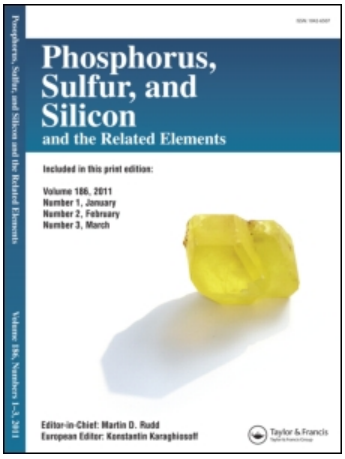


## Phosphorus, Sulfur, and Silicon and the Related Elements

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# Dithioesters in Organic Synthesis

Andre Thuillier[a]
[a] Laboratoire de Chimie des Composes Thioorganiques (Equipe de Recherche associee au CNRS n° 391), Universite de Caen, Caen, France

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# DITHIOESTERS IN ORGANIC SYNTHESIS

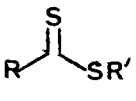
ANDRE THUILLIER
Laboratoire de Chimie des Composes Thioorganiques
(Equipe de Recherche associee au CNRS n° 391),
Universite de Caen, 14032 Caen, France

Abstract: After a short survey of the methods for the synthesis of dithioesters, reactions of these compounds with Grignard reagents, of their enethiolates, of α-oxodithioesters, conjugated and α-oxoketene dithioacetals as well as of N-phenyl-imido thioesters are discussed in detail. Applications of these reactions in syntheses of natural products are given.



## INTRODUCTION

Over the past two decades a large number of synthetic methods involving thioorganic compounds as intermediates for carbon-carbon bond formation have been published [1,2]. Carbanions stabilized by one or two adjacent sulfur atoms appeared particularly useful, one of the most significant examples being the use of 1,3-dithianes as masked carbonyl compounds, allowing an inversion of polarity of a carbonyl group (umpolung)[2]. I shall present here various methods allowing the formation of carbon-carbon bonds, using dithioesters 1 and the corresponding enethiolates 2 as well as some closely related compounds such as ketenedithioacetals 3, β-oxodithioesters 4, α-oxoketenedithioacetals 5 and N-phenylimidothioesters (thioimidates) 6, all readily available.



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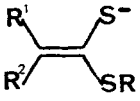

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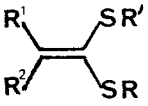

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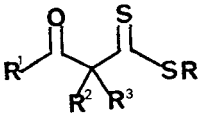

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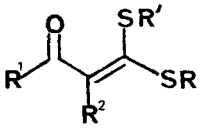

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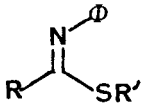

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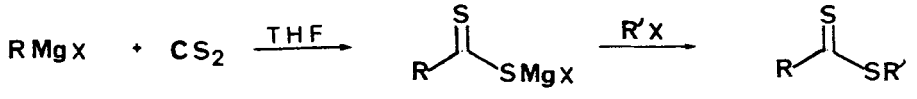
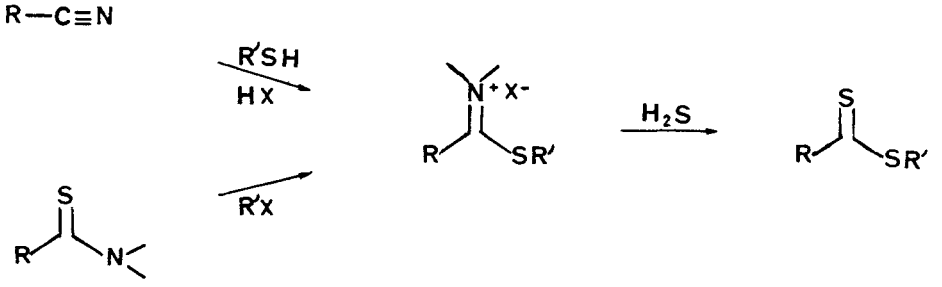
## 1. DITHIOESTER SYNTHESES

Scheithauer and Mayer's comprehensive book on thio and dithiocarboxylic acids and their derivatives [3] and a recent review [4] cover exhaustively synthetic methods for dithioesters and only some of the main routes to these compounds are mentioned below:

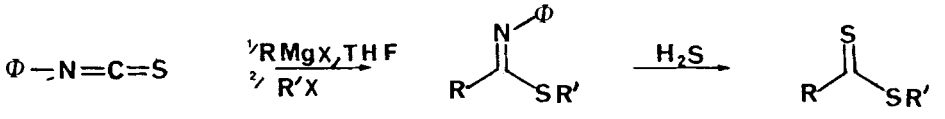
Addition of Grignard reagents to carbon disulfide followed by alkylation; this old reaction has been very much improved by the use of THF as solvent [5,6] or copper (I) catalysis [7].



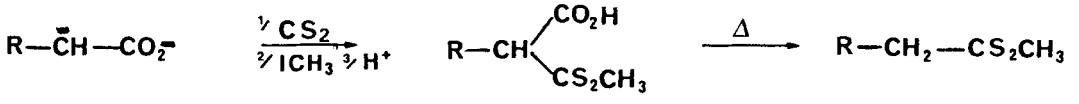
Sulfhydrolysis of (1-alkylthio-alkylidene) ammonium salts obtained either by addition of thiols to nitriles or by alkylation of thioamides [8,9]



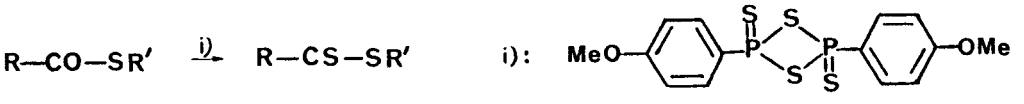
Sulfhydrolysis of N-phenylimidothioesters prepared by addition of organometallic reagents to phenylisothiocyanate and alkylation [10].



Addition of carbon disulfide to the dianion of a carboxylic acid followed by alkylation and decarboxylation [11].



Sulfuration of S-alkyl thioesters by Lawesson's reagent [12].

