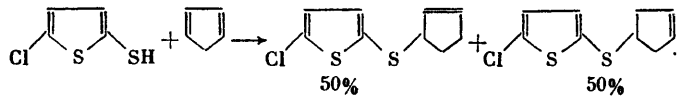


As in the previously described reaction, the allyl group is unaffected.

The addition of 5-chloro-2-thiophenethiol to 1,3-butadiene affords predominantly the 1,4-adduct:[52]

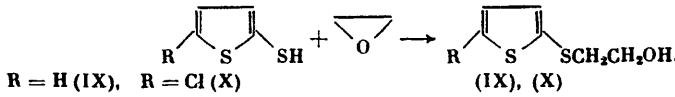


The reaction takes place in the presence of ethanesulphonic acid. In contrast, cyclopentadiene reacts with 5-chloro-2-thiophenethiol to give a 1 : 1 mixture of 1,2- and 1,4-adducts in 79% yield:



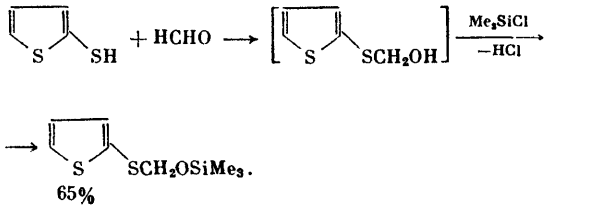
The fact that rate of the addition and the yield of adducts increases in the presence of a protic acid, e. g. ethyl sulphate, confirms the nucleophilic character of the addition of 5-chloro-2-thiophenethiol to such 1,3-dienes.

A study has been made of the interaction of ethylene oxide with heteroaromatic thiols in the liquid phase at 20 °C in the presence of an acid catalyst.[53] It was shown that the yield of the resulting heterocyclic β-hydroxyethyl sulphides increases with increase in the acidity of the thiols. The addition of an acid catalyst, e. g. aluminium oxide or an aluminosilicate, also increases the yield of product. Under optimum conditions, the yield of sulphides (IX) and (X) was 75% and 91%, respectively:

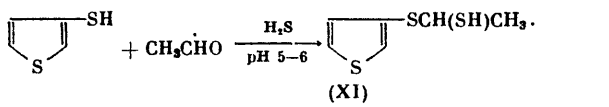


The observed effect of thiol acidity and addition of catalyst confirms that the mechanism of the epoxide ring opening involves protonation of the oxygen atom. The products obtained by addition of thiols to expoxides possess fungicidal and acaricidal activity.[54]

Simple aldehydes react with thiophenethiols to give monothiohemiacetals. Thus, the interaction of 2-thiophenethiol with paraformaldehyde in the presence of an excess of trimethylsilyl chloride affords 2-thienyl trimethylsiloxymethyl sulphide, as follows:[55]

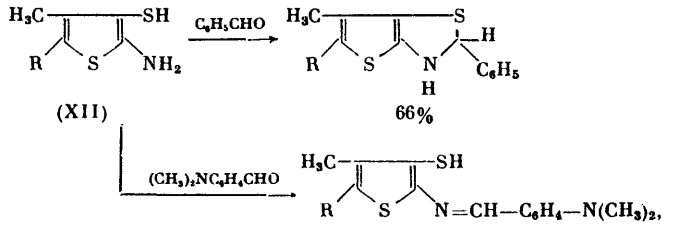


Bis(2-thienylthio)methane is formed as a by-product in 20% yield. The condensation of 3-thiophenethiol with acetaldehyde in the presence of H_2S affords the hemimercaptal (XI):[34]



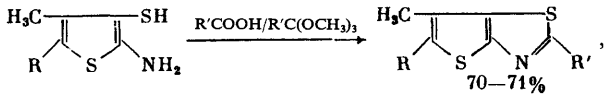
The interaction of 2-amino-3-thiophenethiols (XII) with benzaldehyde leads to substituted thieno[2,3-*d*]thiazolines.[19] It is likely that the reaction involves intramolecular addition of the thiol group across the C=N bond arising by initial condensation of the aldehyde with the amino group. It is interesting that the cyclisation

does not take place with *p*-dimethylaminobenzaldehyde. This has been supported by IR- and ^{1}H NMR spectroscopic data.



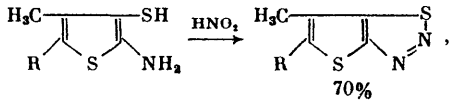
R = $CO_2C_2H_5$.

