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The Swern Oxidation: First example of direct oxidation of 2-pyrazolines with "activated" DMSO

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ABSTRACT

1,3-Dipolar cycloaddition of 2-diazopropane to conjugated dienes **1** and α,β-unsaturated ketones **5** is taking place regiospecifically to give cycloadduit **4** and **6**. The reaction of 2-pyrazolines derivatives with dimethylsulfoxide and oxalyl chloride under Swern conditions led to a pyrazolenines **7** and **8**.

1. Introduction

Pyrazoles derivatives are synthetic targets of utmost importance in the pharmaceutical industry, since such a heterocyclic moiety represents the core structure of numerous drugs. Furthermore, recent reports indicate of a number of highly potent inhibitors of coagulation factors Xa [1]. A number of compounds containing the pyrazole core have been examined for antidepressant activity through screening against monoamine oxidases [2], treatment of obesity as cannabinoid-1 antagonists [3], antiviral activity against the West Nile virus [4], and multidrug resistance modulators in tumor cells [5]. Pyrazoles are extremely powerful reagents for the construction of nitrogen containing substance [6]. The 1,3-dipolar cycloaddition reaction is a classical and widely used method for the construction of 2-pyrazolines [7]. The oxidation of 2-pyrazoline, is, in fact, the pyrazolenine which applies to many synthetic strategies. A large number of procedures are found in the literature to accomplish this fundamental setup. Several reagents are available for oxidation of the 2-pyrazolines, the nature of the product depending on the choice of oxidant and the substitution pattern of the substrate. Various efforts have been made previously in the oxidation of 2-pyrazolines with a variety of reagents including $Zr(NO_3)_4$ [8], Pd/C [9], Co(II) and oxygen [10], iodobenzene diacetate [11], lead tetraacetate [12], MnO_2 [13], potassium permanganate [14] and NBS [15], for the preparation of pyrazolenines. The combination of dimethylsulfoxide (DMSO) with an electrophilic species to form "activated DMSO" [16] has been widely exploited for the oxidation of alcohols to their respective carbonyl compounds. However, many of these methods are subject to certain drawbacks such as longer reaction times, low yields and toxicity due to the presence of some elements embodied in the reagents utilized. So still there is need for development of new catalysts which overcome all these drawbacks.

2. Results and discussion

The reaction of dienes **1a–d** and 2-diazopropane furnishes the 1-pyrazoline **3a–d**, which undergoes spontaneous tautomerization to afford the 2-pyrazoline **4a–d** in good yield (Scheme 1) [17].

The FAB mass and two-dimensional NMR spectra of these adducts are identical and correspond to diastereoisomeric