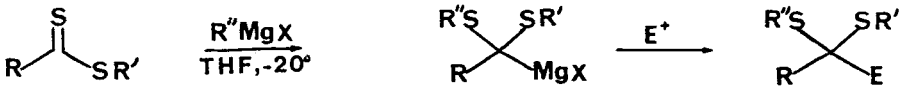


Sulfuration of carboxylic acids with Davy's reagent [13]

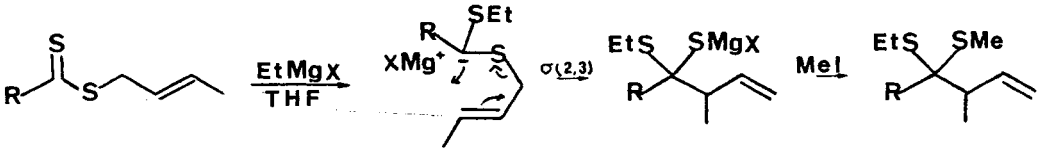
This one-step conversion of carboxylic acids into dithioesters is particularly convenient.

## 2. THIOPHILIC OR CARBOPHILIC ADDITION OF GRIGNARD REAGENTS ON DITHIOESTERS: APPLICATIONS

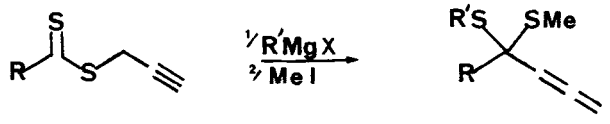
In THF as solvent, under controlled temperature (~below $-15^{o}C$) alkylmagnesium halides (except methylmagnesium halides) were found to give exclusively a thiophilic addition on the thiocarbonyl group of dithioesters; the resulting megnesiodithioacetal can be hydrolysed, alkylated [14], or condensed with other electrophiles [15] ($R^1COR^2$, ClCOOEt, $CO_2$, $R_2NCHO$).



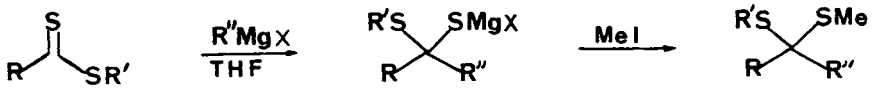

As the dithioacetal group is easily converted into a carbonyl or a methylene group, the starting dithioester can be considered as equivalent to the synthons: R-C=O and R-$CH_2$. Starting from an allylic dithioester, the thiophilic addition was followed by a [2,3] sigmatropic shift and the dithioacetal of a β-unsaturated ketone was obtained after alkylation at sulfur as in the following example [16]:



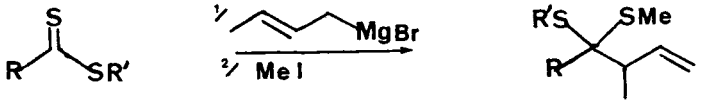
Olsson used this sequence to prepare allenic dithioacetals from propargylic dithioesters [17].




In contrast with the thiophilic addition of alkyl Grignard reagents, unsaturated Grignard reagents (allylic, propargylic, benzylic or vinylic) reacted with dithioesters by carbophilic addition [18,19].



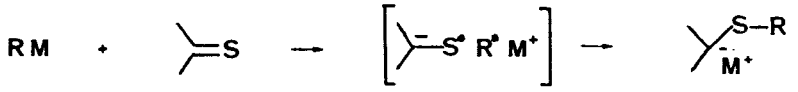

With allylic Grignard reagents inversion of the allylic chain occured




By carrying out cross reactions with allylic dithioesters we were able to show that the S-allylic chain of such compounds is not involved in the process and a thiophilic addition followed by a [2,3] sigmatropy could be ruled out [18]; a $S_N2'$ mechanism was proposed. The carbophilic addition allows the use of dithioesters as R-$\overset{+}{C}$=O and R-$\overset{+}{C}H_2$ synthons.

The difference of reactivity between saturated and unsaturated organometallics towards a thiocarbonyl group has been interpreted by Fukui [20] and the thiophilic addition mechanism has been discussed in terms of radical reactions [21,22,23]; it has been suggested that a charge transfer complex formed in a solvent cage underwent radical

coupling:

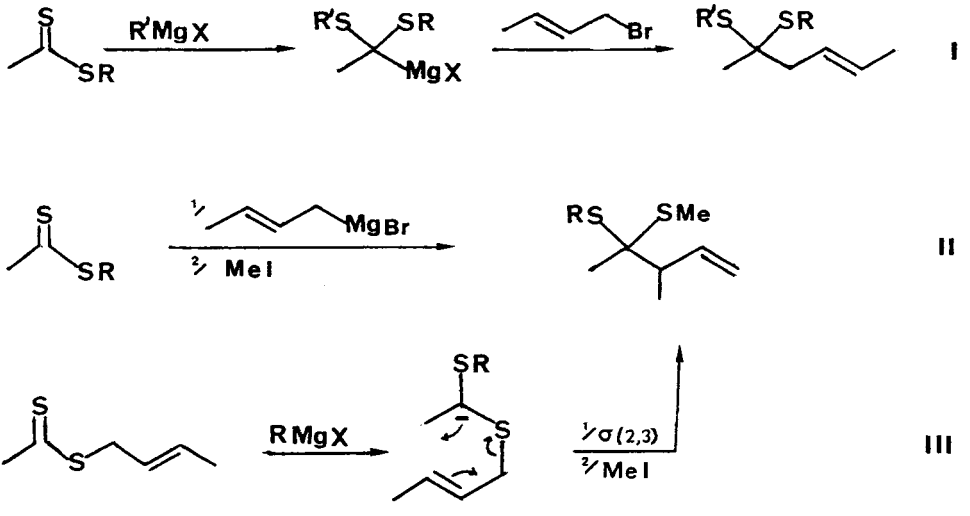


From a synthetic point of view thiophilic and carbophilic additions on dithioesters afford pathways for carbon-carbon bond formation and a straighforward application is the preparation of β-ethylenic ketones through their dithioacetals [19]. Three complementary routes can be envisaged as illustrated in scheme 1 with a dithioacetate and a crotyl chain:

Thiophilic addition of an alkylmagnesium halide, alkylation with an allylic halide (route I).

