

## 3-Arylazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones as Azodisperse Dyes for Dyeing Polyester Fabrics

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**Summary.** Diazotized 3-amino-2-thioxo-2,3-dihydro-1H-quinazolin-4-one was coupled with various hydroxy aromatics to give the corresponding 3-arylazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones in reasonable yields. The dyes produced were applied to polyesters as disperse dyes and their fastness properties were elevated.

**Keywords.** 3-Amino-2-thioxo-2,3-dihydro-1H-quinazolin-4-one; Diazonium salt; Disperse dyes; Fastness properties; Polyester.

### Introduction

The unique biological activities and characteristic chemical structures have made synthesis studies of the quinazoline ring system attractive over the years. Synthesis of quinazoline derivatives provides the backbone for the synthesis of numerous compounds with pharmacological activities [1–9].

As part of our continuing interest in heterocyclic chemistry, we have reported simple and convenient approaches for the synthesis of various quinazolines [10–21]. We now report on the successful synthesis of some new 3-arylazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones and their application as disperse dyes for dyeing polyester fabrics.

### Results and Discussion

3-Amino-2-thioxo-2,3-dihydro-1H-quinazolin-4-one (**1**) was prepared according to Ref. [22]. Diazotization of **1** was performed by using a mixture of sodium

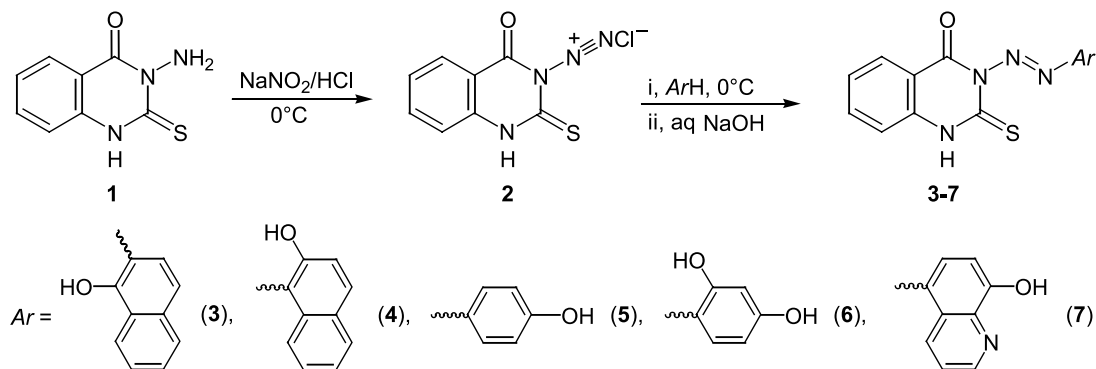
nitrite and hydrochloric acid to produce the corresponding diazonium salt **2**. Coupling of **2** with several phenols, such as  $\alpha$ -naphthol,  $\beta$ -naphthol, phenol, resorcinol, and 8-hydroxyquinoline in aqueous sodium hydroxide solution (10%) afforded the corresponding 3-arylazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones **3–7** in moderate yields (50–54%; Scheme 1).

The structures of diazo dyes **3–7** were confirmed by various spectroscopic techniques including IR, <sup>1</sup>H NMR, mass, and accurate mass data. The IR spectra of **3–7** are characterized by the presence of absorption bands within the  $\bar{\nu}$  = 3167–3300 cm<sup>–1</sup> region corresponding to the stretching vibrations of the NH group. The bands within the  $\bar{\nu}$  = 1661–1666 cm<sup>–1</sup> region are due to the stretching vibration of the C=O group of the quinazolinone moiety. The absorption bands at the  $\bar{\nu}$  = 1389–1410 cm<sup>–1</sup> region are due to the symmetric stretching vibrations of the azo group. Thus absorption bands within the  $\bar{\nu}$  = 3386–3544 cm<sup>–1</sup> region are attributed to the phenolic OH group of the coupler moieties. The <sup>1</sup>H NMR spectra showed a characteristic NH signal in the  $\delta$  = 13.11–13.22 ppm region. The structures of diazo dyes **3–7** were confirmed further by mass and accurate mass data. The electron impact mass spectra indicated the presence of molecular ion peaks in all cases.

