

## Synthesis of Heterocyclic Azo Dyes from Quinolin-8-ol

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### Abstract

Six derivatives of heterocyclic azo dyes were synthesized by coupling diazotized aniline and 4-nitroaniline with quinolin-8-ol, 5-nitrosoquinolin-8-ol and 5-nitroquinolin-8-ol respectively. Benzene diazonium salt and 4-nitrobenzene diazonium salt were prepared by treating aniline and 4-nitroaniline respectively with HNO<sub>2</sub> generated in situ from NaNO<sub>2</sub>/HCl at 0-5°C. 5-nitrosoquinolin-8-ol was prepared by treating a solution of quinoline-8-ol in conc. HCl/H<sub>2</sub>O with solution of sodium nitrite at 0-4° in an ice-salt bath. Oxidation of 5-nitrosoquinolin-8-ol with conc. HNO<sub>3</sub> at 17° gave 5-nitroquinolin-8-ol. The dyes were of intense colouration. They were characterized by FT-IR, NMR and elemental analysis.

**Keywords:** Aniline; 4-nitroaniline; quinolin-8-ol; diazotization; nitrosation; oxidation; coupling reaction

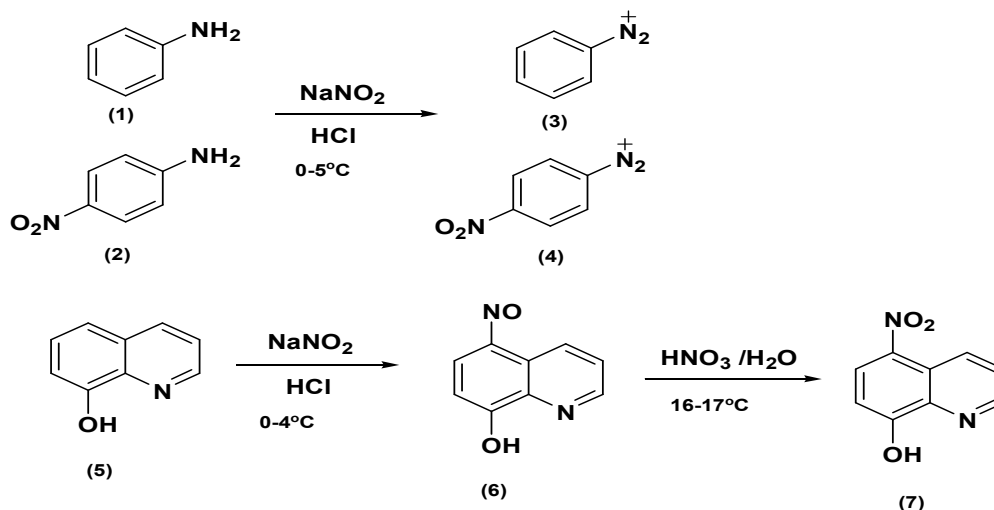
### 1.0. Introduction

Azo dyes are by far the most important class of dyes accounting for about or more than 50 % of all commercial dyes or colourants. They have been applied so much in various fields of science and technology<sup>1-2</sup>. The ease of preparation by diazotization and azo coupling reactions has made it possible for azo dyes to have been studied more than any other class of dyes. Azo dyes containing heterocyclic rings have also been studied as a result of their superior properties. They have wide applications as good dyeing agents in textile industries<sup>3</sup>. The introduction of heterocyclic ring as coupling components has made possible the synthesis of good colourants with chromophoric strength and brilliant colours that has high level dyeing property and excellent fastness properties<sup>4-6</sup>. They are used in dyeing hydrophobic textile material such as polyamide and polyester. The increasing use of heterocyclic dispersed azo dyes by electronic industries as colorimetric sensors, nonlinear optical devices, liquid crystalline display and potential sensitizers has been reviewed<sup>7</sup>. They are also used as photodynamic therapy and reprography<sup>8</sup>, metallochromic indicators<sup>9</sup> to mention but a few. Benzothiazolyl and thiazolyl compounds were the popular ones in the production of red azo dyes that has better fastness properties<sup>10, 11</sup>. Benzothiazolyl dispersed azo dyes are especially used for the dyeing polyester materials because of their better fastness and high level dyeing properties<sup>12-14</sup>. Very much attention has been focused on the heterocyclic derivatives of azo dyes containing nitrogen in the aromatic rings due their divers' biological activities as antitumor<sup>15</sup>, anti-inflammatory<sup>16</sup>, antibacterial<sup>17</sup> and antifungal agents<sup>18</sup>. It has been observed that the activity of azo linkage increases when heterocyclic rings are incorporated.