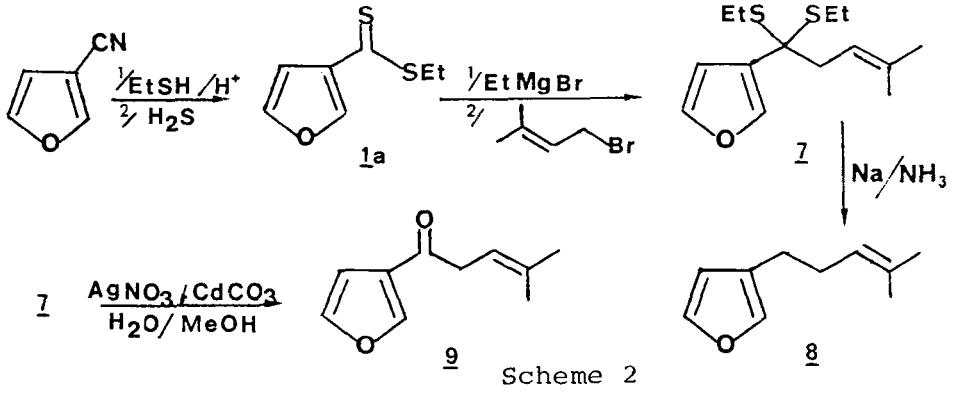
Carbophilic addition of an allylic Grignard reagent, alkylation at sulfur (route II).

Thiophilic addition of an alkylmagnesium halide to an allylic dithioester followed by a [2,3] sigmatropy and alkylation at sulfur (route III).



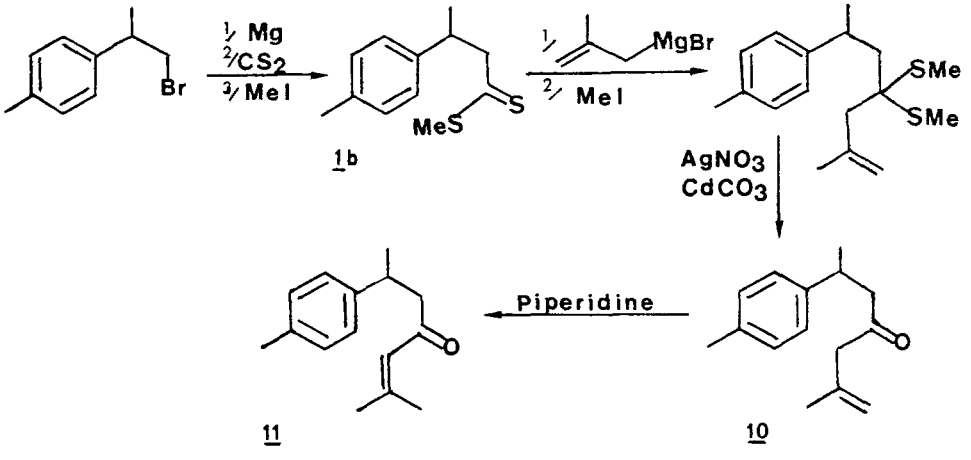
Scheme 1

These dithioacetals can be converted into the corresponding ketones without reconjugation of the double bond, or reductively desulfurized into alkenes. We applied these reactions to the synthesis of some terpenic compounds [24]. Using route I, perillene 8 and egomaketone 9 were easily obtained from 3-cyanofuran through the dithioacetal 7 (scheme 2).



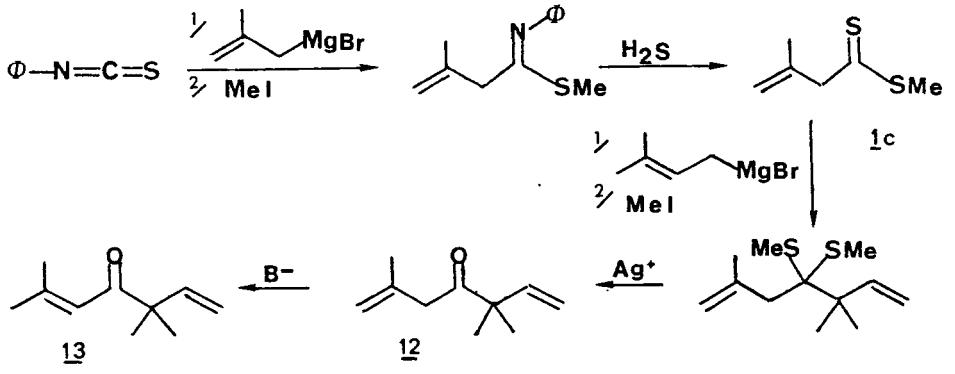

Scheme 2

Egomaketone 9 was also synthesized via Corey-Seebach dithiane method [25]. The synthesis of ar-turmerone 11 and its unconjugated isomer 10 illustrates route II (scheme 3); routes I or III could have been used as well.




Scheme 3

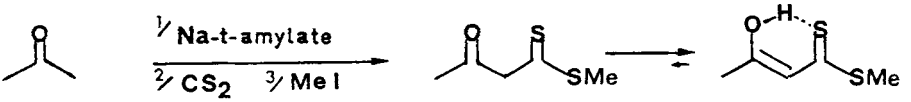
As shown in scheme 4 a β-unsaturated dithioester 1c was used for a synthesis of isoartemisiaketone 12 and artemisiaketone 13 [26].



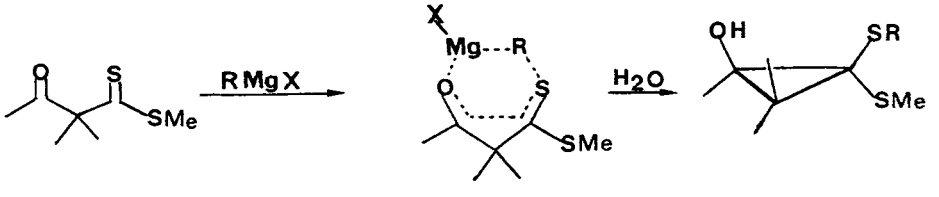

Scheme 4

## 3. REACTIONS OF β-OXODITHIOESTERS

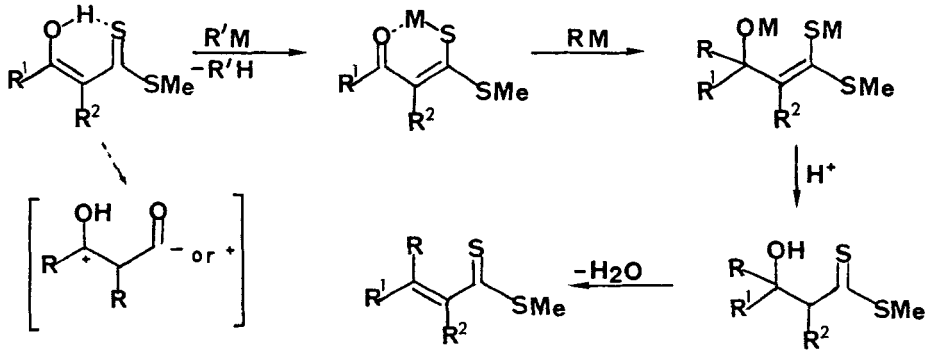
The most common access to these compounds is the addition of carbon disulfide to ketone enolates and monoalkylation [27]; for example:




When there is at least one hydrogen in the 2 position the enol form is generally predominant; when this is not the case they were shown to react with Grignard reagents by 1,4 homoaddition leading stereospecifically to cyclopropane derivatives [28].

