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Reaction of Carbenes with Divalent Sulfur Compounds

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REACTION OF CARBENES WITH DIVALENT SULFUR COMPOUNDS

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(Received November 19, 1991)

The first extensive treatment of this subject by Ando¹ appeared about two decades ago and dealt primarily with stabilized sulfur ylides formed by a carbene-sulfide reaction and the different reaction pathways that followed. A more recent treatment by Anisimov and coworkers² focuses on the reaction of carbenes with unsaturated sulfides. The current endeavor will deal primarily with the reaction of carbenes with sulfides and polysulfides. Although literature prior to 1976 will be mentioned, emphasis will be on material published between 1976 and spring of 1991. Due to the bias already present in the literature, halo- and carboalkoxycarbenes form the core of the reactions. Other carbenes and carbenoid reactions are mentioned wherever appropriate.

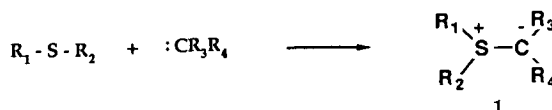
Key words: Carbenes, disulfides, sigmatropic rearrangements, Stevens rearrangement, sulfides, trisulfides.

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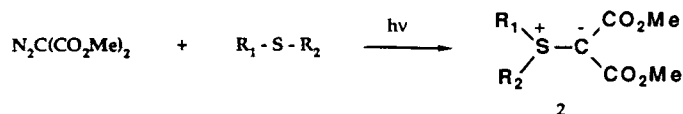
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1. REACTION WITH SULFIDES

Generation of a carbene in the presence of a sulfide yields the corresponding ylide **1**. If the carbenic carbon bears electron-withdrawing groups (e.g., carboalkoxy) that can



stabilize the developing negative charge, the ylide will be stable. Thus photolysis of dimethyl diazomalonate in the presence of alkyl and aryl sulfides gave the stable sulfonium bis carbomethoxymethylide **2**.³



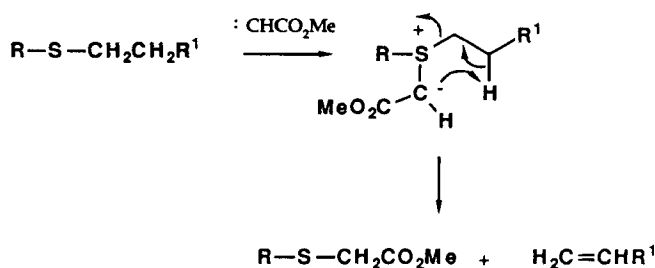
Once formed, the ylides can undergo three types of molecular rearrangements to furnish products. These rearrangements have often been utilized very successfully in organic synthesis. The three molecular rearrangements possible are (i) β -elimination, (ii) [2,3] sigmatropic rearrangement, and (iii) Stevens rearrangement.

Regarding the multiplicity of the carbenes involved in the reactions to be described, all involve singlet carbenes giving ylides directly. Only in the reaction of diphenylcarbene with sulfides and disulfides^{4a} does the electron paramagnetic spectrum indicate the reaction to proceed partially or completely by a radical-like mechanism. This, and the reaction of diphenylcarbene with 1,2,3-benzothiadiazole,^{4b,4c} are the only examples where unequivocal evidence has been advanced for triplet carbenes.

1.1. β -Elimination

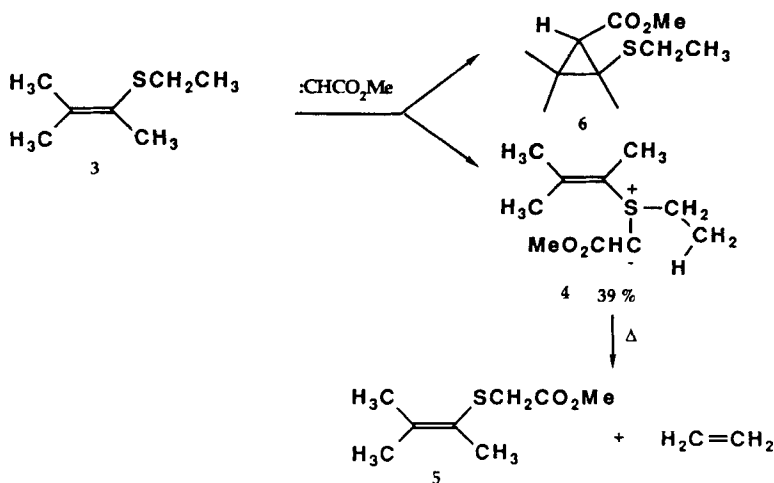
Compared to the other two rearrangement modes β -elimination traverses a higher energy surface and hence is observed when the molecule has no other choice. This accounts for the relatively few examples of this reaction.

