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$Y(NO_3)_3.6H_2O$ catalyzed Four-Component Reaction for the Synthesis of Highly Functionalized Pyrano[2,3-c]pyrazoles in Aqueous Medium

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Abstract

An efficient one-pot four-component protocol for the synthesis of pyrano[2,3-c]pyrazole derivatives has been demonstrated using $Y(NO_3)_3.6H_2O$ as catalyst under mild condition. This is a general synthetic protocol which could be applicable to a wide range of carbonyl compounds including aromatic aldehydes, isatins and acenaphthenequinone. All The reactions proceeded smoothly, high-yielding and purity via an easy work-up procedure.

Keywords: Pyranopyrazoles, Spiro[indoline-3,4-pyrano[2,3-c]pyrazole], Spiro[acenaphthylene-1,4-pyrano[2,3-c]pyrazole]

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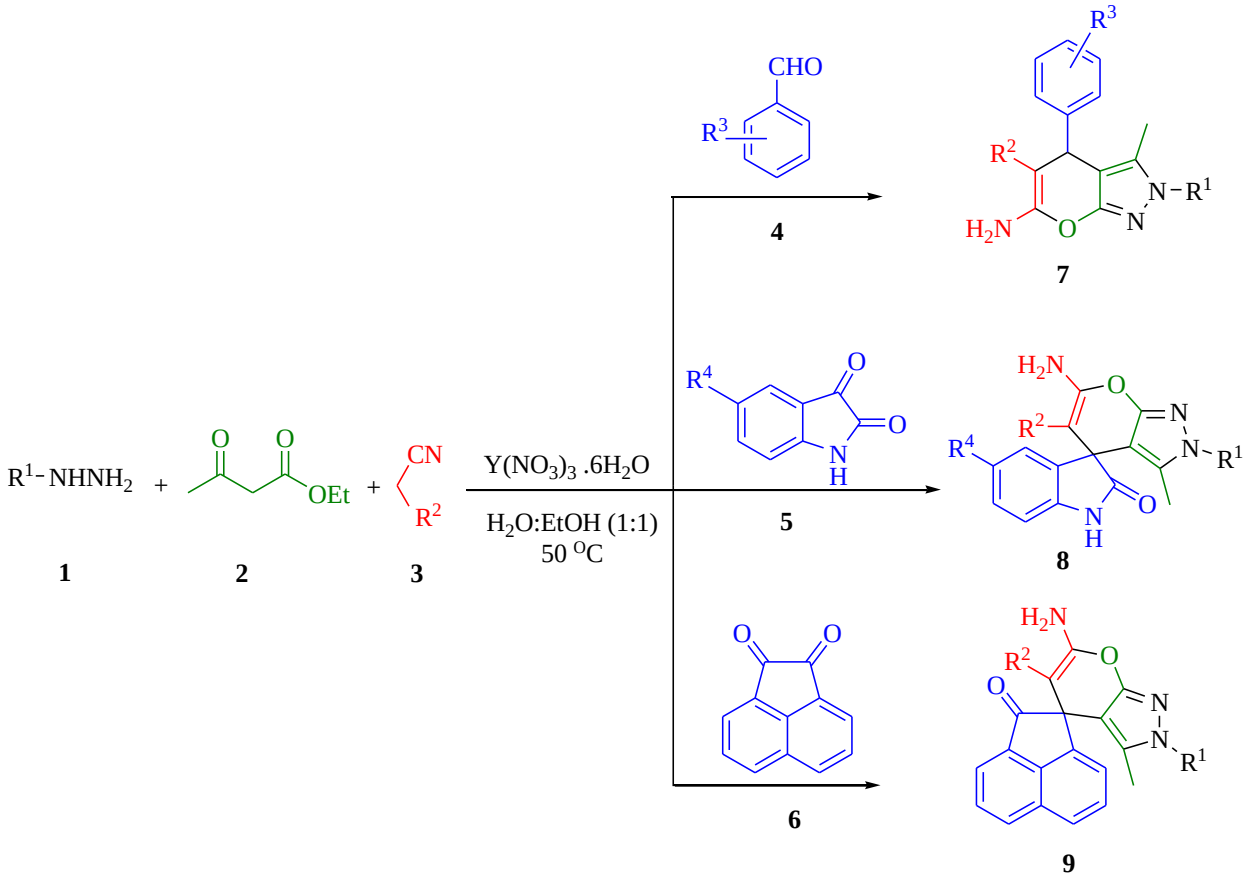
Introduction