In view of this, the authors describe the synthesis of heterocyclic azo dyes from quinoline-8-ol as the precursor.

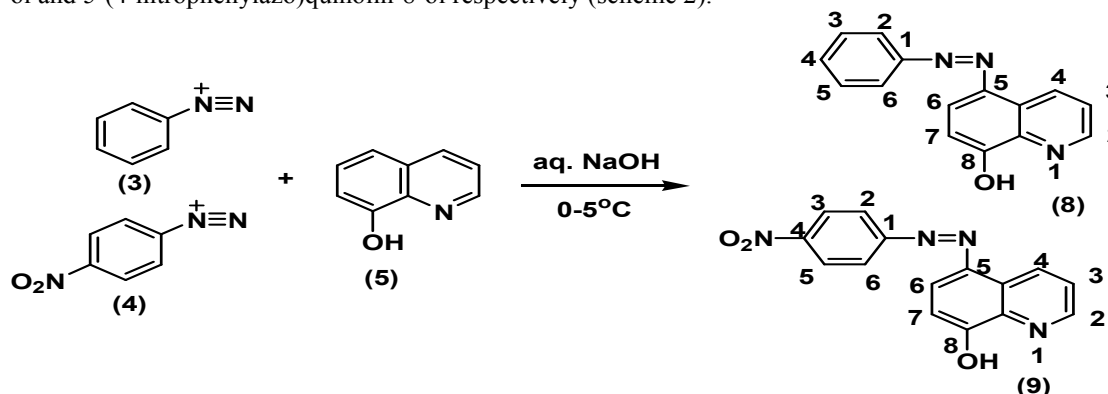
### 2.0. Results and discussion

Aniline **1** and 4-nitroaniline **2** were converted to their respective stable benzene diazonium salt **3** and 4-nitrobenzene diazonium salt **4** when they were treated respectively with HNO<sub>2</sub> generated in situ from NaNO<sub>2</sub>/HCl at 0-5°C. On the other hand, quinolin-8-ol **5** when treated with a solution of NaNO<sub>2</sub> gave 5-nitrosoquinolin-8-ol **6**. Oxidization of **6** with HNO<sub>3</sub> furnished 5-nitroquinolin-8-ol **7** as yellow solid (scheme 1).