Ando and coworkers reported in 1971⁵ that the reaction of carbomethoxycarbene with alkyl sulfides bearing a β hydrogen gave the alkene and $\text{RCH}_2\text{CO}_2\text{Et}$. A cis eliminative decomposition of the initially formed ylide via a five-membered transition state (Scheme 1) explained the formation of the observed products.

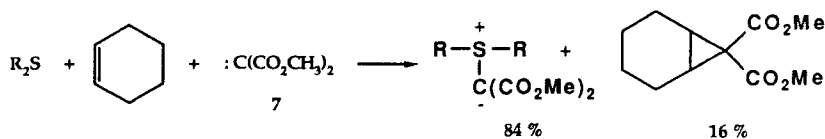


Scheme 1.

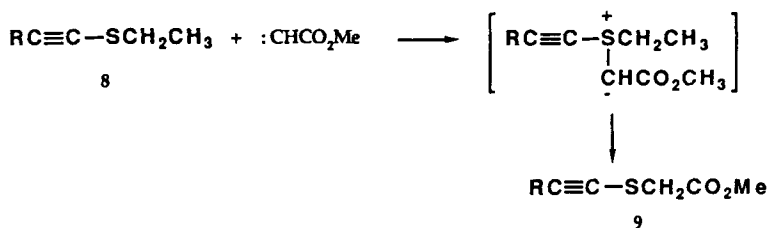
With the vinyl sulfide **3** as a substrate, where reaction can occur at both the double bond and/or the sulfur atom, it was found that reaction at the sulfur atom was favored by a factor of 7 to give the rearranged vinyl sulfide **5** via the ylide **4**. A competition



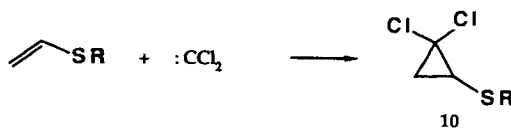
experiment between the reactivity of alkyl sulfides and cyclohexene towards the electrophilic dimethylmalonylcarbene **7** further illustrated the wide difference in reactivity.⁶



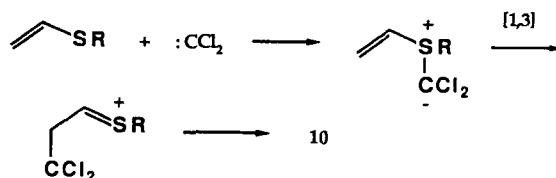
Alkynyl sulfides (**8**), like vinyl sulfides, react with carbomethoxycarbene to afford the sulfide **9**, probably via the acetylenic sulfonium ylide, followed by elimination of alkene.⁶



It is important to note that dibromo- and dichlorocarbene react with vinyl sulfides to give exclusively the corresponding dihalocyclopropanes (**10**).⁷



That the cyclopropanes are not formed by a direct insertion of carbene into the double bond was evident from the fact that vinyl sulfides add dichlorocarbene 5 times faster than vinyl ethers.⁸ Thus direct participation of sulfur is clearly evident and the mechanism that has been proposed (Scheme 2) takes this into account.



Scheme 2.

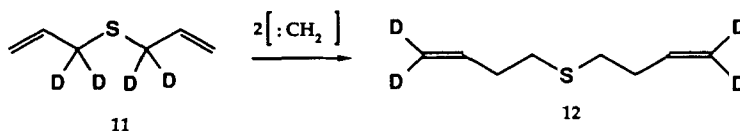
Dialkyldithioketals can be readily converted to vinyl sulfides by treatment with "methylene" generated from diethylzinc and diiodomethane.⁹

1.2. [2,3] Sigmatropic Rearrangement

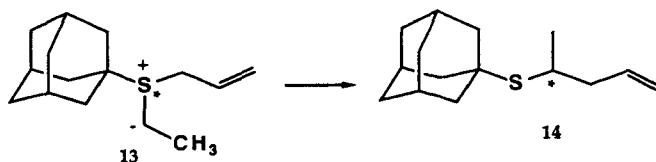
Allyl sulfides react with carbenes to give the corresponding sulfur ylid which invariably undergoes a [2, 3] shift to furnish new sulfides. This orbital symmetry-allowed operation proceeds, as expected, with complete allylic inversion which was demonstrated¹⁰ clearly



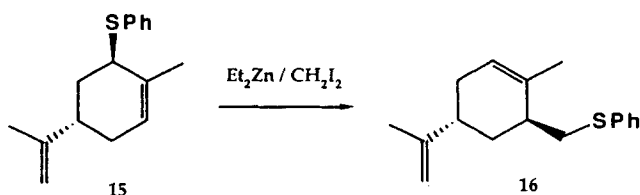
by the rearrangement of the tetradeuterated sulfide **11**. Reaction of **11** with 2 equivalents of methylene gave the sulfide **12** with all the deuterium residing on the terminal olefinic carbon.



