Synthesis and Application of Novel Styryl Dyes Derived from 1,4-Diethyl-1,2,3,4-tetrahydro-6-methoxyquinoxaline

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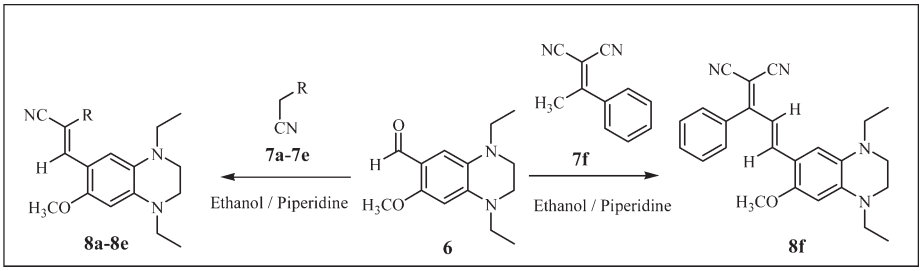

The novel 1,4-diethyl-1,2,3,4-tetrahydro-7-methoxyquinoxalin-6-carboxaldehyde was synthesized, characterized, and condensed with suitably substituted active methylene compounds by classical Knoevenagel condensation reaction to obtain novel monostyryl dyes **8a-8e** and a bis-styryl dye **8f**. These novel styryl dyes have hue varying from orange to violet. The dyes were applied to polyester fibers. The spectral and thermal characteristics of the dyes and fastness properties of the dyed polyester fabric were investigated.

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INTRODUCTION

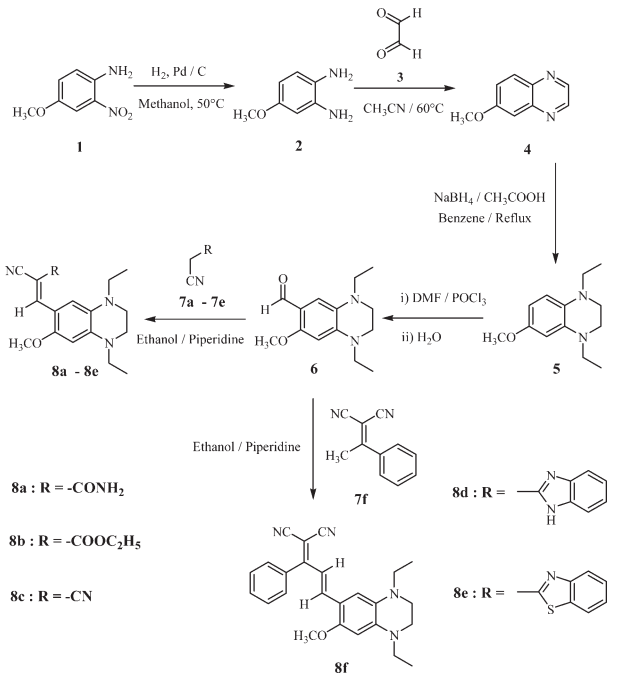
Heterocyclic compounds are of interest as functional materials in the emitters of electroluminescence devices and in molecular probes used for biochemical research, as well as in the traditional textile and polymer fields [1–3]. Heterocyclic chromophores are useful materials in the search for new biologically active compounds and diagnostic methods [4]. They are generally known to have planar and rigid π-conjugation system, and many of them are based on rigid ring systems such as coumarin, naphthalimide, perylene, pyrromethene, and rhodamine. Our research group has been interested in the chemistry of nitrogen containing heterocyclic molecules for many years. Various heterocyclic chromophores based on quinoxaline nucleus have been studied in our laboratory. Fluorescent compounds based on 2-methylthiazolo[4,5-*b*]quinoxaline [5,6], 2-methyl-(1,3)-dithiolium[4,5-*b*]quinoxaline [7] have been reported. In this contribution, we want to report synthesis and application of some new 1,4-diethyl-1,2,3,4-tetrahydroquinoxaline derivatives with different electron donating abilities.