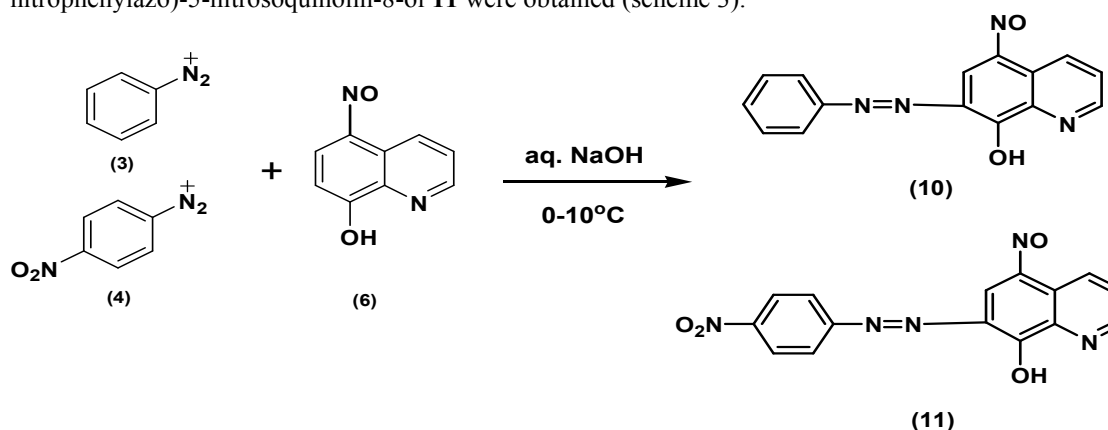
Scheme 1

Coupling of quinolin-8-ol **5** with benzene diazonium salt **3** and 4-nitrobenzene diazonium salt **4** in alkaline solution of NaOH at 0-5°C gave the expected heterocyclic azo dyes **8** and **9** identified as 5-phenylazoquinolin-8-ol and 5-(4-nitrophenylazo)quinolin-8-ol respectively (scheme 2).



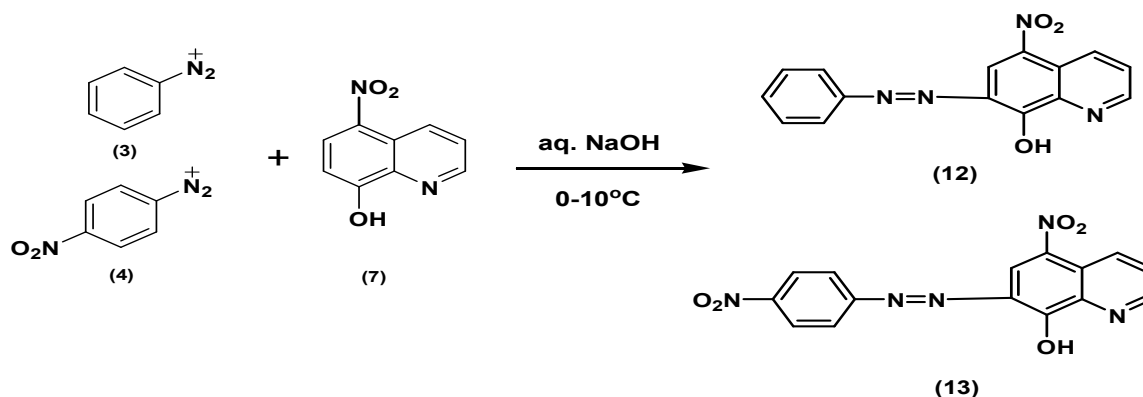
Scheme 2

When nitroso derivative of quinolin-8-ol **6** was treated with the diazonium salts **3** and **4** in the same alkaline condition as for scheme 2, two more heterocyclic azo dyes 5-nitroso-7-phenylazoquinolin-8-ol **10** and 7-(4-nitrophenylazo)-5-nitrosoquinolin-8-ol **11** were obtained (scheme 3).



Scheme 3

Further reactions of the diazonium salts **3** and **4** with 5-nitroquinolin-8-ol **7** gave another two heterocyclic azo dyes 5-nitro-7-phenylazoquinolin-8-ol **12** and 7-(4-nitrophenylazo)-5-nitroquinolin-8-ol **13** respectively (scheme 4).