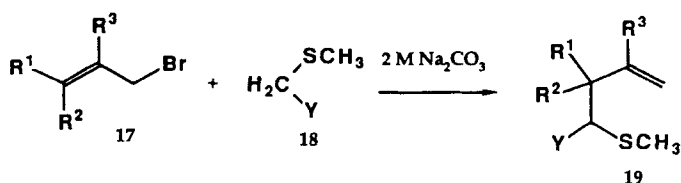
This symmetry controlled rearrangement has been effectively used to transfer a C-S chirality to C-C chirality. Thus the adamantyl substituted ylid **13** undergoes a [2, 3]



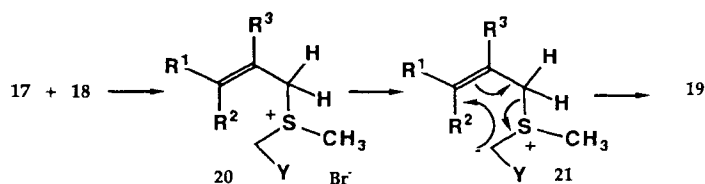
shift to provide **14** with 94% retention of optical activity.¹¹ More recently a modified Simmons-Smith reaction has been used to transform allyl to homoallylic sulfides.¹² Optically active sulfide **15** underwent the manipulation with complete retention to give **16**.



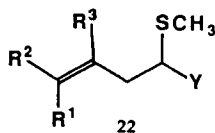
The reaction of allyl bromides and sulfides in the presence of mild bases (this process can be viewed as a carbene synthon strategy) affords products of the type **19** arising from [2, 3] sigmatropic rearrangement.¹³ The authors have suggested the initial formation of



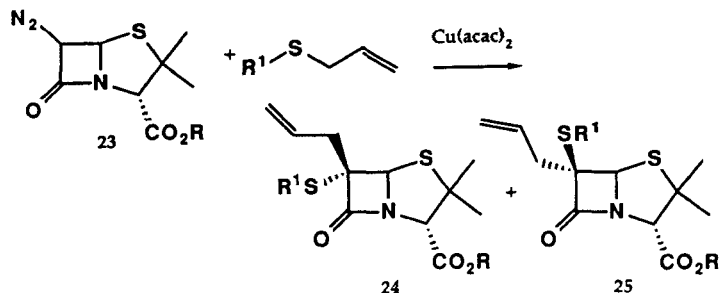
the sulfonium salt **20** which undergoes deprotonation to give **21**, followed by a [2, 3] sigmatropic rearrangement to give the product.



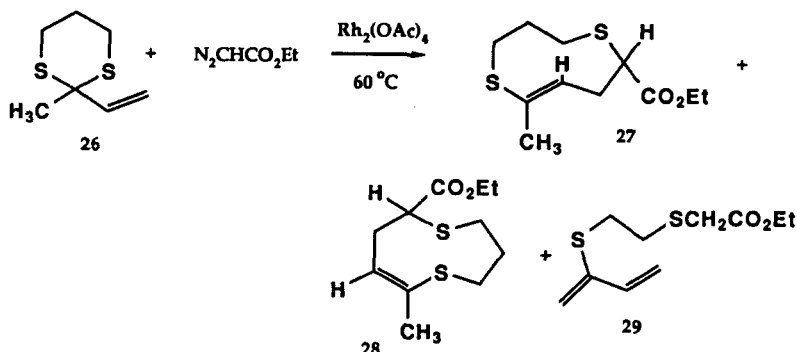
Direct $\text{S}_{\text{N}}2$ displacement by the anion of **18** was dismissed by the following observations: (i) **18** is too weak an acid to be deprotonated by Na_2CO_3 . (ii) Even if the carbanion of **18** is formed in equilibrium, the regioselectivity observed in the reaction cannot be explained since the sodiomalonic ester reacts with crotyl halides exclusively by $\text{S}_{\text{N}}2$ attack. (iii) It was observed that the sodio derivative of **18** ($\text{Y} = \text{COPh}$) affords a mixture of **19** and **22**.



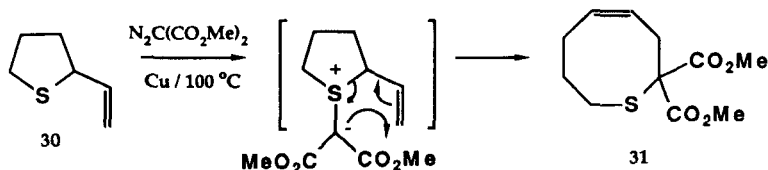
In recent years the use of carbenoids (metal complexed carbenes) to form sulfur ylids has gained prominence. Reaction of 6-diazopenicillanates (**23**) with allyl sulfides and selenides in the presence of catalytic amounts of $\text{Cu}(\text{acac})_2$ gave moderate yields of the 6-substituted 6-allylpenicillanates **24** and **25** with **25** as the major product.¹⁴ Absence of crossover products when isomeric allyl sulfides were used indicated that the [2, 3] sigmatropic rearrangement was the exclusive mode of product formation.



