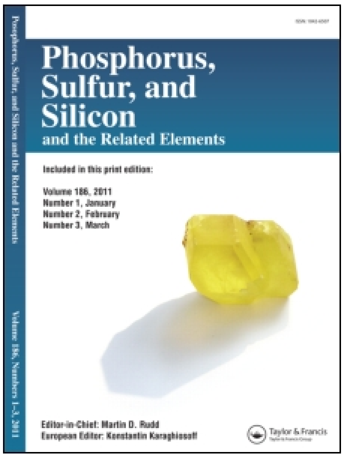

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The Pericyclic Reaction of the Derivatives of Dithiocarboxylic Acids

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THE PERICYCLIC REACTION OF THE DERIVATIVES OF DITHIOCARBOXYLIC ACIDS

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Abstract Review of pericyclic reactions of the derivatives of dithiocarboxylic acids. 1. Anionic [3+2]-cycloaddition of dithioate anions to activated multiple bonds. 2. The esters of dithiocarboxylic acids in cycloaddition and sigmatropic shift reactions.

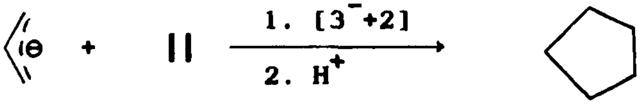
INTRODUCTION

The pericyclic reactions are of great importance both in theory and in practice among the reactions of organosulfur compounds. Without considering the trivial cases of influence of the substituents containing sulfur upon these reactions one could note different aspects of such using of organosulfur compounds. One of the above aspects consists in mechanical substitution of one or several sulfur atoms (as a rule, bivalent) into the general scheme of the system for which pericyclic reaction is possible [isoatomic (isoionic) substitution]. Different examples of the thio-Claisen rearrangement, [4+2]-cycloaddition of thiocarbonyl compounds, numerous photochemical pericyclic reactions of these systems could be mentioned.

It should be noted that including sulfur atom into carbon system of atoms, in general case, according to the experiment leads to lesser deformation of its pericyclic features as compared to replacement of carbon by oxygen which is the upper element of the sixth period. Double bond C=S possesses an increased reactivity resulting from its low stability (overlapping of $2p_\pi$–$3p_\pi$ orbitals). For instance, π-bond order of thioacetone is equal to 0.78, that of acetone being equal to 0.93.[1] The ionization potential of π-electrons of thioformaldehyde (11.76 eV) is more closely to that of ethylene (10.51 eV), than that of formaldehyde (14.5 eV).[2,3] The double bond C=S is much less polarized than C=O, according to ab initio calculations for thioformaldehyde the charge on the sulfur atom being equal to zero and carbon has small negative charge.[4] Under the electrocyclic reactions in contrast to purely carbon systems there is no necessity in turning of the C=S bond to 90°.

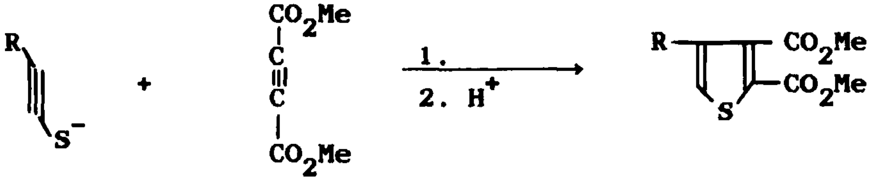
ANIONIC [3+2]-CYCLOADDITION OF DITHIOATE ANIONS TO ACTIVATED MULTIPLE BONDS

R.B.Woodward and R.Hoffmann were the first to point out the possibility of pericyclic reactions for charged systems. T.Kauffmann investigated the problem of ionic cycloaddition of allylic anions and its aza-analogues by general scheme [3^-+2].

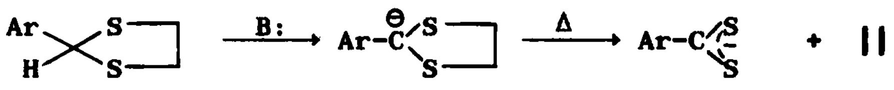


which is, principally, the convenient way to five-membered systems.[5] However, it turned out that these reactions are not of general character and are highly sensitive both to the nature of the second cycloaddend and to the nature of substituents, the alternative being the Michael addition to multiple bond.

