



ISSN 0975-413X
CODEN (USA): PCHHAX

Der Pharma Chemica, 2022, 14(5): 24-27
(<http://www.derpharmachemica.com/archive.html>)

Synthesis of Substituted 1, 3- Dipropanone Containing Phenol Group Synthesized From 4-Bromophenol

Dongapure A.C^{1*}, Choudhari P.P²

¹Department of Chemistry, Shankarlal Agarwal Science College Salekasa Dist Gondia, M.S., India

²Department of Chemistry, G.S Tompe College, Amravati, M.S., India

*Corresponding author: Dongapure A.C, Department of Chemistry, Shankarlal Agarwal Science College Salekasa Dist Gondia, M.S., India, E-mail: avinashdongapure888@gmail.com

Received: 30-Mar-2022, Manuscript no: dpc-22-59067, Editor assigned: 02- Apr-2022, PreQC No: dpc-22-59067, Reviewed: 16- Apr -2022, QC No: dpc-22-59067, Revised: 26-Apr -2022, Manuscript No: dpc-22-59067, Published: 03-May-2022, DOI: 10.4172/0975-413X.14.5.24-27

ABSTRACT

A 1, 3- Dipropanone is a molecule containing two ketone groups. It is used as a ligand for the preparation of various coordination complexes. In the present investigation bromo substituted diketone namely 1-(5-bromo-2-hydroxyphenyl)-3-p-tolylpropane- 1,3- dione was prepared by using a generally known method [1]. The purity of synthesized compounds was checked by the thin layer chromatography.

Keywords: 1, 3- Dipropanone; 4-Bromophenol

INTRODUCTION

In chemistry synthesis of 1,3-diketones is more important because it is key intermediates synthesis of Carbon-Carbon bonds in a vast quantity of biologically and pharmaceutically products. Their various properties like availability, stability make β -diketones useful in a number of areas of the human being. Various β -diketones were very reactive with those β -diketones which are structurally similar to natural products. The researchers who are doing research in numerous fields of medicinal chemistry and coordination chemistry are interested in β -diketones moieties [2,3]. In multistage reactions for the synthesis of biologically active compounds, β -diketones are used as intermediates reactions, in mainly regio- and stereoselective synthesis [4,5]. In coordination, chemistry compare to other ligands the β -diketones ligand is used most frequently [6,7]. Because of tautomerism (keto- enol) and twice oxygen donor atoms and facile [8], hence β -diketones as a ligand simply coordinate with metal ions and form a six-membered chelate complex [9]. Last few years, in physical chemistry studies metal complexes synthesized by β -diketones as a ligand, have been used as model complexes. β -diketones and their moieties have been used as chelating ligands for the lanthanides and transition metals [10]. They are bidentate ligands with the possibility of complexation with almost all the metal ions [11]. Volatility [12], lewis acidity or aggregation state [13], standard molar enthalpies of sublimation and formation, vapor pressures [14], and enantioselective catalytic have been reported of various moieties of β -diketones [15, 16]. Biological activities of β -diketones particularly antioxidants [17], antitumor [18,19], and antibacterial activities [20], and β -diketone act as a vital mediator to synthesize numerous heterocyclic compounds [21,22]. It was therefore thought interestingly to use 4-Bromophenol to synthesize new and novel substituted 1, 3- Dipropanone. Hence, the present work deals with the synthesis of a new substituted 1, 3- Dipropanone from 4-Bromophenol, its characterization by IR, PMR, and Mass spectral data.

EXPERIMENTAL

The melting points were recorded using Thiel's tube containing parafine oil and a thermometer. The IR spectrum was recorded in KBr pellets. The NMR spectra were recorded using DMSO as solvent and TMS as an internal standard. The product was purified by recrystallization and their purity was tested by thin-layer chromatography on silica layers. Synthesis of Substituted 1, 3- Dipropanone involves the following preparatory steps:

Preparation of 4-Bromophenyl acetate (I)

Initially, acetic anhydride in presence of anhydrous sodium acetate is heated with 4-Bromophenol (A) for an hour and allow to cool at room temperature and then pour ice-cold water. Separate the water organic layer using a separating funnel which was purified by distillation to obtain 4-Bromophenyl acetate (I).

Preparation of 1-(5-bromo-2-hydroxyphenyl)ethanone (II)

When 4-Bromophenyl acetate (I) was going through the Fries rearrangement in presence of anhydrous AlCl_3 gives 1-(5-bromo-2-hydroxyphenyl)ethanone (II).