Using rhodium acetate as the catalyst, Doyle and coworkers¹⁵ have shown that 2-allyldithianes such as **26** undergo reaction with diazoacetates to provide the nine-membered ring compounds **27** and **28** regioselectively. Some of the acyclic counterpart **29** is also formed.



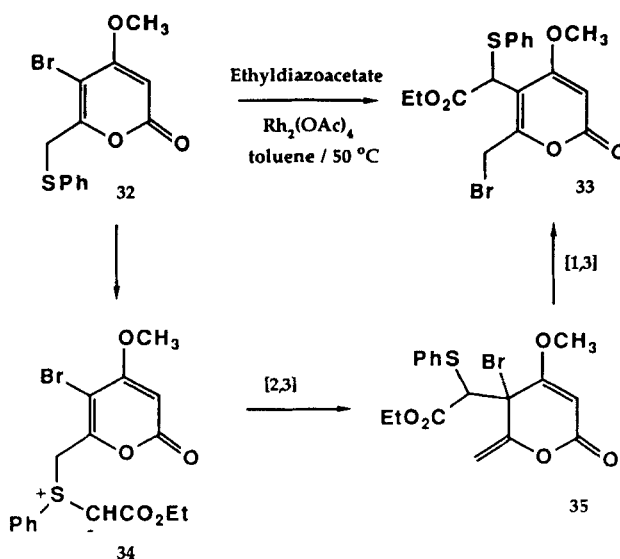
The conversion of five-membered into eight-membered heterocycles via ring expansion by [2, 3] sigmatropic shift has been studied extensively by Vedejs and coworkers. Thus, treatment of **30** with dimethyl diazomalonate and copper bronze in toluene at 100°C gave the *cis*-thiacyclooctene **31** as the only isolable product.¹⁶ Since this carbenoid approach gave very low yields with other diazomalonates and could not be used for



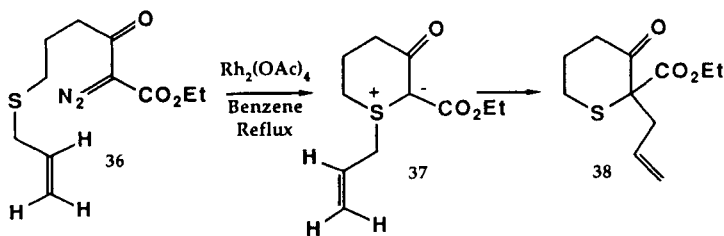
other substrates, sulfonium salts (prepared by direct alkylation of sulfides) were converted to the ylides by strong bases (e.g. Na_2CO_3 and LDA).¹⁷

During the same period a similar approach was developed independently by Cere and coworkers in Italy.¹⁸ The starting sulfur ylides were all generated by proton abstraction from sulfonium salts with strong bases.

Treatment of the pyrone **32** with diethyl diazoacetate under rhodium(II) acetate catalysis gave **33**.¹⁹ This one-pot transformation is the overall result of a [2, 3] sigmatropic shift of the ylid **34** to give **35** which, under the reaction conditions, rearranges via a [1, 3] shift of a bromine atom to the more stable tautomer **33**.

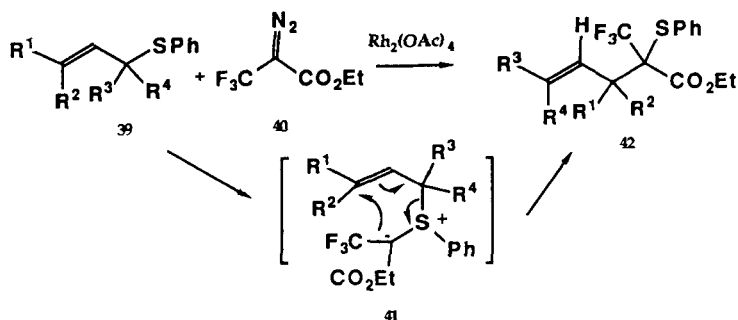


Rhodium carbenoid mediated cyclization of sulfonium ylides has been exploited elegantly by Moody and coworkers for the synthesis of 6- and 7-membered heterocycles.²⁰ Heating the diazo allyl sulfide **36** with a catalytic amount of $\text{Rh}_2(\text{OAc})_4$ in boiling benzene gave the rearranged thiopyran **38** in 59% yield.

