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Effect of Microwave Irradiation on the Condensation of 6-Substituted 3-Formylchromones with Some Five-membered Heterocyclic Compounds

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Abstract: Different types of 3-substituted 4*H*-4-oxobenzopyrans were prepared by microwave irradiation as well as by a classical method. The beneficial effect of microwave irradiation on the aldol condensation of 3-formylchromones with 2-imino-1-methylimidazolidine-4-one (creatinine), 2-thioxoimidazolidine-4-one (thiohydantoin) and 2-ethyl-2-thioxothiazolidin-4-one (3-ethylrhodanine) in different reaction media is described. Our results show that the effect of microwave irradiation on the reactions studied was a shortening of the reaction times and a smooth increase in the yields. The subsequent reactions of the product with some nucleophiles are discussed. The structure of the products was proven by elemental analysis, IR and NMR spectra.

Keywords: Creatinine, thiohydantoin, rhodanine, carbamoic acids, guanidine derivatives, Diels - Alder reaction, IR, ^{1}H NMR, ^{13}C NMR.

Introduction

This study is a continuation of our earlier publications [1-7], in which we described the theoretical, spectral and biological properties of newly synthesized chromone and chromanone derivatives. The

aim of this work was the preparation of some new five-membered nitrogen heterocyclic derivatives of chromone as potentially useful intermediates for synthesis.

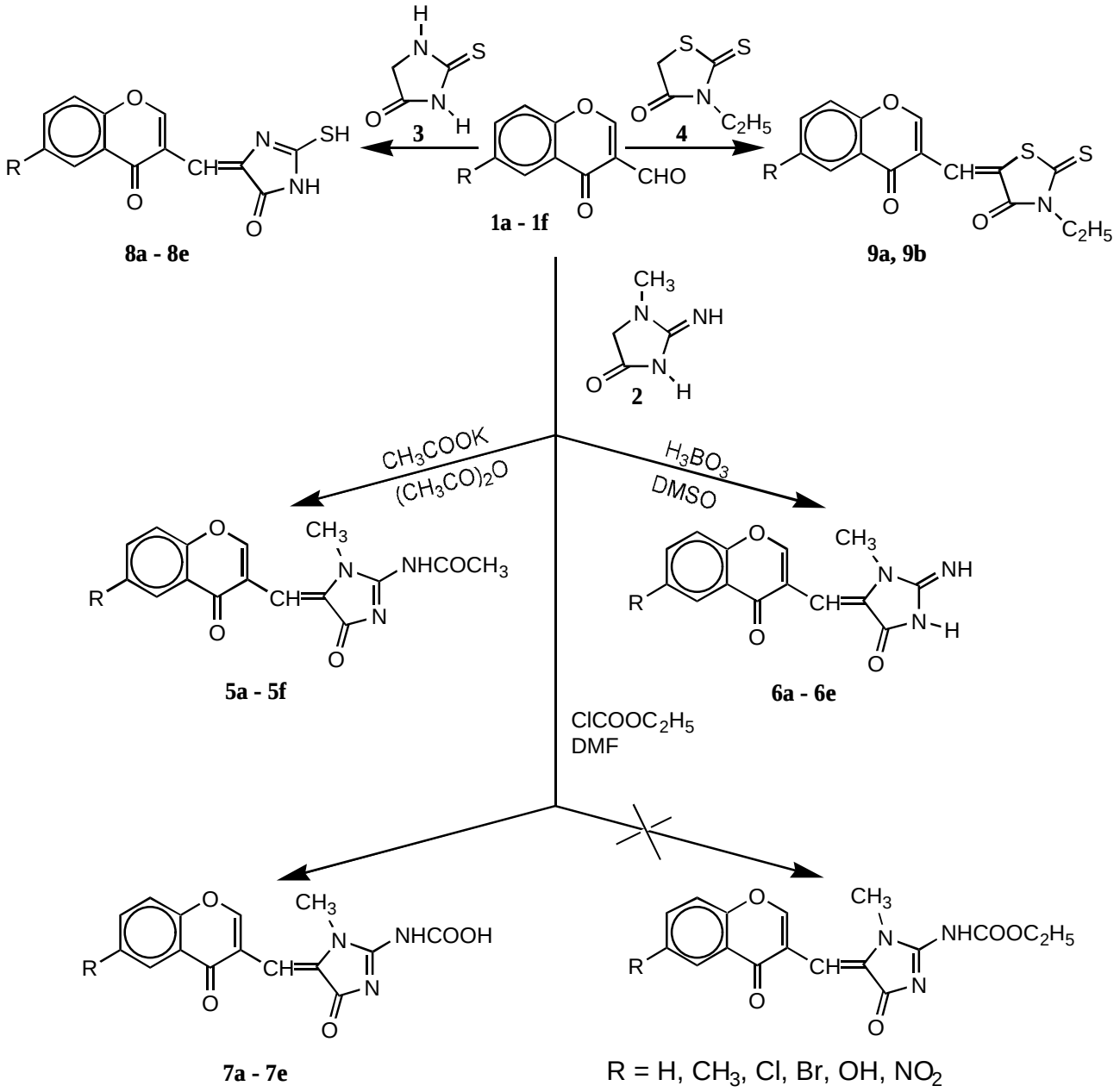
3-Formylchromones **1** were chosen as being synthetically versatile molecules with a reactive carbonyl group. They have considerable significance for their biological activities [8-10] and for their reactivity towards nucleophiles which allows the synthesis of a wide variety of heterocycles. A study of the influence of microwave irradiation on the condensation reactions was the next goal of this paper. Five-membered ring heterocycles **2-4** are known as precursors of α-amino acids and they can be condensed easily with aldehydes. It is known that condensations of creatinine with aldehydes [11, 12] as well as the Gränacher synthesis [13] with rhodanine under classical conditions take several hours at high temperatures (160–180°C).