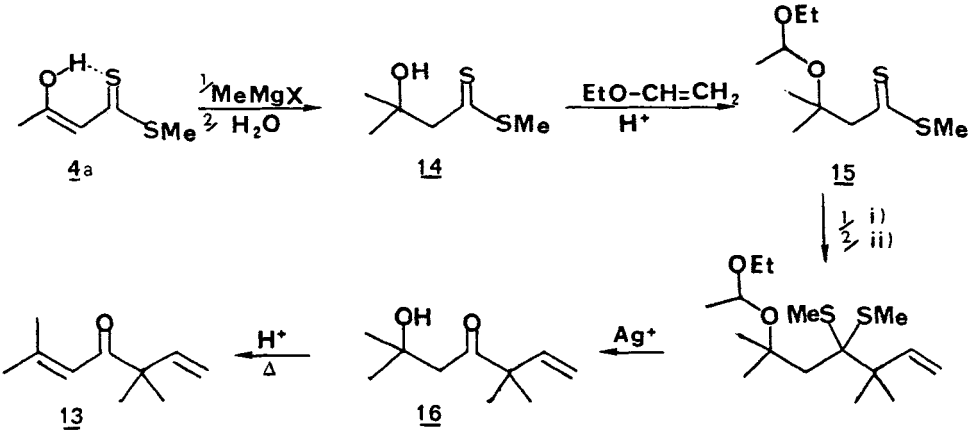


The reaction was quite different for enolizable β-oxodithioesters; the first mole of organometallic reacted with the enol proton; a second mole (not necessarily the same reagent) added exclusively to the carbonyl function providing β-hydroxydithioesters in good yields. After protection of the OH group it is possible to use the dithioester function for formation of another carbon-carbon bond and these β-oxodithioesters can be viewed as synthetic equivalents $a^3d^1$ or $a^3a^1$. Dehydration of β-hydroxydithioesters into α-unsaturated dithioesters could also be accomplished (scheme 5) [29].




Scheme 5

As an application of these synthons, artemisia ketone 13 was synthesized, via a β-hydroxyketone precursor 16, starting from methyl 3-oxobutanedithioate 4a and introducing methyl and transposed prenyl chains by two succes-



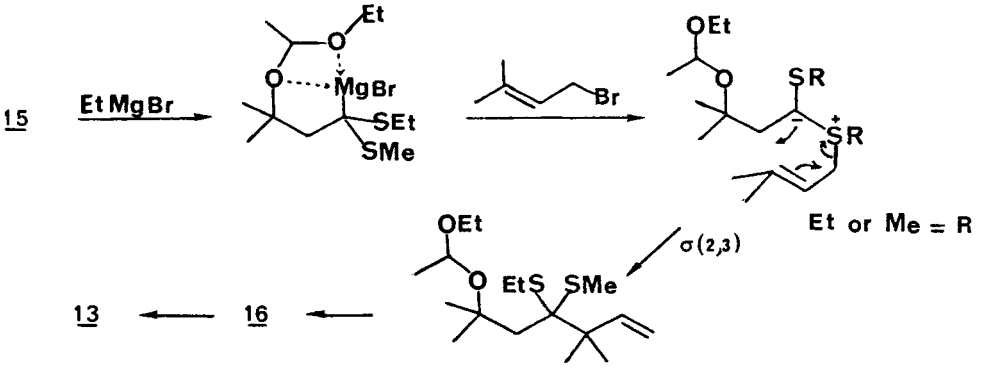

i) prenylmagnesium bromide ii) methyl iodide

Scheme 6

sive nucleophilic additions on carbons 3 and 1 (scheme 6).

On the protected acetal 15 of the β-hydroxydithioester 14, thiophilic addition of ethylmagnesium bromide was also realized; attempted alkylation with prenylbromide led to an unexpected result: inversion of the allylic chain was observed and artemisia ketone was again obtained by dethioacetalization and dehydration: actually such an inversion has been previously reported in alkylation of enolates stabilized by an alkylthio or alkylseleno group [30,31]; in the present case, stabilization of the organometallic intermediate by the oxygen atoms of the acetal group could lead to alkylation on sulfur with ylide formation and inversion of the allyl chain would result from a [2,3] sigmatropy (scheme 7).

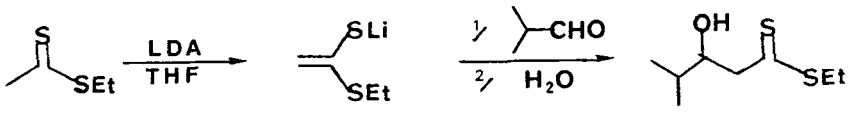




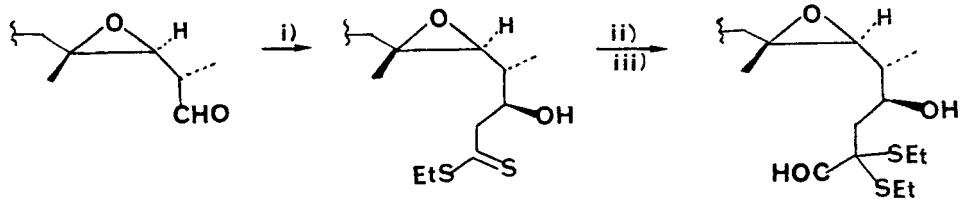

Scheme 7

## 4. REACTIONS OF THE ENETHIOLATES OF DITHIOESTERS

Meyers and co-workers [15] have shown that the lithio derivative of ethyl dithioacetate reacts with isobutyraldehyde [15] to give a β-hydroxydithioester. The use of the dithioester group as acyl anion equivalent allows two electrophilic additions on two vicinal carbons of the starting dithioester which appears as a $d^2d^1$ synthon: $\underline{C}H_2\text{-}\underline{C}{=}O$; the sequence was applied in Meyers' synthesis




of maytansinoids such as (-) maysine [32]; a key fragment was obtained as depicted in scheme 8.

