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"Synthesis of hemicyanine dyes through ethyl iodide of five benzothiazole salts and ten suitable complex ketones"

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Abstract

For the present investigation of Synthesis of hemicyanine dyes, ethyl iodide of five benzothiazole salts and ten suitable complex ketones have been chosen and synthesised in quantitative yield. The use of above mentioned ketones, not only provide a means of weighining the dye molecule but also furnishes a means to study the role of steric and electronic effect of substitutions in the prime chromophoric methane chain. Keeping in view the multidimentional applications of cyanine and hemicyanine dyes, it was through to synthesise some new hemicyanine dyes by condensing some suitable auxochromic chalcones and benzophenons with quarternised benzothiazolium salts in presence of suitable basic catalyst and study their optical characteristics i.e. U. V. absorption.

***Keyword**: Cyanine and Hemicyanine dyes, benzothiazolium salts and ketones*

Introduction

The first cyanine dye was discovered by Greville Williams but the cyanine dye were not used as a textile dyes because their poor fastness properties. The cyanine and hemicyanine dyes have been used in various purpose. E.g. Photosensitisers, in space photography, as organic photoconductor for electrophotography, as infrared absorbing material for optical recording system in the absorvity and in semiconductor laser. For the present investigation only five quaternised heterocyclic (aromatic) salt (benzothiazole) ten complex auxochromic ketones have been selected for synthesis of dyes.

All the five benzothiazole base have been synthesized by adopting the method of Jacobson (1886), mills (1992), Beilenson (1936) by procedural alteration suggested by J. C. Banerji (Banerji and Sanyal (1968) and quaternised with ethyl iodide by following the method of Johnson and Adams (1921) with slight modification made by Jha et al (Jha and Banerji 1983, 1985) and Ansari et al (1914, 1995).

Experimentals

Synthesis of hemicyanine dyes, ethyl iodide of five benzothiazole salts are ;-

(1) 6 – Iodo – 2 - methyl banzothiazole,

(2) 6 – Chloro – 2 – methyl banzothiazole,

(3) 2 - methyl banzothiazole,

(4) 2,6 - Dimethyl banzothiazole,

(5) 6 – Methoxy -2 - methyl banzothiazole,

In the present study all the require chalcones were obtained by a simpler Novel method reported by Jha et al (1987, 1988, 1990, 1995) with some procedural alteration which afforded better yield with high purity. In this developed method Ehrlich's Reagent and substituted acetophenones were dissolved in minimum volume of dry methanol in equimolar quantities and a few pellets of caustic alkali were added. The container was tightly corked and shaken vigorously for about one hour on a magnetic stirrer to get crude coloured chalcone. The product was filtered washed with water and then recrystalised from aqueous ethanol. All prepared chalcones with their physical data are given as follows.

(1) 4 – Dimethylaminophenyl – 4^1 nitrophenyl ketone

The crude product was crystallized from ethanol as scintillating orange leaflets.

Yield : 51% m.p. 206-207^{0}C

Lit* : 54% m.p. 207^{0}C

(2) 4 – Dimethylaminophenyl phenyl ketone

The crude product was crystallized from ethanol as light bluish green soft needles.

Yield : 46% m.p. 90-91^{0}C

Lit* Yield : 54% m.p. 92-93^{0}C

(3) 4, 4^1 – Bis (Dimethylamino) phenyl ketone

The crude product was recrystallized from ethanol as dirty green crystalline solid.

Yield : 45% m.p. 174^{0}C

Lit* Yield : 51% m.p. 174^0 -193^{0}C

(4) 4 - Dimethylaminostyryl-3' - nitrophenyl ketone (New)

The afforded crude ketone was recrystalised from ethanol as deep red crystals

Yield : 80% m.p. 152-154^{0}C

Found : C, 68.88 H, 5.42 , N, 9.44%

$C_{17}H_{16}N_2O_3$ requires : C, 68.90 H, 5.44 N, 9.45%

(5) 3' Chlorophenyl, - 4 – Dimethylaminostyryl ketone : (New)

The crude product was recrystallized from ethanol as dirty yellow sandy crystals.

Yield : 77% m.p. 77^{0}C

Found : C, 71.44 H, 5.64 , N, 4.99%

$C_{17}H_{16}ClNO$ requires : C, 71.47 H, 5.64 N, 4.90%

I.R. Spectra (cm^{-1}) : 1626(CH = CH), 1690 (C = O), 1616 (C = N), 710 (Cl)

(6) 3' - Bromophenyl, - 4 – Dimethylaminostyryl ketone : (New)

The crude product was recrystallized from ethanol as deep bright yellow sandy crystals.

Yield : 75% m.p. 83^0C

Found : C, 61.80 H, 4.85 , N, 4.22%

$C_{17}H_{16}BrNO$ requires : C, 61.83 H, 4.88 N, 4.24%

I.R. Spectra (cm^{-1}) 1625(CH = CH), 1685 (C = O), 1615 (C = N), 560 (Br)

(7) 4 – Dimethylaminostyrylphenyl ketone

The ketone was recrystallized from ethanol as bright yellow leaflets.

Yield Lit* : 74%, m.p. Lit* 110^0c

Found : 70%, Found 110^0c

(Lit:Jha, B. N.; Jha, R.K.; & Banerji, J. C. ; 1986)

(8) 4 - Dimethylaminostyryl-3' - nitrophenyl ketone (New)

The crude product was recrystalised from ethanol as turmeric yellow sandy crystals

Yield : 60% m.p. 71^0c

Found (%) : C, 81.45 H, 7.20 , N, 5.25

$C_{18}H_{19}NO$ requires (%) : C, 81.47 H, 7.21 N, 5.27

I.R. Spectra (cm^{-1}) 1620(CH = CH), 1680 (C = O), 1617 (C = N), 3010 (CH_3)

(9) 3' – Aminophenyl - 4 - Dimethylaminostyryl ketone : (New)

The afforded crude ketone was recrystalised from ethanol as deep red crystals.

Yield : 58% m.p. 142-144^0c

Found (%) : C, 79.63, H, 7.05 , N, 10.90%

$C_{17}H_{18}N_2O$ requires (%) : C, 79.65, H, 7.07 N, 10.92%

I.R. Spectra (cm^{-1}) 1610(CH = CH), 1670 (C = O), 1620 (C = N), 3390 (N - H)

(10) 4 - Dimethylaminostyryl methyl ketone: (New)

The crude afforded ketone was recrystalised from toluene as deep orange crystals.

Yield : 79% m.p. 120^0c

Found (%) : C, 76.18, H, 7.89 , N, 7.38

$C_{12}H_{15}NO$ requires : C, 76.19, H, 7.90 N, 7.40

I.R. Spectra (cm^{-1}) : 1610(CH = CH), 1670 (C = O), 1620 (C = N), 3390 (N - H)

Discussion on dye intermediates

Synthesis of hemicyanine dyes, ethyl iodide of five benzothiazole salts are ;-

- 6 – Iodo – 2 - methyl banzothiazole,
- 6 – Chloro – 2 – methyl banzothiazole,
- 2 - methyl banzothiazole,
- 2,6 - Dimethyl banzothiazole,
- 6 – Methoxy -2 - methyl banzothiazole,

The substitution have been affected at 6-position only in all cases except (3)

Besides weighing (Which is found associated with bathochromic shifts in absorption maxima of resultant dyes), it is expected that substituents would affect the reactivity of banzothiazoles as well as the properties of the dyes due to their inductive and mesomeric effects.

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