Quinoxalines are, in general, comparatively easy to prepare and numerous derivatives thereof have been designed and synthesized for potential use as biologically active molecules. The classical synthesis of quinoxalines involves condensation of aromatic 1,2-diamines with 1,2-dicarbonyl compounds. The reaction is facile and is the most widely used synthetic method for both quinoxaline itself and its derivatives. They can be easily reduced to 1,2,3,4-tetrahydroquinoxalines by reducing agents such as lithium aluminum hydride [8] and sodium borohydride [9] in excellent yields. Sequential reduction and alkylation of N-heterocycles such as indole to N-alkylated indoline and quinoline to N-alkylated-1,2,3,4-tetrahydroquinoline by sodium borohydride and trifluoroacetic acid is well known [10–13]. Quinoxalines can also be sequentially reduced and dialkylated using sodium borohydride and carboxylic acids. 6-Nitroquinoxaline has been subjected to reductive alkylation using sodium borohydride and glacial acetic acid to obtain 1,4-diethyl-1,2,3,4-tetrahydro-6-nitroquinoxaline [14]. These results led us to explore the utility of substituted tetrahydroquinoxalines to prepare novel styryl dyes.

In this study, 6-methoxyquinoxaline was subjected to alkylative reduction to afford 1,4-diethyl-1,2,3,4-tetrahydro-6-methoxyquinoxaline. This key intermediate was then used to prepare novel red to violet styryl dyes by convenient method of synthesis. We have varied the electron accepting groups to study the influence of the nature of substituents on spectral characteristics of the dyes. To increase the bathochromicity further and study the effect of incorporation of another styryl group, 1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-carboxaldehyde was condensed with

Scheme 1

Synthetic pathway of the dyes **8a-8f**



1,1-dicyano-2-methyl-2-phenylethene. The extended styryl dye thus obtained showed violet hue and displayed absorption maxima at 585 nm. The spectral properties of these novel heterocycles in various solvents differing considerably in polarity and their application as disperse dyes for polyester fabric are reported latter. The thermogravimetric analysis was also carried out to study thermal stability of the dyes.

RESULTS AND DISCUSSION

Synthesis of styryl dyes. The novel styryl dyes were prepared by classical Knoevenagel condensation of 1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-carboxaldehyde **6** and various active methylene compounds **7a-7f** as shown in Scheme 1. In the first stage, 4-methoxy-2-nitroaniline **1** was hydrogenated over palladium charcoal catalyst in methanol to obtain 4-methoxy-1,2-phenylenediamine **2**, which was subsequently condensed with glyoxal **3** in acetonitrile to afford 6-methoxyquinoxaline **4** in excellent yield. Reductive alkylation of 6-methoxyquinoxaline with sodium borohydride and glacial acetic acid in dry benzene yielded 1,4-diethyl-6-methoxy-1,2,3,4-tetrahydroquinoxaline **5**. The electron rich 1,2,3,4-tetrahydroquinoxaline derivative **5** was subjected to Vilsmeier–Haack reaction to yield 1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-carboxaldehyde **6**. To obtain styryl dyes **8a-8f**, a mixture of this aldehyde **6** and the corresponding active methylene compound was refluxed in absolute ethanol and catalytic amount of piperidine. The structures of the dyes were confirmed by IR, ^{1}H NMR, and elemental analysis. The results are summarized in experimental section.

Spectral characteristics of the dyes. Basic absorption characteristics of the compounds such as the absorption maxima (λ_{max}), extinction coefficient (ε) were measured in different solvents and are presented in Table 1. The electronic absorption spectra of the dyes **8a**-**8f** in methanol displayed absorption maxima in the visible region from 480 to 585 nm, which underwent bathochromic shift depending on the extent of conjugation as well as electron withdrawing nature of the acceptor group. The values of molar extinction coefficients were in the range from 12,477 L/mol cm to 31,899 L/mol cm. In the case of monostyryl dyes, introduction of heterocyclic ring as electron acceptor caused a strong bathochromic shift. Especially, dye containing benzothiazole ring showed remarkable shift towards longer wavelength with ε_{max} value of 25,514. This is due to high electronegativity of sulphur atom in the ring and increase in π-delocalization conjugation in the dye. The bis-styryl dye **8f** showed well pronounced maxima at 585 nm, longest among the reported dyes in this communication. This further increase in bathochromicity was attributed to incorporation of another styryl group which produced dye based on 1,1-dicyano-2,4-

Table 1

Spectral properties of the dyes **8a-8f** in different solvents.

