



ISSN (E): 2277- 7695

ISSN (P): 2349-8242

NAAS Rating: 5.03

TPI 2020; 9(4): 73-74

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www.thepharmajournal.com

Received: 12-02-2020

Accepted: 14-03-2020

Dr. Pushpa Kumari

W/o Shri S.K. Suman,
Vishwanath Bhavan,
Road No.-3, Moh. &
P.O. : Lakshmisagar,
P.S. : LNMU Campus,
Dist. : Darbhanga (Bihar)-
846009, Mob.: 9800082118,
8210540143, E-mail :
sumannhpc@gmail.com

Study of some complex compounds of dibenzoylmethane

Dr. Pushpa Kumari

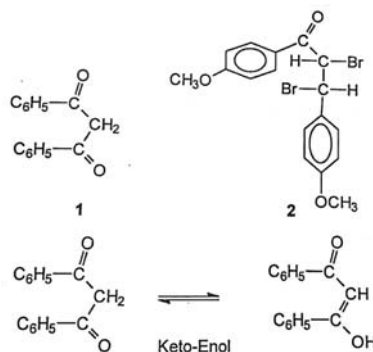
Abstract

In this paper, some novel complex compounds were synthesized from the reaction of dibenzoylmethane (1) and p. p-dimethoxybenzalacetophenone bromine (2) with some transition metal salts. From the reaction of dibenzoylmethane with FeSO_4 , CuSO_4 , CoCl_2 and NiCl_2 the compounds of $[\text{Fe}(\text{C}_{15}\text{H}_{12}\text{O}_2)]\text{SO}_4$, $[\text{Co}(\text{C}_{15}\text{H}_{12}\text{O}_2)_2]\text{Cl}_2$, $[\text{Cu}(\text{C}_{15}\text{H}_{12}\text{O}_2)]\text{SO}_4$ and $[\text{Ni}(\text{C}_{15}\text{H}_{12}\text{O}_2)_2]\text{Cl}_2$, were obtained respectively. Reaction of 2 and FeSO_4 have given the compound of $[\text{Fe}(\text{C}_{17}\text{H}_{16}\text{O}_3\text{Br}_2)_3]\text{SO}_4$.

Keywords: Some complex compounds, dibenzoylmethane

Introduction

Dibenzoylmethane (1) is starting compound for heterocyclic compounds. 1 Establishes keto-enol tautomerism and very resolution properties for direct reactions. p. p-dimethoxybenzalacetophenone bromine (2) is a very new compound in synthetic organic chemistry. Lots of heterocyclic compounds had been synthesized using this compound. Both 1 and 2 are bidentate ligand. In this research, the complex reaction ability of 1 and 2 investigated and good results were obtained. Interesting chemical shifts were observed in IR and NMR spectrums, as shown below,



Material and method

The compounds were routinely checked for their homogeneity by TLC using Kieselgel GF₂₅₄ 60 as absorbent. The IR spectra were recorded on a Shimadzu spectrophotometer model 435 V-04 as pellets. The ¹H-NMR ¹³C-NMR spectra were recorded with Varian H-100 FT and Varian 200 instruments using TMS as internal reference. The elemental analysis were determined with Carlo-Erba 1106 of Hewlett Packard model 105. Mass spectrums were recorded at Tibutak Marmara Research Centre.

Mono dibenzoyl methane-iron-(II)- sulphate 3

Dibenzoylmethane 0.672g (4.46mmol) and 0.347g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ were dissolved in 1:1 ratio alcohol-water mixture and heated for 5h at 75°C. Reaction mixture was cooled at room temperature and fine and precipitate was filtered and washed with diethylether (m.p. 240°C and yield: 64%)

Anal. For $[\text{Fe}(\text{C}_{15}\text{H}_{12}\text{O}_2)]$

Calc: C:72.80; H:4.20

Corresponding Author:

Dr. Pushpa Kumari

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Vishwanath Bhavan,
Road No.-3, Moh. &
P.O. : Lakshmisagar,
P.S. : LNMU Campus,
Dist. : Darbhanga (Bihar)-
846009, Mob.: 9800082118,
8210540143, E-mail :
sumannhpc@gmail.com

Found: C:72.17; H:4.56

IR (KBr, cm^{-1}): 3326, 1547, 1520-1493, 739-712

$^1\text{H-NMR}$ (400MHz, DMSO-d_6): δ 7.26 (m.10H. ArH), 5.71-5.31 (s.2H, $-\text{CH}_2$)

Bis- Dibenzoylmethane-cobalt-(II)-chloride 4

Dibenzoylmethane 0.448 (2mmol) and 0.25g $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ were dissolved 20ml ethylalcohol-5ml distilled water mixture. pH was regulated to 4-5 with ammonia. Reaction mixtures was heated on magnetic stirrer at 50°C for 3h. The yellow precipitate was filtered and crysallized from CHCl_3 , mp 70°C and yield 55%.