As we have shown before [14], microwave irradiation is a suitable method for shortening the duration of condensation reactions. Condensations of creatinine [15] and thiohydantoin [16] with aromatic aldehydes without a solvent under microwave irradiation and giving good yields of products were described by *Villemin and al*. The starting 3-formylchromones used in this work are accessible via Vilsmeier-double formylation of appropriate o-hydroxyacetophenones [17]. Commercially available nitrogen heterocycles were used for the reactions. The reactions are outlined in Schemes 1 and 2. Details of the experimental results are listed in Table 1.

Results and Discussion

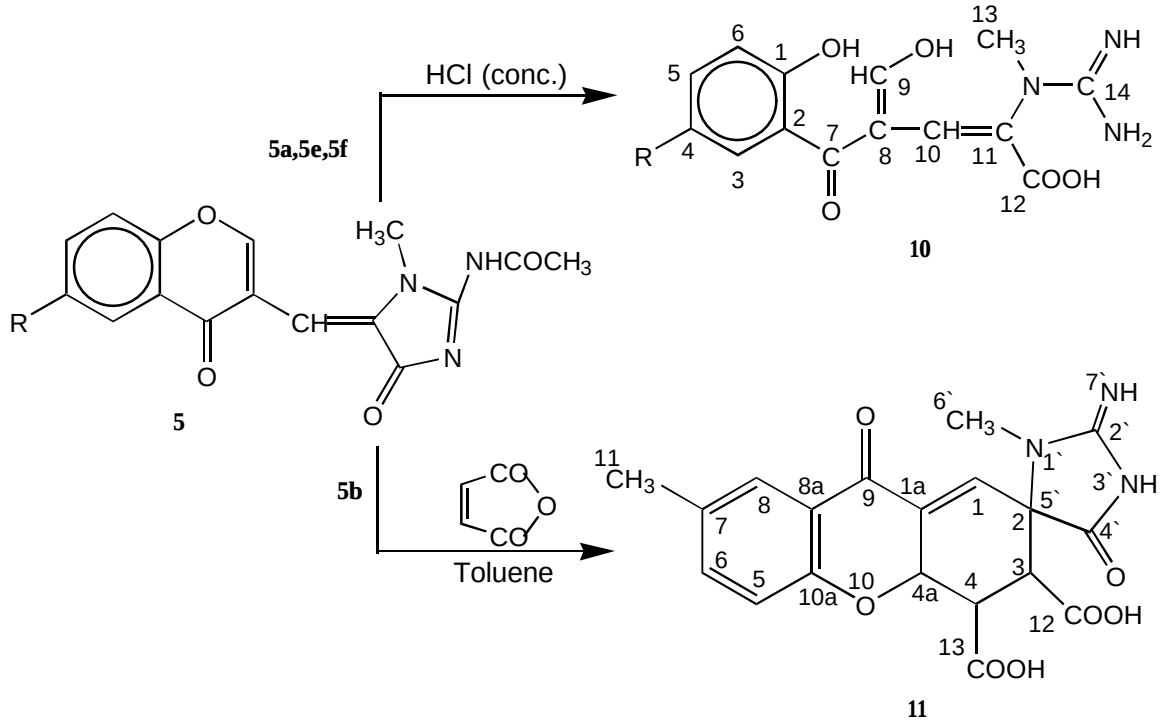
2-Acetamido-1-methyl-5-[(6-R-4-oxo-4*H*-benzopyran-3-yl)methylidene]-4,5-dihydroimidazol-4-ones **5a**-**5f** were obtained by condensations of **1a**-**1f** with creatinine **2** in acetic anhydride, both under microwave irradiation (*A*) and classical (*B*) conditions. Although the yields by both methods were almost the same (46-84%), the reactions in a microwave oven were considerably faster (Table 1). Imino derivatives **6a**-**6e** (2-imino-1-methyl-5-[(6-R-4-oxo-4*H*-[1]-benzopyran-3-yl) methylidene]- imidazolidin-4-ones) were obtained using dimethylsulfoxide as a solvent and boric acid as a catalyst by both methods *A* and *B*.