Interesting examples of similar cycloaddition for 1-thiopropargyl anions were found.[6]

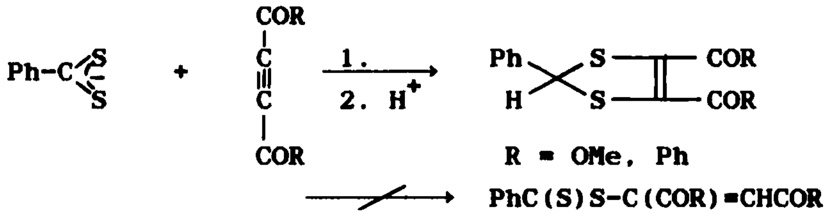


In 1979 M.P.Cava and co-workers suggested the method of synthesis of aromatic dithiocarboxylic acids based on the reaction of anionic [3+2]-cycloreversion of 1,3-dithioles under action of bases.[7]



This allowed us to hope that under the proper conditions and appropriate selection of addends it would be possible to carry out the reverse reaction of anionic cycloaddition with the participation of 1,3-dithiaallylic systems.

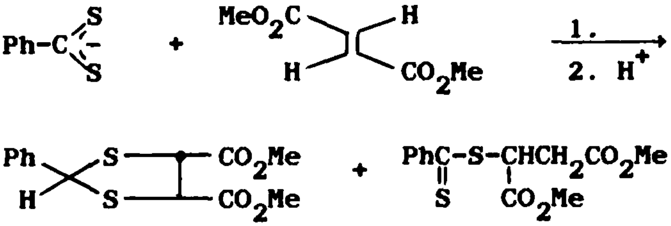
In fact, under the interaction of triethylammonium dithiobenzoate with ADM (dimethyl acetylenedicarboxylate) in acetonitrile or with dibenzoylacetylene in methanol the adducts are produced with good yield which have 1,3-dithiole structures rather than alternative structures of Michael addition products.



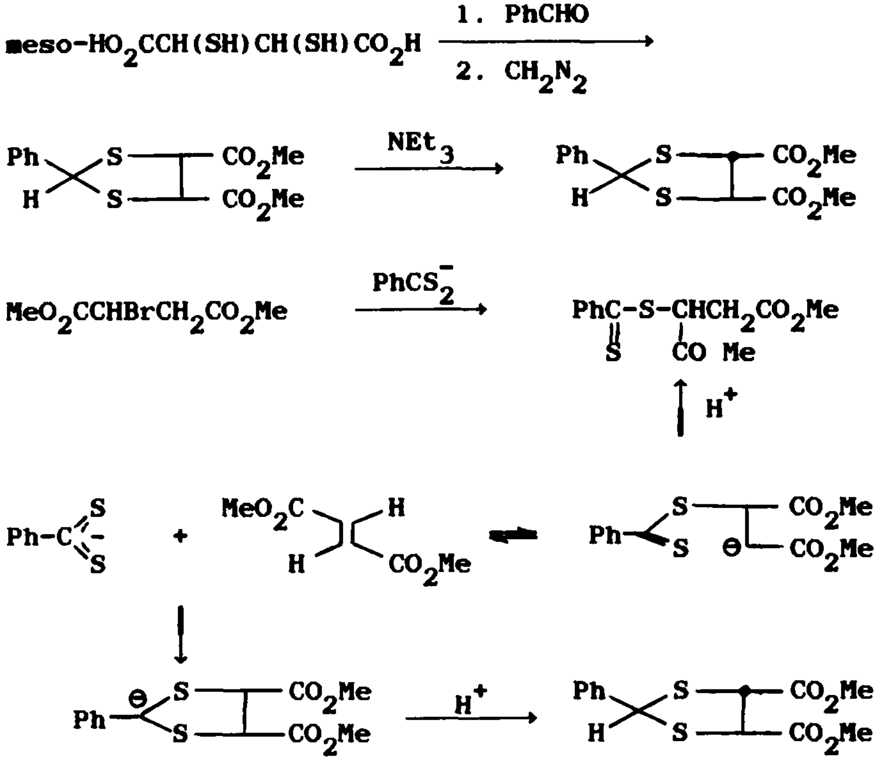
Mass spectra are characterized with intensive peaks $[M-H]^+$ and $[M-Ph]^+$. This type of fragmentation is not characteristic for esters of the dithiobenzoic acid. The isolated adducts have no characteristic absorption of $n-\pi^*$ transition of the dithiobenzoyl group (near 500 nm) in their electron spectra. This result in bright red colouring of dithiobenzoates. The qualitative chemical test for thiocarbonyl group, i.e., the oxidation by mercuric acetate in acetic acid with mercuric sulfide formation also gave the negative result. The signal of carbon atom connected with hydrogen is observed at 55.6 ppm (doublet, $^{1}J_{C-H}$ 160.0 Hz) upon ^{13}C NMR spectrum of 4,5-dimethoxycarbonyl-2-phenyl-1,3-dithiole (the value of chemical shift of corresponding proton in PMR spectrum being 6.06 ppm). This value of chemical shift in ^{13}C NMR spectrum agrees only with C^{2} atom of 1,3-dithiole structure since in the case of alternative dithioester structure the signal of double bond carbon atom would be observed significantly in a weak field in the range of approximately 140 ppm. Besides, ^{13}C NMR spectrum quite definitely results to equivalence of the both methoxycarbonyl groups: only one signal of groups C=O (161.2 ppm) and one signal of Me (53.0 ppm, $^{1}J_{C-H}$ 148.1 Hz) shall be observed.[8,9]

