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THE COMPATIBILITY OF THE CYANO AND ALKYL CHLORIDE FUNCTIONS IN PHENYLTHIOALKYLATION REACTIONS OF O-SILYLATED ENOLATES

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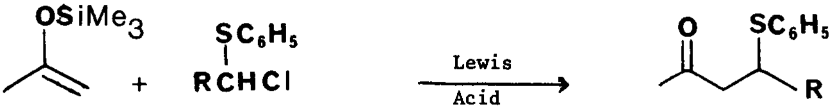
Summary: The compatibility of the cyano group and of chlorides with the conditions of phenylthioalkylation of O-silylated enolates has been demonstrated.

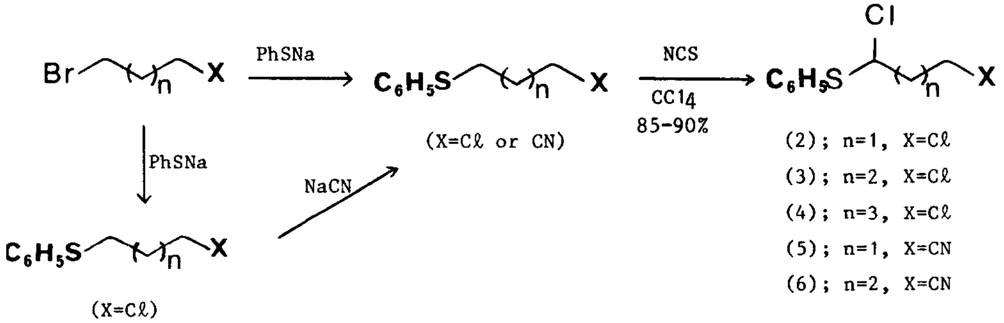
The alkylation of O-silylated enolates with phenylthioalkyl chlorides (1) is now established as an excellent means of regiospecifically alkylating ketones and enones. The scope of the R group in (1) has been investigated by a number of groups and it has been shown that the conditions of the reaction are compatible for R being an alkyl group, a ketone function[2], a carbomethoxy group[3] and an alkenyl function[4], amongst others.



(1)

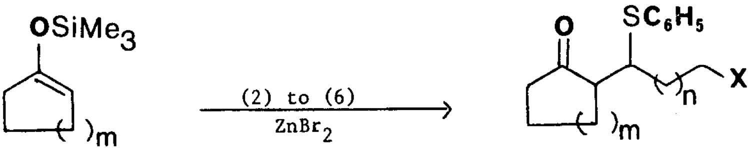
As part of a study aimed at developing novel annulation procedures we needed to assess the compatibility of the cyano function and the alkyl chloride moiety with the conditions of phenylthioalkylation. We now report that the α-phenylthioalkyl-α,ω-dihalides (2,3,4) and the phenylthiochloronitriles (5,6), prepared as shown in the SCHEME, do react with O-silylated

SCHEME



enolates in the presence of zinc bromide, to provide the ketones (7 to 15) in good yields of 65-95%.

Prior to this study there had been one example of the reaction of (2) with an O-silylated



m=1 or 2

(7); m=1, n=1, X=Cℓ
(8); m=1, n=2, X=Cℓ
(9); m=1, n=3, X=Cℓ
(10); m=2, n=1, X=Cℓ
(11); m=2, n=2, X=Cℓ
(12); m=1, n=1, X=CN
(13); m=1, n=2, X=CN
(14); m=2, n=1, X=CN
(15); m=2, n=2, X=CN

enolate[5], as a footnote to a larger synthetic sequence. We have now demonstrated more clearly the compatibility of alkyl halides in phenylthioalkylation reactions and extended them to include the cyano function, so enhancing the procedure.[6] Furthermore we now have ready access to ketones (7) to (15) as required for our novel annulation studies, which we will describe in due course.

REFERENCES AND NOTES.

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6. All new products gave satisfactory spectral and analytical data.

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