	Acetone		Methanol		Acetonitrile		DMF	
Dye No.	λ_{max} (nm)	ε (L/mol cm)	λ_{max} (nm)	ε (L/mol cm)	λ_{max} (nm)	ε (L/mol cm)	λ_{max} (nm)	ε (L/mol cm)
8a	475	24,554	480	18,499	484	21,859	478	18,918
8b	490	26,511	489	31,899	493	29,347	499	31,041
8c	499	15,801	498	12,477	502	19,980	508	20,794
8d	496	29,939	501	29,218	502	23,916	502	30,702
8e	529	35,498	528	25,514	529	28,235	538	33,065
8f	583	39,123	585	24,306	586	42,354	598	46,035

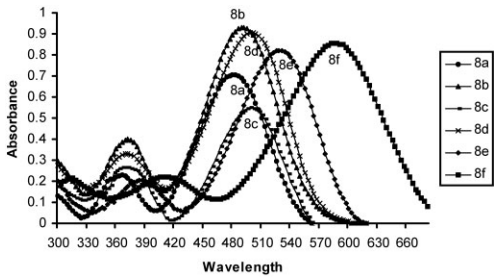


Figure 1. Absorption maxima of the dyes **8a-8f** in methanol.

diarylbutadiene system. The ε_{max} value of the dye was 24,306. The dye exhibited intense violet hue, which is usually difficult to obtain with small styryl structures.

To investigate the influence of solvents on the absorption maxima of the dyes, their absorption spectra were measured in different solvents such as dimethyl formamide, acetonitrile, methanol, and acetone. The solvents differ considerably in polarity and ability to form H-bonding. The electronic absorption spectra of the styryl dyes showed absorption maxima in the visible region at 475–583 nm in acetone, 484–586 nm in acetonitrile, and 478–598 nm in dimethyl formamide (DMF). From the presented values in Table 1, it is evident that practically no solvent effect was observed. Only in case of DMF slight bathochromicity was noticed. The dyes reported in this study exhibit no fluorescence in solvents; however, very weak fluorescence was observed when dissolved in DMF. Figure 1 displays absorption maxima of the dyes **8a-8f** in methanol.

Thermal properties of the dyes. The dyes were subjected to the thermogravimetric analysis to investigate their thermal stability. The change in weight of the dye was measured as a function of temperature. Figures 2 and 3 display thermograph of the representative dyes **8b** and **8f**, respectively. The thermogravimetric curve for the monostyryl dye **8b** (Fig. 2) shows a clear plateau fol-

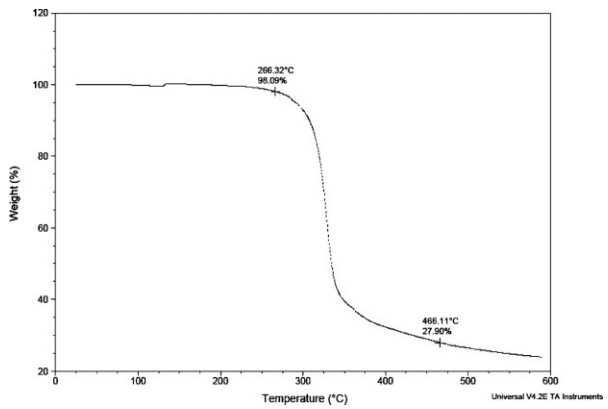


Figure 2. Thermogravimetric curve for the monostyryl dye **8b**.

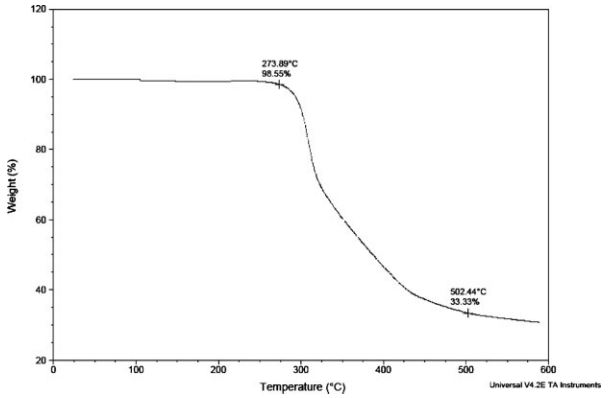


Figure 3. Thermogravimetric curve for the bis-styryl dye **8f**.

lowed by a sharp decomposition curve. The loss in weight of the dye is rapid when heated above 266°C. These results indicate that the dye is stable up to 266°C after which it decomposes rapidly and decomposition completes above 466°C. Almost similar pattern of thermogravimetric curve was obtained for the bis-styryl dye **8f** (Fig. 3) which is stable up to 274°C then starts decomposing slowly and completely decomposes above 502°C. It is clear from the presented data that both monostyryl and bis-styryl dyes have good thermal stability.