Scheme 4

Elemental analysis of the synthesized heterocyclic azo dyes was in agreement with the molecular formulas: **8** ( $C_{15}H_{11}N_3O$ ), **9** ( $C_{15}H_{10}N_4O_3$ ), **10** ( $C_{15}H_{10}N_4O_2$ ), **11** ( $C_{15}H_9N_5O_4$ ), **12** ( $C_{15}H_{10}N_4O_3$ ), **13** ( $C_{15}H_9N_5O_5$ ). The FT-infrared spectrum of all the dyes showed a broad band at the region of  $3480-3225\text{ cm}^{-1}$  confirming the presence of hydroxyl group (-OH). Also there are bands at  $\text{cm}^{-1}$ : 1610-1590 for (C=N), 1575-1520 (-N=N-), 1515-1480 (C=C aromatics).  $^1\text{H-NMR}$  spectrum gave further evidence of the structures: **5-phenylazoquinolin-8-ol 8** exhibited the following  $^1\text{H-NMR}$  signals in ppm: 9.50 (OH), 9.01 (d, H-2 of quinoline ring), 8.20 (d, H-4 of quinoline ring), 7.31 (t, H-3 of quinoline ring), 8.10 (d, H-6 of quinoline ring), 7.20 (d, H-7 of quinoline ring), 7.75 (m, 5H, Ar-H azo benzene). **5-(4-nitrophenylazo)quinolin-8-ol 9** gave  $^1\text{H-NMR}$  signals in ppm: 9.47 (br, OH), 9.07 (d, H-2 of quinoline ring), 8.10 (d, H-4 of quinoline ring), 7.35 (t, H-3 of quinoline ring), 8.05 (d, H-6 of quinoline ring), 7.23 (d, H-7 of quinoline ring), 8.45 (d, H-3, H-5 of azo benzene ring), 8.25 (d, H-2, H-6 of azo benzene ring). **5-nitroso-7-phenylazoquinolin-8-ol 10** gave  $^1\text{H-NMR}$  signals in ppm: 9.55 (br, OH), 9.05 (d, H-2 of quinoline ring), 8.17 (d, H-4 of quinoline ring), 7.21 (dd, H-3 of quinoline), 8.65 (s, H-6 of quinoline ring), 7.80 (m, 5H, Ar-H azo benzene). The following  $^1\text{H-NMR}$  signals were observed in the spectrum for **7-(4-nitrophenylazo)-5-nitrosoquinolin-8-ol 11**: 9.51 (br, OH), 9.11 (d, H-2 of quinoline ring), 8.13 (d, H-4 of quinoline ring), 7.30 (dd, H-3 of quinoline ring), 8.60 (s, H-6 of quinoline ring), 8.45 (d, H-3, H-5 of azo benzene ring), 8.23 (d, H-2, H-6 of azo benzene ring). **5-nitro-7-phenylazoquinolin-8-ol 12** exhibited the following  $^1\text{H-NMR}$  signals: 9.49 (br, OH), 9.21 (d, H-2 of quinoline ring), 8.80 (d, H-4 of quinoline ring), 7.68 (t, H-3 of quinoline ring), 8.83 (s, H-6 of quinoline ring), 7.78 (m, 5H of azo benzene ring), **7-(4-nitrophenylazo)-5-nitro-quinolin-8-ol 13** gave  $^1\text{H-NMR}$  signals in ppm at 9.48 (br, OH), 9.23 (d, H-2 of quinoline ring), 8.81 (d, H-4 of quinoline ring), 7.70 (t, H-3 of quinoline ring), 8.85 (s, H-6 of quinoline ring), 8.42 (d, H-3, H-5 of azo benzene ring), 8.23 (d, H-2, H-6 of azo benzene ring).

#### Dyeing of polyester fabric with the synthesized azo dyes:

As a result of the dyes poor solubility in water the dyeing was carried out in aqueous solution of DMF/Acetone/acetic acid. They gave well to excellent shades on the polyester fabric and showed excellent wash fastness and good sublimation fastness base on the international geometric gray standard (1 and 5 for poor and excellent respectively) as presented in the table.