Multi-component reactions (MCRs) are one of the best tools for modern organic synthesis because they can use most of the constituent atoms of several reactant molecules to form a product molecule [1]. Their leadsto interesting heterocyclic scaffolds are particularly useful for the creation of diverse chemical libraries of ‘‘druglike’’ molecules for biological screening, as the combination of three or more small molecular weight building blocks in a single operation leads to a high combinatorial efficacy [2, 3]. Subsequent MCRs include Biginelli [4], Mannich [5], Robinson [6] and Passerini [7] reactions which have been widely used in synthetic, medicinal, and combinatorial chemistry [8, 9]. Pyranopyrazoles are an important class of heterocyclic compounds. Theirs derivatives have showed a wide variety of biological activities, such as counting anti HIV [10], anticancer [11], anti-inflammatory [12], antifungal [13], insecticidal [14], analgesic [15] and inhibit human Chk1 kinase [16].



Scheme 1. Synthesis of dihydropyrano[2,3-c]pyrazole, spiro[indoline-3,4-pyrano[2,3-c]pyrazole] and spiro[acenaphthylene-1,4-pyrano[2,3-c]pyrazole].

Several MCRs have been used in various syntheses of spiro[indoline-3,4-pyrano[2,3-c]pyrazole] derivatives in the presence of trimethylamine [17], piperidine [18], L-proline [19], per-6-amino-β-cyclodextrin [20], hexadecyl dimethyl benzyl ammonium chloride [21], basic ionic liquids [22], disulfonic acid imidazolium chloroaluminate [23], meglumine [24] and sodium benzoate [25]. However, most of these methods are associated with limitations such as low yields, high temperature, long reaction time and special conditions (microwave and ultrasonic irradiation). In most cases, the substrate of carbonyl compounds limited to aromatic aldehydes. In continuation of our work on MCRs [26-30], herein, we explored the development of a general protocol leading to the pyranopyrazole derivatives by four-component reactions of β-ketoester, hydrazines and malononitrile with different carbonyl compounds is highly desirable (Scheme 1).

Experimental

Material and methods

Melting points and IR spectra of all compounds were determined using an Electro thermal 9100 apparatus and FT-IR-JASCO-460 plus spectrometer. The ^{1}H NMR spectra of compounds were recorded on a Bruker DRX-300 Avance instrument in DMSO at 300 MHz. We purchased all chemicals from chemical producer Merck (Darmastadt, Germany) and Fluka (Buchs, Switzerland) and they were used without further purification.

*General procedure for preparation of **7a-u**, **8a-e**, **9a-c**.*

An equimolar amounts of hydrazines (1.0 mmol), ethyl acetoacetate (1.0 mmol), alkylcyanide (1.0 mmol) and arylaldehyde/isatin/acenaphthenequinone (1.0 mmol) was added to a vial containing a magnetic stirring bar and $Y(NO_3)_3.6H_2O$ (0.1 mmol, 0.038g). The mixture was heated at 50 ºC in an oil bath and stirring was continued until disappearance of the starting materials (monitored by TLC). The reaction mixture was cooled and washed with water to extract the $Y(NO_3)_3.6H_2O$. The solid obtained was recrystallized from absolute EtOH to furnish the desired pure products. The products have been characterized by melting points and ^{1}H NMR spectroscopy. Spectra data of selected and known products are represented below:

6-Amino-3-methyl-4-phenyl-2,4-dihydro-pyrano[2,3-c]pyrazole-5-carbonitrile (**7a)**

IR (KBr ν_{max}, cm^{-1}): 3373, 3310 (NH_2), 3267 (N-H), 3170 (Ar–H), 3022 (C–H), 2193 (CN), 1649 (C=N), 1611, 1656 (C=C), 1152 and 1215 (C–O–C); ^{1}H NMR (300 MHz, DMSO-d_6): 1.79 (s, 3H, CH_3), 4.60 (s, 1H, CH), 6.91 (s, 2H, NH_2), 7.16–7.35 (m, 5H, Ar–H) and 12.12 (s, 1H, NH).

6-Amino-3-methyl-2,4-diphenyl-2,4-dihydro-pyrano[2,3-c]pyrazole-5-carbonitrile (**7n)**
IR (KBr ν_{max}, cm^{-1}): 3434 (NH_2), 3105 (Ar–H), 2934 (C–H), 2222 (CN), 1601 (C=N), 1581, 1567, 1507, 1468 (C=C), 1143 and 1272 (C–O–C); ^{1}H NMR (300 MHz, $CDCl_3$): 1.91 (s, 3H, CH_3), 4.69 (s, 1H, CH), 4.71 (s, 2H, NH_2), 7.26–7.69 (m, 10H, Ar–H).