Preparation of 2-acetyl-4-bromophenyl 4-methylbenzoate (III)

1-(5-bromo-2-hydroxyphenyl) ethanone (II) and 4-methylbenzoic acid were dissolved in pyridine and POCl_3 was added drop by drop with constant stirring till the viscous mass is obtained. Maintain the temperature below 10°C during the addition of POCl_3 to the reaction mixture. The reaction mixture was kept overnight at room temperature and then decomposed by 10% HCl. The product thus separated and was filtered, washed with water followed by 10% NaHCO_3 solution, and then again with water. Finally, it was crystallized from ethanol to obtain 2-acetyl-4-bromophenyl 4-methylbenzoate (III).

Preparation of 1-(5-bromo-2-hydroxyphenyl)-3-p-tolylpropane-1,3-dione through Baker-Venkataraman transformation (IV)

2-acetyl-4-bromophenyl 4-methylbenzoate (III) was dissolved in pyridine (40 ml). The solution was warmed up to 60°C and pulverized KOH was added slowly with constant stirring. After 4 hours the reaction mixture was acidified by ice-cold dil.HCl (1:1). The solid product thus separated was filtered, washed with 10% NaHCO_3 , and finally several times with water. It was then recrystallized from ethanol to obtain 1-(5-bromo-2-hydroxyphenyl)-3-p-tolylpropane-1,3-dione (IV). The reaction scheme for the synthesis of substituted 1, 3- Dipropanone from 4-Bromophenol is shown below in Figure-1.

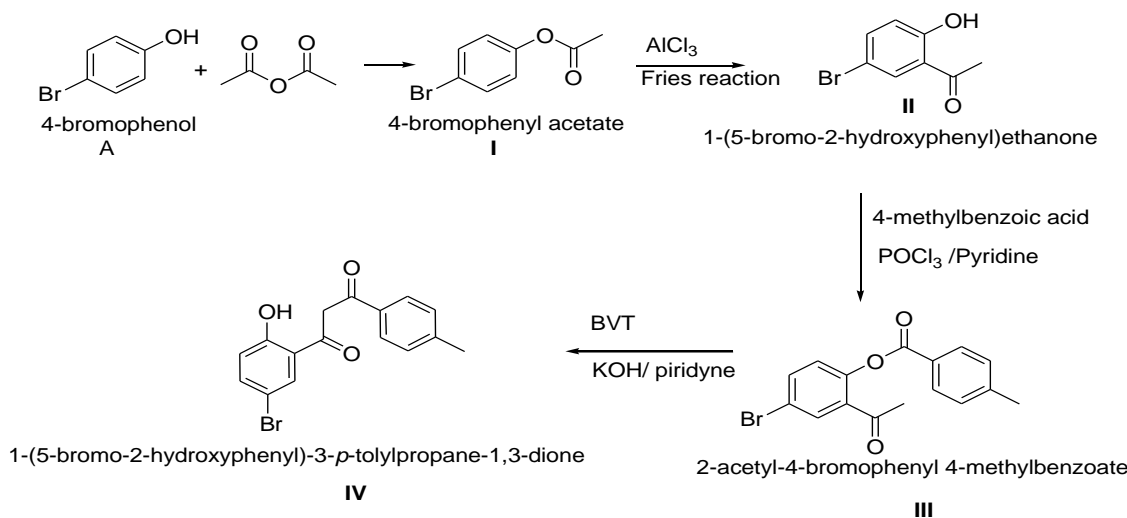


Figure-1.

Figure 1: Reaction scheme for the synthesis of substituted 1, 3- Dipropanone from 4-Bromophenol

RESULTS AND DISCUSSION

The physical and analytical data of synthesized substituted 1, 3- Dipropanone (IV) is given in Table-1, and their IR, PMR, and Mass spectral data are illustrated in Table-2, 3, 4 respectively.

Table 1: Physical and analytical data of compound IV

Formula	Exact Mass	Mol. Wt.	Melting Point	% Yield	Elements (%)	C	H	O
$\text{C}_{16}\text{H}_{13}\text{BrO}_3$	332	333.18	618.84 [K]	70	Observed	54.90	3.69	18.26
					Calculated	55.01	3.75	18.28

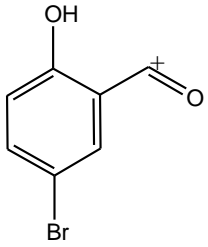
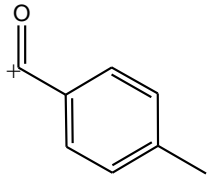
Table 2: IR Spectral data of compound IV

