Diversity-Oriented Synthesis of Novel 2′-Aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] Derivatives *via* a One-Pot Four-Component Reaction

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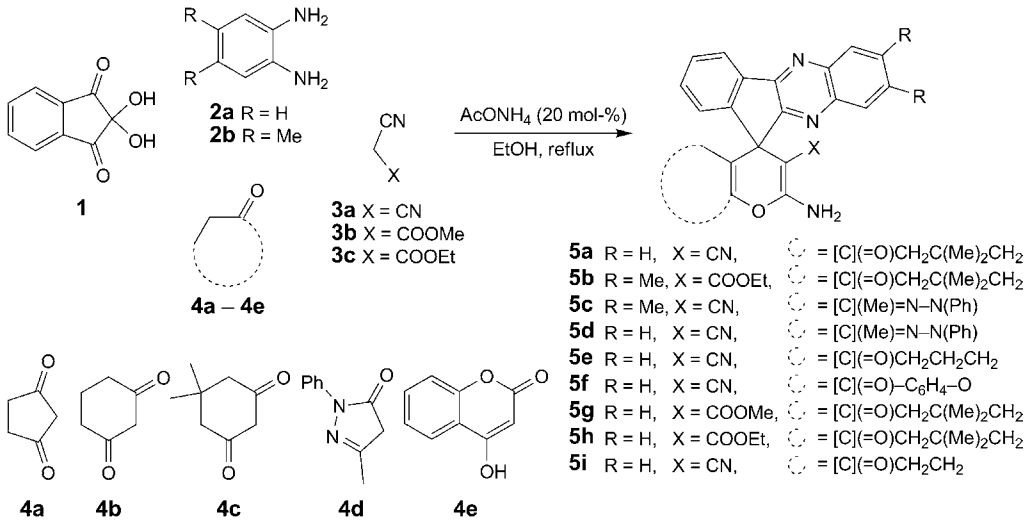
A sequential one-pot four-component reaction for the efficient synthesis of novel 2′-aminospiro-[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] derivatives **5** in the presence of $AcONH_4$ as a neutral, inexpensive, and dually activating catalyst is described (*Scheme 1*). The syntheses are achieved by reacting ninhydrin (**1**) with benzene-1,2-diamines **2** to give indenoquinoxalines, which are trapped *in situ* by malono derivatives **2** and various α-methylenecarbonyl compounds **4** through cyclization, providing the multifunctionalized 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] analogs **5**. This chemistry provides an efficient and promising synthetic way of proceeding for the diversity-oriented construction of the spiro[indenoquinoxalino-pyran] skeleton.

Introduction. – Quinoxaline derivatives are an important group of aza-polycyclic compounds which have many biological and pharmaceutical applications such as antibacterial [1], antifungal [1], antidepressant [2], and antitumor agents [3]. On the other hand, pyrans and their derivatives are of considerable interest because of their wide range of biological properties [4] such as spasmolytic [5], diuretic [6], and anticancer activity [7]. The 4*H*-pyrans also constitute the structural framework of many natural products [8]. Moreover, spiro compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties. The spiro functionality has been found to be present in phytochemicals such as alkaloids, lactones, or terpenoids [9]. The biological activities of spiro compounds containing pyran moieties have also been reported [10]. They also show good activity as hypertensive agents [11] and have been the subject of significant interest as potential novel analgesic agents [12].

In continuation of our research on the synthesis of quinoxalines [13] and the development of multicomponent reactions [14], we report herein a one-pot, four-component method for the diversity-oriented synthesis of novel 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] derivatives **5** from ninhydrin (=2,2-dihydroxy-1*H*-indene-1,3(2*H*)-dione; **1**), benzene-1,2-diamines **2**, malono derivatives **3**, and α-methylenecarbonyl compounds **4** in the presence of ammonium acetate as a neutral, inexpensive, and dually activating catalyst (*Scheme 1*).