### Dyeing of Polyester Fabrics and Dyeing Properties

Most influences that can affect fastness are light, washing, heat, perspiration, and atmospheric pollution.

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Scheme 1

Table 1. Fastness properties of dyes 3–7

Compd.	Washing	Perspiration		Rubbing		Sublimation		Light (40 h)
		Acid	Alkali	Dry	Wet	Alt	St	
3	5–6	4	4–5	4	5	4	4	6
4	5–6	4–5	5	5	4	5	4	6
5	5	5	4	5	4	5	5	5
6	5–6	4–5	5	4	4	4	5	6
7	4–5	4	4	4	5	4	4	5

Alt Alteration; St staining; rate for light fastness: 5–8 (acceptable), 1–4 (not acceptable); rate for different fastness: 4–5 (acceptable), 1–3 (not acceptable)

Conditions of such tests are chosen to correspond closely to the treatments employed in manufacture and of ordinary use conditions [23]. Results are given after usual matching of the tested samples against standard reference (the *Grey* scale) [23]. The results obtained are shown in Table 1. The results revealed that these dyes have good fastness properties.

In conclusion, a set of five useful disperse dyes 3–7 was synthesised by azo coupling. The dyes were investigated for their dyeing characteristics on polyester and showed good washing, perspiration, rubbing, sublimation, and light fastness. The remarkable degree of brightness after washings is indicative of good penetration and the excellent affinity of these dyes for the fabric due to the accumulation of polar groups.

## Experimental

Melting points were determined on an electrothermal melting MEL-TEMP II apparatus.  $^1\text{H}$  NMR spectra were recorded on  $\text{CF}_3\text{COOD}$  solutions using a Bruker AC400 spectrometer operating at 400 MHz. Low resolution mass spectra were recorded on a Quattro II triple quadrupole mass spectrometer

(electron impact: 70 eV). Accurate mass data were obtained on a MAT 900 instrument. IR spectra were recorded on a Perkin-Elmer 1430 spectrometer. Compound 1 was prepared according to Ref. [22]. Its analytical data agreed with the published work.

### Synthesis of 3-Arylaazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones 3–7

A cold ( $0^\circ\text{C}$ ) solution of 0.7 g sodium nitrate in  $10\text{ cm}^3$   $\text{H}_2\text{O}$  was added gradually to a cold ( $0^\circ\text{C}$ ) suspension of 1.93 g 1 (0.01 mol) in  $5\text{ cm}^3$  conc. HCl. The diazonium salt thus obtained was added with continuous stirring to a cold ( $0^\circ\text{C}$ ) solution of hydroxyaryl compounds (0.01 mole) in  $20\text{ cm}^3$  10% NaOH solution. The mixture was stirred at  $0^\circ\text{C}$  for 1 h, filtered, washed with  $\text{H}_2\text{O}$ , dried, and crystallized from glacial acetic acid to give the corresponding 3-arylaazo-2-thioxo-2,3-dihydro-1H-quinazolin-4-ones 3–7.

### 3-(4-Hydroxynaphthylazo)-2-thioxo-2,3-dihydro-1H-quinazolin-4-one (3, $\text{C}_{18}\text{H}_{12}\text{N}_4\text{O}_2\text{S}$ )

Yield 54%; yellow; mp  $140\text{--}142^\circ\text{C}$ ; IR (KBr):  $\bar{\nu} = 3386$  (OH),  $3224$  (NH),  $1661$  (C=O),  $1400$  (N=N),  $1251$  (C=S)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR:  $\delta = 6.72\text{--}7.58$  (m, 10H),  $7.81$  (dd,  $J = 8.1, 2.0$  Hz, 1H),  $13.20$  (s, exch, 1H) ppm; EI-MS:  $m/z$  (%) =  $348$  ( $\text{M}^+$ , 7),  $316$  (3),  $272$  (3),  $143$  (14),  $128$  (16),  $115$  (34),  $105$  (100),  $90$  (16),  $76$  (20),  $63$  (30); HREI-MS:  $\text{C}_{18}\text{H}_{12}\text{N}_4\text{O}_2\text{S}$  ( $\text{M}^+$ ), calcd  $348.0681$ , found  $348.0682$ .