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Synthesis Of Hydantoins Under Microwave Irradiation By The Environmentally Benign Method

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Abstract:- The environmentally benign method includes microwave induced organic transportation which needs a little or no solvent. Uses of solvent like pyridine, hydrochloric acid, etc. are dangerous to the environment. Keeping this thing in mind, microwave irradiation has been chosen for heating purpose so as to carry out organic reactions and no any solvent. To activate the organic reactions, the household microwave oven has been used in place of traditional method of heating. The present work is based on household microwave heating and avoiding solvents.

Under microwave irradiation, hydantoins have been synthesized by a general procedure which is very simple and fast. Syntheses have been achieved only in few minutes instead of 3-4hours.The unique feature of the work is the denial of use of solvent. The yield of the products under solvent free condition was satisfactory and effective also.

Key Words: - Hydantoins, Microwave Irradiation, Solvent free condition, TLC, Open glass capillary, Elico melting point apparatus.

Introduction: -

Nowadays, epilepsy is one of the most common disorders of the central nervous system, worldwide. There is still an increasing need of research intothe newer molecules for treatment of epileptic seizures. As Primary aid, anticonvulsant drugs are effective to prevent and to control the epileptic seizures. For the treatment of epileptic seizures, the most common choice is Hydantoin.

To afford products in higher yields and in shorter reaction periods, microwave irradiation is an

efficient and environmentally benign method which activates various organic transformations. It also increases in the purity of the products and involves very little amount of solvent or no solvent[4-13]. Further, the use of microwave acceleration eliminates the need for heating baths, reaction flasks, and reflux condensers with ground glass joints.

Substituted hydantoins have been prepared by conventional method[14] as well as under microwave irradiation[15]. The objective of this present work is to synthesise the various hydantoins of medicinal value[15] under microwave irradiation, even in solvent free condition to minimise the cost of the product. In the presence of 30% aqueous sodium hydroxide,disubstituted -1, 2-diketones 1(a-c) are heated with ureaunder microwave irradiation, intermediate heterocyclic pinacols, 2(a-c), are obtained. Now this reaction mixture is cooled to room temperature and diluted with water., On acidification of the diluted intermediateswith concentrated hydrochloric acid, give hydantoins 3(a-c) as the result of a pinacolic rearrangement.

Experimental:-

A compound, 1a-c (0.025 mol) was mixed with 3.0 g (0.05 mol) of urea and 15ml of 30 percent aqueous sodium hydroxide solution. The reaction mixture was irradiated with microwaves at 40% (320 W) level in a kenstar OM-20 ESP (800 W), unmodified domestic oven operating at 2450 MHz for the time indicated in Table 1. Now the reaction mixture is cooled to room temperature, diluted with 125 ml of distilled water and then mixed well. After allowing it to stand for 15 minutes, the insoluble byproduct is filtered off. The filtrate is rendered strongly acidic with concentrated hydrochloric acid. Under suction the precipitated product is filtered. From industrial spirit, the product is recrystallised, finally.

5, 5-Diphenyl - 2, 4-imidazolidinedione (3a) :

IR (Nujol, υ_{max} , cm^{-1}) ; 730 (C-H deformation), 1490-1430 (phenyl ring breathing), 1720 (C=O stretching - C_4), 1780 (C=O stretching - C_2), 3200 (N-H stretching).

5, 5-Dimethyl -2, 4-imidazolidinedione (3b) :

IR (Nujol, υ_{max} cm^{-1}) ; 1150 (C-CH_3 stretching), 1450 (C-H deformation), 1710 (C=O stretching - C_4), 1780 (C=O

stretching - C_2), 3030 (C-H stretching), ^{1}H NMR (DMSO - d_6, δ ppm) ; 1.22 (s, 6H), 3.50 (s, 1Ha), 8.00 (s, 1Hb).

