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PEG-400 assisted Kröhnke synthesis of 2-(2-hydroxyphenyl)-4-arylpyridines annulated by C_5-C_6 cycles with substituted benzylidene group

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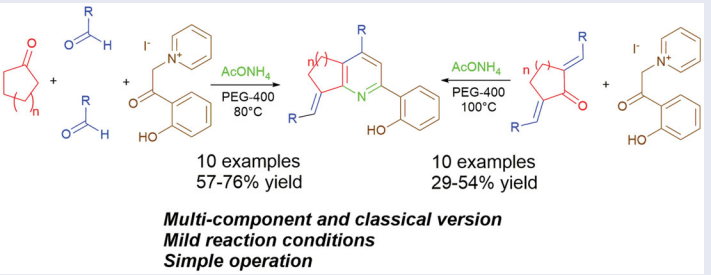
ABSTRACT

A simple and efficient method has been developed for the synthesis 2-(2-hydroxyphenyl)-4-arylpyridines annulated by C_5-C_6 cycles with substituted benzylidene group is achieved by multi-component Kröhnke-type reaction with moderate to good yields in PEG-400. The classical version was considered using cross-conjugated dienones as substrates by counter synthesis.

GRAPHICAL ABSTRACT



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KEYWORDS
Cross-conjugated dienones; 2-(2-hydroxyphenyl)pyridines; Kröhnke reaction; PEG-400

Introduction

In recent years, the development of highly efficient, selective, green, safe, atomic and stepwise-economic strategies for the synthesis of target compounds became one of the most prominent areas of organic chemistry. Special attention is paid to multi-component reactions (MCR) as they allow simplifying product separation, reducing reaction time and usually result in higher overall yield of final product compared to step-by-step synthesis.[1] It should be also noted that polyethylene glycol 400 has become a common solvent used for various organic reactions as it is inexpensive, thermally stable and easy to use. It also has low toxicity, thus making it preferable for "green" chemistry applications.[2] Thus PEG-400 and derivatives of PEG are an effective

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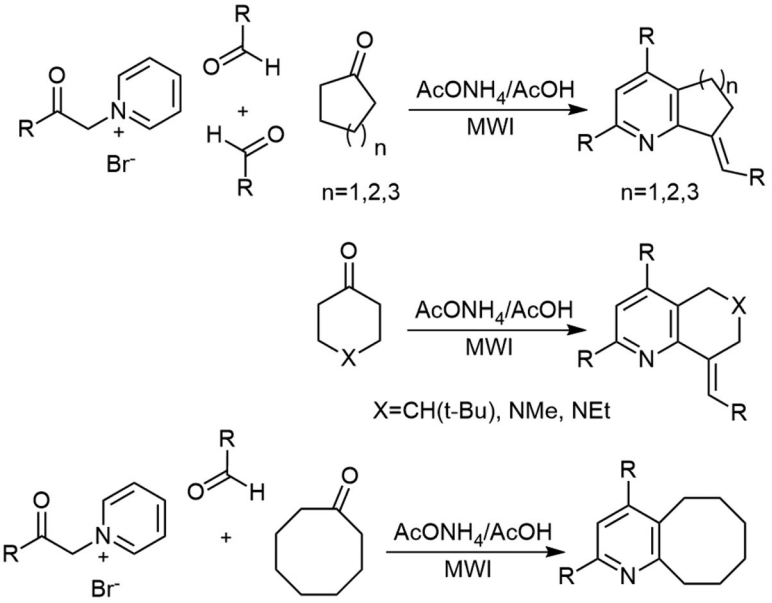


Scheme 1. Microwave assisted synthesis of annulated pyridines.

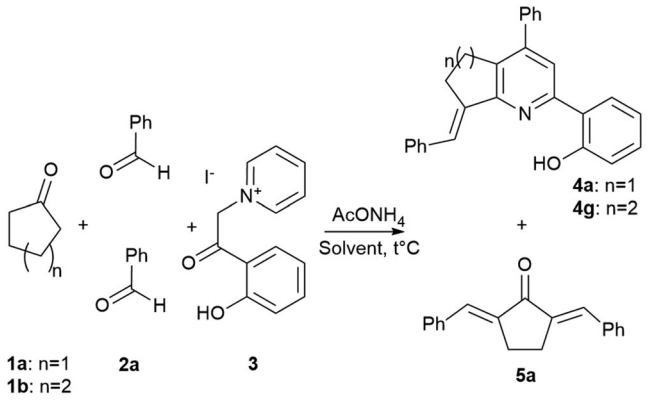
alternative medium for various multi-component transformations and can be used for such reactions as Biginelli reaction,[3] 1,3-dipolar cycloaddition,[4] Hantzch synthesis[5] and other multi-component reactions.[6]