Substituted thieno[2,3-*d*]thiazoles are obtained by interaction of (XII) with formic or acetic acid and their orthoester derivatives:



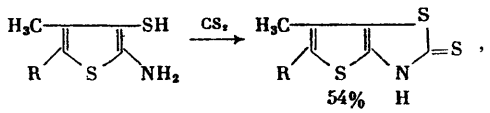
R = CO_2CH_3, $CO_2C_2H_5$; R′ = H, CH_3.

Diazotisation of the amino group in compound (XII) also leads to ring closure:



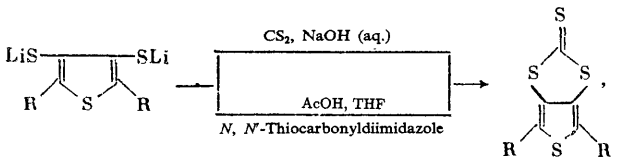
R = $CO_2C_2H_5$.

The reaction of (XII) with carbon disulphide exemplifies nucleophilic addition of a mercaptan across a C=S bond:



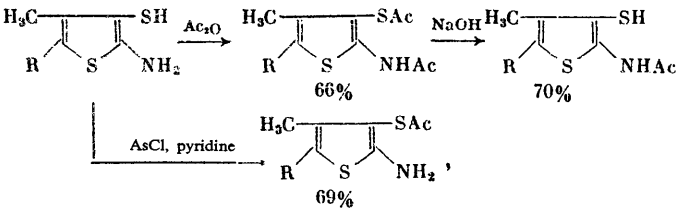
R = $CO_2C_2H_5$.

A similar reaction with thiophenedithiolates leads to $\Delta^{2,2'}$-bithieno-[3,4-*d*][1,3]dithioles, which have been used to prepare "organic metals" in the tetrathiafulvalene series:[56,57]



R = H, Me.

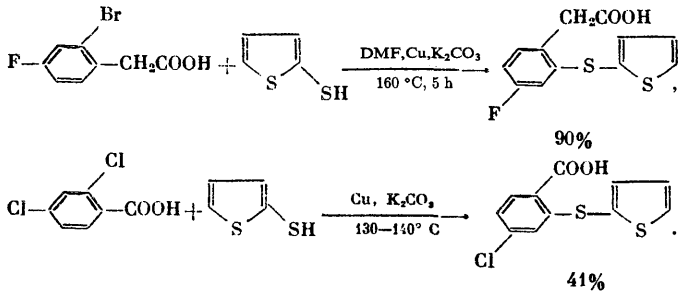
Acylation is also characteristic of thiophenethiols, as exemplified below with acetic anhydride or acetyl chloride as acylating agent:



R = $CO_2C_2H_5$.

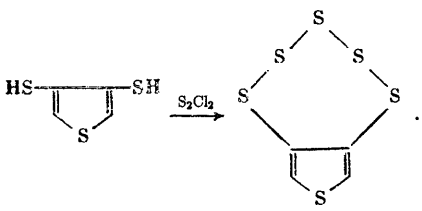
2. Nucleophilic substitution

2-Thiophenethiol participates in nucleophilic substitution of halogen in activated aromatics by heating in the presence of copper as catalyst:

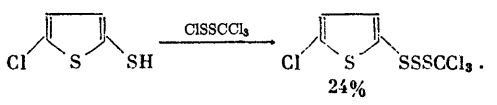


The resulting products have been used as intermediates in the synthesis of neuroleptic compounds.[58–60] The dye, 1-amino-5,8-bis-(2-thienylthio)anthraquinone, has been prepared in a similar way from the corresponding dichloroanthraquinone and 2-thiophenethiol.[61]

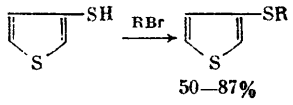
The formation of thieno[3,4-*f*]-1,2,3,4,5-pentathiepin from 3,4-thiophenedithiol and sulphur monochloride in dichloroethane also involves a nucleophilic substitution:[15]



Trichloromethylthiosulfenyl chloride reacts similarly with 5-chloro-2-thiophenethiol to give the corresponding trisulphide:[62]



Thiophenethiols are readily *S*-alkylated to give a wide variety of thienyl sulphides. Thus, *n*-alkyl 3-thienyl sulphides may be obtained by reaction of 3-thiophenethiol with alkyl bromides, RBr, in the presence of potassium tert-butoxide.[63]

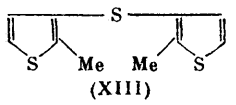


50—87%

R = C_nH_{2n+1}, where $n = 1$—6, 8, 10, 12.

