

Efficient Synthesis of 6-Methyl-4-phenyl-4,7-dihydrotetrazolo[5,1-c][1,2,4]triazine under Solvent-free Conditions

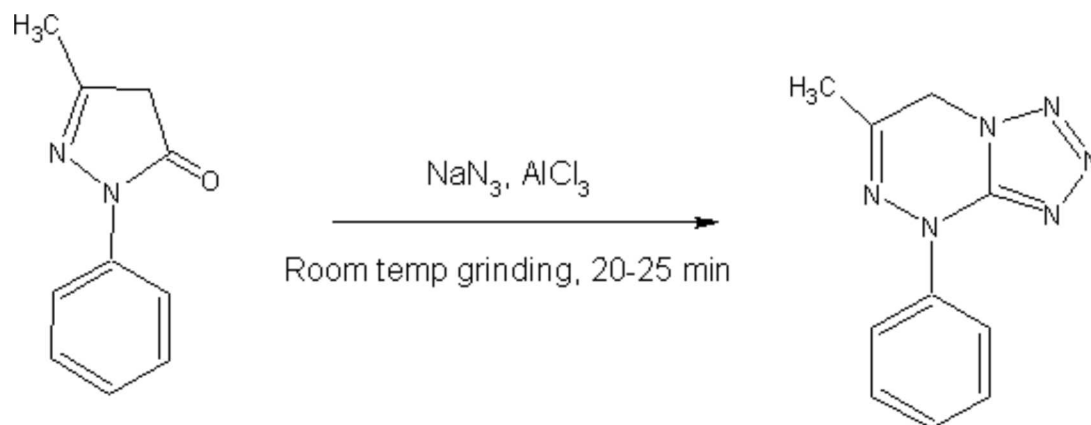
M. Raghavendra, Halehatty S. Bhojya Naik ^{*}, Tangali R. Ravikumar Naik and Bailure S. Sherigara

Department of PG Studies and Research in Industrial Chemistry, School of Chemical Sciences, Kuvempu University, Shankaraghatta- 577-451, Karnataka, India

^{*} Author to whom correspondence should be addressed. E-mail: hsb_naik@rediffmail.com

Received: 11 January 2006 / Accepted: 16 January 2006 / Published: 31 May 2007

Keywords: tetrazolotriazine, sodiumazide, solvent free conditions



In recent years reaction under solvent free conditions gaining much popularity because under solvent free conditions products obtains in high yield, avoids the use of hazardous and expensive solvents, and makes the manipulation much easier.

Tetrazoles and triazines posses wide class of biological activities [1-6]. It is known that fused tetrazoles can be prepared by employing Schmidt reaction of ketones with hydrazoic acid or inorganic azides [1], but inorganic azides are poorly soluble in organic solvent results increase in reaction time and decrease in yield. So, we wished to carry out above reaction under solvent media using anhydrous AlCl_3 as catalyst.

Mixture of 5-methyl-2-phenyl-2,4-dihydro-3H-pyrazol-3-one (0.696 g, 0.004 mol) 1, and Sodium azide (1.3 g, 0.020 mol) was ground portion-wise with anhydrous aluminium chloride (0.012 mol) taken in the mortar, continued the grinding for 20 min, During grinding considerable heat liberates which is sufficient to complete the reaction. After the completion of the reaction, reaction mixture was poured into water obtained solid was filtered, washed with water, recrystallised from aqueous DMF.

Melting Point: 151-152 °C

MS (m/z, %): 214 ($[\text{M}+\text{H}]^+$, 100%).

IR (KBr, cm^{-1}): 1525, 1480, 1425, and 1245 (tetrazole region), 1650 (C=N)

$^1\text{H-NMR}$ (400 MHz, DMSO-d_6): 4.35 (2H, s, $-\text{CH}_2-$); 2.54 (3H, s, CH_3); 6.90-7.10 (5H, ArH.).

References

1. Wittenberger, S.; *J. Org. Prep. Proced. Int.* **1994**, *26*, 499.
2. Butler, R.N.; *Comprehensive Heterocyclic Chemistry II*; Storr, R. C., Ed.; Elsevier: Oxford, UK, 1996, *4*, 621
3. Juby, P.F.; Hudyma, T.W.; Brown, M. *J. Med. Chem.* **1968**, *11*, 111
4. Herbst, R.M.; Roberts, C.W.; Givens, H.T.F.; Harvill, E.K. *J. Org. Chem.* **1952**, *17*, 262
5. Segarra, V.; Crespo, M.I.; Pujol, F.; Beleta, J.; Domenech, T.; Miralpeix, M.; Palacios, J.M.; Castro, A.; Marinez, A. *Bioorg. Med. Chem. Lett.* **1998**, *8*, 505
6. O'Brien, P.M.; Sliskovich, D.R.; Bernabei, A.; Hurley, T.; Anderson, M.K.; Bousley, R.F.; Krause, B.R.; Stanfield, R.L. *Bioorg. Med. Chem. Lett.* **1995**, *5*, 295
7. Neunhoffer, H.; and wiley P. F.; *Chemistry of 1,2,3-triazines and 1,2,4-triazines, tetrazines and pentazines*, ed. A. Weissberger and E. C. Taylor, 1978, *J. Wiley and Sons New York, Brisbane, Toronto* 1001

© 2007 by MDPI (<http://www.mdpi.org/>). Reproduction is permitted for noncommercial purposes.