Successful cycloaddition of dithiobenzoate anion to electrophilic acetylenes made us to try to carry out the analogous reaction with activated alkenes. Red oil was isolated as a result of interaction of triethylammonium dithiobenzoate and dimethyl fumarate in acetonitrile at ambient temperature, yield being equal to 48%. The data obtained by element analysis and the presence of the molecular ion (m/z 298) in mass spectrum allow us to consider the structure of 1:1 adduct. The isolated adduct reacts with mercuric acetate to produce mercuric sulfide, the absorption in the range of $n-\pi^{*}$ transition of thiocarbonyl group being observed in its electron spectrum.

PMR spectrum unambiguously testifies to the formation of the both possible types of adducts during the reaction, i.e., dimethyl 2-thiobenzoylthiosuccinate and trans-4,5-dimethoxycarbonyl-2-phenyl-1,3-dithiolane in ratio 2:1 which was further confirmed by the data of NMR spectroscopy and gas-liquid chromatography (GLC).



We have fulfilled the synthesis of the both compounds by the below schemes.



Benzaldehyde reacts with meso-2,3-dimercaptosuccinic acid in the presence of anhydrous zinc chloride followed by methylation by diazomethane to produce the mixture of two diastereomeric cis-4,5-dimethoxycarbonyl-2-phenyl-1,3-dithiolane with cis- and trans-configuration of phenyl group in ratio 2:3 (in accordance with the PMR data). Isomerisation of cis-isomer into trans-isomer proceeds easily in the presence of triethylamine approximately for twenty-four hours.

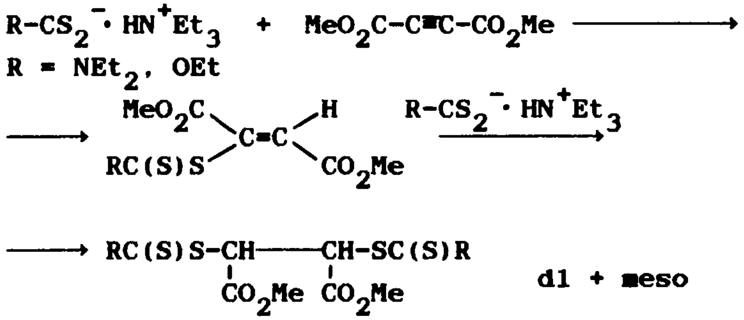
What is the formation mechanism of these adducts?

When both compounds are treated in acetonitrile with triethylamine in the cold, i.e., under the conditions of addition reaction the mutual conversion of these substances is not observed (test carried out by means of GLC) although deuterium exchange of all protons of 1,3-dithiolane ring and protons of CH_2-CH group of acyclic compound in the presence of methanol-d_4 takes place. This observation testifies to the fact that the mutual conversion of intermediate acyclic and cyclic carbanions is unlikely to be possible. That is why, in our opinion, the Michael nucleophilic addition and 1,3-dithiolane ring formation resulting from anionic [3+2]-cycloaddition should be regarded as two parallel reactions, the cycloaddition by itself being regarded as concerted process. Apparently, considering the case of interaction of dithiobenzoate anion with activated acetylenes one might assume the analogous concerted mechanism of cycloaddition although in this very case there are no arguments against stepped mechanism.

It should be noted that under the heating of the Michael adduct in acetonitrile solution in the presence of triethylamine the decomposition of the above dithioester take place with the formation of dimethyl fumarate and dithiobenzoate anion. The latter was converted into methyl dithiobenzoate upon the action of methyl iodide, reaction products being identified by means of GLC. The formation of the corresponding 1,3-dithiolane is observed which might have been the result of the subsequent interaction between dimethyl fumarate and dithiobenzoate anion by the scheme of cycloaddition. One couldn't observe the formation of the Michael adduct under the treating of 1,3-dithiolane with triethylamine within the temperature range from 20°C to 100°C, dithiobenzoate anion being the only to be discovered among the products of fragmentation.[9]

It would be highly tempting to use the revealed reaction of anionic cycloaddition for adjacent easily accessible analogues of dithiobenzoate anion which are dialkyldithiocarbamate and alkylxanthogenate anions. However, the radical's nature at the central atom of 1,3-dithiaallylic system exerted the determining influence upon the reaction character.