Results and Discussion. – Our initial efforts focused on the search for a suitable catalyst for the synthesis of 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-

Scheme 1. *One-Pot-Four-Component Synthesis of 2′-Aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] Derivatives* **5a**–**5i**



[4*H*]pyran] derivatives **5**. The condensation reaction of equimolar amounts of ninhydrin (**1**), benzene-1,2-diamine (**2a**), malononitrile (**3a**), and dimedone (= 5,5-dimethylcyclohexane-1,3-dione; **4c**) to yield compound **5a** was selected as a model reaction in the presence of different catalytic systems, and the results are summarized in *Table 1*. After expansive screening, we found that optimum yields and reaction times were obtained in the presence of 20 mol-% of $AcONH_4$ in EtOH under reflux, which

Table 1. *One-Pot Four-Component Synthesis of Compound* **5a** *from* **1**, **2a**, **3a**, *and* **4c** (each 1 mmol) in the Presence of Various Catalysts

Entry	Catalyst	Reaction conditions	Time [h]	Yield [%][a])
1	SiO_2/SO_3H (0.5 g)	EtOH, reflux	24	81
2	P_2O_5/SiO_2 (5% w/w)	EtOH, reflux	24	57
3	$NaHSO_4 \cdot SiO_2$ (20 mol-%)	EtOH, reflux	36	46
4	$Bi(NO_3)_4$ (20 mol-%)	EtOH, reflux	18	50
5	$(NH_4)_6Mo_7O_{24} \cdot 4\ H_2O$ (20 mol-%)	EtOH, reflux	18	26
6	[Bmim]HSO_4[b]) (20 mol-%)	EtOH, reflux	24	50
7	NH_4Cl (20 mol-%)	EtOH, reflux	24	trace
8	$(NH_4)_2CO_3$ (20 mol-%)	EtOH, reflux	24	20
9	$AcONH_4$ (20 mol-%)	EtOH, reflux	12	91
10	$AcONH_4$ (20 mol-%)	H_2O, 80°	24	trace
11	$AcONH_4$ (15 mol-%)	EtOH, reflux	24	65
12	$AcONH_4$ (20 mol-%)	MeCN, reflux	24	76
13	$AcONH_4$ (20 mol-%)	$CHCl_3$, reflux	24	52
14	$AcONH_4$ (20 mol-%)	EtOAc, reflux	24	43
15	–	EtOH, reflux	48	trace

[a])Yield of isolated **5a**. [b]) 1-Butyl-3-methyl-1*H*-imidazolium hydrogen sulfate.

furnished 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran]-3′-carbonitrile **5a** in 91% yield within 12 h (*Table 1*, *Entry 9*). Increasing the amount of $AcONH_4$ to more than 40 mol-% did not substantially improve the yield, whereas the yield decreased when the amount of the catalyst was 15 mol-%. Moreover, the reaction did not proceed efficiently in the absence of $AcONH_4$ even after 48 h.

Having optimized the conditions, various malono derivatives **3a**–**3c** and α-methylenecarbonyl compounds **4a**–**4e** were condensed with ninhydrin (**1**) and benzene-1,2-diamines **2** in the presence of $AcONH_4$ to afford the corresponding products **5b**–**5i** (*Scheme 1*). The results showed (*Table 2*) that the reactive malononitrile (**3a**) and the less reactive methyl and ethyl cyanoacetate (**3b** and **3c**, resp.), and five different α-methylenecarbonyl compounds were successfully applied in this process to afford the corresponding 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] derivatives in excellent yields.