6-Amino-3-methyl-2-oxo-1H-spiro[indoline-3,4-pyrano[2,3-c]pyrazole]-5-carbonitrile (**8a)**
IR (KBr ν_{max}, cm^{-1}): 3338 (NH_2), 3137 (N–H), 2923 (C–H), 2183 (CN), 1712 (C=O), 1608, 1584 (C=C);^{1}H NMR (DMSO-d_6, 300 MHz): 1.53 (s, 3H, CH3), 6.90 (d, 1H, Ar–H), 6.98-7.06 (m, 2H, Ar–H), 7.22-7.28 (m, 3H, Ar–H, NH_2), 10.6 (s, 1H, NH), 12.3 (s, 1H, NH).

6-Amino-3-methyl-2-oxo-4-phenyl1H-spiro[indoline-3,4-pyrano[2,3-c]pyrazole]-5-carbonitrile **(8b)** IR (KBr ν_{max}, cm^{-1}): 3460, 3292 (NH_2), 3176 (N–H), 3069 (Ar-H), 2921 (C–H), 2196 (CN), 1699 (C=O), 1653, 1614, 1595, 1580 (C=C); ^{1}H NMR (DMSO-d_6, 300 MHz): 1.58 (s, 3H, CH_3), 6.97-7.60 (m, 9H, Ar–H), 7.81-7.83 (d, 2H, NH_2), 10.77 (s, 1H, NH).

6-amino-3-methyl-2-oxo-2H spiro[acenaphthylene-1,4-pyrano[2,3-c]pyrazole]-5-carbonitrile **(9a)**
IR (KBr ν_{max}, cm^{-1}): 3423, 3323 (NH_2), 2189 (CN), 1712 (C=O), 1608, 1406 (C=C);^{1}H NMR (DMSO-d_6, 300 MHz): 1.08 (s, 3H, CH3), 7.34 (s, 2H, NH2), 7.47 (d, 1H, Ar–H), 7.76 (t, 1H, Ar–H), 7.90 (t, 1H, Ar–H), 8.05 (d, 2H, Ar–H), 8.40 (d, 1H, Ar–H), 12.29 (s, 1H, NH).

Results and discussion

In continuation of our recent interest in the synthesis of pyrano[2,3-c]pyrazoles and spiro annulated pyrano[2,3-c]pyrazoles to provide privileged scaffolds for the generation of target compounds for drug discovery. In our preliminary studies in this protocol, pyrano[2,3-c]pyrazoles were synthesized by an efficient one-pot four-component approach under aqueous medium conditions. In order to seek an optimal solvent, temperature and amounts of catalyst, the model reaction was explored using different solvents such as water, ethanol, methanol, tetrahydrofuran (THF), acetonitrile, dichloromethane, chloroform, and mixture of water/ethanol at room temperature (Table1, entries1-10). It was found that polarity of solvent play an important role for the success of the reaction. In the organic solvents such as dichloromethane, THF or chloroform, the yield of **7a** were lower and longer reaction times were required, whereas the reaction using Water:EtOH (1:1) resulted in good yields. Based on the results, Water:EtOH (1:1) was chosen to be the best in terms of the yield of the product and reaction time in comparison to common organic solvents. In the next step, we tried to

optimize the temperature on the model reaction in the presence of 0.038g $Y(NO_3)_3.6H_2O$ in diverse temperature conditions. As shown in Table1, we reached the shorter time and excellent yields when the reaction was carried out at 50 °C (Table1, entry 12). Also, in order to optimize the $Y(NO_3)_3.6H_2O$ loading, the model reaction was performed with different amounts of catalyst at ambient temperature. From Table1, we observed that the yield of product 7a was improved and the reaction time was relatively shortened when the amount of catalyst was increased from 3 mol % to 10 mol % (Table 1, entries 16-20).