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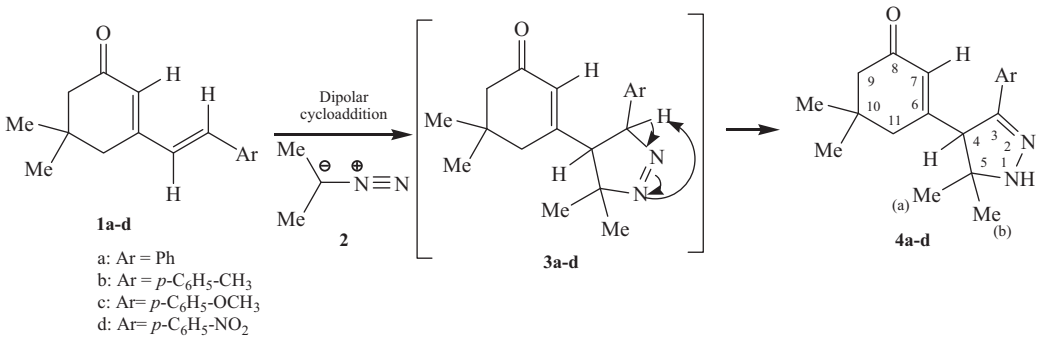
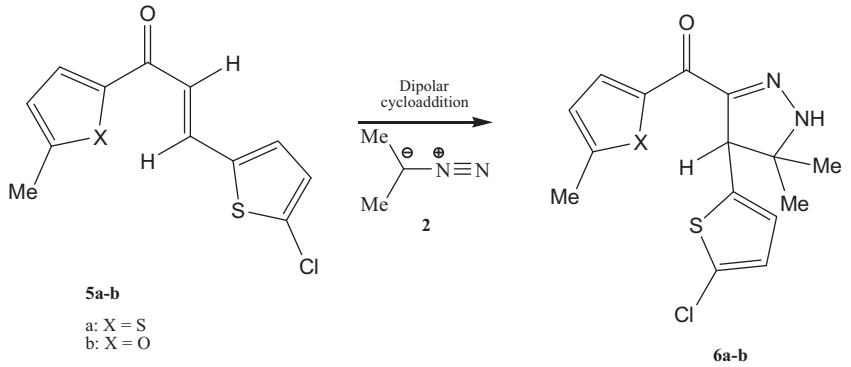
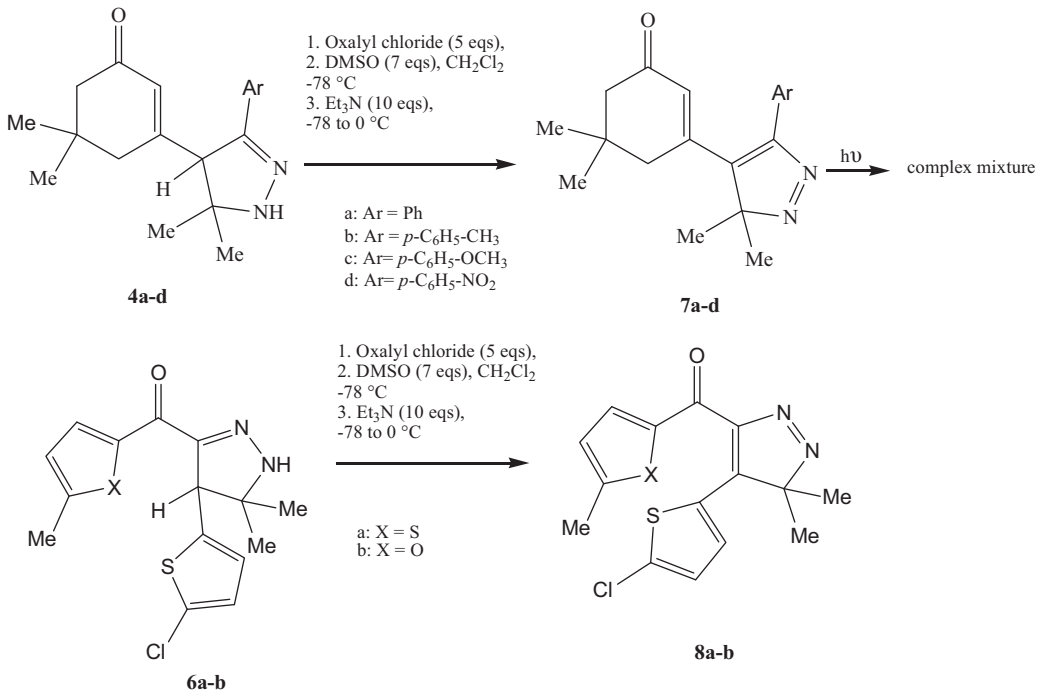


