One-Pot Reaction of Aldehydes, α-Haloketones and (Phenylsulfonyl)acetonitrile Promoted by SmI₃

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Abtract: One-pot reaction of aldehydes, α -haloketones and (phenylsulfonyl)acetonitrile promoted by SmI₃ proceeded smoothly to give 1-cyano-1-phenylsulfonyl-2-aryl-3-aroyl-propane derivatives in moderate to good yields.

Keywords: One-pot reaction, sulfonyl group, cyano group, samarium (III) triiodide.

Recently the reports of using samarium (III) in mediating carbon-carbon bond formation reactions have rapidly increased. For example, promoted by SmI₃, α -haloketones can react with aldehydes to give α , β – unsaturated ketones¹; mediated by SmI₃, α -diketones can condense with aldehydes to form benzylidene-substituted α -diketones in fair yields². More recently we found that SmI₃ can efficiently promote Michael addition of active methylene compounds to α , β – unsaturated ketones to give poly-functionalized compounds³.

It is well known that compounds bearing sulfonyl and/or cyano group(s) are important intermediates in organic synthesis since cyano group can be easily transformed into other functionalities and sulfonyl group can be used as a good leaving group for the substitution reactions. In addition, both cyano and sulfonyl group have remarkable activation effect on the substrates and thus enable the subtrates to undergo a variety of reactions. Our previous work on the reactions^{2, 3} promoted by SmI₃ led us to investigate the one-pot preparation of compounds bearing both sulfonyl and cyano groups, such as 1-cyano-1-phenylsulfonyl-2-aryl-3-aroyl-propane derivatives **4**, directly from one-pot reaction of aldehydes **1**, α -haloketones **2** and (phenylsulfonyl)acetonitrile **3** promoted by SmI₃ (as shown in Scheme 1). Herein we wish to report our preliminary results of this novel preparation (Table 1).

Scheme 1

$$Ar^{l}CHO + Ar^{2}COCH_{2}Br + PhSO_{2}CH_{2}CN \xrightarrow{SmI_{3}} Ar^{l}CHCH_{2}COAr^{2}$$

$$\downarrow PhSO_{2}CHCN$$

$$1 \qquad 2 \qquad 3 \qquad 4$$

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Entry	Ar^1	Ar ²	Yield (%) ^a
1	C_6H_5	C ₆ H ₅	69
2	$3-BrC_6H_4$	C_6H_5	71
3	$4-ClC_6H_4$	C_6H_5	70
4	$4-CH_3C_6H_4$	C_6H_5	66
5	C_6H_5	$4-CH_3C_6H_4$	64
6	$4-ClC_6H_4$	$4-CH_3C_6H_4$	65
7	$3-BrC_6H_4$	$4-CH_3C_6H_4$	68
8	C_6H_5	$4-BrC_6H_4$	71
9	$4-CH_3C_6H_4$	$4-BrC_6H_4$	68
10	$4-ClC_6H_4$	$4-BrC_6H_4$	75

 Table 1
 Preparation of 1-cyano-1-phenylsulfonyl-2-aryl-3-aroyl-propanes promoted by SmI₃

a: Isolated yields

Experimental

General procedure for the synthesis of compound 4: To a pale yellow suspension of SmI_3 (1 mmol) in THF was added 1 (1 mmol) and 2 (1 mmol) and stirred until 1 and 2 were almost consumed (monitored by TLC), then a solution of 3 (1 mmol) was added. The mixture was refluxed for 10-12 h, then water was added and the product was extracted with diethyl ether. The organic phase was collected, dried over Na_2SO_4 and evaporated to afford the crude product. The product was purified by preparative TLC on silica gel using cyclohexane and ethyl acetate (5:1) as eluent. The products were characterized by their ¹H NMR, IR and Mass spectra⁴.

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- 4. The spectra data have been sent to the Editorial Department.

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