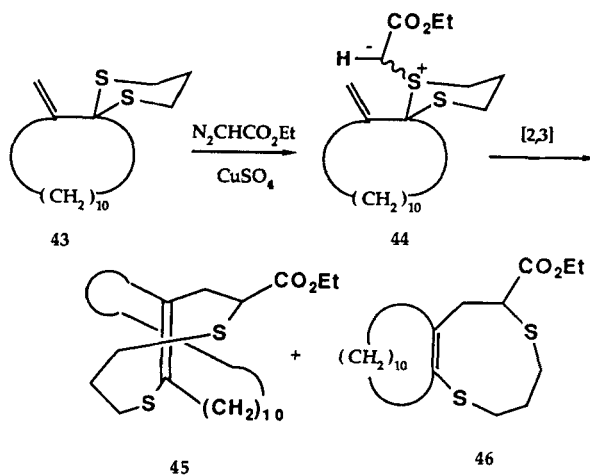


The intermediate sulfonium ylid **37** was not detected. Seven-membered heterocycles were readily synthesized by the same methodology.

With the [2, 3] sigmatropic rearrangement the biologically important trifluoromethyl group can be readily introduced.²¹ Treating an allyl sulfide **39** with the carbene precursor ethyl 3,3,3-trifluoro-2-diazopropionate **40** in the presence of rhodium(II) acetate gave a quantitative yield of the trifluoromethylated product **42** resulting from the expected [2, 3] sigmatropic shift of the sulfur ylid **41**. As shown by the authors a large variety of substituents in the sulfide can be tolerated in this reaction sequence.

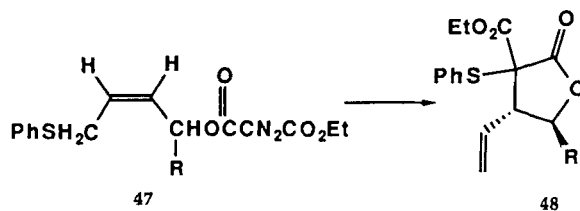


Nickon and coworkers have applied this strategy to a spirocyclic system for the synthesis of betweenanes.²² Treating the spirocyclic dithioketal **43** with diethyl diazoacetate in the presence of CuSO₄ gave the ylid **44** which gave the ring enlarged isomers **45** and **46** in a 4:1 ratio via a [2, 3] sigmatropic process.

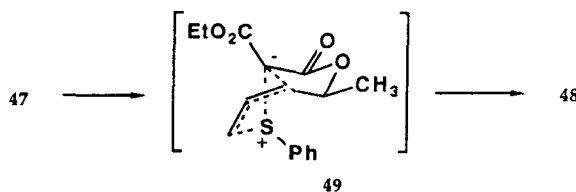


By incorporating the sulfide moiety, the carbenic center, and the ene system in an acyclic substrate Kido and coworkers were able to synthesize a variety of vinyl substituted butyro- and valerolactones in a stereoselective manner.²³ When **47** was heated in boiling

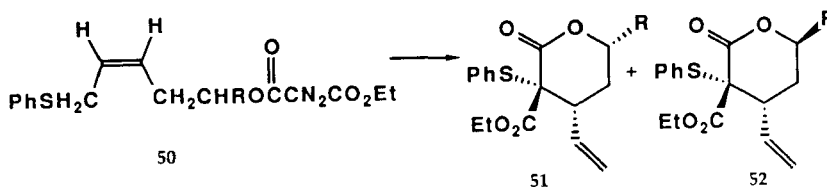
benzene with rhodium(II) acetate the [2, 3] rearrangement proceeded in a stereoselective fashion giving the butyrolactone **48** (52% yield). Neither insertion nor fragmentation



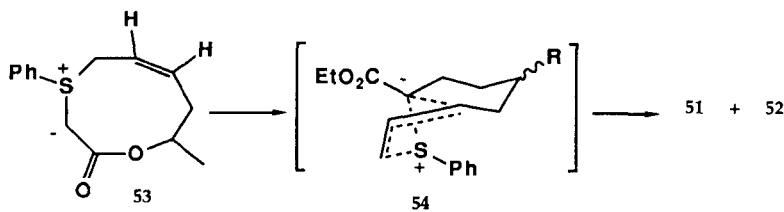
products were observed. The energetically most favorable conformation during bond rearrangement would be as shown in **49** which leads to the stereochemistry as obtained.



Similarly when **50** was treated with rhodium(II) acetate in boiling benzene the diastereoisomeric valerolactones **51** and **52** were isolated in a ratio of 4 : 1

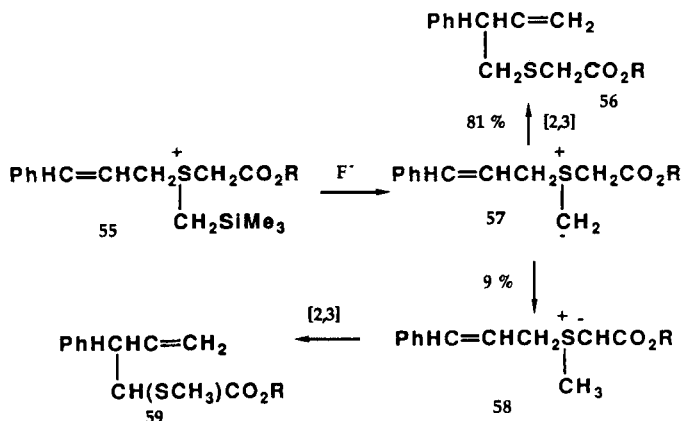


Formation of the minor isomer **52** indicates that the nine-membered cyclic allylsulfonium ylid **53** rearranges through a transition state **54** that is less rigid than **49**.