Table 2. *One-Pot Four-Component Synthesis of Compounds* **5a**–**5i**

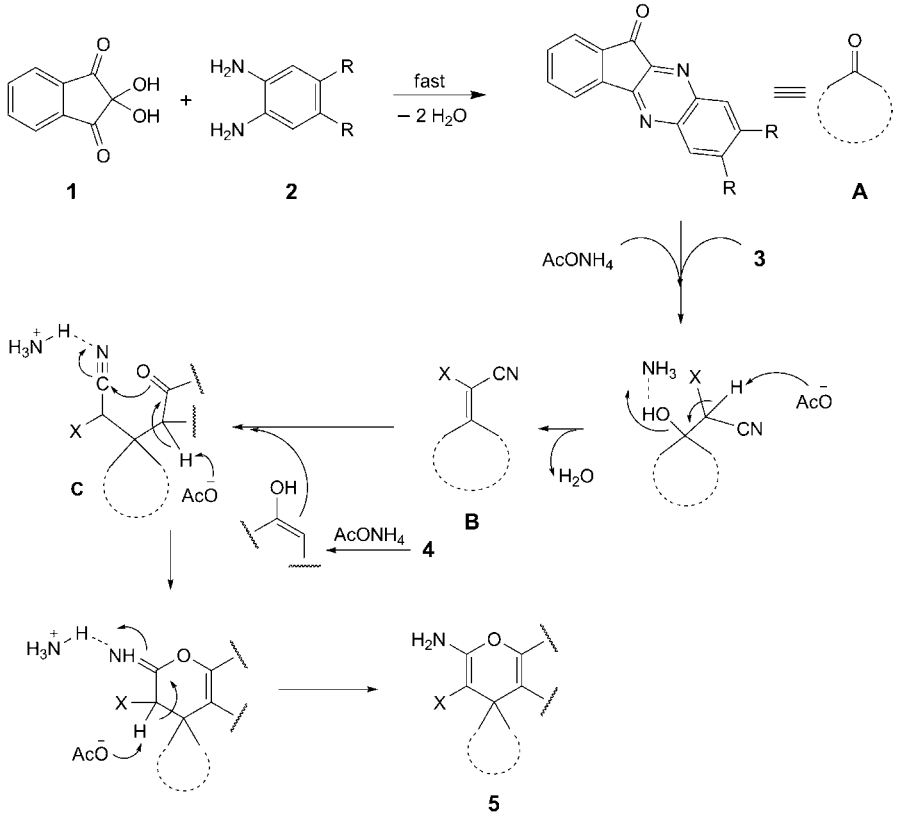
Benzene-1,2-diamine	Malono derivative	α-Methylenecarbonyl compound	Product[a])	Time [h]	Yield [%][b])
2a	**3a**	**4c**	**5a**	12	91
2b	**3c**	**4c**	**5b**	15	90
2b	**3a**	**4d**	**5c**	12	92
2a	**3a**	**4d**	**5d**	12	93
2a	**3a**	**4b**	**5e**	14	91
2a	**3a**	**4e**	**5f**	15	91
2a	**3b**	**4c**	**5g**	15	92
2a	**3c**	**4c**	**5h**	15	91
2a	**3a**	**4a**	**5i**	14	91

[a]) Reaction conditions: **1** (1 mmol), **2** (1 mmol), **3** (1 mmol), **4** (1 mmol), $AcONH_4$ (20 mol-%), and EtOH (15 ml) under reflux. [b]) Yield of isolated **5**.

The proposed mechanism for the synthesis of 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] compounds **5** in the presence of $AcONH_4$ is shown in *Scheme 2*. We envisioned that this reaction could be realized in a one-pot, two-step manner. Initially, ninhydrin (**1**) and the benzene-1,2-diamine **2** react to form the corresponding indenoquinoxalinone **A** in the presence of ammonium acetate. *Knoevenagel* condensation of **A** with **3** affords an intermediate **B**, which undergoes *Michael* addition with the enolate form of carbonyl compound **4**. The enolate O-atom of the formed intermediate **C** attacks the CN group, and subsequent H-atom shift leads to compounds **5**. $AcONH_4$ plays a dual role in this reaction. Activation of the C=O group occurs *via* H-bond formation between one H-atom of NH_4^+ and the O-atom of the C=O group. Moreover, the malono compounds **3** are activated through deprotonation by the acetate ion derived from $AcONH_4$ resulting in a strong nucleophile.

In conclusion, a highly efficient one-pot four-component method was developed for the synthesis of novel 2′-aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] derivatives in the presence of $AcONH_4$ as a neutral and inexpensive catalyst. This

Scheme 2. *Proposed Mechanism for the Synthesis of 2′-Aminospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] Derivatives* **5** *in the Presence of* $AcONH_4$



method has several advantages including high conversions, cost efficiency, and simple procedures, thus contributing to ecologically valuable chemistry.

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Experimental Part

General. All chemicals were purchased from *Merck* or *Fluka* and used without further purification. M.p.: *Electrothermal-9100* apparatus; uncorrected. ^{1}H- and ^{13}C-NMR Spectra: *Bruker 500 DRX Avance*; at 500 and 125 MHz, resp.; in (D_6)DMSO; δ in ppm rel. to Me_4Si as internal standard, J in Hz.