A convenient synthesis of carbamoic acid derivatives **7** (1-methyl-4-oxo-5-[(6-R-4-oxo-4H-[1]-benzopyran-3-yl)methylidene]-4,5-dihydroimidazol-2-carbamoic acids) was accomplished by reaction of creatinine with ethylchloroformate in N,N-dimethylformamide followed by the subsequent addition of aldehydes **1a**-**1e**. The hydrolyzed products **7a**-**7e** were obtained in 69-71% yields by both the classical and microwave irradiation condensation methods, even under anhydrous conditions. It is evident that compounds **7** resulted from the utilization of the water of condensation for the hydrolysis process. In the ^{1}H NMR spectra of compounds **7** no signals for the ethyloxy group were observed (Table 2).



Scheme 1.

3-Formylchromone condensations with thiohydantoin **3** and 3-ethylrhodanine **4** were carried out in acetic anhydride in the presence of potassium acetate under both irradiation and classical conditions. The yields of 2-thioxo-5-[(6-R-4-oxo-4*H*-[1]-benzopyran-3-yl) methylidene]imidazolidin-4-ones **8a**-**8e** and 2-thioxo-5-[(6-R-4-oxo-4*H*-benzopyran-3-yl)methylidene]thiazolidin-4-ones, **9a** and **9b**, respectively, were comparable by both methods.



Scheme 2.

Attempts to hydrolyze the condensation products with diluted mineral acids to prepare ring opened heterocycles were unsuccessful. The 5-(2-hydroxyphenyl)-4-(hydroxy-methylidene)-2-(1-methyl-guanidino)-2-pentenoic acid hydrolysis products (**10**) were obtained only by refluxing compounds **5** in concentrated hydrochloric acid. We propose the structure of compounds **10**, which contain guanidinyl, carboxyl and enolic groups on the basis of ^{1}H NMR, ^{13}C NMR spectra and elemental analysis. Compounds **10** could be regarded as a mixtures of isomers with a very fast tautomeric equilibrium of both enolic and oxo groups. The assumption of this fast tautomeric equilibrium is supported by the data for the shift signals of C-9 in the ^{13}C NMR spectra.