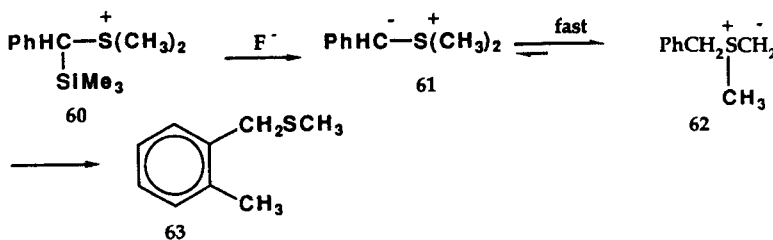


Apart from the reaction of a carbene with divalent sulfur and the action of strong bases on a sulfonium salt, sulfur ylids have been generated recently from (trimethylsilylmethyl-

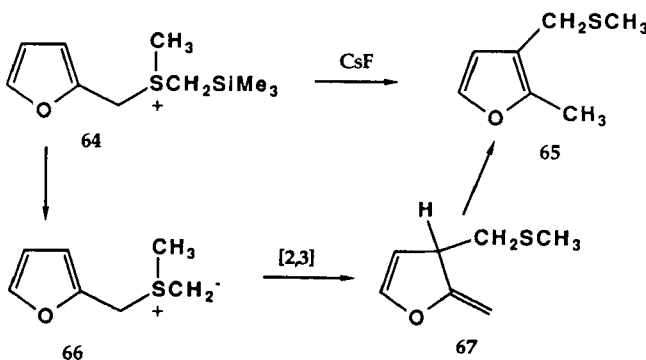
sulfonium salts by fluoride ion induced desilylation.²⁴ Thus the reaction of **55** with fluoride ion gave 81% of **56** derived by a [2,3] sigmatropic shift from the kinetic ylid **57** even though a more stabilized ylid **58** could have been produced by proton transfer.



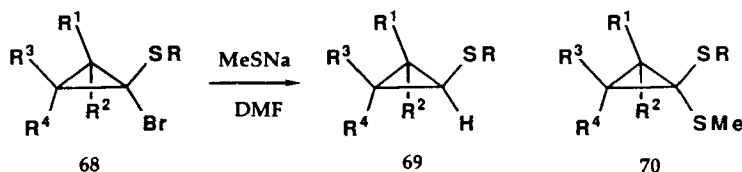
On the other hand, Padwa and Gasdaska have shown²⁵ that the initially formed sulfur ylid **61** (formed by fluoride ion displacement from **60**) rapidly rearranges to the more stable ylid **62** which undergoes a facile [2,3] sigmatropic shift to give **63** as the only



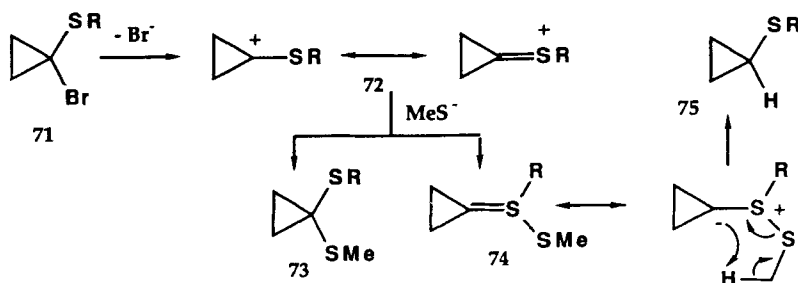
isolable product. This mechanistic postulate was supported by desilylation of labeled precursors. Similarly, the reaction of the 2-furfurylsulfonium salt **64** with CsF gave the **65** as the major product.



When 1-bromocyclopropyl methyl sulfides **68** are treated with sodium methanethiolate the substrates are quantitatively converted into cyclopropyl sulfides **69** and the 1,1-bis(alkylthio)cyclopropanes **70** in a ratio of 20:1.²⁶ A detailed kinetic analysis together



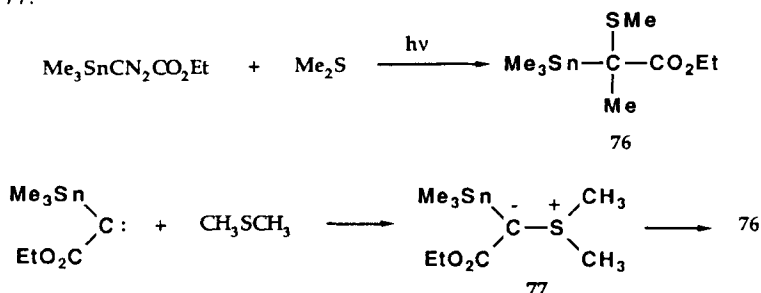
with extensive labeling studies suggested that the most probable mechanism leading to the reduct product **75** involved a [2, 3] sigmatropic shift of the disulfur ylid **74**, formed by attack of methanethiolate anion on the sulfur of the ambidentate cation **72**.