Fastness properties of the dyes. Fastness properties of the dyes under study were determined from the standpoint of their application as disperse dyes for polyester fabrics. The dyes **8a-8f** were applied to polyester fiber at 2% shade by high temperature-pressure technique and generally gave bright intense hue ranging from orange to violet. The fastness properties of the dyed fabric such as wash fastness, light fastness, and sublimation fastness were evaluated and assessed on 1 (poor) to 7 (outstanding) rating. The light fastness, wash fastness, and sublimation fastness properties were evaluated as per AATCC standard test methods [15–17]. Table 2 presents values of the fastness properties of dyed polyester fabric. For sublimation fastness determinations, the dyed

Table 2
Fastness properties of the dyes **8a–8f** on polyester fabric.

Dye No.	Light fastness (1–7)	Wash fastness (1–5)	Sublimation fastness (1–5)
8a	4	4	3
8b	3	5	2
8c	3	3	4
8d	5	4	4
8e	6	5	3
8f	4	5	3

1, Poor; 2, Fair; 3, Fairly good; 4, Good; 5, Very good; 6, Excellent; 7, Outstanding.

polyester fabrics were stitched between two pieces of undyed polyester fabrics (stain cloths) and treated at 200°C for 1 min. Any staining on the undyed piece, change in tone, or loss in depth was assessed on a 1 (poor) to 5 (very good) rating.

All the dyes built up well on polyester to give strong, bright colors. The light fastness of the dyes was of generally good order and varied from 3 to 6, dye **8e** exhibited excellent light fastness. The sublimation fastness of the dyes was in the range of 2–4, thus showed good sublimation fastness properties on polyester fabric. The dyes showed good wash fastness, as it is usual with disperse dyes.

EXPERIMENTAL

All melting points were uncorrected and in degree Celsius (°C). IR spectra were recorded on a Bomem Hartmann and Braun MB-Series FIIR spectrometer (KBr). ^{1}H NMR spectra were recorded on Varian 300 MHz mercury plus spectrometer, and chemical shifts are expressed in δ ppm using TMS as an internal standard. Microanalysis for C, H, and N were performed on Thermofignnin Elemental analyzer. Electronic spectra were recorded on Spectronic spectrophotometer from dye solutions in DMF, methanol, acetone, and acetonitrile. Thermogravimetric analysis was carried out on SDT Q600 v8.2 Build 100 model of TA instruments.

Synthesis of 6-methoxyquinoxaline (4). 4-Methoxy-2-nitroaniline (16.8 g, 0.1 mol) was dissolved in methanol (200 mL) and hydrogenated in Parr hydrogenator using 10% Pd/C catalyst at 60°C for 6 h (reaction monitored on TLC). After cooling, the reaction mass was filtered to separate catalyst and concentrated in roto-evaporator. Residue obtained was dissolved in dry acetonitrile (350 mL) and to this solution was added glyoxal (40%, 32.0 mL, 2.6 mol). Reaction mixture was then stirred at 60°C for 6 h and cooled. Solvent was removed in roto-evaporator and the dark brown sticky solid obtained was passed over silica gel column to remove base impurities (Eluent: 70% ethyl acetate in hexane). 6-Methoxyquinoxaline was obtained as white solid in excellent yield, 13.6 g (85%), mp 58–60°C (Lit mp 60°C [18]).

Synthesis of 1,4-diethyl-6-methoxy-1,2,3,4-tetrahydroquinoxaline (5). 6-Methoxyquinoxaline (5.5 g, 0.034 mol) was dissolved in dry benzene (350 mL) and cooled to 5°C. To this cold solution was added sodium borohydride (13.2 g, 0.35 mol) over a period of 15 min. Pale yellow slurry thus obtained was stirred for 10 min. Glacial acetic acid (57.3 mL, 60 g, 1.0 mol) was added to it drop wise over a period of 1 h maintaining the temperature 5–10°C. The brownish slurry that formed was stirred for another 1 h at 10°C and allowed to attain room temperature. It was then heated to gentle reflux for 5 h (reaction monitored on TLC). On cooling, thick red resinous mass was obtained to which water (250 mL) was added. The benzene layer formed was separated and the aqueous layer was extracted with ethyl acetate (3 × 100 mL). Combined extracts and benzene layer were washed repeatedly with dilute sodium carbonate solution and water, dried over anhydrous sodium sulfate, filtered and vacuum evaporated. The dark brown oil obtained was purified by vacuum distillation to afford golden yellow oil, 6.35 g (84%), bp 142–144°C at 2 mm.