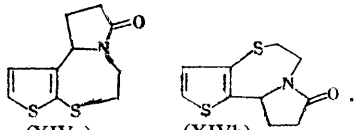
Dimethylformamide acetals may also be used as alkylating agent.[18]

3-Bromo- or 3-tosyloxy-2-methyltetrahydrothiophene has been converted to the bis(thienyl) sulphide (XIII). The latter is a component of certain natural aromas, having a smell of roasted nuts:[33]



(XIII)

The isomeric tricyclic compounds, (XIVa) and (XIVb), are obtained by condensation of 2- and 3-thiophenethiol, respectively, with *N*-(2-chloroethyl)succinimide, followed by partial reduction with sodium borohydride and ring closure of the resulting lactamol in the presence of formic acid.[64]

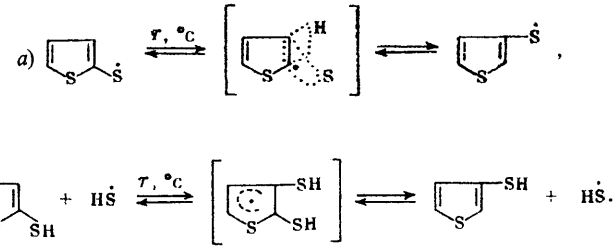


(XIVa) 38% (XIVb) 44%

These products are analogues of biologically active compounds.

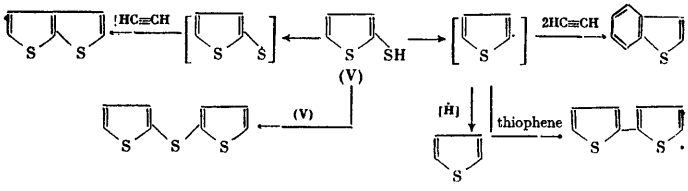
3. Radical reactions

Radical reactions take place on thermolysis or hydrothiolysis of thiophenethiols (at 400—600 °C) to give a mixture of products containing thiophene, dithienyls, and condensed systems.[5] The conversion of 2-thiophenethiol to 3-thiophenethiol also takes place under these conditions and is worthy of note.[5,6,8] Two mechanisms, *a*) and *b*), have been considered:



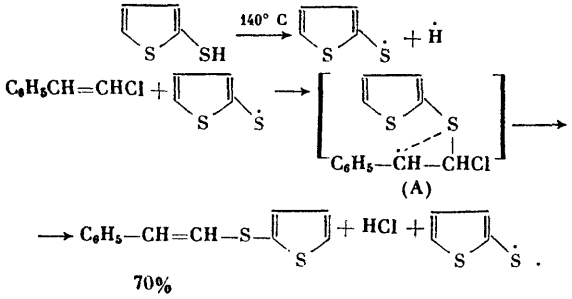
Reaction is favoured by the increased thermal stability of the 3-substituted thiophene, relative to the 2-substituted derivative.

It has been established that 2-thiophenethiol reacts with acetylene at 550—650 °C to give a mixture of thieno[*b*]thiophene, bis(2-thienyl) sulphide, thiophene, and benzo[*b*]thiophene.[65] Dithienyls are also obtained above 530 °C. The yield of each product varies according to the temperature and the proportion of the two reactants. The complexity of the reaction is a result of competing S—H and C—S cleavage in the initial phase of the heterolysis. The acetylene molecule then traps the resulting thiyl and 2-thienyl radicals:



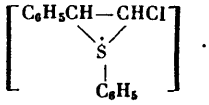
Thiophenol reacts under the same conditions to give benzo[*b*]thiophene as the only product.

The formation of β-styryl 2-thienyl sulphide from 2-thiophenethiol and β-chlorostyrene exemplifies radical substitution of halogen bonded to an unsaturated carbon atom.[66]

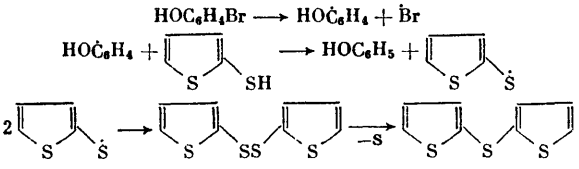


70%

By comparison with substitution by thiphenol, the reaction is much slower and stereo-random as a consequence of steric hindrance in the transition state (A). In the case of thiophenol, there is a clearly defined intermediate (below) which determines the stereospecificity of the reaction.

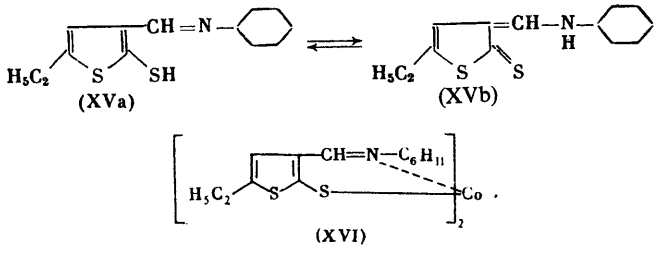


At 180 °C 2-thiophenethiol reduces bromophenols to the corresponding phenols while being oxidised to the disulphide.[67]



The reaction takes palce below the temperature at which the C—Br bond of bromophenols normally dissociates.