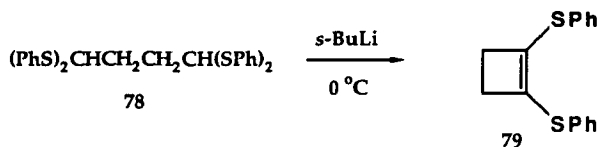
1.3. Stevens Rearrangement

In 1932 it was shown that heating phenylmethylbenzylsulfonium bromides gave α -methylthio- β -phenylpropiophenone, probably via an ylid intermediate.²⁷ About thirty years later Ando and coworkers carried out a detailed study of this rearrangement. Based on kinetic analysis and CIDNP spectroscopy they were able to show that the reaction proceeded through a homolytic dissociation followed by a [1, 3] migration. In general the Stevens rearrangement is limited in scope and only a few recent examples of this pathway can be found.

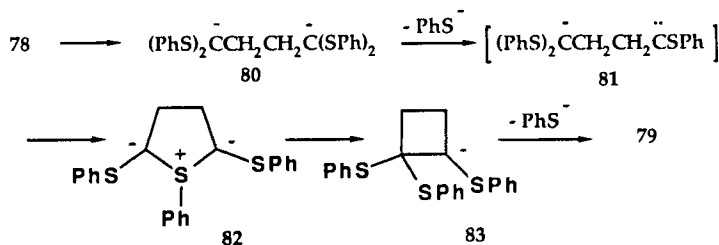
Irradiation of ethyl trimethylstannyldiazoacetate in excess dimethyl sulfide gave **76** as the major product.²⁸ The formation of **76** was explained by a Stevens rearrangement of the ylid **77**.



Ring closure reactions of dianions from bis(diphenyl thioacetals)²⁹ have been proposed to involve a Stevens rearrangement as the key step. The reaction of **78** with 2 eq of *sec*-butyllithium gave a 40% yield of the cyclobutene **79**. By labeling the two phenyl rings



of one of the thioacetal functions of **78** the authors were able to distinguish between the two quenching pathways available for the carbene **81** formed from the dianion **80**. The results indicated that **81** forms the ylid **82** which undergoes a Stevens rearrangement to **83**. Loss of a benzenethiolate anion from **83** yields **79**.

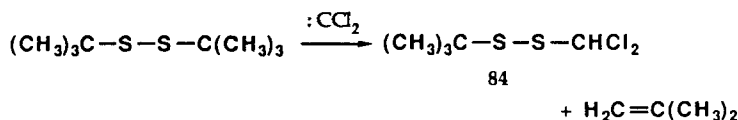


2. REACTION WITH POLYSULFIDES

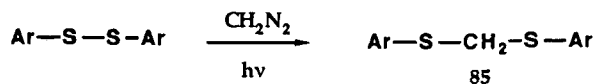
In contrast to the reaction of carbenes and carbenoids with sulfides the reaction with polysulfides has been investigated in much more limited fashion. With a renewed interest in the synthesis and properties of polysulfides one expects more research activity in this area in the near future. During the literature search it was surprising to find that no attempt has been made to let a carbene react with S_8 !

2.1. Disulfides

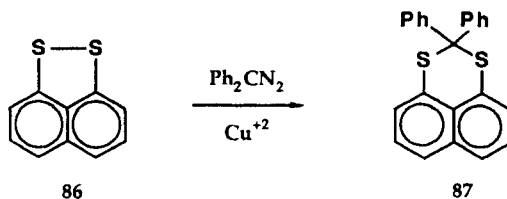
In the earliest study in this area, Searles and Wann found that the reaction of dichlorocarbene with disulfides led to dichloromethylalkyl disulfides (**84**) and alkenes.³⁰



Photolysis of diazomethane in the presence of a diaryl disulfide resulted in the insertion of methylene in the S-S bond.³¹

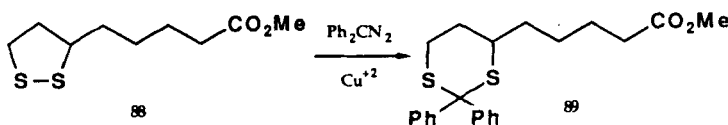


A little later Tamagaki and Oae³² showed that the reaction of 1,2-dithiaacenaphthene (**86**) with diaryldiazomethane in the presence of copper salts gave the corresponding insertion product.