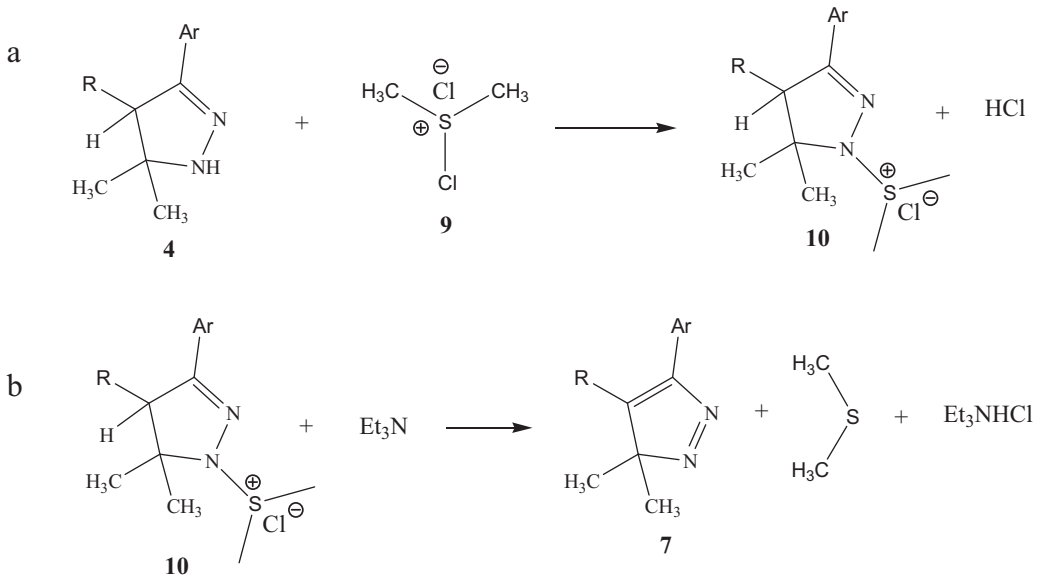
Scheme 1. Preparation of 2-pyrazolines.



Scheme 2. Synthesis of 2-pyrazolines.



Scheme 3. Formation of pyrazolenines.



Scheme 4. Swern oxidation reaction steps.

structures indiscernible by spectroscopic analyses. However, regiochemical assignments of all adducts were deduced from their HMBC 2D-NMR spectra. H-7 and H-11 protons correlate only with the carbon atom C-4 (58.9–59.5 ppm). Also, methyl protons (a) and (b) correlate only with two carbon atoms C-4 and C-5 and each other suggesting that they are directly linked to the quaternary carbon C-5. In a similar manner, aromatic protons correlate with the carbon C-3 which is directly bonded to the aryl group. Consequently, this latter correlation shows Ar-C3-C4-C5-(Me(a),Me(b)) linkages indicative of a "inverse" regiochemistry which is generally observed in 1,3-dipolar cycloaddition reactions of simple diazoalkanes with α,β-unsaturated ketones [18].

On the other hand the same reaction is carried out under similar operating conditions with α,β-unsaturated ketones **5** has led to a 2-pyrazoline cycloadduits **6** (Scheme 2) [19].

As shown in Scheme 3, the reaction between 2-pyrazoline derivatives **4a–d** and **6a–b** and dimethylsulfoxide under Swern conditions gave good yields of pyrazolenine derivatives **7a–d** and **8a–b** which can be considered as suitable precursors to *gem*-dimethylcyclopropenes after photochemical nitrogen extrusion [20].

In the approach to the Swern oxidation, oxalyl chloride is added to a solution of the 2-pyrazoline **4** and dimethylsulfoxide, while maintaining the temperature at −78 °C. Under these conditions the "activated" DMSO intermediate **9** is formed as a transient species which is rapidly consumed by the 2-pyrazoline **4** to form the alkoxysulfonium chloride **10**. Addition of triethylamine afforded the pyrazolenine **7** [21] (Scheme 4).

Irradiation of an ethereal solution of the pyrazolenine **7** at 0–5 °C using 350 nm lamps gave a complex mixture of products.