We next attempted to carry out Diels - Alder reactions with various dienophiles, using e.g. maleic anhydride, diethyl maleate and tetracyanoethylene, using both the classical and microwave irradiation procedures. Only heating and stirring a mixture of **5b** in toluene with an excess of maleic anhydride at 40^{o}C over 15 hr. was successful and the spiroheterocyclic adduct spiro[(1´-methyl-2´-imido-4´-oxo)-1´, 3´-diazolane-5´, 2-(7-methyl-9-oxo-2, 3, 4, 4a tetrahydroxantene)]-3, 4-dicarboxylic acid (**11**) was formed in 64% yield. The proposed structure was confirmed by ^{13}C NMR. Similar Diels - Alder reactions were reported previously [18]. The use of microwave irradiation for the Diels - Alder experiments was unsuccessful.

All condensation products are stable solid compounds, rather insoluble in common solvents, with high melting points. Because of their poor solubility in DMSO we had to measure their ^{1}H NMR spectra at elevated temperatures (Table 2). The resonance signals and their multiplicity confirmed the pro-

posed structures. The infrared spectra of the prepared compounds **5-9** showed strong absorption bands of the C=O stretching vibrations in two very well distinguished regions 1645 - 1668 cm^{-1} and 1688 - 1745 cm^{-1} (Table 3). The absorption bands in the lower region of the spectra belong to the ν(C=O) of the γ-pyrone ring. The higher region was attributed to the azole heterocyclic part of the prepared compounds. Compounds **10** lacked the ν(C=O) band at 1640 - 1660 cm^{-1}. Strong bands around 1740 cm^{-1} confirmed the presence of unsaturated aldehyde groups (Table 4).

Experimental

General

Products were characterized by elemental analyses (Table 1), NMR spectra (Table 2) and IR spectra (Tables 3-4). The melting points were determined on a Kofler block and are uncorrected. Infrared spectra of nujol suspensions were recorded in 400 - 4000 cm^{-1} region on a Specord IR 75 spectrometer (Zeiss Jena). ^{1}H and ^{13}C NMR spectra were measured on a 300 MHz spectrometer VARIAN GEMINI 200 in deuterated DMSO at 50-80^{O}C. All microwave assisted reactions were carried out in a Lavis - 1000 multi Quant microwave oven. The apparatus was adapted for laboratory applications with magnetic stirring and an external reflux condenser.

Synthesis of **5a-5f** *,* **8a-8e** *and* **9a**, **9b**

Method A

A mixture of 6-R-3-formylchromones **1a-1f** (2.87 mmol), creatinine **2** (or thiohydantoin **3** or 3-ethylrhodanine **4**) (2.87 mmol) in dry acetic anhydride (2 cm^{3}) in the presence of freshly fused potassium acetate was stirred and irradiated in a microwave oven for the time given in Table 1. The solid was filtered off. The products were recrystallized from dioxane or toluene.

Method B

A mixture of the same composition as in method *A* was heated at 110-120°C for the time given in Table 1. Isolation of products was accomplished as described in method *A.*

Synthesis of **6a**–**6e**

Method *A*

A mixture of 6-R-3-formylchromone **1** (2.87 mmol), creatinine **2** (2.87 mmol), a catalytic amount of H_3BO_3 (20 mg) in 1cm^{3} of dry dimethyl sulfoxide was stirred and irradiated at 270 W in a microwave oven. The solid product was filtered off and recrystallized from dioxane or toluene.

Method *B*

A mixture of the same composition as in method *A* was heated in 1cm^3 of dry dimethyl sulfoxide at 120°C over 3 h. The solid product was filtered off and recrystallized from dioxane.

Synthesis of **7a**, **7b**

Method *A*

Creatinine **2 (**2.87 mmol) was dissolved in 1 cm^3 of dry dimethylformamide and then ethyl chloroformate was added to the solution. The mixture was stirred and irradiated in a microwave oven at 270 W. The solid product was filtered off and recrystallized from dioxane or toluene.

Method *B*

The mixture of creatinine **2** (2.87 mmol) and ethyl chloroformate (3.0 mmol) in dry dimethylformamide (1 cm^3) was stirred at room temperature for 3h and then 6-R-3-formylchromone **1** (2.87 mmol) was added to the mixture and heated at 90°C for 6 h. The isolation of products was the same as described above.