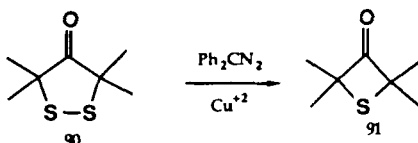


Field and Banks have carried out a systematic study of the insertion of carbenes into disulfides.³³ One interesting observation was the inability of dichlorocarbene to react with diaryl disulfides.

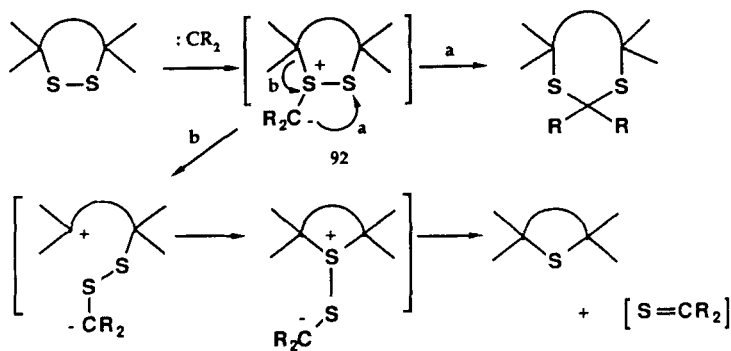
More recently Ando and coworkers have studied the reaction of cyclic disulfides with carbenes generated both photochemically and thermally.³⁴ This study showed that sterically uncrowded disulfides (e.g. **88**) reacted with carbenes



to give the corresponding 1,3-dithianes **89** via S-S insertion. In contrast sterically crowded disulfides (e.g., **90**) gave **91** via selective desulfurization.

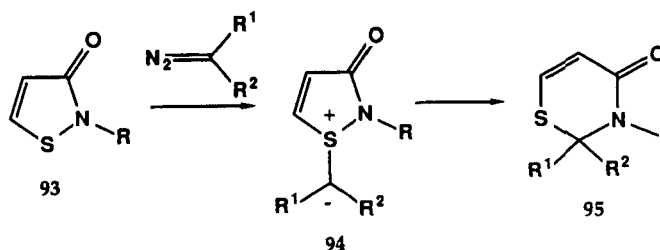


The authors have proposed that these reactions involve the sequence of steps shown in Scheme 3. The sulfur ylid **92** can rearrange by carbon sulfur attack on sulfur, followed by S-S bond cleavage (path a) to give insertion or by C-S bond cleavage (path b) to give a tioketone extrusion. In a crowded ylid path b becomes predominant.



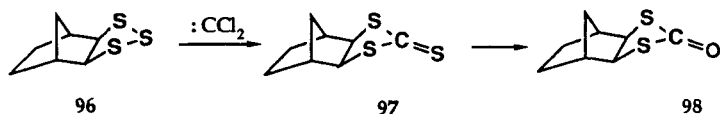
Scheme 3.

Insertion into an S–N bond has been reported. Rhodium catalyzed reaction of diazo compounds with 2-substituted isothiazol-3(2*H*)-ones, **93**, gave the ring-expanded six-membered product **95** via the intermediate sulfonium ylid **94**.³⁵

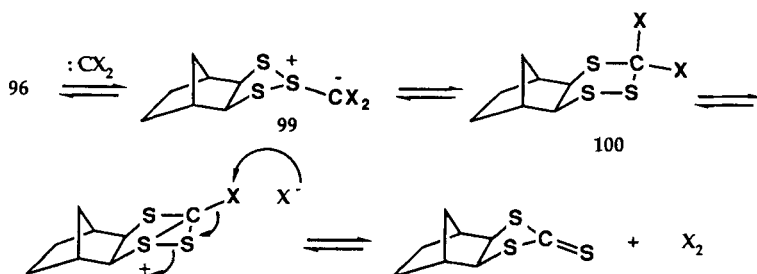


2.2. Trisulfides

In the only example involving a trisulfide, the reaction of *exo*-3,4,5-trithiatricyclo-[5.2.1.0^{2,6}]decane (**96**) with dichlorocarbene (generated under phase transfer conditions) gave the trithiocarbonate **97** which underwent further reaction to give the thionocarbonate **98**.³⁶ A similar reaction was observed when the reaction was performed with dibromocarbene. The formation of **97** from **96** was explained by a reaction pathway



(Scheme 4) involving the ylid **99** which undergoes ring expansion to **100** followed by ring contraction with the expulsion of a halogen molecule. In the dibromocarbene reaction the bromine generated was trapped *in situ* with cycloheptene.



Scheme 4.

CONCLUSION

It is clearly evident from this review that the reaction of carbenes (and carbenoids) with sulfides has received much greater emphasis over the years compared to that of polysulfides. With the renewed interest in the chemistry of polysulfides, it is hoped that in the

future a systematic investigation of polysulfides with carbenes will be carried out. If the past is any indication then one can be certain that the future holds a great many synthetic and mechanistic surprises.

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