Synthesis of 1,4-diethyl-7-methoxy-1,2,3,4-tetrahydro-6-quinoxalinecarboxaldehyde (6). Phosphorous oxychloride (8.0 mL, 0.09 mol) was added to dimethyl formamide (10.1 mL, 0.13 mol) at 5°C under stirring. After 15 min, 1,4-diethyl-6-methoxy-1,2,3,4-tetrahydroquinoxaline (11.0 g, 0.05 mol) was added to the cooled reagent under stirring. The mixture was then heated at 70–80°C for 4 h and poured on ice water. The clear solution was neutralized by adding cold sodium hydroxide solution (15%) maintaining the temperature at 10–15°C. The sticky mass obtained was extracted in ethyl acetate (4 × 100 mL). Ethyl acetate extracts were combined, dried over anhydrous sodium sulfate, and vacuum evaporated. Brown sticky solid showing dark yellow spot on TLC was purified by column chromatography using neutral activated aluminum oxide (Eluent: 40% ethyl acetate in hexane), 8.43 g (68%), bp 146–148°C, IR: CO 1712 cm^{-1}, ^{1}H NMR: δ 1.16 (t, 6.9Hz, 3H, CH_3), δ 1.23 (t, 6.9Hz, 3H, CH_3), δ 3.13–3.18 (m, 2H), δ 3.31 (q, 6.9Hz, 2H, CH_2), δ 3.41 (q, 6.9Hz, 2H, CH_2), δ 3.49–3.54 (m, 2H), δ 3.93 (s, 3H, OCH_3), δ 6.0 (s, 1H, phenyl proton), δ 7.0 (s, 1H, phenyl proton), δ 10.20 (s, 1H, aldehydic proton), *Anal.* Calcd for $C_{14}H_{20}N_2O_2$: C, 67.71; H, 8.12; N, 11.28. Found: C, 67.81; H, 8.14; N, 11.34.

Synthesis of styryl dyes (8a–8f).

Synthesis of 2-cyano-3-(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)-2-propenoic acid ethylester (8b). 1,4-Diethyl-7-methoxy-1,2,3,4-tetrahydro-6-quinoxalinecarboxaldehyde (2.48 g, 0.01 mol) and ethyl cyanoacetate 1.13 g (0.01 mol) were dissolved in dry ethanol. Piperidine (0.1 mL) was added to it and reaction mixture was refluxed for 2 h. Ethanol was removed by distillation and reddish crystals were washed with water and dried. Dye **(8b)** thus obtained was further purified by column chromatography using neutral activated aluminum oxide (Eluent: 30% ethyl acetate in hexane), 2.7 g, 78%, mp 128–132°C, IR: CO 1755, CN 2210 cm^{-1}, ^{1}H NMR**:** δ 1.21 (t, 6.9Hz, 3H, CH_3), δ 1.26 (t, 6.9Hz, 3H, CH_3), δ 1.36 (t, 6.9Hz, 3H, CH_3), δ 3.16–3.22 (m, 2H), δ 3.34 (q, 6.9Hz, 2H, CH_2), δ 3.44 (q, 6.9Hz, 2H, CH_2), δ 3.53–3.59 (m, 2H), δ 3.92 (s, 3H, OCH_3), δ 4.44 (q, 6.9Hz, 2H, CH_2), δ 6.0 (s, 1H, phenyl proton), δ 7.7 (s, 1H, phenyl proton), δ 8.6 (s, 1H, olefinic proton), *Anal.* Calcd for $C_{19}H_{25}N_3O_3$: C, 66.45; H, 7.34; N, 12.24. Found: C, 66.56; H, 7.35; N, 12.31.

Synthesis of 2-cyano-3-(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)-2-propenamide (8a). 2.54 g (81%), mp 172–175°C, IR: CO 1685, CN 2218 cm^{-1}, ^{1}H NMR: δ 1.20 (t, 7.1Hz, 3H, CH_3), δ 1.25 (t, 7.1Hz, 3H, CH_3), δ 3.18–3.23 (m, 2H), δ 3.35 (q, 6.9Hz, 2H, CH_2), δ 3.44 (q, 6.9Hz, 2H, CH_2), δ 3.54–3.59 (m, 2H), δ 3.90 (s, 3H, OCH_3), δ 5.3 (s, 2H, $CONH_2$ proton), δ 6.0 (s, 1H, phenyl proton), δ 7.6 (s, 1H, phenyl proton), δ 8.5 (s, 1H, olefinic proton), *Anal.* Calcd for $C_{17}H_{22}N_4O_2$: C, 64.95; H, 7.05; N, 17.82. Found: C, 64.83; H, 7.07; N, 17.99.

