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Synthesis of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone using different heterogeneous catalysts in dry media under microwave ir-radiation.

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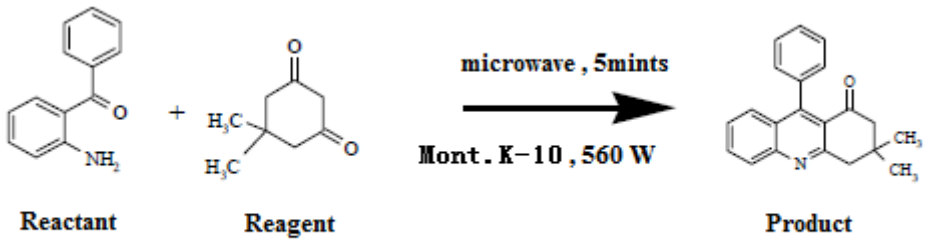
Abstract:
The3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinoneis synthesized in dry media using heterogeneous catalyst in high yield in shorter reaction time under microwave ir-radiations.
Key Words: *Indole, Microwave, Aryl, heterocyclic.*

I. Introduction:

The trisubstituted quinolines have wide range of biological activities as anti-malarial, anti-bacterial, anti-asthmatic, anti-hypertensive, anti-inflammatory, anti-platelet activity and as tyro-kinase PDGF-RTK inhibiting activity.[1-3] The synthesis of trisubstituted quinolines under conventional refluxing conditions require longer reaction time and tedious work up so, there existed a need for alternative methods to carry out the synthesis of trisubstituted quinolines. Microwave assisted reactions are gaining much more importance in synthetic organic chemistry due to dramatic reduction in time from days to hours and hours to minutes or seconds.[4-24] The conventional heating reaction conditions are modified by changing media and catalyst. The present work reports the synthesis of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone in dry media using heterogeneous catalyst in high yield in shorter reaction time under microwave irradiations (Scheme-I).



(Scheme)

We initiated our investigations by condensing2-aminobezophenone with3,3-dimethyl-5-oxo-cyclohexanone at 80 W,160 W,240 W,320 W,400 W,480 W and 560 W in the presence of Mont.K-10. The results obtained are shown in Table-1 below. As can be seen from the Table-1 that when 2- aminobezophenone react with3-oxo-cyclohexanone to give 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone, 560 W power level proved to be the best from the yield point of view.

Table-1: Synthesis of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone using Mont.K-10under various ir-radiation (power levels).

Sr.N.	Power Levels (watts).	Yield (%).	Time (mints.)
1	80	62	5
2	160	66	5
3	240	69	5
4	320	73	5
5	400	76	5
6	480	80	5
7	560	83	5

We next carried out the formation of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone by condensing 2-aminobezophenone with 3,3-dimethyl-5-oxo-cyclohexanone at 560W in the presence of Mont. KSF, silica gel, $MgSO_4$(anhyd), Na_2SO_4(anhyd), $Yb(OTf)_3$, $Sc(OTf)_3$, $Dy(OTf)_3$, $Gd(OTf)_3$, $InCI_3$,$Y(OTf)_3$ and $Bi(OTf)_3$ catalysts. The results obtained have also been collected in the Table 2.

Table-2: Synthesis of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone using different catalysts at 560W under various ir-radiation.

Sr.No.	Catalyst	Time (in min.)	Yield (%)
1	Mont. K-10	5	83
2	Mont. KSF	5	82
3	Silica gel	5	80
4	$MgSO_4$(anhyd)	5	79
5	Na_2SO_4(anhyd)	5	78
6	$Yb(OTf)_3$	5	77
7	$Sc(OTf)_3$	5	76
8	$Dy(OTf)_3$	5	75
9	$Gd(OTf)_3$	5	74
10	InCl3	5	73
11	$Y(OTf)_3$	5	72
12	$Bi(OTf)_3$	5	71

II. Experimental:

All the melting points reported are uncorrected. Infrared spectra (V_{max} in cm^{-1}) were recorded in nujol mull or KBr on a Perkin-Elmer 842/Beckman IR-20 / Hitachi 215 spectrometers. The proton magnetic resonance spectra were recorded on a VXR-200 MHz or R-32 Perkin-Elmer 90 MHz spectrometer in $CDC1_3$ or DMSO-d_6 using tetramethylsilane (TMS) as internal reference standard. The chemical shifts are expressed in δ (ppm) units downfield from TMS. Mass spectra were scanned on a Jeol JMX-DX-300 spectrometer operating at 70 eV. Carbon, hydrogen and nitrogen analyses were carried out on a Yanaco MT-3 (JAPAN) instrument. Thin layer chromatography (TLC) were performed on silica-gel plates using acetone-benzene (1 : 3 or 1 : 2) as solvent system and iodine chamber as visualizing agent.

Typical procedure for the synthesis of 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone**:**Amixture of 2-aminobezophenone(1mmole),3,3-dimethyl-5-oxo-cyclohexanone(1mmole), catalyst(1g) was taken in an Erlenmeyer flask(100 ml) and was irradiated for 5 minutes at 70% power level (560 W) in an unmodified domestic microwave oven operating at 2450 MHz. After cooling to room temperature, the crude product was extracted and recrystallised with ethanol to yield pure 3,3-Dimethyl-9-phenyl-1,2,3,4-tetrahydro-1-acridinone.Mp observed : 191°C , reported:190-192°C: 1H-NMR(δ in ppm in CDCl3): δ = 1.1(s, 6H), 2.59 (s, 2H), 3.3 (s, 2H), 7.1-7.2 (m, 2H),7.3-7.55 (m, 5H), 7.78 (t, 1H), 8.1(d, J = 8Hz, 1H)and IR (KBr, cm-1) : 3061, 2956, 1712,1602, 1573, 1206, 735.

