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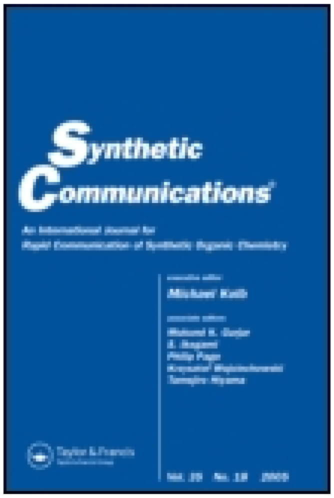

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ONE-POT SYNTHESIS OF 1,2,4-TRIAZINES

Shahnaz Rostamizadeh[a] & Kamran Sadeghi[a]

[a] Department of Chemistry , K. N. Toosi University of Technology , P.O. Box 15875-4416, Tehran, Iran

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ONE-POT SYNTHESIS OF 1,2,4-TRIAZINES

Shahnaz Rostamizadeh* **and Kamran Sadeghi**

Department of Chemistry, K. N. Toosi University of
Technology, P.O. Box 15875-4416 Tehran, Iran

ABSTRACT

1,2,4-Triazines have been prepared from the one-pot condensation reaction of acid hydrazide (**1**), ammonium acetate and dicarbonyl compounds (**2**) on the surface of silica gel in the presence of triethylamine under microwave irradiation.

In recent years there has been considerable interest in the use of dry media as nonconventional reaction medium for the preparation of organic compounds.[1]

Microwave irradiation has become a very useful tool in organic synthesis and a great number of classical organic reactions have been successfully carried out under microwave heating technique.[2]

1,2,4-Triazines are a representative class of heterocyclic compounds with a wide range of interesting properties which are used in medicine and agriculture.[3–6] These compounds have been prepared by different methods: a) Condensation of acid hydrazides with benzil in acetic acid containing

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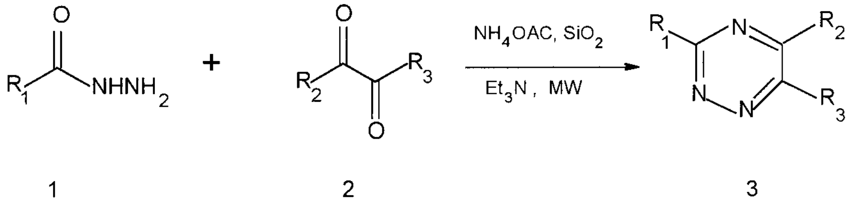
*Corresponding author.

ammonium acetate to give 5,6-diphenyl-1,2,4-triazine with various aromatic and heterocyclic groups attached at 3 position.[7] b) Cyclization of aliphatic, aromatic and heterocyclic acid hydrazide with 1,2-diketone monoacylhydrazones in alcoholic ammonia under pressure.[8,9] c) Condensation of monohydrazones of aromatic (but not aliphatic) 1,2-diketones with formation to give 5,6-disubstituted 1,2,4-triazines.[10] d) Condensation reaction of acid hydrazide and α-halo acetophenone in the presence of NaOAc or AgOAc.[11,12] e) Reaction of α-diketones with amidrazones and *S*-methylthiosemicarbazide were also reported in the literature.[14–16]

In this work, 1,2,4-triazines (**3a–i**) were obtained from the one-pot Condensation reaction of acid hydrazides (**1**), ammonium acetate and dicarbonyl compounds (**2**) on the surface of silica gel in the presence of triethylamine under microwave irradiation (Scheme 1, Table 1).

High yield, short reaction time, products and easy work up are advantages of this procedure in comparison to other methods. The data for the synthesis of 1,2,4-triazines are shown in Table 2.



Scheme 1.

Table 1. Cyclization of α-Diketone with Acid Hydrazide

Product	R_1	R_2	R_3
3a	Ph	Ph	Ph
3b	Ph	Et	Et
3c	Ph	Me	Me
3d	4-MeO-C_6H_4	Ph	Ph
3e	4-MeO-C_6H_4	Me	Me
3f	4-Me-C_6H_4	Ph	Ph
3g	Me	Ph	Ph
3h	Me	Et	Et
3i	Me	Me	Me

Table 2.

Product	Time (min)	Yield (%)	M.P. (°C)	Lit. M.P. (°C)	Ref.
3a	5	93	146–147	148	12
3b	11	88.5	56	56	8
3c	11	74	70–81	82	8
3d	7	71	158–159	159–160	20
3e	12	73.5	142–144	144–146	20
3f	7	78	138–140	138–140	19
3g	7	74	89–91	92	19
3h	12	61	18–21	19	8
3i	7	63	48–51	49–51	8

EXPERIMENTAL

All products are known compounds and were characterized by comparision of their spectral and physical data with those of known samples.

Melting points were taken on an Electrothermal 9100 apparatus. IR spectra were recorded with a Shimadzu IR-480 spectrometer (KBr). The ^{1}H-NMR spectra were determined in chloroform-d on a FT-NMR Bruker AC-80 (80 MHz).

Microwave irradiation was carried out using commercial microwave oven Moulinex (2735A). Merck silica gel 60GF254 was used for analytical and preparative TLC. Acid hydrazides were prepared according to the literature method.[17,18]

General Procedure for Synthesis of 3,5,6-Trisubstituted-1,2,4-triazines

A mixture of acid hydrazide (2 mmol), α-diketone (2 mmol) and silica gel (2 g) was well ground with a pestle and introduced to microwave irradiation in an open pyrex beaker for appropriate time (Table 1). After complete conversion as indicated by TLC, the mixture was extracted with petroleum ether (3 × 50 ml) and washed with water (3 × 50 ml). Then the solvent was evaporated in vacuum and the product was purified by chromatography on a short silica gel column with proper solvent or recrystallized directly from appropriate solvent.

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