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Green synthesis of some novel N-(1-(10H-phenothiazin-8-yl) ethylidene)-3-(10H-phenothiazin-10-yl)propanehydrazides

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ABSTRACT

A new series of title compounds (3a-f) were synthesized under microwave irradiation by using various acetyl phenothiazine (1a-b) and various phenothiaziyl propanehydrazides (2a-c). The structures of these compounds were confirmed by spectral and elemental analysis.

KEYWORDS

Phenothiazine;
Hydrazides;
Microwave synthesis;
Green synthesis.

INTRODUCTION

Phenothiazine compounds have shown various biological activities including as anti-inflammatory[1], antimalarial[2], antipsychotropic[3], antimicrobial[4-7], antitumor[8,9] antihistamine and analgesic[10] properties.

In a recent time, there has been much interest for the use of microwave irradiation in synthesis due to substantial reduction in time as well as eco-friendly. Apart from being environmentally friendly technique, microwave irradiation has carved its importance in the field of pharmaceutical chemistry for the synthesis of new potent drugs.

In view of these above, our project aimed to synthesis some novel phenothiazines by using green methodology.

MATERIALS AND METHODS

All chemicals were purchased from s.d fine chemicals, Mumbai (India), and were used without further purification. Melting points were determined in open capillaries using a Toshniwal melting point apparatus and are uncorrected. Formation of compounds was routinely checked by TLC using Silica G, and spots were exposed to iodine vapor for visualization.

The IR spectra in KBr were recorded on a Perkin - Elmer FT-IR spectrometer (vmax in cm-1); and 1H NMR spectra were obtained in CDCl3 on a Brucker 300 MHz instrument using TMS as internal standard (chemical shifts in δ, ppm), Mass spectra on LCQ davantage Therma Finiger spectrometer. Elemental analysis was performed on Carlo Erba 1108 analyzer.

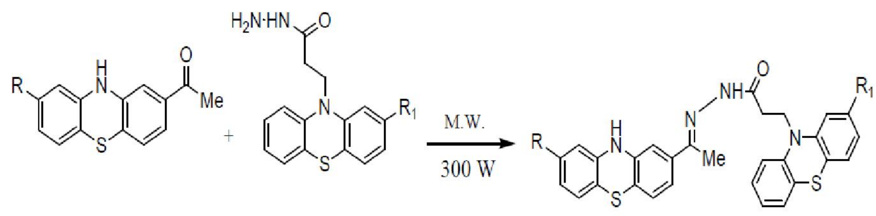
General procedure for 3a-f

(E)-N-(1-(10H-phenothiazin-8-yl)ethylidene)-3-(10H-phenothiazin-10-yl)propanehydrazide (3a)

To the solution of 2-acetyl Phenothiazine 1a (10 mmol) in absolute ethanol (20ml), 3-(10H-phenothiazin-10-yl)propanehydrazide 2a (10 mmol) was added and the reaction was carried out under microwave irradiation (300W) for 3-6 minutes. The solid obtained was washed with cold water finally recrystallised from ethanol to give 3a.

TABLE 1 : Physical data of compounds 3a-f

Comp. no	R	R1	M.F.	% Yield	M.P
3a	H	H	$C_{29}H_{24}N_4S_2O$	80	181-183
3b	H	Cl	$C_{29}H_{23}N_4S_2OCl$	78	178-180
3c	H	CF_3	$C_{30}H_{23}N_4S_2OF_3$	83	205-207
3d	Cl	H	$C_{29}H_{23}N_4S_2OCl$	83	217-219
3e	Cl	Cl	$C_{29}H_{22}N_4S_2OCl_2$	82	208-210
3f	Cl	CF_3	$C_{30}H_{22}N_4S_2OF_3Cl$	81	202-205



Scheme 1

IR (KBr) cm^{-1}:3355 (NH), 3050 (Ar-H), 1596 (C=N); ^{1}H NMR ($CDCl_3$): δ 8.32 (s,1H,NH), 8.03 (s, 1H, NH), 7.86 – 6.69 (m, 15H, Ar-H), 2.31 (t, 2H, CH_2), 2.18 (t, 2H, CH_2), 1.10 (s, 3H); Mass (m/z) : 508. Anal.cald.for $C_{29}H_{24}N_4S_2O$: C, 68.48; H, 4.76; N, 11.01. Found: C, 68.29; H, 4.88; N, 11.03%.

Similarly, 3b-f were synthesized by using various 2-acetyl Phenothiazine(1a-b) with various phenothiazyl hydrazines 2b-c.

RESULTS AND DISCUSSION

Phenothiazinyl propane hydrazides 3a–f were obtained by under microwave irradiation at 300 w in ethanol by treating various 2-acetylphenothiazine (1a-b) with various phenothiazyl propanehydrazides (2a-c). various amines and gave above 86% yield. The reaction sequences are outlined in Scheme 1. Formation of products were confirmed on the basis of elemental analysis, IR, 1H NMR, and mass. Compounds 3a showed IR absorption bands in the regions 3355 cm-1 (NH Streching), 3050 cm-1 (Ar C-H stretching), 1596 cm-1 (C=N streching). The 1H NMR spectrum of compound 3a showed two singlets at 8.32 and 8.02 ppm due phenothiazine NH and hydrazide NH protons respectively. It showed multiplets at 7.86–6.69 ppm due to aromatic protons, two triplets at 2.81 and 2.31 ppm due to aliphatic protons and singlet at 1.10 ppm due to methyl proton. The physical data of compounds 3a–f are recorded in TABLE 1.

CONCLUSIONS

A simple, efficient, and convenient method is developed for the synthesis new series of phenothiazyl propane hydrazides.

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