3. Conclusion

In conclusion, we have described the synthesis of new pyrazolines derivatives with complete regioselectivity. The Swern conditions, involving the use of very simple and inexpensive reagents, allow the one-pot transformation of 2-pyrazolines with a pyrazolenines into synthetically valuable. These findings constitute a significant addition to the growing list of synthetic applications of activated dimethylsulfoxide.

4. Experimental

General Methods. Chromatography was performed with silica gel 60 (230–400 mesh), and silica gel F254 plates were used for preparative TLC. The IR spectra frequencies are gives in cm^{-1}. NMR spectra were determined in $CDCl_3$ solutions at 300 and 75.5 MHz for ^1H and ^{13}C NMR, respectively; chemical shifts (δ) were reported in ppm and J values are gives in hertz.

4.1. 1,3-Dipolar Cycloaddition of 2-diazopropane with dienes (1) and α,β-unsaturated ketones (5)

To a solution of dipolarophiles **1a–d** or **5a–d** (1.0 mmol) in diethyl ether, cooled at −20 °C, was added portionwise 2.6 M ethereal solution of 2-diazopropane. The reaction was kept at the same temperature during 1 h. The solvent was removed and chromatography (SiO_2; ethyl acetate/petroleum ether, 1:3) to afford compounds **4a–d** and **6a–d**.

4.1.1. 2-Pyrazoline (4a)

Yield (0.192 g, 65%), colourless solid. M.p 180–181 °C, FAB-MS m/z (%): 297 (MH^+,100), *Anal.* Calcd. For $C_{19}H_{24}N_2O$: C, 76.99; H, 8.16; N, 9.45%; Found: C, 76.87; H, 8.14; N, 9.51%. IR (KBr): N = N 1540; C = O 1680; NH 3250 cm^{-1}. ^1H-NMR (300 MHz, $CDCl_3$) δ: 0.85 (s, 3H, CH_3), 1.00 (s, 3H, CH_3), 1.06 (s, 3H, CH_3), 1.33 (s, 3H, CH_3), 2.21 (s, 2H, H_9), 2.65 (s, 2H, H_{11}), 3.76 (s, 1H, H_4), 5.74 (s, 1H, H_7), 5.83 (br s, 2H, H_1), 6.90–7.30 (m, 5H, H_{arom}). $^{13}C\{^1H\}$NMR (75 MHz, $CDCl_3$) δ: 22.82 (CH_3), 28.29 (CH_3), 29.40 (CH_3), 33. 41(C-10), 38.94 (C-11), 51.50 (C-9), 59.54 (C-4), 67.40

(C-5), 125.40 (C-7), 127.43–136.32 (C_{arom}), 150.15 (C-6), 153.31 (C-3), 200.45 (C-8).

4.1.2. 2-Pyrazoline (4b)

Yield (0.217 g, 70%), colourless solid. M.p 170–171 °C, FAB-MS m/z(%): 311 (MH^+,100), *Anal.* Calcd. For $C_{20}H_{26}N_2O$: C, 77.38; H, 8.44; N, 9.02%; Found: C, 77.30; H, 8.39; N, 9.08%. IR (KBr): C = N 1550; C = O 1690; NH 3250 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.86 (s, 3H, CH_3), 1.01 (s, 3H, CH_3), 1.06 (s, 3H, CH_3), 1.32 (s, 3H, CH_3), 2.22 (s, 2H, H_9), 2.30 (s, 3H, CH_3), 2.66 (s, 2H, H_{11}), 3.73 (s, 1H, H_4), 5.75 (s, 1H, H_7), 5.81 (br s, 2H, H_1), 6.85; 7.08 (AA'BB', 4H, H_{arom}, J = 8.7 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 21.20 (CH_3), 22.90 (CH_3), 28.30 (CH_3), 29.40 (CH_3), 33.50 (C-10), 39.00 (C-11), 51.60 (C-9), 59.30 (C-4), 67.30 (C-5), 125.40 (C-7), 128.00–137.00 (C_{arom}), 150.20 (C-6), 153.50 (C-3), 201.01 (C-8).