It was found that the formation of 1:1 adducts is observed under the slow adding of triethylammonium N,N-diethyldithiocarbamate and O-ethylxanthogenate used in equimolar amounts to ADM in acetonitrile, the temperature being equal to $-17^{o}C$. The combination of phisico-chemical methods showed these adducts to be formed as a result of the exclusively stereospecific Michael trans-addition of the corresponding anions to triple bond of ADM.



The question of adduct configuration is of great interest not only in itself. Usually, the addition of sulfur nucleophiles to activated multiple bonds proceeds by the scheme of trans-addition, although it is not always stereospecific. The reason for such non-stereospecificity might be both the isomerization of primarily formed adduct and realization of the other reaction mechanism. Thus, in our case one might assume the intermediate formation of unstable carbanions of 1,3-dithioles the cycloreversion of which followed by protonation would have produced the adducts of formal cis-addition to triple bond. To determine the configuration of 1:1 adducts which are trisubstituted olefines we made use of the additive scheme of calculation of vinyl proton chemical shifts of compounds with (Z)- and (E)-configuration. The above calculations unambiguously testify to (Z)-configuration of the adducts obtained. The acyclic structure of adducts is confirmed additionally by the fact that the adducts of ADM and corresponding dithioacids with composition 1:2 were obtained by using twofold amount of dithioates and ADM or under interaction between 1:1 adducts and corresponding salts. According to the analysis of PMR and ^{13}C NMR spectra these adducts are the mixtures of dl- and meso-forms of dimethyl 2,3-bis(N,N-diethylthiocarbamoylthio)- and 2,3-bis(ethoxythiocarbonylthio)succinates (the ratio of diastereomers approximately is 1.8:1 and 1.3:1 in the first and in the second case, respectively).[10]

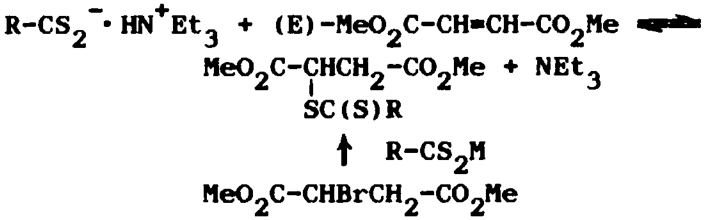
Also, we have investigated the interactions of acetylenes with only one electron-withdrawing substituents. We have used the example of methyl propiolate with triethylammonium dithiobenzoate, N,N-diethyldithiocarbamate and O-ethylxanthogenate. In these cases the character of multiple bond polarization was expected to promote the Michael addition of dithioate ions. The reaction was carried out in acetonitrile at the temperature -17°C in the presence of the acetic acid in equimolar amount which inhibits the futher conversion of primary products of addition.

$$RCS_2H + HC{\equiv}C\text{-}CO_2Me \longrightarrow (Z)\text{-}RCS_2\text{-}CH{=}CH\text{-}CO_2Me$$

$$R = Ph,\ NEt_2,\ OEt$$

The results of the analysis of PMR spectra of reaction mixtures allow us to state that in all cases irrespective of the radical nature added to the central carbon atom of 1,3-dithiaallylic system the Michael addition proceeds stereoselectively as trans-addition.

We also investigated the interaction of diethyl fumarate with triethylammonium N,N-diethyldithiocarbamate and O-ethylxanthogenate in methanol, the concentration of reactants being 0.2 M (25°C). We have found out that only the Michael addition could be observed under these conditions and the nascent triethylamine prevents the reaction to proceed to the end. In the first case the reaction is stopped with the accumulation of the reaction product in the mixture in the amount of 82%, in the second case - 67% (test carried out by means of GLC).

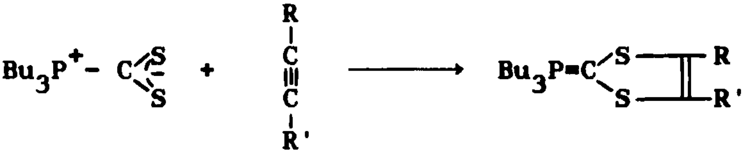


$$\begin{array}{l} R\text{-}CS_2^{-}\cdot HN^{+}Et_3 + (E)\text{-}MeO_2C\text{-}CH{=}CH\text{-}CO_2Me \rightleftharpoons \\ \qquad\qquad MeO_2C\text{-}\underset{\displaystyle SC(S)R}{\underset{|}{C}}HCH_2\text{-}CO_2Me + NEt_3 \\ \qquad\qquad\qquad\quad \big\uparrow\ R\text{-}CS_2M \\ \qquad\qquad MeO_2C\text{-}CHBrCH_2\text{-}CO_2Me \end{array}$$