Acid hydrolysis of compounds **5a**, **5e** *and* **5f**

The solution of 0.3 g (1 mmol) creatinine derivative **5a** (or **5e**, **5f**) in 10 ml of concentrated hydrochloride acid was heated at 90-100°C for 4 h. After cooling the resulting white crystals were filtered off, washed with cold water and recrystallized from dioxane. Thus prepared were compounds **10a**, **10e** and **10f**

^{13}C NMR spectral data for compound **10e** [δ(ppm); DMSO - d_6, 300 MHz]

C-1	**C-2**	**C-3**	**C-4**	**C-5**	**C-6**	**C-7**
155.45	123.77	123.65	154.87	107.95	120.11	174.53
C-8	**C-9**	**C-10**	**C-11**	**C-12**	**C-13**	**C-14**
114.78	157.60	109.79	130.04	163.32	28.69	149.23

Diels - Alder reaction of compound **5b** *with maleic anhydride (***11***)*

The mixture of 1g (2.51 mmol) of compound **5b** and 0.52 g (5.02 mmol) of maleic anhydride in toluene (30 cm^3) was heated at 40°C for 15 h. The solid adduct after cooling was filtered off, washed with 10 cm^3 of toluene and dried. The product was suspended in 30 cm^3 water was then stirred at 40°C for 3 h. The solid acid was removed by suction and recrystallized from ethanol. Yield 64%.

^{13}C NMR spectral data for compound **11** [δ(ppm); DMSO - d_6, 300 MHz]

C-1	**C-1a**	**C-2**	**C-3**	**C-4**	**C-4a**	**C-5**
131.1	131.6	107.8	39.2	39.2	135.8	116.0
C-6	**C-7**	**C-8**	**C-8a**	**C-9**	**C-10a**	**C-11**
135.7	130.9	124.6	153.8	190.0	118.5	20.5
C-12	**C-13**	**C-2'**	**C-4'**	**C-6'**		
166.8	166.8	157.6	174.8	28.4		

Analytical Data

Table 1. Characterization of the prepared compounds.

Comp.	R	Formula M_r	m.p. °C	w_i (calcd)/ % w_I (found)/ %			Yield %	t_r min
				C	H	N		
5a	H	$C_{16}H_{13}N_3O_4$	246 - 248	61.73	4.21	13.50	75	3
		311.29		61.31	4.14	13.41	71	60
5b	CH_3	$C_{17}H_{15}N_3O_4$	277 - 279	62.76	4.65	12.92	60	4
		325.32		62.15	4.66	12.59	57	120
5c	Cl	$C_{16}H_{12}ClN_3O_4$	268 - 270	55.58	3.50	12.15	76	2
		345.74		55.58	3.59	12.13	72	60
5d	Br	$C_{16}H_{12}BrN_3O_4$	270 - 272	49.25	3.10	10.77	84	1
		390.19		48.91	3.02	10.54	-	-
5e	AcO	$C_{18}H_{15}N_3O_6$	259 - 262	58.54	4.09	11.38	46	3
		369.33		58.06	4.07	11.28	40	90
5f	NO_2	$C_{16}H_{12}N_4O_6$	285 - 286	53.94	3.39	15.72	84	3
		356.29		53.83	3.29	15.43	-	-
6a	H	$C_{14}H_{11}N_3O_3$	250 - 252	62.45	4.12	15.61	70	3
		269.26		62.38	3.96	15.72		
6b	CH_3	$C_{15}H_{13}N_3O_3$	294 - 297	63.60	4.63	14.83	67	3
		283.28		63.24	4.68	14.06		
6c	Cl	$C_{14}H_{10}ClN_3O_3$	356 - 360	55.36	3.32	13.83	92	3
		303.7		55.21	3.49	13.22		
6d	Br	$C_{14}H_{10}BrN_3O_3$	276 - 278	48.30	2.89	12.07	68	3
		348.16		48.25	2.96	12.44		
6e	NO_2	$C_{14}H_{10}N_4O_5$	237 - 240	53.51	3.21	17.83	98	3
		314.26		52.94	2.99	17.39		

Continuation of the Table 1.

