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SOLID-LIQUID PHASE TRANSFER CATALYTIC SYNTHESIS VIII: THE RAPID ALKYLATION OF ETHYL PHENYLSULFONYLACETATE UNDER MICROWAVE IRRADIATION

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ABSTRACT: Rapid alkylations of ethyl phenylsulfonylacetate with a series of halides were performed in 960 W domestic microwave oven, and the isolated yield of α-monoalkylated product varying from 76 to 86%.

In previous papers [1-6] we have reported the solid-liquid phase transfer catalytic alkylation, carbonyl addition, Michael addition and 1.3-dipolar cycloaddition. It is a convenient synthetic method for large scale preparation in the organic synthesis. Recently the rapid heating capablity of microwave oven has

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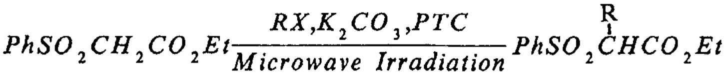
Table 1: Alkylation of ethyl phenylsulfonylacetate.

Entry	alkyl halide	mol. ratio (RX:Subst.)	irradiation time (min)	isolated yield (%)
1	$C_6H_5CH_2Cl$	1.4:1	3	76
2	$p-ClC_6H_4CH_2Br$	1:1	2	76
3	$\beta-C_{10}H_7CH_2Br$	1:1	2	86
4	n–OctylBr	1:1	3	79
5	n–BuBr	2:1	3	83

been used in the rapid organic synthesis [7-11], one of the major focuses for the phase transfer catalytic synthesis is to carry out some rapid organic reactions without solvent in only a few minutes. Here we would like to describe the procedure to perform rapid and efficient alkylation of ethyl phenylsulfonylacetate in microwave oven, using TEBA as phase transfer catalysis without solvent.



$$PhSO_2CH_2CO_2Et \xrightarrow[Microwave\ Irradiation]{RX, K_2CO_3, PTC} PhSO_2\overset{R}{C}HCO_2Et$$

1 **2**

The alkylation of **1** was simply achieved by microwave irradiation to the mixture of ethyl phenylsulfonylacetate, alkyl halide, potassium carbonate and TEBA for 2–3 minutes, followed by isolation and purification gave monoalkylated ethyl phenylsulfonylacetate in 76–86% yield(Table 1). All the products were characterised by IR, ^1H–NMR and MS (Table 2).

Table 2: Analytical data of compounds **2**

Products **2a–e**	IR (cm^{-1})	^{1}H–NMR($CDCl_3$) δ,J(Hz)	MS m / z(%)
2a	1740, 1306, 1140.	7.96–7.26(5H,m);7.07(5H,s); 4.10(1H,m);3.82(2H,q.J = 7.0Hz); 3.30(2H,d);0.87(3H,t,J = 7.0Hz)	273(M^+–OEt,1.06)
2b	1738, 1322, 1146.	8.15–6.60(9H,m);4.10(1H,m); 3.82(2H,q,J = 7.0Hz);3.78(2H,d); 0.88(3H,t,J = 7.0Hz)	353(M^++1,0.34); 355(M^++3,0.18); 307(M^+–OEt,7.03)
2c	1738, 1322, 1148.	8.18–6.95(12H,m); 4.50–3.06(3H,m); 3.78(2H,q,J = 7.0Hz), 0.82(3H,t,J = 7.0Hz).	369(M^++1,1.86); 323(M^+–OEt,1.87)
2d	1740, 1328, 1150.	7.86–7.26(5H,m);4.00(2H,q,J = 7.0Hz);3.80(1H,m);1.95–1.65 (2H,m);1.65–0.62(18H,m).	295(M^+–OEt,0.70)
2e	1738, 1311, 1150.	7.82–7.20(5H,m);3.95(2H,q,J = 7.0Hz);3.78(1H,m);2.05–1.65 (2H,m);1.05(3H,t,J = 7.0Hz); 1.35–0.60(7H,m).	285(M^++1,0.56); 239(M^+–OEt,6.14)

Experimental

General Considerations: IR spectra were recorded on a Nicolet MX–1 infrared spectrometer; ^{1}H–NMR spectra were recorded on a Varian FT–80A spectrometer using TMS as internal standard; MS data were obtained using a HP–5988A instrument; K_2CO_3 was grounded and dried at 500℃ for 3h.

General Procedure for the Alkylation of Ethyl Phenylsulfonylacetate: A mixture of ethyl phenylsulfonylacetate(1.14g, 5.0mmol),benzyl chloride(0.89g,7.0mmol),triethylbenzylammonium chloride (TEBA,0.23g, 1.0mmol) and potassium carbonate (3.0g,22mmol) was introduced into a FEIYUE 960W domestic microwave oven in an open container, microwave irradiation was carried out for 3 minutes at defrost power. Then 50ml ether was poured into cooled mixture,filtration, concentration and followed by purification through column chromatography (silica gel 100–200mesh; eluent:petroleum ether:EtOAc = 4:1) to give 2a. yield: 1.25g (79%).

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