R = NEt_2, OEt

When the pure adducts in methanol solution of the same concentration are treated by equimolar amount of triethylamine the equilibration is obtained with the contents of dimethyl fumarate in reaction mixtures approximately 7% and 21%, respectively.[10]

As found by H.D.Hartzler, the peculiar 1,3-dithiaallylic systems which can realize anionic [3+2]-cycloaddition are betaines produced as a result of interaction between phosphines and carbon disulfide.[11] The adduct of carbon disulfide and tributylphosphine reacts with acetylenes possessing electron-withdrawing substituents already at the temperature from -30° to 0°C, arylacetylenes slowly react at the ambient temperature and the acetylene itself reacts at the temperature higher than 100°C to produce the corresponding phosphoranes.[11]



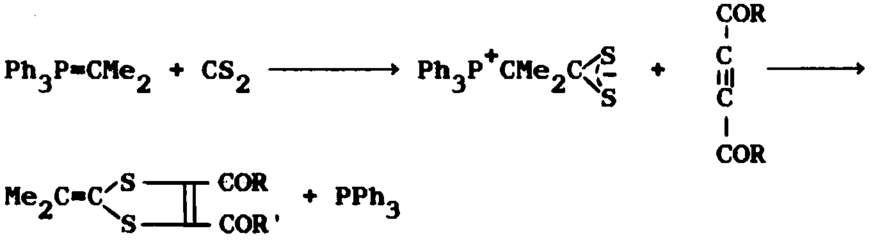
The phosphoranes produced could be further used for the fulfilment of different reactions, in particular, for the syntheses of tetrathiafulvalenes.

Formamidinium- and imidazolidiniumcarbodithioates similary react with twice activated acetylenes.[12]

It is interesting that dipolarophiles with activated double bonds (tetracyanoethylene, diethyl dicyanofumarate, dibenzyl azodicarboxylate) don't react.

The betaines of which 1,3-dithiaallylic anion system is separated from positively charged centre through one carbon atom were produced as a result of interaction of triphenylphosphine ylides with carbon disulfide.[13] In the case of disubstituted ylides the betaines which are stable enough were synthesized from isopropylidene and cyclohexylidene triphenylphosphoranes, other dialkyl and diaryl derivatives being thermally unstable.

We have assumed that 1,3-dithiaallylic system of α-(triphenylphosphonium)alkanedithiocarboxylates can react with dipolarophiles activated enough by substituents according to anionic [3+2]-cycloaddition scheme. The factor determining this path of the reaction is futher stabilisation of intermediate cyclic zwitter-ion in consequence triphenylphosphine fragment elimination and exocyclic double bond formation.[14]



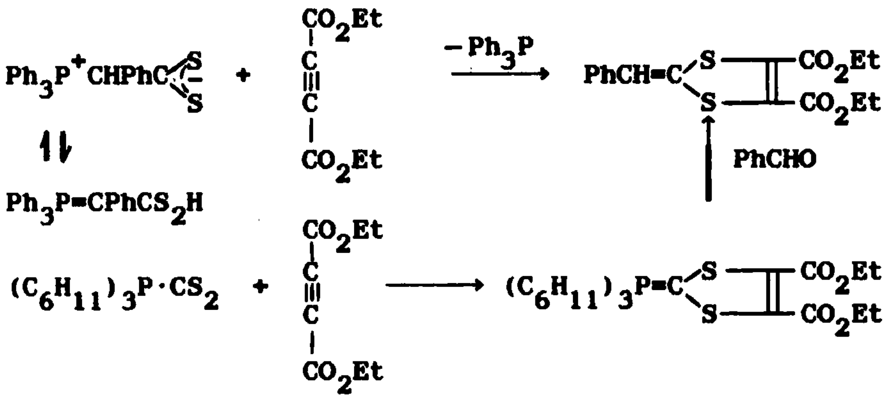
The most active addends as regards to

α-(triphenylphosphonium)alkanedithiocarboxylates turned out to be acetylenes twice activated by electron-withdrawing substituents: dimethyl and diethyl acetylenedicarboxylates and dibenzoylacetylene, the latter being reacted more vigorously. The reactions were carried out in acetonittrile solution whithin the temperature range of 0°-20°C, as a rule, the most stable α-(triphenylphosphonium)dithioisobutyrate being used as the second cycloaddend.