Comp.	R	Formula M_r	m.p. °C	w_i (calcd)/ % w_I (found)/ %			Yield %	t_r min
				C	H	N		
7a	H	$C_{15}H_{11}N_3O_5$	253 - 255	57.82	3.51	13.41	69	6
		313.3		57.50	3.90	13.08		
7b	Cl	$C_{15}H_{10}ClN_3O_5$	356 - 360	54.34	3.76	11.18	71	4
		375.76		54.69	3.23	11.23		
7c	CH_3	$C_{16}H_{13}N_3O_5$	299 - 301	58.71	3.97	12.84	58	7
		327.3		58.44	4.30	12.75		
7d	Br	$C_{15}H_{10}BrN_3O_5$	350	45.92	2.55	10.71	60	7
		392.2		46.28	2.36	10.48		
8a	H	$C_{13}H_8N_2O_3S$	292 - 295	57.35	2.96	10.29	79	8
		272.3		56.98	2.91	10.05	72	60
8b	CH_3	$C_{14}H_{10}N_2O_3S$	315 - 317	58.73	3.52	9.78	70	6
		286.3		58.50	3.52	9.04	66	30
8c	Cl	$C_{13}H_7ClN_2O_3S$	319 - 321	50.91	2.30	9.13	74	4
		306.7		51.03	2.34	9.05	71	30
8d	Br	$C_{13}H_7BrN_2O_3S$	329-331	44.46	2.01	7.98	96	9
		351.2		44.53	2.02	7.16	88	60
8e	AcO	$C_{15}H_{10}N_2O_5S$	303 - 305	54.54	3.05	8.48	62	10
		330.3		53.82	2.98	7.99	59	120
9a	H	$C_{15}H_{11}NO_3S_2$	215 - 217	56.77	3.49	4.41	70	5
		317.4		57.07	3.48	4.45	74	60
9b	Cl	$C_{15}H_{10}ClNO_3S_2$	231 - 233	51.21	2.86	3.98	67	5
		351.8		50.96	2.82	3.79	65	60
10a	H	$C_{14}H_{15}N_5O_5$	325 - 327	55.08	4.95	13.76	0	20
		305.3	decomp.	55.35	4.59	13.65	52	240
10e	OH	$C_{14}H_{15}N_3O_6$	315 - 317	52.33	4.70	13.08	0	20
		321.3	decomp.	52.12	4.59	12.72	47	260
10f	NO_2	$C_{14}H_{14}N_4O_7$	>360	48.00	4.02	15.98	0	20
		350.3	decomp.	48.09	3.855	15.77	56	250
11	CH_3	$C_{19}H_{17}N_3O_7$	221 - 223	57.16	4.25	10.51	0	20 - 30
		399.3		57.10	4.22	10.41	89	15 hr.

[a]The upper yield and reaction time (t_r) data are given for the condensation in microwave oven, the lower data for the classic condensation.

Table 2. ^{1}H NMR spectra data of prepared compounds.