4.1.3. 2-Pyrazoline (4c)

Yield (0.261 g, 80%), colourless solid. M.p 210–211 °C, FAB-MS m/z(%): 327 (MH^+,100), *Anal.* Calcd. For $C_{20}H_{26}N_2O_2$: C, 73.59; H, 8.03; N, 8.58%; Found: C, 73.50; H, 8.11; N, 8.51%. IR (KBr): C = N 1550; C = O 1680; NH 3330 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.75 (s, 3H, CH_3), 0.93 (s, 3H, CH_3), 0.99 (s, 3H, CH_3), 1.21 (s, 3H, CH_3), 2.22 (s, 2H, H_9), 2.66 (s, 2H, H_{11}), 3.72 (s, 1H, H_4), 3.78 (s, 3H, OCH_3), 5.76 (s, 1H, H_7), 5.80 (br s, 1H, H_1), 6.81; 6.88 (AA'BB', 4H, H_{arom}, J = 9 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 22.80 (CH_3), 28.00 (CH_3), 28.20 (CH_3), 29.20 (CH_3), 33.30 (C-10), 39.00 (C-11), 51.60 (C-9), 55.19 (OCH_3), 58.90 (C-4), 67.30 (C-5), 125.40 (C-7), 114.00–158.90 (C_{arom}), 150.10 (C-6), 153.50 (C-3), 201.20 (C-8).

4.1.4. 2-Pyrazoline (4d)

Yield (0.238 g, 70%), colourless solid. M.p 202–203 °C, FAB-MS m/z(%): 342 (MH^+,100), *Anal.* Calcd. For $C_{19}H_{23}N_3O_3$: C, 66.84; H, 6.79; N, 12.31%; Found: C, 66.76; H, 6.82; N, 12.39%. IR (KBr): C = N 1550; C = O 1680; NH 3330 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.76 (s, 3H, CH_3), 0.94 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 1.26 (s, 3H, CH_3), 2.23 (s, 2H, H_9), 2.64 (s, 2H, H_{11}), 3.70 (s, 1H, H_4), 5.78 (s, 1H, H_7), 5.81 (br s, 1H, H_1), 7.81; 8.12 (AA'BB', 4H, H_{arom}, J = 8.7 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 22.81 (CH_3), 27.01 (CH_3), 28.22 (CH_3), 29.16 (CH_3), 33.27 (C-10), 38.50 (C-11), 51.62 (C-9), 58.91 (C-4), 67.29 (C-5), 125.39 (C-7), 124.10–171.5 (C_{arom}), 150.09 (C-6), 152.97 (C-3), 202.19 (C-8).

4.1.5. 2-Pyrazoline (6a)

Yield (0.287 g, 85%), yellow solid. M.p 140–141 °C, *Anal.* Calcd. For $C_{15}H_{15}ClN_2OS_2$: C, 53.16; H, 4.46; N, 8.27%; Found: C, 53.23; H, 4.35; N, 8.21%. IR (KBr): C = N 1520; C = O 1640; NH 3300 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.87 (s, 3H, CH_3), 1.29 (s, 3H, CH_3), 2.41 (s, 3H, CH_3), 4.10 (s, 1H, H_4), 6.15 (br s, 1H, H_1), 6.67; 7.09 (2H, H_{th}, J = 3.6 Hz); 6.82; 7.51 (2H, H_{th}, J = 3.9 Hz). ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 15.90 (CH_3), 22.51 (CH_3), 29.02 (CH_3), 57.94 (C-4), 68.00 (C-5), 152.55 (C-3), 125.2–153.04 (C_{th}), 187.80 (C = O).