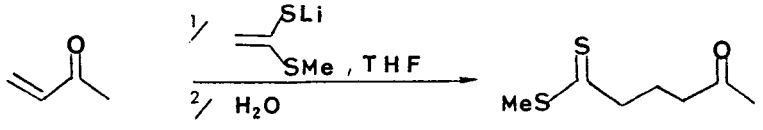



i) lithium ethyl dithioacetate; $H^+$

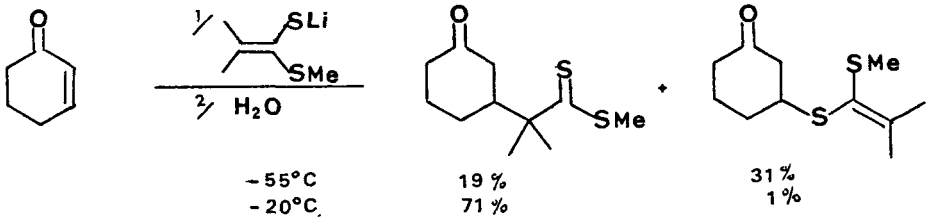
ii) $EtO-CH=CH_2$ iii) EtMgBr; formylation; $H^+$

Scheme 8

In our research group we have established that the lithium enethiolate of methyl dithioacetate undergoes a selective 1.4-addition with various enones to yield 5-oxodithiocarboxylates [33], versatile 1,5-dicarbonyl presursors with functional differentiation as shown below:

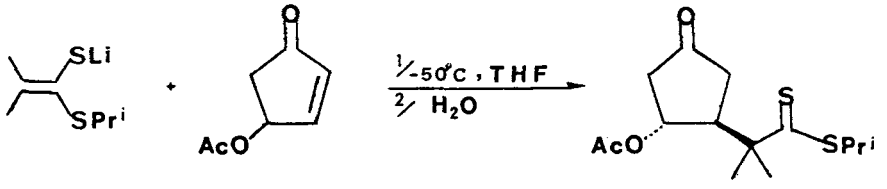



With a more hindered enethiolate, namely the lithium enethiolate of methyl 2-methyldithiopropanoate, a kinetic sulfur 1,4-addition and a thermodynamic carbon 1,4-addition was generally observed as shown with cyclohexenone from the temperature dependent results [34].

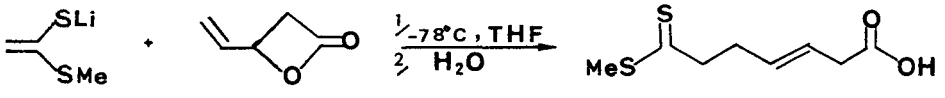



Similar results were obtained with pent-3-en-2-one but with cyclopent-2-en-1-one only the C-1,4 addition could be detected.

Bertz and co-workers [35] have studied the reaction of lithium isopropyl 2-methyldithiopropanoate with cyclopent-2-en-1-ones substituted with a leaving group in the 4 position; they reported two interesting features of the 1,4 C-addition on 4-acetoxycyclopent-2 en-1-one: the addition is stereospecific and the acetoxy group is preserved.

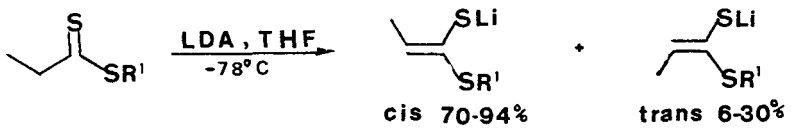



Lithium dithioester enethiolates were also found to react regio and stereoselectively at the terminal carbon of β-vinyl β-propiolactones [36].

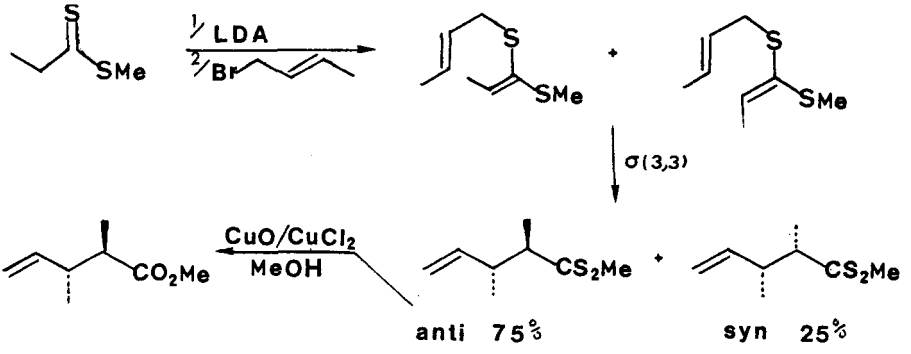



Recently we found that the kinetic deprotonation of dithiopropanoates affords predominantly the cis lithium enethiolates [37] (as for amides and thioamides but in contrast with the trans enolization of most carbonyl compounds); the best cis selectivity observed was 94% ($R^1$=

$CH_2OMe$).

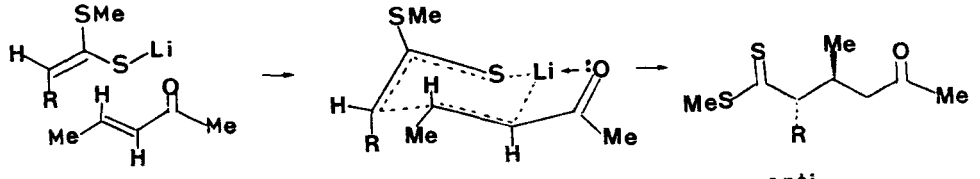



In order to establish the geometries of the lithium enethiolates we used the thio-Claisen rearrangement [38] of their S-crotyl derivatives which led selectively to anti (erythro) and syn (threo) methyl 2,3-dimethyl-4 pentenedithioates; these could be converted, without epimerization, into the corresponding methyl esters which were identified by a direct synthesis according to Ireland's procedure [39] (scheme 9).




Having established the geometries of the enethiolates, we looked at the stereochemical aspects of their conjugate additions to enones [40]. With acyclic enones (pent-3-ene-2-one, chalcone) two diastereoisomers were formed in essentially the same ratio as that of the starting thioenolates and the addition appears to be stereospecific. A transition state such as the one shown in scheme 10 could explain the stereospecificity observed and would lead to the anti isomer as the major product from the cis thioenolate (although this has yet to be proved); that transition state implies the possibility of a s-cis con-

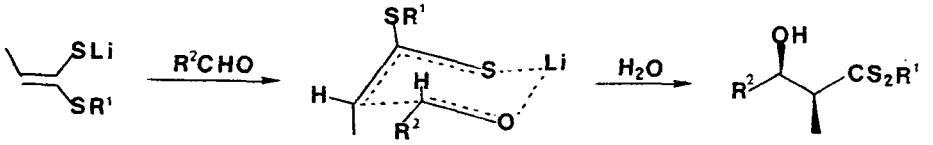
formation for the enone and indeed no or only low stereoselectivity was observed with cyclic enones.