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References:

[1]. Larsen, R. D. ; Corley, E. G.; King, A. O. ; Carrol. J. D.; Davis, P.; Verhoeven , T. R; Reider, P. J. ; Lablle, M. ; Gauthier, J. Y. ; Xiang, Y. B.; Zamboni, R. J. J. Org. Chem. **1996**, 61, 3398. (b) Chen, Y. L.; Fang, K. C.; Sheu, J. Y.; Hsu, S. L.; Tzeng, C. C. J. Med. Chem. **2001**, 44, 2374. (c) Roma, G.; Braccio, M. D.; Gmattioli, G. F.; Ghia, M. Eur. J. Med. Chem. **2000**, 35, 1021.
[2]. Kalluraya, B.; Serrnivasa, S.; Farmaco **1998,** 53, 399. (b) Doube, D.; Blouin, M.; Brideau, C.; Chan, C.; Desmarais, S.; Eithier, D.; Fagueyret, J. P; Friesen, R. W.; Girard, M.; Girard, Y.; Guay, J.; Tagari, P.; Young, R. N., Bioorg. Med. Chem. Lett. **1998**, 8, 1255.
[3]. Ko, T. C.; Hour, M. J.; Liem, J. C.; Teng,C. -M.; Lee, K -H.; Kuo, S.-C.; Huang, L.-J.; Bioorg. Med. Chem. Lett. **2001**, 11, 279. (b) Ferrarini, P. L.; Mori, C.; Badwneh, M.; Manera, C.;Martinelli, A.; Miceli, M.; Ramagnoli, F.; Saccomanni, G. J. Hetetocycl. Chem. **1997**, 34, 1501. (c) Maguire, M. P.; Sheets, K. R.; Mcverty, K.; Spada, A. P.; Zilberstain, A. J. Med. Chem. **1994**, 37, 2129.
[4]. Anastas, P.T. and Farris, C.A. (Eds.), Benign by Design: Alternative Synthetic Design for Pollution Prevention, ACS symposium, Ser. N. 557. Washington DC, 1994.
[5]. Collins, T. Towards sustainable chemistry, Science, 2001, 291, 5501, 48.
[6]. Anastas, P. and Warner, J.C. Green Chemistry: Theory and Practice, Oxford Science Publicatons, Oxford, 1998.
[7]. Collins, T.J. Green Chemistry, Macmillan, Encyclopedia of Chemistry, New York, 1997.

[8]. Wilkinson, S.L. "Green" Is practical, Even Profitable. No longer a luxury. Green Chemistry becomes a central strategy for sustainable firms', Chem. Eng. News, 1997, 75, 35-43.
[9]. Sanghi, R. 'Better living through sustainable Green chemistry', Current Science, 2000, 79, 12, 1662.
[10]. Tundo, P. and Selva, M. Green Chemistry: Designing Chemistry for the Environment, Williamson Eds. ACS Sym Series No. 626, 81, 1996.
[11]. Tundo, P. and Anastas, P.T. (Eds.), Green Chemistry: Challenging Perspectives, Oxford University Press, Oxford 2000.
[12]. Goehl, T.J. 'Green Chemistry', Env. Health Perspectives, 1997, 105, 3.
[13]. Anastas, P.T. and Williamson, T.C. (Eds.), Green Chemistry: Frontiers in Chemical Synthesis and Processes, Oxford University press, Oxford, 1988.
[14]. Anastas, P.T. 'Green Chemistry and the Role of Analytical Methodology Development', Critical Rev. Anal. Chem. 1999, 29, 3, 167-175.
[15]. Strauss, C.R., Aust. J. Chem. 1999, 52, 83.
[16]. Caddick, S. Microwave assisted organic reactions. Tetrahedron, 1995, 51, 10403.
[17]. Bose, A.K., Banik, B.K., Lavlinskaia, N., Jayaraman, M., Manhas, M.S., MORE chemistry in a microwave. Chemtech, September 1997, 18.
[18]. Krstenansky, J.L., Cottrerill, I. Recent advances in microwave-assisted organic synthesis. Curr. Opin. Drug Discovery Dev. 2000, 4, 454-461.
[19]. Wilson, N.S., Roth, G.P. Recent trends in mcrowave-assisted synthess. Curr. Opin. Drug Discov. Dev. 2002, 5, 620-629.
[20]. Kappe, C.O., Stadler, A. Microwave-assisted combinatorial chemistry. In "Microwaves in Organic Synthesis"; Loupy, A., ed, Wiley-VCH, 2002, in press.
[21]. Loupy, A., Petit, A., Hamelin, J., Texier-Boullet, F., Jacquault, P., Mathe, D. New solvent-free organic synthesis using focused microwaves. Synthesis 1998, 1213.
[22]. Varma, R.S., Solvent-free organic syntheses using supported reagents and microwave irradiation. Green Chem. 1999, 43-55.
[23]. Kidawi, M. Dry media reactions. Pure Appl. Chem. 2001, 73, 147-151.
[24]. Varma, R.S. Solvent-free accelerated organic syntheses using microwaves. Pure Appl. Chem. 2001, 73, 193-198.