**Table;** fastness properties of synthesized heterocyclic azo dyes

Dye no	Wash fastness (1-5)	Stain on undyed fabric after washing (1-5) Polyester, cotton	Sublimation fastness (1-5)	Stain on undyed fabric after sublimation (1-5) Polyester, cotton
8	4	4 4-5	4-5	4-5 4
9	4-5	5 4-5	5	5 4-5
10	4-5	4-5 5	4-5	4 4
11	4-5	4-5 4	4-5	4-5 4
12	4-5	5 4	4	4 5
13	5	4-5 5	4-5	4-5 5

### 3.0. Experimental

All the solvents and reagents were of analytical grade purity and were purchased from Zigma-zayo chemical company. Melting points were recorded on Fischer Jone's melting point apparatus and are uncorrected. Infrared spectra were recorded on an FTIR-8400S infrared spectrophotometer using KBr discs.  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  were recorded on Bruker instrument 400 MHz in India; chemical shifts were reported on ppm scale.

Microanalysis was obtained on Heraous CHN-O rapid analyzer in Cairo Egypt.

### 3.1. 5-Nitrosoquinolin-8-ol

A solution of quinoline-8-ol (40 g, 0.27 moles) in 120 mL of water and 45 mL of concentrated hydrochloric acid and 120 g of ice in 500 mL beaker was immersed in ice-salt bath. It was then treated with a cold solution of sodium nitrite (20 g) in 75 mL of water for a period of 2 h at 0-4 °C as previously reported<sup>19</sup> to give a bright yellow solid.

### 3.2. 5-Nitroquinolin-8-ol

A suspension of (45 g, 0.25 moles) of 5-nitrosoquinolin-8-ol in a solution of 150 mL of nitric acid and 104 mL of water was in 1 litre beaker was stirred vigorously for 45 min at 16-18 °C. It was later stirred occasionally for an additional 1 h and 15 min, diluted with equal amount of cold water and cooled to 0°. It was made alkaline with aqueous potassium hydroxide and later neutralized acetic as previously reported<sup>19</sup> to give yellow powder.

### 3.3. Diazotization method

A solution of one of the aromatic primary amine in concentrated hydrochloric acid was cooled to 0° in an ice-salt bath. Calculated amount of sodium nitrite dissolved in cold water was added drop wise to the solution of the aromatic amine in an ice-salt bath with vigorous stirring for a period 40 min. Little quantity of ice blocks was also added to the mixture to ensure that the temperature does not exceed 5 °C and then to avoid decomposition of the diazonium salt. The reaction mixture was stirred for additional 30 min to allow for complete diazotization.

### 3.4. Coupling method:

Quinoline-8-ol (3.0 g, 0.02 moles) was dissolved in 25 mL of 10 % NaOH and then cooled to 0 °C in an ice-salt bath. Calculated amount of diazotized primary aromatic amine from the above experiment was added very slowly to the cold solution of quinolin-8-ol with vigorous stirring and then ensuring that the temperature does not exceed 5 °C. Little amount of ice was also added to avoid rise in temperature. After all the diazonium salt solution has been added, the mixture was allowed to stand in an ice bath for an additional 1 h with occasional stirring. It was filtered under suction, washed with water and air dried and purified from chloroform/methanol.

#### 3.4.1. 5-Phenylazoquinolin-8-ol (8)

Quinoline-8-ol (3.0 g, 0.02 moles) was coupled with benzene diazonium salt (2.91 g, 0.02 moles) to give glittering red crystals, mp 224-226 °C; FT-IR (KBr)  $\text{cm}^{-1}$  3460-3330 (br, OH), 1585(-C=N), 1555 (-N=N-), 1506 (C=C Ar), <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) ppm: 9.50 (br, OH), 9.01 (d, 1H), 8.20 (d, 1H), 7.31 (t, 1H), 8.10 (d, 1H), 7.20 (d, 1H), 7.75 (m, 5H, Ar-H azo benzene). <sup>13</sup>C-NMR (CDCl<sub>3</sub>) ppm: 156.2, 154.6, 151.3, 147.4, 140.2, 135.1, 130.3, 129.1, 126.5, 123.2, 122.4, 121.7, 115.6. Elemental analysis calculated for (C<sub>15</sub>H<sub>11</sub>N<sub>3</sub>O), %: C (72.28), H (4.45), N (16.86); Found C (72.25), H (4.47), N (16.84).