*One-Pot Synthesis of 2′-Aminospiro[indeno[1,2-*b*]quinoxaline-11,4′-[4*H*]pyran] Derivatives* **5***: General Procedure.* Ninhydrin (**1**; 1 mmol), benzene-1,2-diamine **2** (1 mmol) and $AcONH_4$ (0.2 mmol) were added at r.t. to EtOH (15 ml) while stirring. After *ca.* 10 min, malono derivative **3** (1 mmol) and α-methylenecarbonyl compound **4** (1 mmol) were added. The mixture was stirred under reflux for the appropriate amount of time (*Table 2*). Then, the mixture was cooled to r.t. and allowed to stand at 25° for 1 h. During this time, crystals of the product formed and were collected by filtration. To obtain pure product, the crystals were washed with EtOH and Et_2O (2×20 ml) and then dried.

*2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxospiro[4*H*-1-benzopyran-4,11′-[11*H*]indeno[1,2-*b*]quinoxaline]-3-carbonitrile* (**5a**): White powder. M.p. 280° (dec.). ^{1}H-NMR: 1.00 (*s*, 3 H); 1.03 (*s*, 3 H); 1.97–2.08 (*m*, 2 H); 2.61–2.75 (*m*, 2 H); 7.33 (*s*, 2 H); 7.51–7.55 (*m*, 2 H); 7.59–7.60 (*m*, 1 H); 7.75–7.78 (*m*, 1 H); 7.81–7.84 (*m*, 1 H); 8.05 (*dd*, *J* = 1.5, 8.2, 1 H); 8.08 (*d*, *J* = 7.5, 1 H); 8.15 (*dd*, *J* = 1.0, 8.0, 1 H). ^{13}C-NMR: 27.3; 29.4; 32.7; 34.7; 50.7; 51.4; 77.3; 109.6; 114.8; 121.6; 125.8; 124.6; 128.6; 129.0; 129.4; 129.5; 129.3; 132.2; 138.6; 141.4; 141.7; 153.8; 157.3; 160.2; 164.2; 167.8. Anal. calc. for $C_{26}H_{20}N_4O_2$: C 74.27, H 4.79, N 13.33, found: C 74.31, H 4.82, N 13.36.

*Ethyl 2-Amino-5,6,7,8-tetrahydro-7,7,7′,8′-tetramethyl-5-oxospiro[4*H*-1-benzopyran-4,11′-[11*H*]indeno[1,2-*b*]quinoxaline]-3-carboxylate* (**5b**): White powder. M.p. 287° (dec.). ^{1}H-NMR: 0.94 (*s*, 3 H); 0.98 (*s*, 3 H); 1.88 (*d*, *J* = 18.0, 1 H); 2.42 (*s*, 3 H); 2.46–2.53 (*m*, 6 H); 2.60 (*d*, *J* = 18.5, 1 H); 2.68 (*d*, *J* = 18.5, 1 H); 3.20–3.21 (*m*, 2 H); 7.35–7.46 (*m*, 3 H); 7.69 (*s*, 1 H); 7.87 (*s*, 1 H); 7.95–7.97 (*m*, 3 H). ^{13}C-NMR: 14.2; 18.1; 18.2; 26.3; 27.1; 31.3; 34.9; 44.1; 50.2; 63.1; 77.2; 109.0; 123.3; 126.1; 127.6; 127.7; 128.4; 128.9; 135.7; 137.6; 138.2; 141.4; 141.7; 153.8; 157.4; 160.2; 164.2; 167.8; 169.5; 196.6. Anal. calc. for $C_{30}H_{29}N_3O_4$: C 72.71, H 5.90, N 8.48; found: C 72.74, H 5.88, N 8.54.