4.1.6. 2-Pyrazoline (6b)

Yield (0.226 g, 70%), yellow solid. M.p 149–150 °C, *Anal.* Calcd. For $C_{15}H_{15}ClN_2O_2S$: C, 55.81; H, 4.68; N, 8.68%; Found: C, 55.89; H, 4.59; N, 8.47%. IR (KBr): C = N 1525; C = O 1650; NH 3320 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.89 (s, 3H, CH_3), 1.28 (s, 3H, CH_3), 2.41 (s, 3H, CH_3), 4.09 (s, 1H, H_4), 6.35 (br s, 1H, H_1), 6.13; 6.63 (2H, H_{fu}, J = 3 Hz); 6.69; 7.13 (2H, H_{th}, J = 3.6 Hz). ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 15.79 (CH_3), 22.47 (CH_3), 28.99 (CH_3), 57.87 (C-4), 67.93 (C-5), 151.94 (C-3), 108.15–153.65 ($C_{fu,th}$), 186.41 (C = O).

4.2. Dehydrogenation of 2-Pyrazolines

To a solution of oxalyl chloride (5 equiv) in dry CH_2Cl_2 (10 mL), at −78 °C under an argon atmosphere, was added DMSO (7 equiv). The solution was stirred for 15 min, until effervescence ceased. A solution of the 2-pyrazolines **4a–d** or **6a–d** (1 mmol) in dry CH_2Cl_2 (5 mL) was added dropwise, and the solution was stirred for 10 min at −78 °C. Triethylamine (10 equiv) was then added and the solution was left to warm to 0 °C for 30 min, while stirred. The reaction mixture was diluted with CH_2Cl_2 (20 mL) and washed with saturated aqueous NH_4Cl (3 × 20 mL). The organic layer was dried ($MgSO_4$) and evaporated, and the residue was purified by chromatography (SiO_2; ethyl acetate/petroleum ether, 1:4) to afford compounds **7a–d** and **8a–d**.

4.2.1. Pyrazolenine (7a)

Yield (0.235 g, 80%), colourless solid. Mp 121–122 °C, *Anal.* Calcd. For $C_{19}H_{22}N_2O$: C, 77.52; H, 7.53; N, 9.52%; Found: C, 77.44; H, 7.56; N, 9.45%. IR (KBr): C = C–N = N 1615; C = O 1690 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.95 (s, 6H, CH_3), 1.25 (s, 6H, CH_3), 2.20 (s, 2H, H_9), 2.63 (s, 2H, H_{11}), 5.71 (s, 1H, H_7), 6.89–7.35 (m, 5H, H_{arom}); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 20.50 (CH_3), 27.62 (CH_3), 33. 35 (C-10), 38.91 (C-11), 51.51 (C-9), 147.34 (C-4), 163.20 (C-5), 125.37 (C-7), 127.36–136.47 (C_{arom}), 150.14 (C-6), 153.28 (C-3), 201.21 (C-8).

4.2.2. Pyrazolenine (7b)

Yield (0.277 g, 90%), colourless solid. M.p 110–111 °C, *Anal.* Calcd. For $C_{20}H_{24}N_2O$: C, 77.89; H, 7.84; N, 9.08%; Found: C, 77.91; H, 7.81; N, 9.06%. IR (KBr): C = C–N = N 1610; C = O 1690 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.91 (s, 6H, CH_3), 1.25 (s, 6H, CH_3), 2.31 (s, 3H, CH_3), 2.20 (s, 2H, H_9), 2.59 (s, 2H, H_{11}), 5.71 (s, 1H, H_7), 6.85; 7.08 (AA'BB', 4H, H_{arom}, J = 8.7 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 20.53 (CH_3), 21.09 (CH_3), 26.98 (CH_3), 33.51 (C-10), 38.55 (C-11), 51.61 (C-9), 147.33 (C-4), 162.83 (C-5), 125.40 (C-7), 128.11–138.01 (C_{arom}), 151.13 (C-6), 154.10 (C-3), 200.03 (C-8).