#### 3.4.2. 5-(4-nitrophenylazo)quinolin-8-ol (9)

Quinoline-8-ol (3.0 g, 0.02 moles) coupled with 4-nitrobenzene diazonium salt (3.82 g, 0.02 moles) to give glittering deep orange crystals, mp 247-249 °C; FT-IR (KBr)  $\text{cm}^{-1}$  3475-3410 (br, OH), 1585 (-C=N), 1545 (-N=N-), 1480 (C=C Ar), <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) ppm: 9.47 (br, OH), 9.07 (d, 2H), 8.10 (d, 1H), 7.35 (t, 1H), 8.05 (d, 1H), 7.23 (d, 1H), 8.45 (d, 2H), 8.25 (d, 2H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>) ppm: 159.8, 156.7, 152.3, 151.9, 146.1, 141.3 136.6, 124.7, 123.8, 123.1, 122.6, 121.7, 115.7. Elemental analysis calculated for (C<sub>15</sub>H<sub>10</sub>N<sub>4</sub>O<sub>3</sub>), %: C (62.22), H (3.43), N (19.04); Found C (62.19), H (3.44), N (19.07).

#### 3.4.3. 5-nitroso-7-phenylazoquinolin-8-ol (10)

5-Nitrosoquinoline-8-ol (3.50 g, 0.02 moles) coupled with benzene diazonium salt (2.82 g, 0.02 moles) to give glittering orange crystals, mp 230-232 °C. FT-IR (KBr)  $\text{cm}^{-1}$  3450-3300 (br, OH), 1605(-C=N), 1565 (-N=N-), 1500 (C=C Ar), <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) ppm: 9.55 (br, OH), 9.05 (d, 1H), 8.17 (d, 1H), 7.21 (t, 1H), 8.65 (s, 1H), 7.80 (m, 5H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>) ppm: 159.4, 154.7, 153.6, 141.7, 138.3, 137.1, 131.9, 129.6, 125.2, 123.9, 121.6, 117.4. Elemental analysis calculated for (C<sub>15</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>), %: C (64.74), H (3.62), N (20.13), Found C (64.70), H (3.62), N (20.16).

#### 3.4.4. 7-(4-nitrophenylazo)-5-nitrosoquinolin-8-ol (11)

5-Nitrosoquinoline-8-ol (3.50 g, 0.02 moles) coupled with 4-nitrobenzene diazonium salt (3.72 g, 0.02 moles) to give glittering bright red crystals, mp 255-257 °C. FT-IR (KBr)  $\text{cm}^{-1}$  3470-3320 (br, OH), 1580(-C=N), 1545 (-N=N-), 1490 (C=C Ar); <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) ppm: 9.51 (br, OH), 9.11 (d, 1H), 8.13 (d, 1H), 7.30 (t, 1H), 8.60 (s, 1H), 8.45 (d, 2H), 8.23 (d, 2H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>) ppm: 157.7, 156.1, 155.6, 153.4, 152.9, 141.0, 137.6, 136.5, 124.7, 123.2, 123.0, 121.6, 116.7. Elemental analysis calculated for (C<sub>15</sub>H<sub>9</sub>N<sub>5</sub>O<sub>4</sub>), %: C (55.73), H (2.81), N (21.66); Found C (55.71), H (2.85), N (21.68).

