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Microwave promoted radical cyclization of trihaloacetamides

Julio A. Seijas*, M. Pilar Vázquez-Tato*, Mercedes Jover

Departamento de Química Orgánca. Universidad de Santiago de Compostela.
Facultad de Ciencias. Aptdo. 280. 27080-Lugo. Spain
E-mail: qoseijas@lugo.usc.es, pilarvt@lugo.usc.es

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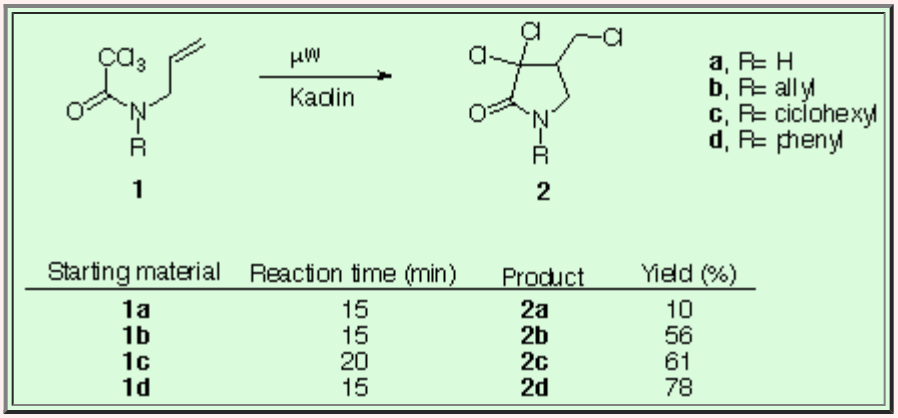
Pyrrolidines bearing bicyclic skeletons are components of various alkaloids or amino acid derivatives with important physiological activities. The efficient construction of the pyrrolidine ring is important in their synthesis. In the last years, most of the synthetic routes via radical cyclization of heteroatom-containing substrates, are based in generating an alkyl radical by treatment of haloalkanes with a radical initiator such as AIBN. One of the disadvantages of this methodology, which requires working with labile reagents, is the formation of a secondary product arising from the reduction of the halogen atom, on the contrary the copper (I) catalyzed radical atom transfer cyclization lack of this problem. We reported previously the synthesis[1] of pyrrolizidines by homolysis of trichloroacetamides in the presence of a catalytic amount of Cu(I). These reactions are usually performed in a sealed tube and heating acetonitrile at temperatures around 150°C[2]. Although less harsh conditions are needed when TMEDA[3]and sometimes bipyridine [4] are used.



1 **2**

Starting material	Reaction time (min)	Product	Yield (%)
1a	15	**2a**	10
1b	15	**2b**	56
1c	20	**2c**	61
1d	15	**2d**	78

Here we describe a new method, carrying out these reactions by irradiating with microwaves. Our first attempt was to irradiate a mixture of CuCl and allyltrichloroacetamides. As we had some sparking on the reaction beaker, we chose to use a solid support with reagents adsorbed on its surface. We tried some supports the better yields were obtained when kaolin was used. The cyclization of N-allyltrichloroacetamide gave poor yield, probably due to its high volatility, but when secondary amides were irradiated we obtained yields ranging between 56-78%.

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