Scheme 1o

anti

We have also shown that the kinetic aldol reactions of these enethiolates are diastereospecific [41]. The cis enethiolate gives the syn aldol and a chair like transition state with the substituent of the aldehyde in equatorial position accounts for this result (scheme 11).

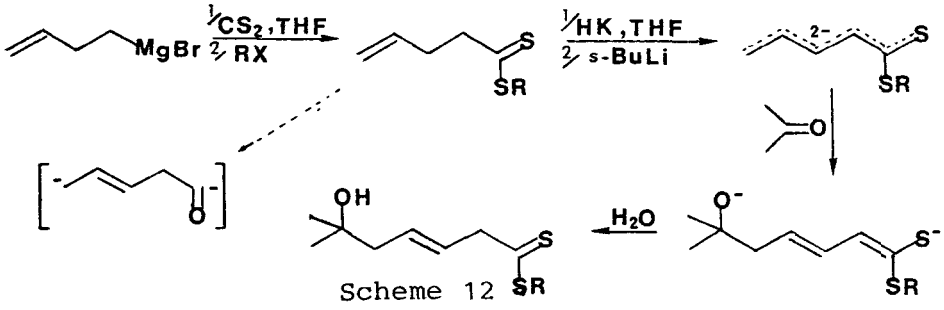


cis

Scheme 11

syn aldol

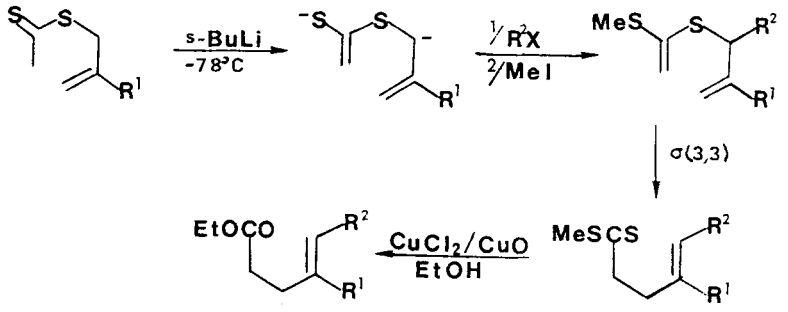
Another regioselective reaction involving a dianion prepared from a γ-ethylenic dithioester was reported by Pomakotr and Seebach [42] (scheme 12), allowing the use of the starting dithioester as a $d^1d^5$ synthon.



Scheme 12

A dianion was also obtained by treating an allyl dithioester with sec.-butyllithium; it could be selectively alkylated first at the allylic site, then at the sulfur atom; a [3,3] sigmatropic rearrangement of the S-allyl ketenedithioacetal thus formed led to a stereoselective

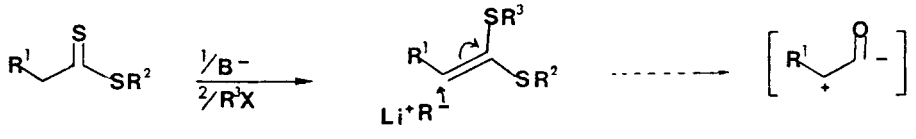
synthesis of trisubstituted olefins [43] (scheme 13).




Scheme 13

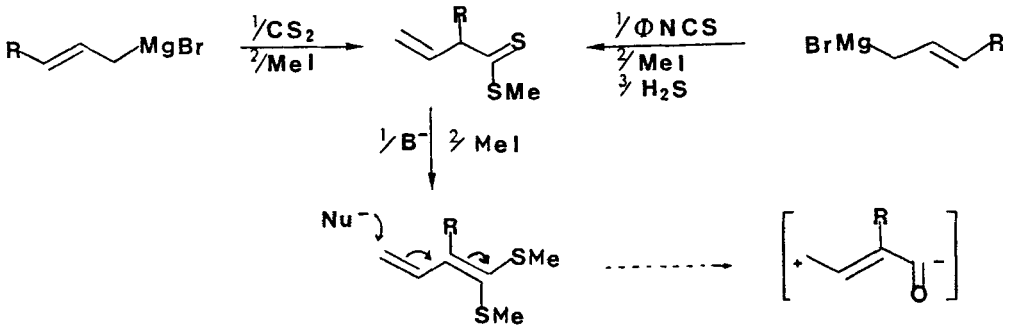
## 5. REACTIONS OF CONJUGATED KETENEDITHIOACETALS

Dithioester enethiolates are alkylated by alkyl halides at the sulfur atom to give ketenedithioacetals which are useful synthetic intermediates [2,5,44-46]; their reactivity towards lithium reagents for example allows their use as $a^2d^1$ synthons:



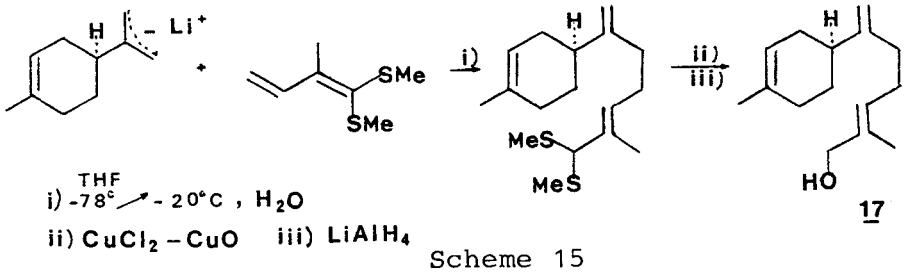

Conjugated ketenedithioacetals are obtained from β-unsaturated dithioesters which are themselves easily prepared from allyl Grignard reagents and carbon disulfide [47] or phenylisothiocyanate [10] (in both cases inversion of the allyl chain occurs).

The conjugate addition of nucleophiles such as organolithium compounds followed by electrophilic addition on carbon 1 offers a possibility to use these conjugated ketenedithioacetals as $d^1a^4$ synthons (scheme 14).