#### 3.4.5. 5-Nitro-7-phenylazoquinolin-8-ol (12)

5-Nitroquinoline-8-ol (3.0 g, 0.015 moles) was coupled with benzene diazonium salt (2.21 g, 0.015 moles) to

give glittering red crystals, mp 252-253 °C FT-IR (KBr)  $\text{cm}^{-1}$  3400-3320 (br, OH), 1585(-C=N), 1535 (-N=N-), 1501 (C=C Ar);  $^1\text{H-NMR}$  (DMSO- $d_6$ ) ppm: 9.49 (br, OH), 9.21 (d, 1H, J), 8.82.0 (d, 1H), 7.68 (t, 1H), 8.83 (s, 1H), 7.78 (m, 5H).  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ ) ppm: 155.3, 153.7, 152.9, 141.6, 141.0, 136.1, 132.7, 132.1, 130.9, 130.4, 126.9, 123.8, 123.0, 118.7. Elemental analysis calculated for **12** ( $\text{C}_{15}\text{H}_{10}\text{N}_4\text{O}_3$ ), %: C (61.22), H (3.43), N (19.04); Found C (61.27), H (3.41), N (19.06).

#### 3.4.6. 7-(4-nitrophenylazo)-5-nitroquinolin-8-ol (13)

5-Nitroquinoline-8-ol (3.50 g, 0.018 moles) was coupled with 4-nitrobenzene diazonium salt (3.40 g, 0.018 moles) to give glittering deep red crystals, mp 286-288 °C FT-IR (KBr)  $\text{cm}^{-1}$  3470-3340 (br, OH), 1585(-C=N), 1545 (-N=N-), 1490 (C=C Ar);  $^1\text{H-NMR}$  (DMSO- $d_6$ ) ppm: 9.48 (br, OH), 9.23 (d, 1H), 8.81 (d, 1H), 7.70 (t, 1H), 8.85 (s, 1H), 8.42 (d, 2H), 8.23 (d, 2H).  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ ) ppm: 155.7, 154.3, 153.1, 150.7, 141.9, 141.3, 134.1, 131.4, 125.6, 123.7, 123.5, 122.9, 119.8. Elemental analysis calculated for ( $\text{C}_{15}\text{H}_9\text{N}_5\text{O}_5$ ), %: C (53.10), H (2.67), N (20.64); Found C (53.17), H (2.69), N (16.84).

#### 3.4.7. Dyeing method:

All the synthesized azo dyes have very poor solubility in water. The synthesized dye **8** (0.20 g) was dissolved initially in dimethylformamide (10  $\text{cm}^3$ ) in 250 mL 2-necked flask attached to a reflux condenser and a thermometer. Acetone 30  $\text{cm}^3$  and acetic acid 5  $\text{cm}^3$  was added and solution was agitated for 10 min and the polyester fabric was inserted. The dye bath was warmed gradually by raising the temperature every 5 min till boiling commenced. It was refluxed at the boiling with occasional agitation for 50 min and quickly cooled to room temperature. The dyed fabric was rinsed with water and dried in air. The same procedure was carried out with other dyes.

#### 3.4.8. Wash fastness test

A specimen of the air dried dyed fabric was sewed between pieces of undyed polyester and cotton fabrics of equal size and were mechanically agitated in wash wheel in soap solution at 60 °C for 30 min. The change in colour of the dyed material and the staining on the undyed materials was accessed according to the international geometric gray scale (1 for poor and 5 for excellent fastness) and recorded as presented in the table.

#### 3.4.9. Fastness to sublimation

A small piece of the dried dyed fabric was sewed between a piece of undyed polyester fabric and cotton fabric of equal size. It was treated with sublimation fastness tester at 160 °C for 40 s and later at 180 °C for another 40 s and any colour change and stains on two undyed fabrics sewed together was examined base on the international geometric gray standard (1 and 5 for poor and excellent respectively) as presented in the table.

### Conclusion

Six derivatives of heterocyclic azo dyes of industrial important as colourants were successfully synthesized from quinolin-8-ol. The dyes have intense colours and gave good shades on polyester fabrics; they have well to excellent wash and sublimation fastness properties.

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