

Synthesis of 4-2,7-Dihydroxy Naphthyl Azo-2,3-Dimethyl-1-Phenyl-3-Pyrazoline-5-one

V.M. SAHAI*, D. JOSHI and M. PANDEY

Department of Applied Chemistry

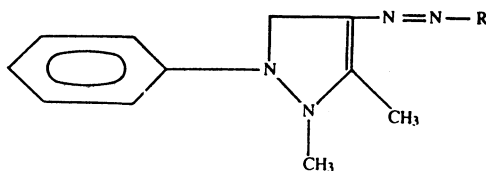
Madhav Institute of Technology and Science, Gwalior-474 005, India

Pyrazoline dye has been synthesised from diazonium salt of 4-amino-2,3-diamino-1-phenyl-3-pyrazoline-5-one by coupling with 2,7-hydroxy naphthalene. Azo dyes are characterised by the presence of one or more azo ($-N=N-$) groups. This class of dye is well known for dyeing potentialities and biological activities. The fastness of azo dyes varies widely with structure and environment.

INTRODUCTION

Besides dyeing potentialities pyrazolone and its derivatives have been found to possess bacteriostatic¹⁻³, anticancerous⁴, antidiuretic⁵, germicidal⁶, fungicidal^{7,8} and therapeutic activities^{9,10}. Pyrazolone dye structure is based on pyrazolone nucleus. The formula (I) represents the probable structure of the dyes of this group.

EXPERIMENTAL



(I)

R = 1,7-dihydroxy naphthalene

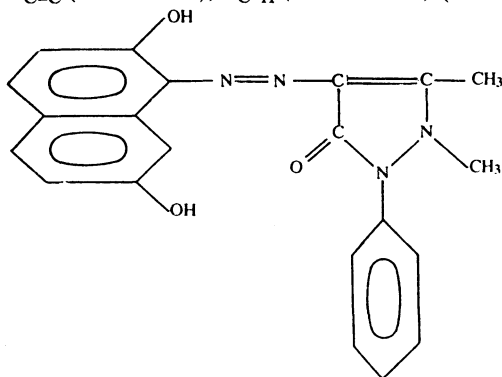
Synthesis of 4-2,7-dihydroxy naphthyl azo-2,3-dimethyl-1-phenyl-3-pyrazoline-5-one

A mono azo dye was synthesised consisting of 2,7-dihydroxy substituted naphthalene ring linked with pyrazolone ring through an azo linkage.

First Step: 1.60 Gm (0.01 M) of 2,7-dihydroxy naphthalene were dissolved in solution and adjusted the pH 8-9 by adding caustic soda solution.

Second Step: 2.03 Gm (0.01 M) 4-amino-2,3-dimethyl-1-phenyl-3-pyrazoline-5-one was diazotized at pH 3 to 5 (using sodium acetate-acetic acid buffer) by dissolving in 20% HCl and adding 0.69 gm (0.01 M) of NaNO₂ solution at 10°C for 1/2 hr. Excess of HNO₂ was destroyed by using urea.

Final Step: Diazo salt of 4-amino-2,3-dimethyl-1-phenyl-3-pyrazoline was added dropwise with stirring at 10°C in alkaline solution of 2-7-dihydroxy naphthalene. After the addition was over, a dark brown coloured dye was precipitated. It was then filtered and washed by using warm water and it was then recrystallised by dissolving it in the least amount of ethyl alcohol. Dark brown crystals of pure azo dye were obtained (yield 89.7%, m. pt. 210°C, soluble in alcohol). IR frequencies (cm^{-1}): $\nu_{\text{C}=\text{O}}$ (1900–1500); ν_{OH} (3000–2750); $\nu_{\text{C}-\text{H}}$ (1450–1375); $\nu_{\text{N}-\text{N}}$ (1556–1429); $\nu_{\text{C}-\text{C}}$ (1600–1580); $\nu_{\text{C}-\text{H}}$ (aromatic str.) (2200 br).



Structure of 4-(2,7-dihydroxynaphthyl)azo-2,3-dimethyl-1-phenyl-3-pyrazolin-5-one

ACKNOWLEDGEMENT

The authors are thankful to Principal, M.I.T.S., Gwalior, for providing necessary research facilities.

REFERENCES

1. G.S. Saharia and H.R. Sharma, *J. Indian Chem. Soc.*, **51**, 351 (1974).
2. Ajay Kabra, G.S. Saharia and H.R. Sharma, *J. Indian Chem. Soc.*, **52**, 987 (1975).
3. P.S. Fernandes, Sandhya Bhate, Philip Gupta and V.V. Nandkarni, *J. Indian Chem. Soc.*, **53**, 30 (1976).
4. W. Wilson and Bottiglieri, *Cancer Chemotherapy*, **21**, 137 (1962).
5. V. Pacovsky and Holecck, *Caropisle Karu-Ceskych*, **195**, 830 (1976); *Chem. Abstr.*, **50**, 7306 (1956).
6. C.N. Anderson, U.S. Pat. 2,107,321 (1938).
7. B. Nandas, Padmanavon, B.Tripathy and A.S. Mitra, *J. Indian Chem. Soc.*, **52**, 533 (1975).
8. G.S. Saharia and H.R. Sharma, *J. Indian Chem. Soc.*, **53**, 504 (1976).
9. C.W. Noell and C.C. Chang, *J. Med. Chem.*, **12**, 545 (1969).
10. E.L. Anderson, J.E. Carey and L.C. Breene, *J. Med. Chem.*, **1**, 259 (1964).

(Received: 26 December 1991; Accepted: 15 October 1992)

AJC-494