Anal. For $[\text{Co}(\text{C}_{15}\text{H}_{12}\text{O}_2)_2]\text{Cl}_2$

Calc: C:67.49; H:4.035

Found: C:68.20; H:4.290

IR (KBr, cm^{-1}): 3460, 2895, 1600,1530, 1493.

$^1\text{H-NMR}$ (400MHz, DMSO-d_6): δ 29.75 (s.1H. enol form), 12.25 (m.ArH), 4.50 (s, CH_2)

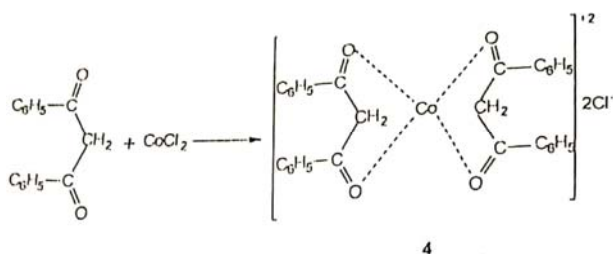
Bis- Dibenzoylmethane-nickel-(II)-chloride 5

Dibenzoylmethane 0.226g (1mmol) and 0.13g (0.55 mmol) $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ were dissolved 30ml ethyl alcohol-25ml distilled water mixture and heated at 60°C dissolved 30min. After cooling, the pale green precipitate was filtered and washed with diethyl ether and alcohol. Pure compound was obtained m.p. 280°C , yield 58%.

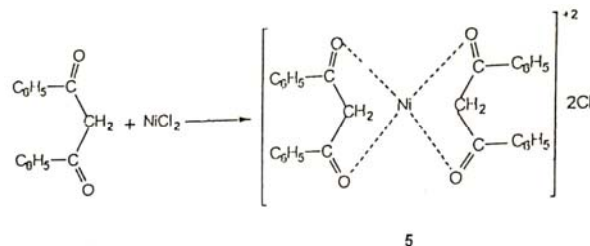
Anal. For $[\text{Ni}(\text{C}_{15}\text{H}_{12}\text{O}_2)_2]\text{Cl}_2$

Calc: C:62.31; H:4.14

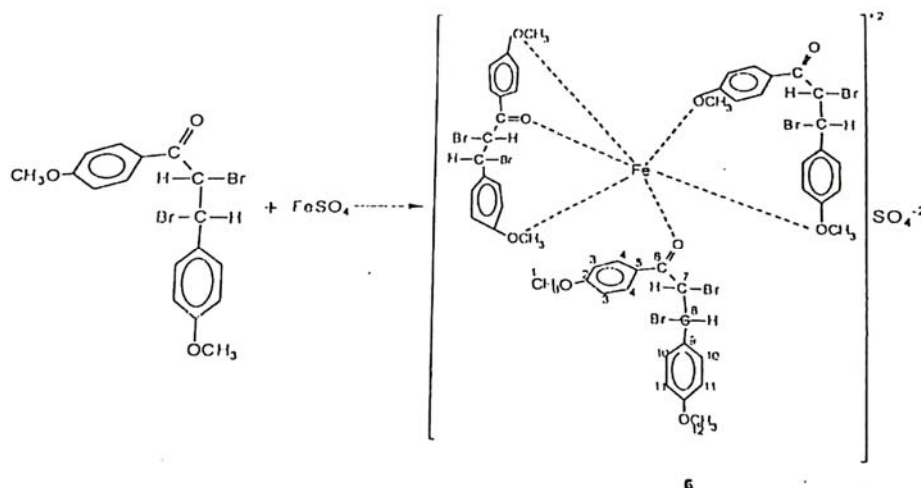
Found: C:68.85; H:4.33



Scheme-2



Scheme-3



Scheme-4

Tris-(p-p - dimethoxy benzalacetophenone dibromide -iron-(II)-sulphate 6, is very new complex compound. The ratio of M:L is 1:3. Thus we can say that compound 2 can be used as bidentad ligand in inorganic chemistry.

Conclusion

The crystal geometry has not been investigated but we think that the molecules of complexes are being hold together with ion- dipol attractives. Further investigations are going on.

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