*6′-Amino-3′,7,8-trimethyl-1′-phenylspiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′(1′*H*)-pyrano[2,3-*c*]pyrazole]-5′-carbonitrile* (**5c**): White powder. M.p. 248° (dec.). ^{1}H-NMR: 1.09 (*s*, 3 H); 2.42 (*s*, 3 H); 2.47 (*s*, 3 H); 7.36 (*t*, *J* = 7.2, 1 H); 7.51 (*t*, *J* = 7.7, 2 H); 7.63–7.64 (*m*, 5 H); 7.85 (*d*, *J* = 9.0, 3 H); 7.97 (*s*, 1 H); 8.14 (*t*, *J* = 3.7, 1 H). ^{13}C-NMR: 12.6; 20.5; 20.6; 48.6; 58.7; 98.3; 118.9; 120.9; 122.2; 126.8; 127.4; 128.9; 129.1; 130.3; 130.5; 133.3; 136.9; 138.2; 140.8; 141.0; 141.5; 141.8; 144.6; 146.0; 150.9; 152.8; 161.9; 163.4. Anal. calc. for $C_{30}H_{22}N_6O$: C 74.67, H 4.60, N 17.42; found: C 74.62, H 4.63, N 17.48.

*6′-Amino-3′-methyl-1′-phenylspiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′(1′*H*)-pyrano[2,3-*c*]pyrazole]-5′-carbonitrile* (**5d**): White powder. M.p. 240° (dec.). ^{1}H-NMR: 1.10 (*s*, 3 H); 7.37 (*t*, *J* = 7.5, 1 H); 7.54 (*t*, *J* = 7.7, 2 H); 7.64–7.71 (*m*, 5 H); 7.81–7.90 (*m*, 4 H); 8.12 (*d*, *J* = 8.5, 1 H); 8.20 (*t*, *J* = 8.5, 2 H). ^{13}C-NMR: 13.8; 43.6; 58.4; 116.9; 118.9; 121.8; 122.7; 125.8; 125.9; 128.8; 128.91; 128.97; 129.0; 129.6; 129.8; 129.9; 135.9; 136.7; 136.9; 139.8; 142.3; 146.1; 154.2; 159.1; 175.9. Anal. calc. for $C_{28}H_{18}N_6O$: C 74.00, H 3.99, N 18.49; found: C 74.10, H 4.09, N 18.56.

*2-Amino-5,6,7,8-tetrahydro-5-oxospiro[4*H*-1-benzopyran-4,11′-[11*H*-indeno[1,2-*b*]quinoxaline]-3-carbonitrile* (**5e**): White powder. M.p. 278° (dec.). ^{1}H-NMR: 1.89–1.94 (*m*, 2 H); 2.07–2.14 (*m*, 2 H); 2.73–2.81 (*m*, 2 H); 7.33 (*s*, 2 H); 7.52–7.61 (*m*, 2 H); 7.75–7.84 (*m*, 2 H); 8.06–8.09 (*m*, 2 H); 8.16 (*dd*, *J* = 1.0, 8.0, 1 H). ^{13}C-NMR: 20.6; 27.8; 37.4; 48.0; 59.5; 113.8; 118.4; 122.2; 125.5; 129.66; 129.69; 129.7; 129.9; 130.5; 136.9; 141.7; 142.4; 152.9; 155.0; 159.6; 166.5; 167.5; 195.9. Anal. calc. for $C_{24}H_{16}N_4O_2$: C 73.46, H 4.11, N 14.28; found: C 73.54, H 4.18, N 14.36.

*2′-Amino-5′-oxospiro[11*H*-indeno[1,2-*b*]quinoxaline-11,4′-[4*H*,5*H*]pyrano[2,3-*b*][1]benzopyran]-3′-carbonitrile* (**5f**): White powder. M.p. 297° (dec.). ^{1}H-NMR: 7.44 (*d*, *J* = 8.0, 1 H); 7.57–7.63 (*m*, 3 H); 7.74–7.81 (*m*, 5 H); 7.84–7.86 (*m*, 1 H); 8.05–8.08 (*m*, 2 H); 8.15–8.20 (*m*, 1 H); 8.19–8.21 (*dd*, *J* = 1.0, 8.5, 1 H). ^{13}C-NMR: 113.5; 117.5; 118.0; 122.5; 123.7; 125.9; 126.3; 129.81; 129.88; 130.3; 130.4; 131.0; 133.4; 134.5; 137.3; 141.8; 142.8; 151.5; 153.0; 154.8; 156.5; 159.1; 159.4; 165.4. Anal. calc. for $C_{27}H_{14}N_4O_3$: C 73.30, H 3.19, N 12.66; found: C 73.36, H 3.25, N 12.69.