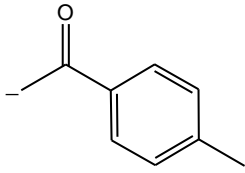
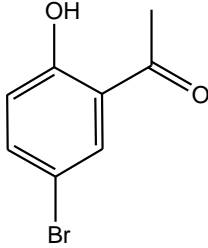
Literature value cm ⁻¹	Frequency cm ⁻¹	Assignment
3650-3600	3648	Ar-OH stretching (free)
3000-2850	2949	C-H stretching (Aliphatic)
1725-1675	1687	C=O (Ketonic)
1600-1500	1558	C=C (Aromatic)
1300-1000	1263	C-O stretch
< 667	616	C-Br stretch

Table 3: PMR Spectral data of compound IV

Peak observed	Multiplicity	No. of Proton	Assignment
2.35	Singlet	3H	Ar-CH ₃
3.7-3.9	Singlet	2H	0
4.7	Singlet	1H	Phenolic -OH
6.73	Doublet	1H	Aromatic-H
7.45	doublet	1H	Aromatic-H
7.86	Singlet	1H	Aromatic-H
7.74	doublet	2H	Aromatic-H
7.17	doublet	2H	Aromatic-H

Table 4: Mass Spectral data of compound IV

m/e value	Probable fragments
331.00	[C ₁₆ H ₁₃ BrO ₃] ⁺
199.90	
120.12	

133.01	
213.00	
15	CH ₃
410	Due to impurity

CONCLUSION

The substituted 1, 3- Dipropanone compound (IV) containing 4-Bromophenolic moiety was successfully synthesized. Which will be used for a ligand for various metal-ligand complex syntheses.

ACKNOWLEDGEMENT

The author is thankful to Principal, G.S Tompe College, Amravati for necessary facilities.

REFERENCES

- [1] Bodkhe PS. Journal of Engee. Science. **2020**, (4)11: p. 1055.
- [2] Kelin AV. Current Organic Chemistry. **2003**, 7: p. 1691-1711.
- [3] Isakova VG. Russian Chemical Reviews. **2010**, 79(10): p. 849-879.
- [4] Gore HA. Current Medicinal Chemistry. **2003**, 10: p. 51-80.
- [5] Simon C. Eur J Org Chem. **2004**, 24: p. 4957-4980.
- [6] Maurya SRRC. J Mol Structure. **2004**, 687: p. 35-44.
- [7] Raphael Meldola. J Chem Soc Trans. 1910: 97: p. 444-456.
- [8] A Z El-Sonbati. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy. **2012**, 99: p. 353-360.
- [9] Valeriia Zozulia, Vladimir Ovchynnikov, Tetiana Slyva, et al., Chem J Mold. **2021**, 16(2): p. 58-65.
- [10] Taydakov IV, VM Korshunov V1, Belousov Y.A et al., Inorganica Chimica Acta. 2020, 513: p. 119922.
- [11] Bereznytska O. Optical Materials. **2021**, 120: p. 111492.
- [12] Dilli S and Robards K. Aust J Chem. **1979**, 32: p. 277-84.
- [13] Célia S. Bonnet, Julien Massue, Susan J Quinn, et al., Org Biomol Chem. **2009**, 7: p. 3074-3078.
- [14] Manuel AV Ribeiro da Silvaa, Gonçalves JM, Pilcher G. J Chem Thermodynamics. **1997**, 29: p. 253-260.
- [15] Sheikh Javed I, Juneja HD, Ingle VN. Hindawi e-Journal of Chemistry. **2010**, 7(1): p. 21-24.
- [16] Samira Poorsadeghi, Katsuki Endo and Satoru Arimitsu. Org Lett. **2022**, 24(1): p. 420-424.
- [17] Noor Al AlBaheley, Asaad A Ali and Tarek A Fahad. Phys Conf Ser. **1999**, p. 012017.
- [18] Jakob Kljun and Iztok Turel. Eur J Inorg Chem. **2017**, p. 1655-1666.
- [19] Liliya A Khamidullina, Igor S Puzyrev, Gennady L Burygin. Molecules. **2021**, 26: p. 6466.
- [20] Tahereh Sedaghat, Marjan Monajjemzadeh and Hossein Motamedi. Coord Chem Rev. **2011**, 64(18): p. 3169-3179.
- [21] Joohee Pradhan and Anju Goyal. Int J Pharm Res Allied Sci. **2015**, 4(2): p. 01-18.
- [22] Kaur Navjeet, Bhardwaj Pranshu, Gupta Mansi. Current Organic Chemistry. **2021**, 25: p. 2765-2790.