



One-Pot Synthesis of 1,8-Dioxo-octahydroxanthene Derivatives

SEYYEDEH NAGHMEH SADAT and FARHAD HATAMJAFARI*

Department of Chemistry, College of Science, Tonekabon Branch,
Islamic Azad University, Tonekabon, Iran.

*Corresponding author E-mail: f_hatamjafari@tonekaboniacu.ac.ir, hatamjafari@yahoo.com

<http://dx.doi.org/10.13005/ojc/310275>

(Received: January 03, 2015; Accepted: February 16, 2015)

ABSTRACT

An efficient, simple and one-pot protocol for synthesis of 1,8-Dioxo-octahydroxanthene derivatives via multi-component reactions between dimedone and various aromatic aldehydes employing barium perchlorate as catalyst is described. The structural features of the synthesized compounds were characterized by IR and ¹H NMR. The presented method is available, environmentally friendly, cheap and highly effective to give the products in good to excellent yields.

Key words: Xanthenes, Solvent-free, Multicomponent reactions, One-pot.

INTRODUCTION

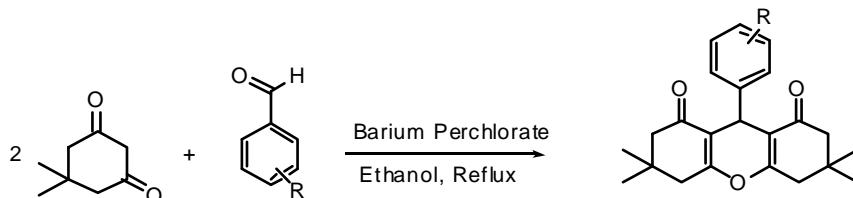
In recent years, the uses of catalysts supported on solid supports have been extensively developed because such catalysts not only cause to simplify the purification processes but also do not release toxic substances residues into the environment. Although some of them are sensitive to moisture and can be easily decomposed, application of them in organic reactions is difficult. This problem can be solved by fixed onto solid supports¹⁻³.

Xanthene derivatives are one of the important classes of organic compounds and there are many applications which are biologically important

drug intermediates in the field of medicinal chemistry for their biologically active properties, such as antinociceptive activities as well as their efficiency in photodynamic therapy antimarial, antibacterial, antiinflammatory, and antiviral properties and have been used as dyes, fluorescent material and in laser technologies⁶⁻¹². Recently, several improved methodologies have been developed that use HClO₄-SiO₂¹³, ZnO¹⁴ triethylbenzyl phosphomolybdic acid supported on silica gel¹⁵, sulfonic acid on silica gel¹⁶, ammonium chloride¹⁷, *p*-dodecyl benzenesulfonic acid¹⁸, and Zn(NO₃)₂¹⁹ among others. Previously, we have synthesized a number of heterocyclic compounds²⁰⁻³⁰.

In this study, we have used of perchlorate as a catalysts to develop a new and easy methodology for the synthesis of xanthene derivatives. The experiments were started with the study of one

pot reaction, available time reaction with high yields, easy separation of product, and a 3-component method, mild and efficient method for the preparation of the xanthenes (Scheme1).



Scheme1

EXPERIMENTAL

All chemicals were obtained from Merck or Fluka without further purification. Silica gel SILG/UV 254 plates were used for TLC. IR spectra were measured on a Shimadzu IR-470 Spectrophotometer. ^1H NMR spectra were determined on Bruker 400 DRX AVANCE instrument at 400 MHz, respectively.

General procedure for preparation of N1

A mixture of benzaldehyde (1 mmol), dimedone (2 mmol), barium perchlorate (15 mol %) as a catalyst with 10 ml ethanol as a solvent was refluxed at 3 hours. The progress of reaction was monitored by TLC. After finishing, recrystallized from ethanol 95% to give pure products (N1)

spectral data for N1

White Crystals, Yield: (91%), m.p 200-204 °C.
FT-IR (Vmax/cm⁻¹) (KBr disc): 3000 (CH arom. Str.); 2940 (CH aliph Str.); 1600 (C=O Str.); 1500 (C=C Str.).

^1H NMR (400 MHz CDCl_3) δ (ppm) = 1.14 (6H, s, 2CH₃); 1.28 (6H, s, 2CH₃); 2.38-2.51 (8H, m, 4CH₂); 5.55 (2H, s, CH), 7.18-7.35 (5H, m, CH arom.).

RESULTS AND DISCUSSION

We have been able to introduce an efficient and environmentally friendly for the synthesis of xanthene derivatives via condensation of dimedone with various aromatic aldehydes. Therefore, reported new catalyst which could provide an efficient, cheap, easy separation, high yield and simple route under solvent-free condition for the synthesis of 1,8-Dioxo-octahydroxanthenes.

ACKNOWLEDGEMENTS

We gratefully acknowledge the financial support from the Research Council of Tonekabon Branch Islamic Azad University.