Synthesis of [(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)methylene]propanedinitrile (8c). 2.22 g (75%), mp 226–228°C, IR: CN 2223 cm^{-1}, ^{1}H NMR: δ 1.22 (t, 7.0Hz, 3H, CH_3), δ 1.28 (t, 7.0Hz, 3H, CH_3), δ 3.16–3.21 (m, 2H), δ 3.33 (q, 7.0Hz, 2H, CH_2), δ 3.42 (q, 7.0Hz, 2H, CH_2), δ 3.52–3.57 (m, 2H), δ 3.93 (s, 3H, OCH_3), δ 6.1 (s, 1H, phenyl proton), δ 7.7 (s, 1H, phenyl proton), δ 8.9 (s, 1H, olefinic proton), *Anal.* Calcd for C17H20N4O: C, 68.89; H, 6.80; N, 18.90. Found: C, 68.99; H, 6.83; N, 18.97.

Synthesis of α-[(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)methylene]-1-H-benzimidazoleacetonitrile (8d). 2.82 g (73%), mp 258–260°C, IR: CN 2220 cm^{-1}, ^1H NMR: δ 1.20 (t, 7.0Hz, 3H, CH_3), δ 1.26 (t, 7.0Hz, 3H, CH_3), δ 3.19–3.25 (m, 2H), δ 3.36 (q, 7.0Hz, 2H, CH_2), δ 3.46 (q, 7.0Hz, 2H, CH_2), δ 3.55–3.61 (m, 2H), δ 3.90 (s, 3H, OCH_3), δ 6.0 (s, 1H, phenyl proton), δ 7.5 (s, 1H, phenyl proton), δ 8.3 (s, 1H, olefinic proton), δ 7.5 (d, 2H, phenyl proton), δ 7.2 (d, 2H, phenyl proton), δ 10.2 (s, 1H, NH proton), *Anal*. Calcd for $C_{23}H_{25}N_5O$: C, 71.29; H, 6.50; N, 18.07. Found: C, 71.41; H, 6.54; N, 18.08.

Synthesis of α-[(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)methylene]benzthiazoleacetonitrile (8e). 3.03 g (75%), mp 170–172°C, IR: CN 2225 cm^{-1}, ^1H NMR: δ 1.20 (t, 7.1Hz, 3H, CH_3), δ 1.26 (t, 7.1Hz, 3H, CH_3), δ 3.19–3.25 (m, 2H), δ 3.33 (q, 7.1Hz, 2H, CH_2), δ 3.43 (q, 7.1Hz, 2H, CH_2), δ 3.55–3.61 (m, 2H), δ 3.93 (s, 3H, OCH_3), δ 6.1 (s, 1H, phenyl proton), δ 7.6 (s, 1H, phenyl proton), δ 8.4 (s, 1H, olefinic proton), δ 7.7 (d, 2H, phenyl proton), δ 7.2 (d, 2H, phenyl proton), *Anal*. Calcd for $C_{23}H_{24}N_4OS$: C, 68.29; H, 5.98; N, 13.85; S, 7.93. Found: C, 68.28; H, 5.96; N, 13.90; S, 7.87.

Synthesis of [3-(1,4-diethyl-7-methoxy-1,2,3,4-tetrahydroquinoxalin-6-yl)-1-phenyl-2-propenylidene]propanedinitrile (8f). 3.2 g (81%), mp 218–220°C, IR: CN 2210 cm^{-1}, ^1H NMR: δ 1.18 (t, 6.9Hz, 3H, CH_3), δ 1.24 (t, 6.9Hz, 3H, CH_3), δ 3.15–3.20 (m, 2H), δ 3.30 (q, 6.9Hz, 2H, CH_2), δ 3.42 (q, 6.9Hz, 2H, CH_2), δ 3.48–3.55 (m, 2H), δ 3.73 (s, 3H, OCH_3), δ 5.99 (s, 1H, aromatic proton), δ 6.63 (s, 1H, aromatic proton), δ 7.28 (m, 1H, phenyl proton), δ 7.33–7.34 (m, 2H, phenyl proton), δ 7.53–7.56 (m, 2H, phenyl proton), δ 7.12–7.17 (d, 15.03Hz, 1H, olefinic proton), δ 7.40–7.45 (d, 15.03Hz, 1H, olefinic proton), *Anal*. Calcd for $C^{25}H^{26}N^4O$: C, 75.35.; H, 6.58; N, 14.06. Found: C, 74.96; H, 6.33; N, 14.28.

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