Compound	Solvent	^{1}H NMR spectrum δ (ppm)
5a	$CDCl_3$	2.25 (s, 3H, CH_3); 3.39 (s, 3H, CH_3-N); 6.86 - 8.32 (m, 5H, H-Ar); 9.68 (s, 1H, H-2); 10.84 (s, 1H, NH).
5b	DMSO-d_6	2.50 (s, 3H, CH_3); 2.54(s, 3H, CH_3); 3.57(s, 3H, CH_3-N); 6.56 (s, 1H, H-9); 7.60 - 7.70 (m, 5H, H-Ar); 7.97 (s, 1H, H-5); 9.56 (s, 1H, H-2).
5c	DMSO-d_6	2.76 (s, 3H, CH_3); 3.52 (s, 3H, CH_3-N); 6.72 (s, 1H, H-9); 8.02 (d, 1H, H-8, ^{3}J=9Hz); 8.11 (d, 1H, H-7, ^{3}J=9Hz); 8.32 (d, 1H, H-5, ^{4}J=2Hz); 9.54 (s, 1H, H-2).
5d	DMSO-d_6	2.78 (s, 3H, CH_3); 3.53 (s, 3H, CH_3-N); 6.74 (s, 1H, H-9); 7.95 (d, 1H, H-8, ^{3}J=9Hz); 8.24 (d, 1H. H-7, ^{3}J=9Hz); 8.49 (d, 1H, H-5, ^{4}J=1.8Hz), 9.55 (s, 1H, H-2).
5e	DMSO-d_6	2.13 (s, 3H, CH_3O); 2.3 (s, 3H, CH_3O); 3.45 (s, 3H, CH_3-N); 6.47 (s, 1H, H-9); 7.6 - 7.85 (m, 3H, H-8, 7, 5); 9.30 (s, 1H, H-2); 11.41 (s (broad), 1H, N-H).
5f	DMSO-d_6	2.13 (s, 3H, CH_3O); 3.21 (s, 3H, CH_3-N); 6.43 (s, 1H, H-9); 8.00(s, 1H, H-8); 8.64 (s, 1H, H-7); 8.85 (s, 1H, H-7); 8.85 (s, 1H, H-5); 9.353 (s, 1H, H-2).
6a	DMSO-d_6	3.35 (s, 3H, CH_3-N); 6.24 (s, 1H, H-9); 7.52 (dd, 1H, H- 7,^{3}J=7.8Hz); 7.69 (dd, 1H, H-8, ^{3}J=7.8Hz); 7.81 (dd, 1H, H-6, ^{3}J=7.8Hz); 8.12 (dd, 1H, H-5, ^{3}J=7.8Hz); 9.88 (s, 1H, H-2); 7.4 - 8.4 (broad, 1H, NH).
6b	DMSO-d_6	2.45 (s, 3H, CH_3); 3.38 (s, 3H, CH_3N); 6.25 (s, 1H, H-9); 7.60 - 7.95 (m, 3H, H-8, 7, 5); 9.86 (s, 1H, H-2); 7.4 - 8.4 (broad, 1H, NH).
7a	DMSO-d_6	3.39 (s, 3H, CH_3CN); 6.19 (s, 1H, H-9); 7.53 (dd, 1H, H-6, ^{3}J=7Hz); 7.69 (d, 1H, H-8, ^{3}J=7Hz); 7.84 (dd, 1H, H-7, ^{3}J=7Hz); 8.12 (d, 1H, H-5, ^{3}J=7Hz); 9.8 (s, 1H, H-2).
7d	DMSO-d_6	3.39 (s, 3H, CH_3CN); 6.19 (s, 1H, H-9); 7.70 (d, 1H, H-8, ^{3}J=8Hz); 7.97 (dd, 1H, H-7, ^{3}J=8Hz, ^{4}J=2.2Hz); 8.17 (d, 1H, H-5, ^{4}J=2.2Hz); 9.895 (s, 1H, H-2).
7e	DMSO-d_6	3.44 (s, 3H, CH_3-N); 6.59 (s, 1H, H-9); 8.00 (d, 1H, H-8, ^{3}J=9Hz); 8.61 (dd, 1H, H-7, ^{3}J=9Hz, ^{4}J=2.5Hz); 8.79 (d, 1H, H-5, ^{4}J=2.54Hz); 9.395 (s, 1H, H-2); 9.64 (s(broad), 1H, NH).

Continuation of the Table 2.