2-Mercapto-5-ethyl-3-thienylmethyleneaminocyclohexane and its cobalt(II) chelate complex are used as inhibitors in the oxidation of hydrocarbons.[68,69]



Mechanistically, these inhibitors would appear to function as radical traps interrupting the chain process. In complex (XVI) it is the cobalt(II) atom which is responsible for the deactivation, while in compound (XV) it is either the SH group (of XVa) or the C=S group (of XVb).

$$RO_2^{\cdot}+RSH\rightarrow RO_2H+RS^{\cdot},$$
$$2RS^{\cdot}\rightarrow RSSR.$$

However, the effectiveness of these compounds as inhibitors is less than what might be expected. This is a result of the competing reaction of (XVa) with peroxides, producing both radicals and unreactive products:

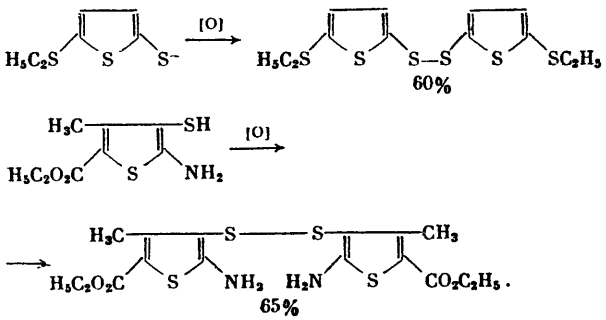
$$RSH+RO_2H\rightarrow RS^{\cdot}+H_2O+RO^{\cdot},$$
$$RSH+RO_2H\rightarrow \text{stable products}.$$

The activity of the inhibitors is thus gradually eroded.

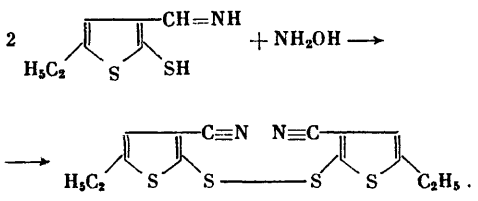
Polymers have been obtained by copolymerisation of 2,5-thiophenedithiol with divinylthiophene or divinylbenzene under UV irradiation or in the presence of radical initiators. These products may be used principally in the manufacture of light-sensitive materials and polaroid films.[70–72]

4. Other properties

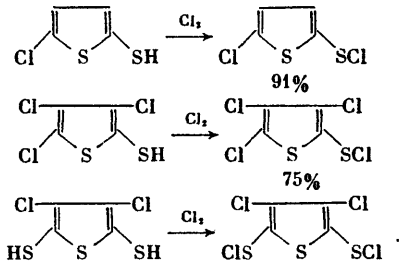
Thiophenethiols are readily converted to the corresponding disulphides in the presence of mild oxidants (air, iodine, iron(III) salts).[73–75] The following conversions are notable:[18,19]



These reactions take place simply on exposing solutions of the mercaptan to air. 2-Mercapto-5-ethyl-3-thienylaldimine is oxidised to bis(3-cyano-5-ethyl-2-thienyl)disulphide in hydroxylic or aqueous alcohol media:[76]

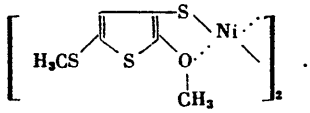


The formation of thienylsulphenyl chlorides from thiophenethiols and chlorine is synthetically very interesting:[14,62]



The reaction takes place in carbon tetrachloride. It is important that the 5-position is blocked. If not, the resulting sulfenyl chlorides react with the starting mercaptans at the unsubstituted 5-position with liberation of HCl. A variety of thienylthio derivatives may be obtained from thienylsulphenyl chlorides, the chlorine atom of which is very labile.

A number of transition metal complexes, e.g. (XVI), may be formed from thiophenethiols and their derivatives. The nickel chelate (below) is formed by heating nickel acetate with a methanolic solution of 2-methoxy-5-methylthio-3-thiophenethiol.[37]



Recently, the synthesis of titanocenethienyl and niobocenethienyl thienylmercaptides was reported,[77,78] and a study made of the stability of the latter towards I_2.

***

Thiophenethiols are an interesting class of heterocyclic compound from both the theoretical and practical viewpoint. From the volume of data published on these compounds in recent years, one can anticipate further developments in this field.

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