4.2.3. Pyrazolenine (7c)

Yield (0.275 g, 85%), colourless solid. M.p 123–124 °C, *Anal.* Calcd. For $C_{20}H_{24}N_2O_2$: C, 74.04; H, 7.46; N, 8.64%; Found: C, 74.00; H, 7.43; N, 8.71%. IR (KBr): C = C-N = N 1615; C = O 1685 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.88 (s, 6H, CH_3), 1.27 (s, 6H, CH_3), 2.19 (s, 2H, H_9), 2.65 (s, 2H, H_{11}), 5.71 (s, 1H, H_7), 3.77 (s, 3H, OCH_3), 6.81; 6.88 (AA'BB', 4H, H_{arom}, J = 9 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 20.45 (CH_3), 26.88 (CH_3), 32.91 (C10), 39.13 (C11), 51.58 (C-9), 55.18 (OCH_3), 146.11 (C-4), 163.01 (C-5), 125.38 (C-7),

115.11–158.87 (C_{arom}), 151.04 (C-6), 152.77 (C-3), 200.82 (C-8).

4.2.4. Pyrazolenine (7d)

Yield (0.254 g, 75%), colourless solid. M.p 98–99 °C, *Anal.* Calcd. For $C_{19}H_{21}N_3O_3$: C, 67.24; H, 6.24; N, 12.38%; Found: C, 67.38; H, 6.17; N, 12.45%. IR (KBr): C = C-N = N 1610; C = O 1685 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.87 (s, 6H, CH_3), 1.28 (s, 6H, CH_3), 2.18 (s, 2H, H_9), 2.63 (s, 2H, H_{11}), 5.68 (s, 1H, H_7), 7.80; 8.22 (AA'BB', 4H, H_{arom}, J = 8.7 Hz); ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 20.38 (CH_3), 26.56 (CH_3), 32.88 (C10), 39.09 (C11), 51.51 (C-9), 146.17 (C-4), 163.21 (C-5), 124.66 (C-7), 123.89–169.57 (C_{arom}), 150.36 (C-6), 151.69 (C-3), 200.54 (C-8).

4.2.5. Pyrazolenine (8a)

Yield (0.218 g, 65%), yellow solid. Mp 101–102 °C, *Anal.* Calcd. For $C_{15}H_{13}ClN_2OS_2$: C, 53.48; H, 3.89; N, 8.32%; Found: C, 53.53; H, 3.99; N, 8.42%. IR (KBr): C = C-N = N 1615; C = O 1690 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.60 (s, 6H, CH_3), 2.39 (s, 3H, CH_3), 6.55; 7.07 (2H, H_{th}, J = 3.6 Hz); 6.67; 7.74 (2H, H_{th}, J = 3.9 Hz). ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 16.02 (CH_3), 21.81 (CH_3), 97.67 (C-3), 138.15 (C-4), 157.29 (C-5), 121.02–153.19 (C_{th}), 190.13 (C = O).

4.2.6. Pyrazolenine (8b)

Yield (0.257 g, 80%), yellow solid. Mp 107–108 °C, *Anal.* Calcd. For $C_{15}H_{13}ClN_2O_2S$: C, 56.16; H, 4.08; N, 8.73%; Found: C, 56.11; H, 4.00; N, 8.58%. IR (KBr): C = C-N = N 1610; C = O 1690 cm^{-1}. ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.63 (s, 6H, CH_3), 2.42 (s, 3H, CH_3), 6.19; 6.62 (2H, H_{th}, J = 3 Hz); 6.55; 7.15 (2H, H_{fu}, J = 3.9 Hz). ^{13}C{^{1}H}NMR (75 MHz, $CDCl_3$) δ: 15.92 (CH_3), 22.03 (CH_3), 97.55 (C-3), 137.87 (C-4), 157.12 (C-5), 114.00–153.11 ($C_{fu,th}$), 190.22 (C = O).

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