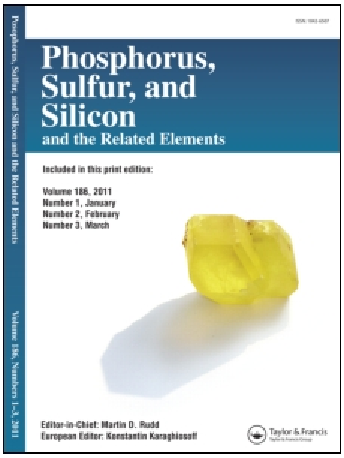

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MICROWAVE-ASSISTED REDUCTION OF β-TRIMETHYLSILYL CARBONYL COMPOUNDS BY SODIUM BOROHYDRIDE

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MICROWAVE-ASSISTED REDUCTION OF β-TRIMETHYLSILYL CARBONYL COMPOUNDS BY SODIUM BOROHYDRIDE

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Several β-trimethylsilyl carbonyl compounds were reduced by sodium borohydride to produce β-trimethylsilylated alcohol under microwave irradiation in short time and good yields.

Keywords: Microwave; Borohydride; Reduction; Trimethylsilyl; Carbonyl; Alcohol

The synthesis of silylated alcohols has found interest due to their synthetic use and for their potential therapeutical effect with less toxicity[1–4]. One approach for the preparation of these compounds is reduction of trimethylsilyl carbonyl compounds by a reducing agent[5]. β-Trimethylsilyl aldehydes and ketones can be easily prepared from the reaction of the corresponding α,β-unsaturated compounds with trimethylsilyl chloride (TMS-Cl) in the presence of Li or Mg in THF[6]. Numerous reagents have been used for reduction of carbonyl compounds to the corresponding alcohols. Relatively inexpensive sodium borohydride, $NaBH_4$, has been extensively used as a reducing agent[7].

Recently, it was shown that microwave-assisted reaction can be applied for many type of organic transformations and offers several advantages[8–9]. In continuation of our interest in development of environmentally benign synthetic method using microwave (MW) irradiation under solvent-free condition[10], we wish to report a mild, convenient and heterogeneous cata-

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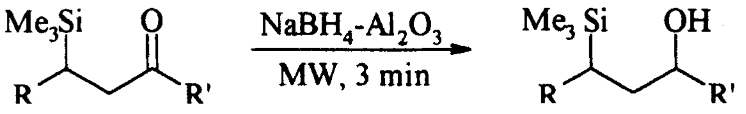
* Corresponding Author.

lytic methodology for the synthesis of β-silylated alcohols. These solvent-free reactions are especially appealing, as they provide an opportunity to work with open vessels. Thus avoiding the risk of high pressure development and a possibility of carrying out the reaction on a preparative scale in a conventional microwave oven.

The process involves simple mixing of β-trimethylsilyl aldehyde or ketone with 10% $NaBH_4$/neutral alumina and irradiation of the mixture in a teflon container in a conventional microwave oven for 2–3 min (Scheme 1, Table I). Although we did not encounter any accident during the reaction, we recommend caution for reactions on large scale.



SCHEME 1

EXPERIMENTAL

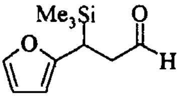
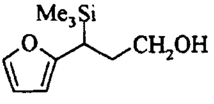
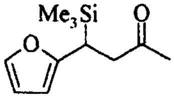
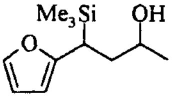
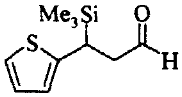
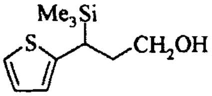
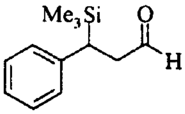
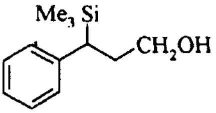
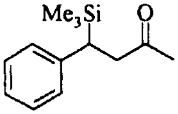
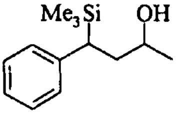
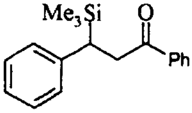
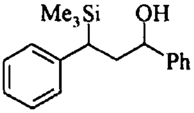
IR spectra were taken on Matt Son 1000 Unicam FTIR, ^1H and ^{13}C-nmr spectra were recorded on Bruker AC 80, ms spectra were obtained on a Varian MAT 311A, and Varian CH 5 spectrometers.

The structure of the new compounds were determined by their ^1H and ^{13}C-nmr, and their ms.

Typical Procedure for the Reduction of β-Trimethylsilyl Aldehyde or Ketone

β-Trimethylsilyl aldehyde or ketone (1 mmol) was added to the mixture of 3 mmol $NaBH_4$ in neutral alumina (1:9) in a teflon container. The mixture was irradiated in an unmodified household microwave oven for the time specified in the table. After cooling, the product was extracted with CH_2Cl_2 (5 mL). Evaporation of the solvent affords the corresponding alcohols in high yields. The structure of the new compounds were determined by their ^1H and ^{13}C-NMR, IR, and MS spectra.

TABLE I Reduction of β-Trimethylsilyl Carbonyl Compounds Using $NaBH_4$-Al_2O_3

Entry	Substrate	Product	$NaBH_4$/ Substrate	Time (min)	Yield (%)
1			3:1	2	100
2			5:1	3	60
3			3:1	2	74
4			3:1	2	100
5			3:1	2	100
6			5:1	2	72

Acknowledgements

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