Table 1. Optimization of solvent, temperature, and catalyst in synthesis of compound **7a**

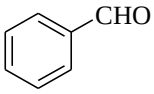
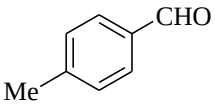
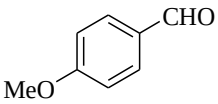
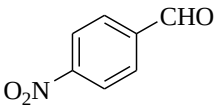
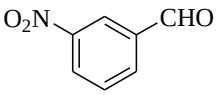
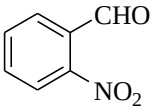
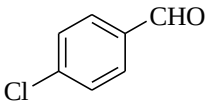
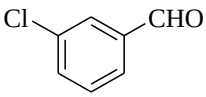
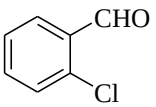
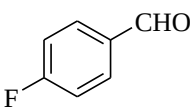
Entry	Solvent	Temperature (ºC)	Catalyst (mol %)	Time (min)	Yield (%)[a]
1	THF	r.t	10	45	63
2	CH_3CN	r.t	10	40	55
3	CH_2Cl_2	r.t	10	50	60
4	$CHCl_3$	r.t	10	45	72
5	EtOH	r.t	10	40	80
6	MeOH	r.t	10	35	75
7	H_2O	r.t	10	30	70
8	H_2O: EtOH (1:1)	r.t	10	35	85
9	H_2O: EtOH (2:1)	r.t	10	32	82
10	H_2O: EtOH (1:2)	r.t	10	35	80
11	H_2O: EtOH (1:1)	40	10	30	87
12	H_2O: EtOH (1:1)	50	10	25	90
13	H_2O: EtOH (1:1)	60	10	22	88
14	H_2O: EtOH (1:1)	70	10	22	87
15	H_2O: EtOH (1:1)	80	10	20	88
16	H_2O: EtOH (1:1)	50	3	30	85
17	H_2O: EtOH (1:1)	50	5	30	87
18	H_2O: EtOH (1:1)	50	15	28	85
19	H_2O: EtOH (1:1)	50	20	30	84
20	H_2O: EtOH (1:1)	50	25	32	85

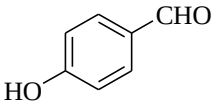
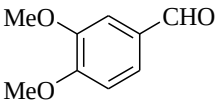
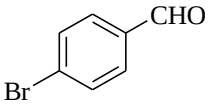
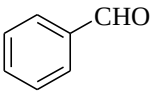
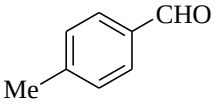
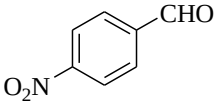
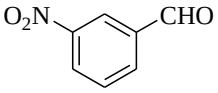
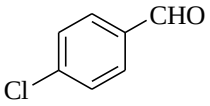
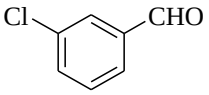
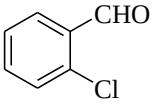
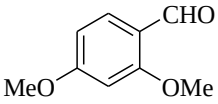
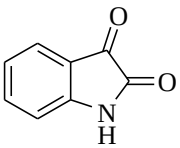
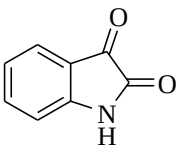
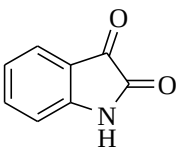
[a] Isolated yields

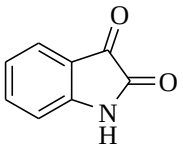
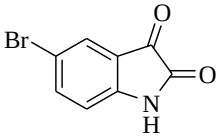
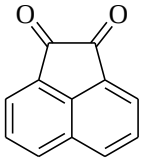
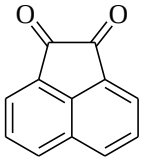
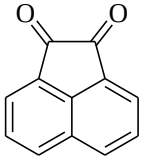
According to the obtained results, $Y(NO_3)_3.6H_2O$ loading of 10 mol % as the optimized reaction conditions was developed to other dihydropyrano[2,3-c]pyrazole derivatives from β-ketoesters, hydrazines, aromatic aldehydes and malononitrile. A large number of aromatic aldehydes with electron withdrawing or electron donating groups were well converted to the corresponding dihydropyrano[2,3-c]pyrazoles in good to excellent yields. Different substituted groups have not shown much effect on the formation of final products. Encouraged by the remarkable results, we

were highly motivated to broaden the scope of this protocol without any further optimization for one-pot synthesis of spiro[indoline-3,4-pyrano[2,3-c]pyrazole] derivatives which contain two different heterocyclic moieties, spirooxindole and dihydropyrano[2,3-c] pyrazole. All reactions smoothly proceeded to provide the products in good to excellent yields without formation of any by-products. Moreover, acenaphthenequinone was employed under the optimized conditions. As expected the reactions proceeded well to afford spiro[acenaphthylene-1,4-pyrano[2,3-c]pyrazole] derivatives in high yields (Table 2).