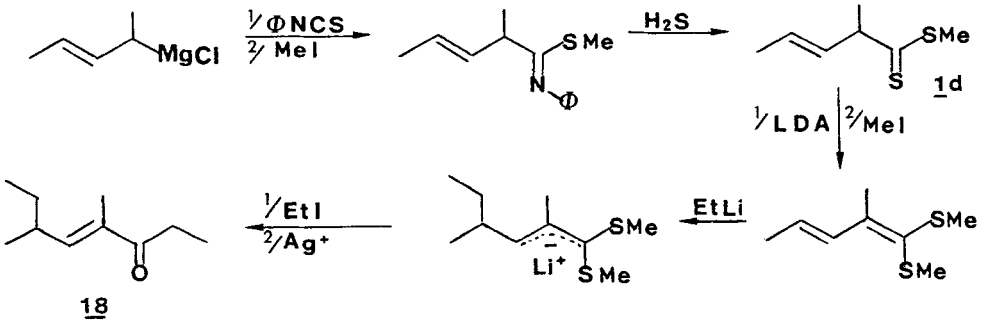
Scheme 14

Julia and al [47,48] have used as isoprenic synthons two $C_5$ conjugated ketenedithioacetals prepared via β-unsaturated dithioesters; an application of one of these in a stereoselective synthesis of E(-) lanceol 17 is given in scheme 15.



Scheme 15

We have applied [49] the addition of ethyllithium to the conjugated ketenedithioacetal derived from methyl 2-methyl-3-pentenedithioate 1d followed by alkylation with



Scheme 16

ethyl iodide, in a stereoselective synthesis of manicone 18, an alarm pheromone of Manica ants (scheme 16).

## 6. REACTIONS OF α-OXOKETENEDITHIOACETALS

These compounds are easily obtained by dialkylation of the condensation products of ketone enolates with carbon disulfide [27,5o-52]; five examples of their use for carbon-carbon bond formation are shown in scheme 17.

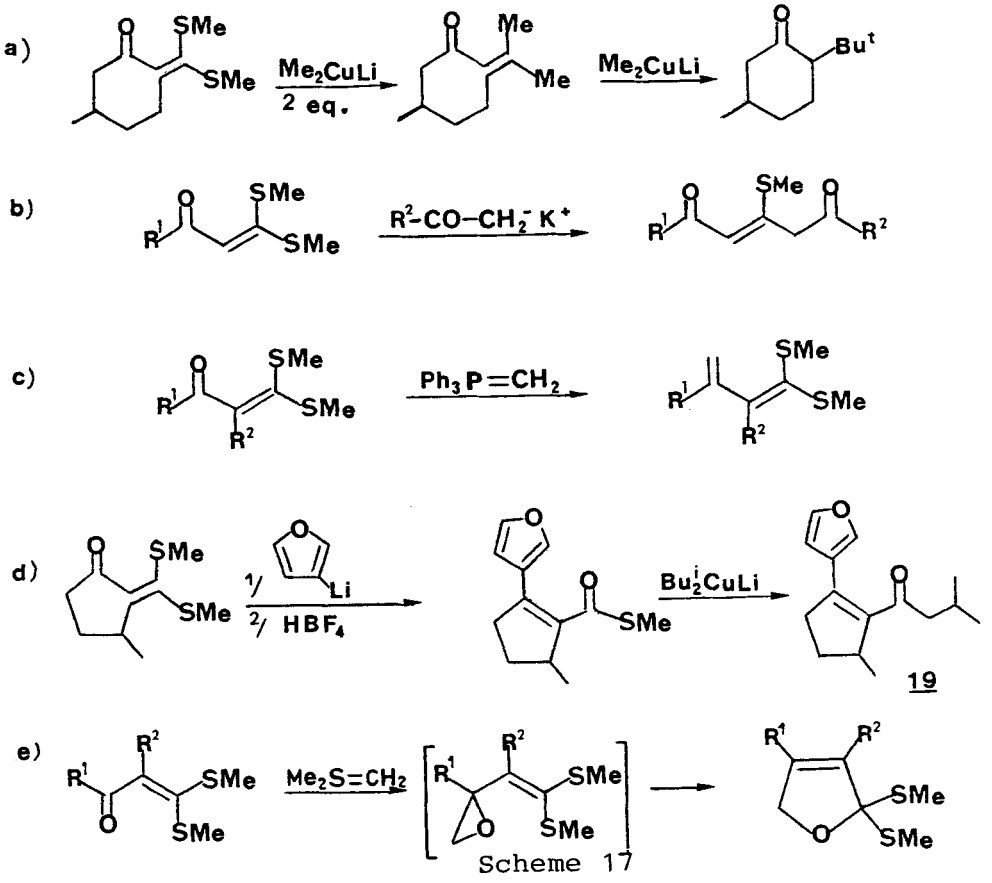
a) 1,4-addition of dimethylcopperlithium; elimination of the methylthio groups occurs and provides a route for introducing a t-alkyl group in α position relative to a carbonyl [5o].

b) an analogous reaction with enolates [53] which leads to 1,5-diketones, with a masked carbonyl group in the 3 position.

c) a Wittig reaction which gives an easy access to conjugated ketenedithioacetals.

d) a nucleophilic addition on the carbonyl group, followed by acid catalysed rearrangement into an α-unsaturated thioester, used for a synthesis of myodesmone 19 [55].

e) methylenation with sulfur ylides which leads to 2,2-bis (alkylthio) dihydrofurans and gives access to furans and butenolides [56,57].