Compound	Solvent	^{1}H NMR spectrum δ (ppm)
8c	DMSO-d_6	6.36 (s, 1H, H - 9); 7.78 (d, 1H, H-8, ^{3}J=10Hz); 7.87 (dd, 1H, H - 7, ^{3}J=10Hz, ^{4}J=2.2Hz); 8.23 (d, 1H, ^{4}J=2.2Hz); 8.94 (s, 1H, H-5); 11.65 (s, 1H, NH); 12.46 (s, 1H, OH).
8d	DMSO-d_6	6.35 (s, 1H, H - 9); 7.74 (d, 1H, H-8, ^{3}J=8.8Hz); 8.02 (dd, 1H, H - 7, ^{3}J=8.8Hz, ^{4}J=2.2Hz); 8.22 (d, 1H, ^{4}J=2.2Hz); 8.92 (s, 1H, H-2);
8e	DMSO-d_6	2.33 (s, 3H, CH_3); 6.38 (s, 1H, H - 9); 7.60 (dd, 1H, H-7, ^{3}J=9Hz, ^{4}J=1.9Hz); 7.83 (d, 1H, H-8, ^{3}J=9Hz); 7.88 (d, 1H, H-5, ^{4}J=1.9Hz); 8.94 (s, 1H, H-2); 11.80 (s, 1H, NH); 12.45 (s, 1H, OH).
9b	DMSO-d_6	2.490 (t, 3H, CH_3); 5.346 (q, 2H, CH_2); 8.84 (s, 1h, H-2); 9.07 (d, 1H, H-8, ^{3}J=9.1Hz); 9.072 (dd, 1H, H-7, ^{3}J=9.1Hz, ^{4}J=2Hz); 9.36 (d, 1H, H-5, ^{4}J=2Hz); 10.27 (s, 1H, H-9).
10a	DMSO-d_6	6.27 (s, 1H, CH); 7.23 - 7.66 (m, H, Ar - H); 9.25 (s, 1H, CH); 10.26 (b., 6H, NH_2, NH, OH).
10e	DMSO-d_6	3.38 (s, 3H, CH_3); 6.70 (s, 1H, CH); 7.29 - 7.39 (dd, 1H, H-5, J=9Hz); 7.39 - 7.41 (d, 1H, H-3, J=3Hz); 7.57 - 7.60 (d, 1H, H-6, J=9Hz); 9.23 (s, 1H, CH); 10.28 (b., 7H, OH, NH, NH_2).
10f	DMSO-d_6	3.68 (s, 3H, CH_3); 7.95 - 8.81 (m, 3H, Ar-H); 9.32 (s, 1H, CH); 10.00 (b., 6H, OH, NH, NH_2).
11	DMSO-d_6	2.46 (s, 3H, CH_3); 2.48 (s, 3H, CH_3); 4.71 (broad, 5H, NH amide, CO_2H, CH); 6.23 - 6.56 (m, 4H, Ar-H, H-2); 7.64 (s, 1H, =CH); 9.47 (s, 1H, NH-imine).

Table 3. IR spectral data of synthesized compounds **5-9**.

	n (cm^{-1})			
Comp.	ν(NH)	ν(C=O)$_{heterocycl.}$	ν(C=O)$_{pyrone}$	ν(C=O)$_{other}$
5a	3078 - 3162	1739	1650	1643
5b	3070 - 3175	1740	1650	1642
5c	3075 - 3179	1735	1659	1636
5d	3075 - 3180	1729	1658	1640

Continuation of the Table 3.

	n (cm^{-1})			
Comp.	ν(NH)	$\nu(C=O)_{heterocycl.}$	$\nu(C=O)_{pyrone}$	$\nu(C=O)_{other}$
5e	3081 - 3181	1739	1652	1630, 1758
5f	3080 - 3177	1745	1660	1641
6a	3070 - 3185	1720	1650	
6b	3095 - 3180	1720	1645	
6c	3070 - 3175	1735	1655	
6d	3090 - 3180	1721	1655	
6e	3094 - 3186	1719	1658	
7a	3080 - 3230	1719	1657	1740
7b	3075 - 3225	1721	1658	1742
8a	3038 - 3180	1740	1665	
8b	3086 - 3185	1739	1668	
8c	3070 - 3190	1742	1665	
8d	3060 - 3180	1736	1668	
8e	3078 - 3185	1745	1663	1750
9a	3069 - 3110	1688	1660	
9b	3060 - 3110	1688	1662	

Table 4. IR spectral data of synthesized compounds **10** and **11**.

	ν (cm^{-1})				
Comp.	ν(OH)	ν(NH)	ν(C=O)	$\nu(C=O)_{acid}$	ν(C=C)
10a	3480-3400	3080-3040	1740	1700	1646
10e	3540-3440	3080-3020	1748	1700	1642
10f	3560-3480	3110-3010	1723	1700	1660
11	3460	3190-3120	1647[a]	1720-1722	1620
	-	-	1708[b]	-	-

[a]$\nu(CO)_{pyr}$, [b]$\nu(CO)_{het}$

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