5-Methyl-5-phenyl-2, 4-imidazolidinedione (3c) :

IR (Nujol, υ_{max} cm^{-1}) ; 1480 (C-C stretching), 1720 (C=O stretching - C_4), 1790 (C=O stretching - C_2) 2980 (C-H stretching), 3300 (N-H stretching). ^1H NMR (DMSO - d_6, δ ppm) ; 1.15 (s, 3H), 6.90 (s, 5H), 3.40 (s.1Ha), 8.10 (s, 1Hb).

RESULT AND DISCUSSION

For few minutes, the reaction was attempted first with microwaves at 20% (160 W) level in a Kenstar OM-20 ESP (800 W) whereupon no any action was observed. With microwaves at 40% (320 W) level of full power, 800W of the oven used, the reaction was successfully attempted. By hit and trial method, the time taken for completion of reaction was decided. By traditional heating,[14] the time required for completion of reaction is about 2 hours but by microwave irradiation[15]is only 2-3.5 minutes using solvent like ethanol in large quantity. We have been successful in carrying out the reaction under microwave irradiation according to Scheme1.The cost of the product may be loweredappreciably by ignoring the use of solvent.

Reactions were carried out under atmospheric pressure in an open vessel adapted to a microwave oven.TLC was used to check the purity of the compounds. Melting points were taken in an open glass capillary using Elico melting point apparatus and are uncorrected. The compounds synthesised (3a-c) are all known compounds and have been characterised on the basis of their melting points, IR and NMR.

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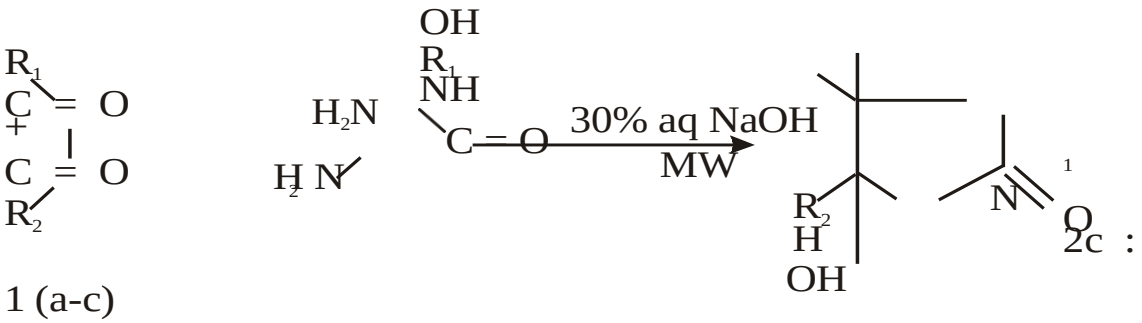
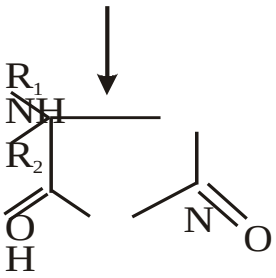


1 (a-c)

1a : $R_1 = C_6H_5$
$R_2 = C_6H_5$ 1b : $R_1 = CH_3$
$R_2 = CH_3$ 1c : $R_1 = CH_3$
$R_2 = C_6H_5$

2a : $R_1 = C_6H_5$
$R_2 = C_6H_5$
2b : $R_1 = CH_3$
$R_2 = CH_3$
2c : $R_1 = CH_3$
$R_2 = C_6H_5$

2 (a-c)



3 (a-c)

3a : $R_1 = C_6H_5$
$R_2 = C_6H_5$ 3b : $R_1 = CH_3$
$R_2 = CH_3$ 3c : $R_1 = CH_3$
$R_2 = C_6H_5$

(Scheme-1)

Table - 1

Results of hydantoins synthesised under microwave irradiation in solvent free condition

Compound	Experimental				Literature[15]	
	Time/ min	Yield%	M.P./^{0}C	Solvent for recrystallisation	Yield%	M.P./ ^{0}C
3a	2.0	90	296-297	Industrial spirit	91.71	297-298
3b	3.0	92	176-177	Industrial spirit	95.23	176-178
3c	3.5	92	146-147	Industrial spirit	94.85	146-148

3.4 **REFERENCES**

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