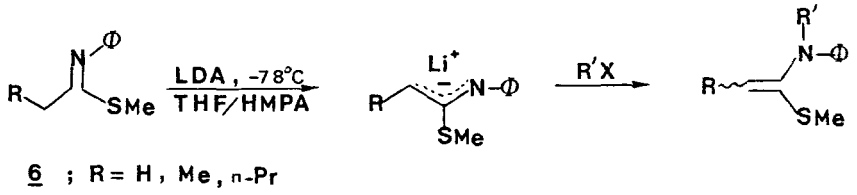



Scheme 17

## 7. REACTIONS OF N-PHENYL IMIDOTHIOESTERS

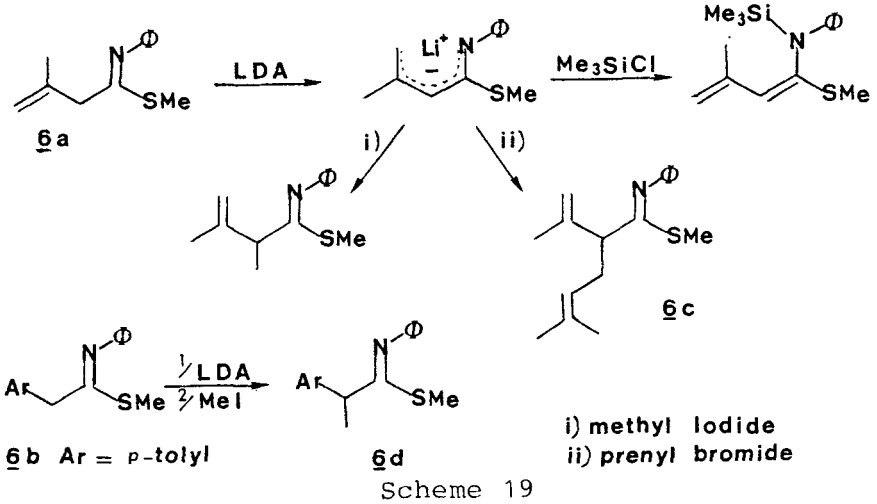
Alkylations on the α-carbon have been reported for heterocyclic imidothioesters [58,60]; thus 2-methylthiazoline [58] and benzothiazole [59] have been used as α-formylated carbanion equivalents. N-phenylimidothioesters 6 are good precursors of dithioesters and their α-alkylation would provide an indirect way for α-alkylation of these dithioesters; we examined first the metallation and alkylation of N-phenylimidothioesters with an alkyl side chain; the anion formed by LDA in THF-HMPA was treated with electrophiles (methyl iodide, prenyl bromide, ethylchloroformate, trimethylsilyl chloride); all gave only N-substitu-

ted products [61] (scheme 18).




Scheme 18

Hoping that the more delocalized anions derived from β-unsaturated imidothioesters could have a different reactivity towards electrophiles we prepared such anions and treated them with electrophiles. With methyl N-phenyl 3-methyl-3 butenethioimidate 6a a N-alkylation was again observed with hard electrophiles (trimethylsilyl chloride and ethylchloroformate), but with methyl iodide, prenyl bromide and dimethyldisulfide a regioselective α-C-alkylation was realized; α-arylated thioimidates gave similar results [61] (scheme 19).

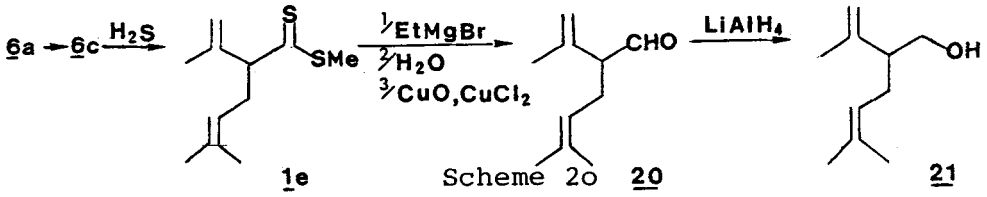



Scheme 19

It became then possible to use the imidothioesters, such as 6a and 6b, to create a carbon-carbon bond in α position and after sulfhydrolysis to use the dithioester function for formation of a second carbon-carbon bond; they are thus equivalents to the following $d^2a^1$ or $d^2d^1$ syn-

thons:

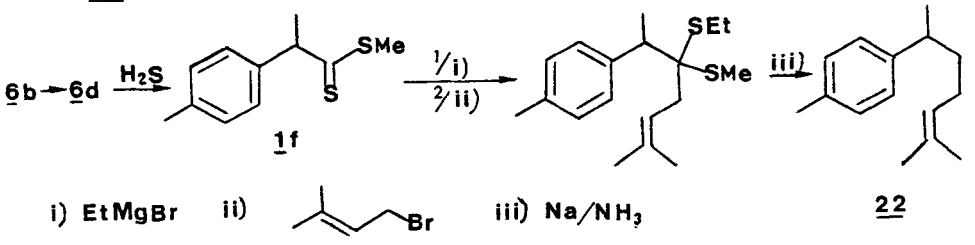
As a first application we synthezized lavandulal 2o and lavandulol 21 (scheme 2o) from 6a.




1e Scheme 2o 20 21

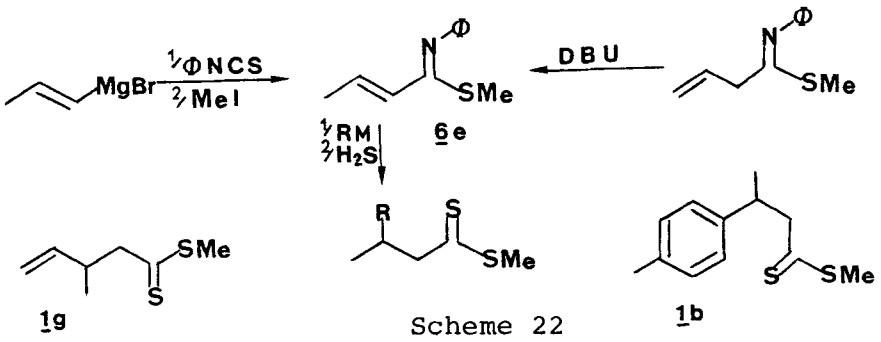
A more direct route to 21 was realized by acid catalyzed hydrolysis of 6c to a S-methylthioester and $LiAlH_4$ reduction.

The synthesis of ar-curcumene 22 (scheme 21) started from 6b (Ar=p-tolyl).



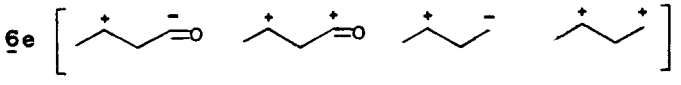

Scheme 21

α-unsaturated thioimidoesters can be prepared from vinylic organometallics and phenylisothiocyanate; they are also available by isomerization of the β-isomers; we found that they undergo 1,4 addition with organometallic compounds and so they permit to realize indirectly a Michael addition to α-unsaturated dithioesters (scheme 22); from p-tolyl magnesium bromide a short route to 1b (used in scheme 3) was obtained; vinylmagnesium bromide led to 1g [62].




Scheme 22

The α-unsaturated thioimidate 6e can consequently be used as $a^3d^1$ or $a^3a^1$ synthon.




Conclusion: It is only recently that dithioesters have received much attention for their potential applications in organic synthesis. Easy to prepare, easy to handle, highly reactive and with some unique features in their reactivity, one can foresee for them, as well as for their precursors and derivatives, an increasing role in the strategy of organic synthesis.

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