*Methyl 2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxospiro[4*H*-1-benzopyran-4,11′-[11*H*]indeno[1,2-*b*]quinoxaline]-3-carboxylate* (**5g**): White powder. M.p. 269° (dec.). ^{1}H-NMR: 0.94 (*s*, 3 H); 0.99 (*s*, 3 H); 1.84–2.01 (*m*, 2 H); 2.55–2.74 (*m*, 2 H); 2.82 (*s*, 3 H); 7.38–7.48 (*m*, 3 H); 7.64–7.74 (*m*, 2 H); 7.88 (*s*, 2 H); 7.92–7.93 (*m*, 1 H); 8.02 (*d*, *J* = 7.0, 1 H); 8.09–8.11 (*m*, 1 H). ^{13}C-NMR: 13.7; 19.4; 27.5; 28.7; 32.6; 47.9; 50.7; 51.2; 56.9; 59.2; 78.0; 114.8; 121.6; 124.6; 128.6; 129.0; 129.45; 129.49; 132.2; 138.6; 141.4; 141.7; 153.8; 157.3; 160.1; 164.2; 167.8; 169.4; 195.7. Anal. calc. for $C_{27}H_{23}N_3O_4$: C 71.51, H 5.11, N 9.27; found: C 71.54, H 5.19, N 9.36.

*Ethyl 2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxospiro[4*H*-1-benzopyran-4,11′-[11*H*]indeno[1,2-*b*]quinoxaline]-3-carboxylate* (**5h**): White powder. M.p. 272° (dec.). ^{1}H-NMR: 0.99 (*s*, 3 H); 1.04 (*s*, 3 H); 1.08 (*t*, *J* = 7.0 H, 3 H); 1.85–2.01 (*m*, 2 H); 2.56–2.74 (*m*, 2 H); 3.32–3.45 (*m*, 2 H); 7.39 (*d*, *J* = 7.5, 1 H); 7.44 (*t*, *J* = 7.2, 1 H); 7.49 (*t*, *J* = 7.2, 1 H); 7.65–7.68 (*m*, 1 H); 7.71–7.74 (*m*, 1 H); 7.93 (*d*, *J* = 8.0, 1 H); 7.98 (*s*, 2 H); 8.02 (*d*, *J* = 7.0, 1 H); 8.10 (*d*, *J* = 8.5, 1 H). ^{13}C-NMR: 13.7; 19.4; 27.5; 28.7; 32.5; 47.8; 51.3; 56.8; 9.2; 77.8; 114.9; 121.5; 124.7; 128.6; 129.0; 129.4; 132.2; 138.8; 141.3; 141.8; 169.4; 195.7. Anal. calc. for $C_{28}H_{25}N_3O_4$: C 71.93, H 5.39, N 8.99; found: C 71.99, H 5.42, N 9.05.

*2-Amino-6,7-dihydro-7′,8′-dimethyl-5-oxospiro[cyclopenta[*b*]pyran-4(5*H*),11′-[11*H*]indeno[1,2-b]quinoxaline]-3-carbonitrile* (**5i**): White powder. M.p. 274° (dec.). ^{1}H-NMR: 2.31 – 2.32 (*m*, 2 H); 2.90 – 2.91 (*m*, 2 H); 7.59 – 7.65 (*m*, 5 H); 7.79 – 7.82 (*m*, 1 H); 7.85 – 7.88 (*m*, 1 H); 8.11 – 8.13 (*m*, 2 H); 8.19 (*d*, *J* = 8.0, 1 H). ^{13}C-NMR: 25.8; 34.1; 47.5; 58.4; 116.9; 118.5; 122.4; 126.3; 129.8; 129.9; 130.3; 131.1; 133.4; 137.0; 141.9; 142.8; 150.8; 154.5; 161.7; 164.1; 178.8; 200.7. Anal. calc. for $C_{25}H_{18}N_4O_2$: C 73.88, H 4.46, N 13.78; found: C 73.93, H 4.40, N 13.81.

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