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Microwave-Mediated Debromination of vicinal-Dibromides

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Abstract: Debromination of *vicinal*-dibromides to E-alkenes is reported using inorganic solid supports (silica gel or acidic alumina) under microwave irradiation.

Keywords: Debromination, inorganic solid support, microwaves, stereoselective, *vic*-dibromides

The olefins can be regenerated from *vicinal*-dibromides with a variety of reagents^[1] and the overall bromination/debromination process represents a means of protection and deprotection^[2] of olefins. Our group has reported solvolytic stereoselective debromination of *vic*-dibromides by N,N-dimethyl-formamide (DMF)^[3,4] and hexamethylphosphoramide (HMPA)^[5] without the aid of any reagent. Different methods^[6,7] reported in literature for debromination have their own advantages and disadvantages.

We report herein the first study on stereoselective debromination of *vic*dibromides (I) adsorbed on inorganic solid support (silica gel or acidic alumina) under microwave irradiation^[8] to give the corresponding E-olefins (II) (Fig. 1). Microwave-mediated debromination has been carried out on a variety of *vic*-dibromides (Table 1) within periods of 1-5 min.

> RCHBr—CHBrR^{stilica} gel/acidic alumina RCH=CHR' I a-1 Microwave, 1-5 min II a-1 II a-1

Figure 1. Debromination of vicinal-dibromides under microwave irradiation.

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S. No.	Substrates	Silica gel		Acidic alumina	
		Time (min)	Isolated yield (%) (II)	Time (min)	Isolated yield (%) (II)
1	<i>meso</i> -Stilbene dibromide (Ia)	2	95	2	96
2	<i>dl</i> -Stilbene dibromide (Ib)	2	$84(89)^a$	2	$84(91)^{b}$
3	meso-4-Chlorostilbene dibromide (Ic)	4	90	4	96
4	meso-4-Methylstilbene dibromide (Id)	3	91	3	95
5	<i>meso</i> -4,4'-Dichlorostilbene dibromide (Ie)	5	92	5	96
6	meso-4-Methoxystilbene dibromide (If)	1	82	1	86
7	<i>meso-</i> α , β -Dibromosuccinic acid (Ig)	1	96	1	98
8	dl - α , β -Dibromosuccinic acid (Ih)	1	95	1	98
9	trans-1,2-Dibromocyclooctane (Ii)	2	81	2	81
10	trans-1,2-Dibromocyclohexane (Ij)	10	84	10	85
11	Dihydrocinnamic acid dibromide (Ik)		—		_
12	Cholesterol dibromide (II)	d	—	d	
13	meso-Stilbene dichloride (Im)	2	21	2	37

Table 1. Reactions of vic-dibromides under microwave irradiation on inorganic solid support

^aYield in bracket is HPLC yield. 11% Z-stilbene was also formed.

^bYield in bracket is HPLC yield. 9% Z-stilbene was also formed.

^cStarting material recovered.

^dDecomposition of starting material was observed.

vicinal-Dibromide Debromination

TYPICAL PROCEDURE

meso-Stilbene dibromide (0.5 g), dichloromethane (5 ml), and silica gel or acidic alumina (2 g) were mixed thoroughly in a beaker. The adsorbed material was dried and then exposed to microwave irradiation. After 2 min, the residue was extracted with ethyl acetate (3×10 ml). The combined ethyl acetate extract was dried over anhyd. MgSO₄, filtered, and concentrated to afford E-stilbene (0.25 g, 95%); mp 122°C, lit. mp^[9] 124°C.

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