The separation of reaction mixtures is a highly difficult process on account of by-reaction of initial products with formed products. The initial betaine itself can decompose in reaction medium with the formation of 3,5-diisopropylidene-1,2,4-trithiolane, triphenylphosphine and its sulfide.[15] Apparently, triphenylphosphine formed can desulfurize both betaine and 2-isopropylidene-1,3-dithioles to produce triphenylphosphine sulfide. Besides, acetilenedicarboxylates and dibenzoylacethyiene are known to react with triphenyylphosphine to produce a wide range of products.

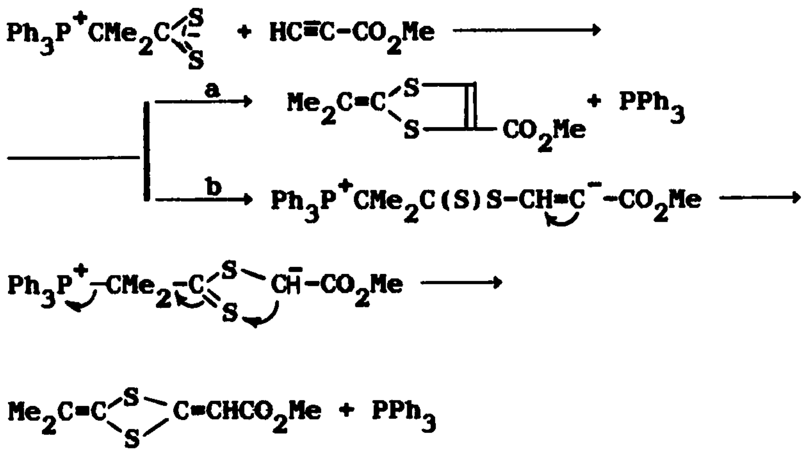
The structures of 1,3-dithioles obtained was proved by the combination of phisico-chemical methods, the most informative having be PMR and ^{13}C NMR spectra which point out to the equivalency of ester and benzoyl fragments and methyl groups in isopropylidene fragment.

α-(Triphenylphosphonium)dithiophenylacetate reacts with ADE to give much less yield (15%). The possibility of prototropic shift from carbon α-atom to sulfur atom must have been the reason for above phenomenon, the tautomer formed being inactive in cycloaddition reaction. In order to prove the structure the synthesis of 4,5-diethoxycarbonyl-2-benzylidene-1,3-dithiole formed the interaction of benzaldehyde with phosphorane produced by cycloaddition of the tricyclohexylphosphine-carbon disulfide complex to ADE was carried out.



While carrying out the reaction of α-(triphenylphosphonium)-dithioisobutyrate with such heterocumulenes as phenyl isocyanate, phenyl isothiocyanate, diphenylketene and carbon disulfide only 3,5-diisopropylidene-1,2,4-trithiolane, being the product of betaine self-condensation was isolated among reaction products. Compounds with double bond activated from both sides by electron-withdrawing substituents do not react. There are: esters of maleic and fumaric acid, N-phenylmaleimide.

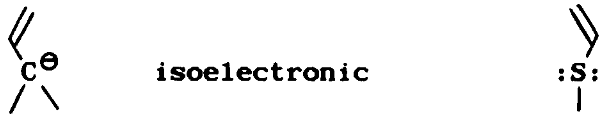
The reaction of α-(triphenylphosphonium)dithioisobutyrate with methyl propiolate, i.e., acetylene of which the triple bond is polarized by only one electron-withdrawing substituent proceeds in two directions.



Direction a) is the above reaction of anionic [3+2]-cyclioaddition with triphenylphosphine elimination and 2-isopropylidene-4-methoxycarbonyl-1,3-dithiole formation. Direction b) is an interesting approach to syntheses of asymmetrically substituted 1,3-dithietanes for which we suggest the following scheme of reaction: nucleophilic attack of dithioate anion at β-carbon atom of triple bond; unusual prototropic rearrangement with hydrogen atom migration from β- to α-carbon atom and, finally, thiophilic attack of carbanionic centre formed at C=S bond with triphenylphosphine elimination results in 2-isopropylidene-4-methoxycarbonyl-1,3-dithietane formation.

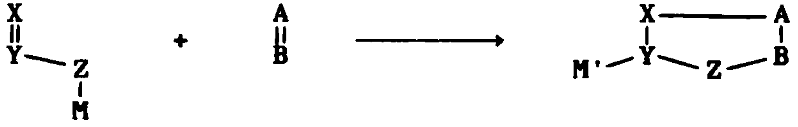
THE ESTERS OF DITHIOCARBOXYLIC ACIDS IN CYCLOADDITION AND SIGMATROPIC SHIFT REACTIONS

We have considered the above examples of anionic [3+2]-cycloaddition reaction when sulfur atom realized isoatomic (isoionic) substitution in allylic anion system. However, in principle, variant could be suggested when sulfur atom realizes isoelectronic substitution of carbanionic atom:



In this case the ylide is to be formed in the process of cycloaddition which will be stabilized by one or another way. One of these variants could be considered is the reactions of sigmatropic shift.