Table2. Synthesis of dihydropyrano[2,3-c]pyrazole, spiro[indoline-3,4-pyrano[2,3-c]pyrazole] and spiro[acenaphthylene-1,4-pyrano[2,3-c]pyrazole] using $Y(NO_3)_3.6H_2O$ in aqueous media.

Entry	Carbonyl compounds	R^1	R^2	Product	Time (min)	Yield (%)[a]	m.p (°C) Found	m.p (°C) Reported[Ref]
1		H	CN	**7a**	25	90	241-243	242-244[31]
2		H	CN	**7b**	30	91	197-199	196-198[22]
3		H	CN	**7c**	35	87	208-210	210-212[31]
4		H	CN	**7d**	22	88	247-249	249-250[31]
5		H	CN	**7e**	25	90	231-233	232-234[22]
6		H	CN	**7f**	22	89	222-223	222–224 [32]
7		H	CN	**7g**	20	94	231-233	230-232[25]
8		H	CN	**7h**	18	94	231-232	231-232[17]
9		H	CN	**7i**	22	92	245-247	(246-247[22]
10		H	CN	**7j**	20	93	245-247	245-246[22]

11		H	CN	**7k**	40	85	225-27	224-226[22]
12		H	CN	**7l**	35	83	190-192	190–191[33]
13		H	CN	**7m**	27	91	180-182	178-180[31]
14		Ph	CN	**7n**	30	88	171-173	170-172[31]
15		Ph	CN	**7o**	32	86	176-178	176-178[34]
16		Ph	CN	**7p**	25	85	194-196	194–196[19]
17		Ph	CN	**7q**	25	84	190-192	190-191[35]
18		Ph	CN	**7r**	22	90	174-175	173-174[25]
19		Ph	CN	**7s**	20	91	148-150	148–150[19]
20		Ph	CN	**7t**	25	90	145-147	144–146[19]
21		Ph	CN	**7u**	38	82	175-177	174-177 [36]
22		H	CN	**8a**	18	93	185-186	285–286 [37]
23		Ph	CN	**8b**	22	91	236-238	236–237 [38]
24		H	CO_2Et	**8c**	25	90	281-282	281–282[39]

25		Ph	CO_2Et	**8d**	30	88	235-236	235–236 [38]
26		H	CN	**8e**	15	94	283-284	283-284[24]
27		H	CN	**9a**	35	92	297-299	298–299 [40]
28		H	CO_2Et	**9b**	40	91	248-249	247–248[41]
29		Ph	CN	**9c**	38	92	194-196	193-195[42]

[a] Yields refer to the pure isolated products

Table 3 indicates the comparison of the activity of different catalysts by considering the yield for the reaction. We observed that the $Y(NO_3)_3.6H_2O$ catalyst gives the best catalytic activity in terms of product yield and reaction time compared to the other catalysts in the literature such as γ-Alumina, Sodium benzoate, DCDBTSD and etc.

Table 3. Comparison of the catalytic efficiency of $Y(NO_3)_3.6H_2O$ with that of reported catalysts in the synthesis of dihydropyrano[2,3-c]pyrazole

Entry	Comp.	Condition	Time(min)	Yield(%)	Ref.
1	HDBAC (30 mol %)	EtOH, reflux	45	73	[21]
2	Sodium benzoate (15 mol %)	Water, r.t.	60	85	[25]
3	Fe-MCM-22	H_2O:EtOH, 60 °C	50	80	[43]
4	Saccharose (20 mol %)	Solvent-free, 100 °C	10	75	[44]
5	Cetyltrimethylammonium	Water, 90 °C	240	89	[45]
6	У-Alumina (30 mol %)	Water, reflux	50	80	[46]
7	DCDBTSD (10 mol %)	Water, 80 °C	60	85	[47]
8	$Y(NO_3)_3.6H_2O$(10 mol %)	H_2O:EtOH, 50 °C	25	90	This work

Conclusion

In summary, we have developed a highly efficient approach for the synthesis of pyrano[2,3-c]pyrazole derivatives using $Y(NO_3)_3.6H_2O$ as inexpensive and available catalyst. This protocol was applicable to a wide range of carbonyl compounds including aromatic aldehydes, isatins and acenaphthenequinone. All the products were purified by simple crystallization. This method also offers several other significant advantages including easy preparation, handling of the catalyst, increased safety and environmental friendly reaction condition.

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