REFERENCES

- Clark, J. H. *Acc Chem Res.*, **2002**, 35, 791-797.
- Salehi, P.; Zolfigol, M. A.; Shirini, F.; Baghbanzadeh, M. *Curr Org Chem.* **2006**, 10, 2171-2189.
- Mohammadizadeh, M.R.; Hasaninejad, A.; Bahramzadeh, M.; Khanjariou, Z. S. *Synth Commun.* **2009**, 39, 1152-1162.
- Hasaninejad, A.; Zare, A.; Sharghi, H.; Shekouhy, M. *Arkivoc*, **2008**, xi, 64-67.
- Hasaninejad, A.; Zare, A.; Balooty, L.; Mehregan, H. *Synth Commun.* **2010**, 40, 3488-3495.
- Iranpoor, N.; Firouzabadi, H.; Jamalian, A.; Kazemi, F. *Tetrahedron*, **2005**, 61, 5699-5704.
- Kalinski, C.; Lemoine, H.; Schmidt, J.; Burdack, C.; Kolb, J.; Umkehrer, M.; Ross, G. *Synlett*, **2008**, 24, 4007-4011.
- Peet, N. P.; Huber, E.W.; Huffman, J. C. *J. Heterocycl. Chem.* **1995**, 32, 33-38.

9. Schumacher, K.; Ravikovich, P.I.; Du Chesne, A.; Neimark, A.; Unger, K. K. *Langmuir*, **2000**, 16, 4648 -4654.
10. Chibale, K.; Visser, M.; Schalkwyk, D.V.; Smith, P. J.; Saravanamuthu, A.; Fairlamb, A. H. *Tetrahedron*, **2003**, 59(13), 2289 -2296.
11. Hideo, T.; *Jpn. Tokkyo Koho JP 56005480., Chem. Abst.*, **1981**, 95, 80922b.
12. Poupelin, J. P.; Saint-Rut, G.; Fussard-Blanpin, O.; Narcisse, G.; Uchida-Ernouf, G.; Lakroix, R. *Eur. J. Med. Chem.* 1978, 13, 67-71.
13. Kantevari, S.; Bantu, R.; Nagarapu, L. *J Mol Catal A: Chem.* **2007**, 269, 53-57.
14. Maghsoodlou, M. T.; Habibi-Khorassani, S. M.; Shahkarami, Z.; Maleki, N.; Rostamizadeh, M. *Chin Chem Lett.* **2010**, 21, 686-689.
15. Srihari, P.; Mandal, S. S.; Reddy, J. S. S.; Srinivasa Rao, R.; Yadav, J. S. *Chin. Chem. Lett.* **2008**, 19, 771-774.
16. Mahdavi, G. H.; Bigdeli, M. A.; Saeidi Hayeniaz, Y. *Chin. Chem. Lett.* **2009**, 20, 539-541.
17. Wang, X. S.; Shi, D. Q.; Li, Y. L.; Chen, H.; Wei, X. Y.; Zong, Z. M. *Synth Commun.* **2005**, 35, 97-104.
18. Jin, T. S.; Zhang, J. S.; Xiao, J. C.; Wang, A. Q.; Li, T. S. *Synlett.* **2004**, 866-870.
19. Vahabi, S. A. H.; Hatamjafari, F.; Pourshamsian, K. *Orient. J. Chem.* **2014**, 30, 849-851.
20. Azizian, J.; Hatamjafari, F.; Karimi, A. R.; Shaabanzadeh, M. *Synthesis*, **2006**, 5, 765-767.
21. Azizian, J.; Shaabanzadeh, M.; Hatamjafari, F.; Mohammadizadeh, M.R. *Arkivoc*, **2006**, xi, 47-58.
22. Hatamjafari, F. *Synthetic Communications.* **2006**, 36, 3563-3570.
23. Azizian, J.; Hatamjafari, F.; Karimi, A. R. *Journal of Heterocyclic Chemistry*, 2006, 43, 1349-1352.
24. Hatamjafari, F.; Montazeri, N. *Turkish Journal of Chemistry*, **2009**, 33, 797-802.
25. Hatamjafari, F. *Orient. J. Chem.* **2012**, 28, 141-143.
26. Hatamjafari, F. *Orient. J. Chem.* **2013**, 29, 93-95.
27. Hatamjafari, F.; Aljanichakoli, F. *Orient. J. Chem.* **2013**, 29, 145-147.
28. Hatamjafari, F.; Hosseiniyan, A. *Orient. J. Chem.* **2013**, 29, 109-111.
29. Hatamjafari, F.; Khojastehkouhi, H. *Orient. J. Chem.* **2014**, 30, 329-331.
30. Hatamjafari, F.; Germani Nezhad, F. *Orient. J. Chem.* **2014**, 30, 355-357.