Cycloaddition reactions followed by sigmatropic shift are mentioned in literature rather seldom. In general aspect in the case of five-membered cycles formation the scheme of such reaction could be written as follows:

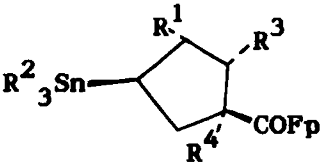


Particular examples of this type of reactions are known. In some cases migrant M has been already connected by σ-bond with Y atom.[16]

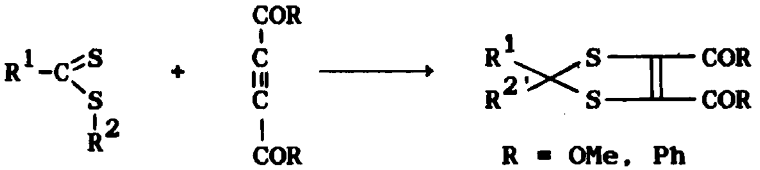
One could regard the reactions of ionic cycloaddition including anionic [3+2]-cycloaddition to be particular case of such reactions.

The reactions of η¹-allylic- and propargylic compounds of transition metals,[17] allylstannanes compounds[18] are related to above reactions.

$R^1CH{=}CHCH_2SnR^2{}_3 + R^3CH{=}CR^4COFp \longrightarrow$



We have discovered that allyl, propargyl and benzyl esters of dithiocarboxylic acids react with ADM or dibehzoylacetylene by the of cycloaddition followed by sigmatropic shift to produce 2,2-disubstituted 4,5-dimethoxycarbonyl(dibenzoyl)-1,3-dithioles[19-21].



R = OMe, Ph

The reaction proceeds in the absence of solvents at ambient temperature during several days. Monoactivated acetylenes are significantly less reactive, namely, esters of propiolic and phenylpropiolic acids, benzoyl- and 1-benzoyl-2-bromoacetylenes.[22] In these cases the reaction could be accelerated by high pressure (1000-1400 MPa).

In accordance with N.S.Zefirov and S.S.Tratch classification based on the formal-logical approach developed by them these are the simplest reaction of isodesmic process of sigmatropic oligoaddition descried as six-centered [2'+2+(1,1)] or eight-centered [2'+2+(1,3)] processes[23].

The question of radical R^2 migration character during 2,2-disubstituted 1,3-dithioles formation is of great interest. In principle, the migration can be realized by scheme both [1,2]- and [2,3]-sigmatropic shift.

On the basis of the experimental data obtained one could come to the following conclusion: in the simple cases of allylic and methyl substituted allylic groups the migration is realized by the scheme of [2,3]-sigmatropic shift. In particular, the reaction of prenyl ester of dithiobenzoic acid with ADM conducted without solvent and in the solvents of different character, namely: hexane, benzene, dichloroethane, acetonitrile is followed by the inversion of γ,γ-dimethylallylic group and results in 2-α,α-dimethylallyl-2-phenyl-4,5-dimethoxycarbonyl-1,3-dithiole formation. Also, a characteristic being the fact that propargyl dithiobenzoate produces 2-allenyl-2-phenyl-4,5-dimethoxycarbonyl-1,3-dithiole resulting from the reaction with ADM.

