

Note

Reaction of α -halosilanes with N-arylidenearylsulfonamides—Attempted synthesis of N-arylsulfonylaziridines

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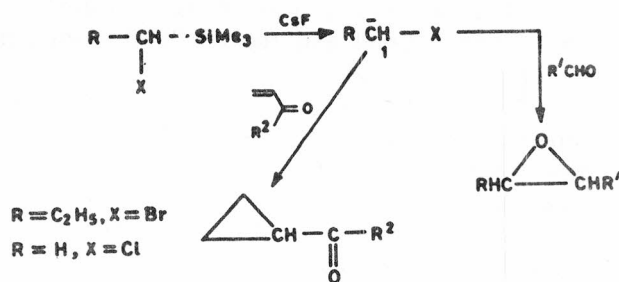
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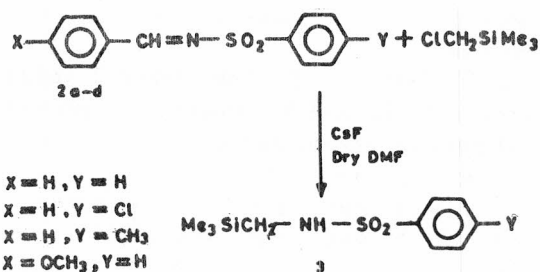
Reaction of α -halosilanes with fluoride ions generates carbanions **1**, through the attack of fluoride ion on the Si atom, and these are reported to transfer methylene to suitable double bonds¹⁻³. Thus Kessar *et al.*⁴ have shown that **1** reacts with aldehydes to give oxiranes and with α,β -unsaturated ketones and esters to yield the corresponding cyclopropanes (Scheme I).

This behaviour of the carbanions **1** parallels that of sulfur ylides⁴; sulfonium and sulfoxonium methylides also transfer⁵ a methylene to $>C=C<$, $>C=O$ and to a lesser extent to $>C=N-$. We report herein the reaction of α -halosilane derived carbanions with N-arylidenearylsulfonamides as a possible route to aziridines and communicate rather unexpected results.

N-Arylsulfonylimines **2a-d**⁶ were reacted with CsF and halomethyltrimethylsilane in dry DMF. In each case a solid product was isolated which in IR spectrum showed a strong signal at 3260 cm^{-1} . The ¹H NMR spectra of these products showed a broad triplet between δ 4.35 and 4.45 integrating for one proton which disappeared on D₂O shake, and a singlet at δ -0.04 (9H) besides aromatic protons at δ 7.4-8.0 integrating for appropriate number of protons. The ¹³C NMR



Scheme I



Scheme II

spectra of the products exhibited a quartet at δ -3.02, a triplet at 32.20 and aromatic carbons between 127.38 and 132.49. On the basis of spectral data the products were assigned structure **3** (Scheme II).

Replacing chloromethyltrimethylsilane with bromo- or iodo-methyltrimethylsilane gave the same product **3**. When the source of fluoride ion was changed from CsF to tetrabutylammonium fluoride, no change in the course of the reaction was observed (Table I). The only other product isolated from the reaction mixture was the corresponding arylsulfonamide, derived probably from the

Table 1—Results of the reaction of α -halosilanes with N-arylidenearylsulfonamides **2**

Reactant	α -Halosilane	Fluoride source	Product	Yield (%)	m.p. °C
2a	ClCH ₂ SiMe ₃	CsF	3a	71	51-53
2b	ClCH ₂ SiMe ₃	CsF	3b	71	96-98
2c	ClCH ₂ SiMe ₃	CsF	3c	70	62-64
2d	ClCH ₂ SiMe ₃	CsF	3c	69	52
2b	BrCH ₂ SiMe ₃	CsF	3b	65	96-97
2b	ICH ₂ SiMe ₃	CsF	3b	64	96-97
2a	ClCH ₂ SiMe ₃	Bu ₄ N ⁺ F ⁻	3a	75	51-52
2b	ClCH ₂ SiMe ₃	Bu ₄ N ⁺ F ⁻	3b	81	96-98
2d	ClCH ₂ SiMe ₃	Bu ₄ N ⁺ F ⁻	3a	68	52

hydrolysis of the unreacted Schiff base. In the absence of CsF, no reaction took place.

Experimental Section

General method for the reaction of N-arylidenearylsulfonamides 2 with α -halosilanes. Cesium fluoride (7.2 mmoles) in a flame dried evacuated r.b. flask was dried by heating with a flame. The vacuum was released into an atmosphere of N_2 and halomethyltrimethylsilane (3.6 mmoles) in dry DMF (10 mL) added to the flask. After stirring for 10 min, the pertinent imine (3.6 m moles) in dry DMF (10 mL) was added and the stirring continued overnight. The reaction mixture was diluted by adding ice-cold water (25 mL) and the whole mixture extracted with ether (3×25 mL).

The combined ethereal extracts were washed with water (1×10 mL) and dried over anhydrous sodium sulfate. Removal of the solvent and crystallization of the residue from benzene-pet. ether gave the product.

References

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