

## Synthesis of 1,2,4-Dinitro Phenyl-3-p-Hydroxy, 3-Methoxy Phenyl-5-2-Naphthol-4-Sulphonyl Formazan Dye

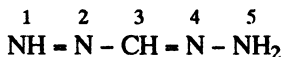
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Formazan dye has been synthesised from diazonium salt of 1-amino, 2-naphthol, 4-sulphonic acid coupling with active hydrogen present in 3-methoxy, 4-hydroxy benzaldehyde phenyl hydrazone.

### INTRODUCTION

Many types of methods have been available for the preparation of formazan dyes<sup>1</sup> of which the most convenient method involves the reaction of a diazonium salt with hydrazone in alkaline medium<sup>2</sup>. Formazan dye belongs to azo dye family which were first discovered<sup>3</sup> in 1892. They are derivatives of hypothetical parent compound



In 1941 Hunter and Roberts<sup>4</sup> established that 1,3,5-triphenyl formazans function as bidentate ligand and form metal complexes with copper(II), nickel(II) and cobalt(II). In the recent years the formazan dyes were used for dyeing and printing acid modified polyamide textiles and carpets to give intense fast shades<sup>5</sup>. Biro and Wizinger investigated some complex-forming formazans carrying donor groups (-OH, -COOH) in the *ortho* position to the formazan chain; various applications of metal complexes of tetradentate formazan as dye stuffs and pigments have been claimed. The copper(II) complex is reported<sup>6</sup> to dye from a natural dye bath in blue shades of good light-fastness. Cobalt complexes<sup>7</sup> of tridentate formazans provide level dyeing on wool from neutral to weakly acid dye baths<sup>8</sup>.

These salts are generally colourless and water-soluble and are readily converted back to the highly coloured formazan. They have therefore been used as biological staining agents.<sup>9,10</sup>

### EXPERIMENTAL

#### Synthesis of 1,2,4-dinitrophenyl-3-p -hydroxy-3-methoxy phenyl-5-2-naphthol-4-sulphonyl formazan dye

The 3-methoxy 4-hydroxy-benzaldehyde and other chemicals of reagent grade quality were purified by standard procedures wherever necessary.

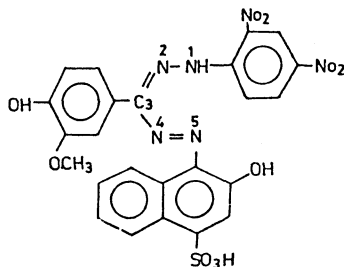
### Preparation of 3-methoxy, 4-hydroxybenzaldehyde phenylhydrazone

Dissolve 5.0 gm of 2,4-dinitrophenylhydrazine and 8 gm of sodium acetate in 50 ml water and add a solution of 2.0 gm to 4 gm 3-methoxy, 4-hydroxybenzaldehyde in a little ethanol. Shake the mixture until a clear solution is obtained and add a little more ethanol, if necessary; warm on water bath for 10 to 15 minutes and cool. filter off the crystalline derivative and recrystallise from dilute ethanol or water<sup>11</sup>.

### Synthesis of 2-2,4-Dinitro-phenyl-3-p-hydroxyphenyl-5-2-naphthol-4-Sulphonyl formazan dye

In the first step the 3-methoxy, 4-hydroxy benzaldehyde phenylhydrazones (4.49 gm) were dissolved in NaOH (10%, 80 ml solution) and the resulting solution was cooled to 5°C. In second step the 1-amino, 2-naphthol, 4-sulphonic acid (3.21 gm) were dissolved in 18 ml HCl and 18 ml distilled water and the resulting solution cooled to 0–4°C. Diazotise this solution by NaNO<sub>2</sub> (4 gm in about 20 ml water cooled to 0–4°C). Check complete coupling by starch iodide paper. Finally 2-naphthol, 4-sulphonyl diazonium chloride solution was added dropwise with stirring at 10°C in alkaline hydrazone solution. After the addition was over the reaction mixture was allowed to stand for 12 hrs. The formazan formed was precipitated by adding distilled water filtered off, washed successively with dilute HCl, hot water and minimum amount of ethyl alcohol. The precipitated dye was dried and recrystallised from ethanol (yield 88.5%, m.pt. 168°C). I.R. frequencies (cm<sup>-1</sup>)  $\nu(\text{NH})$  (3400–3300);  $\nu(\text{OH})$  (3300–2750);  $\nu(\text{C}=\text{N}$  and  $\text{C}=\text{C})$  (1600–1580 cm<sup>-1</sup>); (C–H, aromatic str) (2200 br), (C–H, aromatic str) (1400–1100 cm<sup>-1</sup>);  $\nu(\text{N}=\text{N})$  (1556–1429);  $\nu(\text{OCH}_3)$  (3000);  $\nu(\text{NO}_2)$  (1420).

#### STRUCTURE OF 1-2,4 DINITRO PHENYL-3-p HYDROXY, 3-METHOXY PHENYL-5-2 NAPHTHOL-4 SULPHONYL FORMAZAN DYE



#### ACKNOWLEDGEMENT

Authors are very much thankful to Dr. S.K. Selot (Principal, MITS, Gwalior) for providing necessary research facilities. We are also grateful to Director, Defence Research and Development Establishment, Gwalior (M.P.) for providing

library facilities. Thanks is also due to Dr. Dev (Day Chem. Laboratories, Inc., 1600 N. Broad St. Fairborn, Ohio 45324, America) for I.R. Spectrograph in his laboratory.

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(Received: 25 October 1991; Accepted: 15 July 1992)

AJC-451