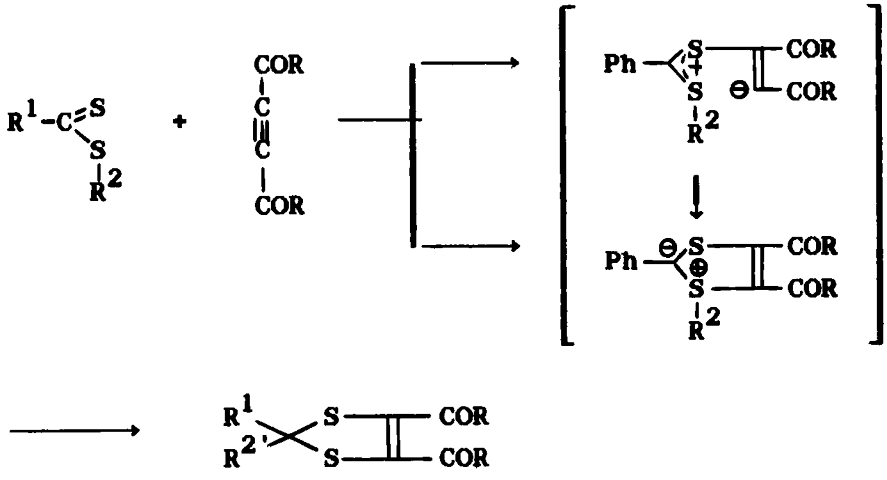
Of the other hand, the reaction of (E)-cinnamyl dithiobenzoate with ADM proceeds with formation of the mixture of 2-α-phenylallyl- and 2-(E)-γ-phenylallyl-2-phenyl-4,5-dimethoxycarbonyl-1,3-dithiole in the ratio 39:61, i.e., both with inversion and without inversion of cinnamyl radical resulting from [2,3]- and [1,2]-sigmatropic shift. The investigation of solvent effect showed that with solvent polarity increase the share of adduct increases which is formed with cinnamyl group inversion. The reactions α,γ,γ-trimethylallyl and β,γ,γ-trichloroallyl dithiobenzoates with ADM also proceed with cycloadducts mixture formation both with inversion of allylic groups and without them.

ADM reacts with p-substituted benzyl dithiobenzoates as easily as it does with allyl esters. As a result, 2-benzyl-2-phenyl-4,5-dimethoxycarbonyl-1,3-dithioles are formed, i.e., the reaction proceed with [1,2]-sigmatropic shift of benzyl group.

The migration of radicals in the course of this reaction is of intramolecular character. At least, the formation of products of cross reactions during the interaction of the mixture of two dithioesters with ADM is not observed.

In general terms, similar character of radical migrations reminds their behavior during sulfur ylide rearrangements.

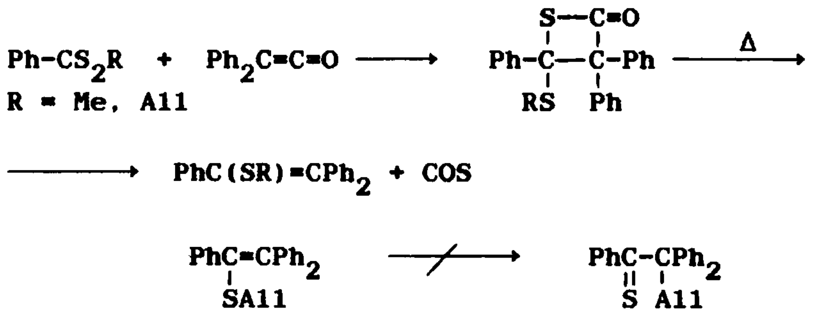
We assume that the above interaction of dithioesters with activated acetylenes is realized by the scheme according to which the cycloaddition precedes the sigmatropic shift.



To make a choice between two ways of cyclic ylide formation, i.e., concerted and stepped ones is not possible now, although calculations by FMO scheme are more favorable for the first of them.

For the purpose of further extension of the sphere of application of the above reaction were made to realize the interaction of allyl and benzyl O-ethylxanthogenates, N,N-diethyldithiocarbamates, and dibenzyl trithiocarbonate. Then, the varying of the reaction triad of atoms in dithiocarboxylates was carried out by replacing of sulfur atoms by other heteroatoms (nitrogen, oxygen) in different combinations, the central carbon atom was replaced by P=S group atom; allyl or benzyl radicals being regarded as a potentially migrating groups. All these attempts turned out to be failure.

Another approach in which we tried to extend the sphere of application of the above reaction consisted in varying of dipolarophiles reacting with dithioesters by the same scheme. However, such polarophiles were not discovered. Thus, the reaction of esters of dithiobenzoic acid with diphenylketene proceeds in other way, i.e., by scheme of [2+2]-cycloaddition with 2-alkylthio-3,3,4-triphenyl-2-thietanone formation.



$$Ph\text{-}CS_2R + Ph_2C{=}C{=}O \longrightarrow Ph\text{-}\underset{RS}{\overset{S\text{—}}{C}}\text{—}\underset{Ph}{\overset{C{=}O}{C}}\text{-}Ph \xrightarrow{\Delta}$$

R = Me, All

$$\longrightarrow PhC(SR){=}CPh_2 + COS$$

$$\underset{SAll}{PhC{=}CPh_2} \not\longrightarrow \underset{S\ \ All}{PhC\text{-}CPh_2}$$

Alkylthiotriphenylethylenes are obtained during the pyrolysis of the thietanones; allylthiotriphenylethylene doesn't enter the thio-Claisen rearrangement by reason of steric hindrance.[24]

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