The pyridine heterocyclic core is widespread in various natural products, pharmaceuticals,[7] and supramolecular assemblies with π–π staking and directional H-bonding.[8] One method used for constructing this heterocycle is a two-step Kröhnke synthesis via reaction of pyridinium salts with α,β-unsaturated ketones in the presence of a mixture of ammonium acetate and acetic acid or other solvent.[9] Recently special attention has been paid to Kröhnke multi-component reaction where α,β-unsaturated intermediate is not isolated from the reaction medium, which was confirmed for one-pot synthesis of 2,4,6-triarylpyridines,[10,11] substituted 5,6-dihydrobenzo[h]quinolines,[11] 5,7-dihydro-1,6-naphtyridines,[12] and annulated by C_5-C_8 cycles pyridines[11–13] were obtained assisted by microwave irradiation (Scheme 1).

Non-symmetric 2-(2-hydroxyaryl)pyridines and their derivatives have some interesting properties, namely, the ability to form coordination compounds with transition metals,[14] a wide spectrum of biological activity[15] and capability of engaging in the excited state intramolecular proton transfer (ESIPT).[16] These systems are usually synthesized via the Kröhnke method under classical conditions.[15,17] To the best of our knowledge, PEG-400 has not yet been used for this chemical transformation in the classical and multi-component versions.

Thus, we would like to report on the application of PEG-400 in multi-component Kröhnke-type reaction between aromatic aldehydes, N-(2-hydroxyphenacyl) pyridinium iodide, C_5-C_6 cycloalkanones and ammonium acetate, and counter synthesis using a cross-conjugated dienones from cyclopentanone and cyclohexanone as substrates by classical version in PEG-400.

Table 1. Optimization of reaction conditions.



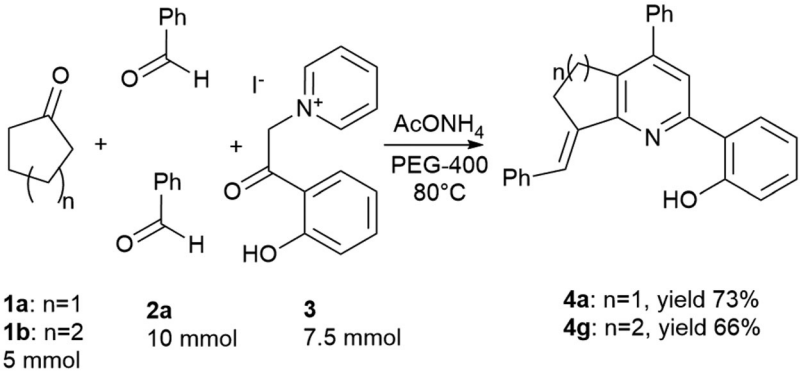
Entry	Substrate	**3a** (eq.)	Solvent	Time (min)	Yield[a] %
1	1a	1.2	PEG-400[b]	15	4a 40 5a 19
2	1a	1.3	PEG-400[b]	10	4a 59
3	1a	1.4	PEG-400[b]	8	4a 67
4	**1a**	**1.5**	**PEG-400[b]**	**8**	**4a 76**
5	1a	1.5	CH_3CN[c]	25	4a 51
6	1a	1.5	AcOH[c]	25	4a 56
7	1a	1.5	EtOH[c]	40	4a 48
8	1b	1.2	PEG-400[b]	16	4g 51
9	1b	1.3	PEG-400[b]	10	4g 58
10	1b	1.4	PEG-400[b]	10	4g 63
11	**1b**	**1.5**	**PEG-400[b]**	**10**	**4g 71**
12	1b	1.5	CH_3CN[c]	30	4g 44
13	1b	1.5	AcOH[c]	30	4g 52
14	1b	1.5	EtOH[c]	45	4g 47

Reaction conditions: cycloalkanone **1** (1 mmol), benzaldehyde **2a** (2 mmol, 2 eq.), N-(2-hydroxyphenacyl) pyridinium iodide **3**, ammonium acetate (3 g) [a]Yields of isolated products **4a** and **4g** after recrystallization from EtOH-$CHCl_3$ mixture (with 3:1 ratio). [b]The reaction was performed at 80 °C. [c]Reflux temperature.

Results and discussion

It's generally known that cross-conjugated dienones are formed as a result of condensation of aldehydes and cycloalkanones at a 2:1 ratio in the presence of a basic[18] or acidic[19] catalyst, and there are studies reporting that aldol condensation occurs in PEG-400 in the presence of potassium carbonate.[20] We hypothesized that cross-conjugated dienones can serve as the intermediate in the one-pot reaction between C_5-C_6-cycloalkanones, aromatic aldehydes and N-(2-hydroxyphenacyl) pyridium iodide that would result in producing pyridine annulated by C_5-C_6 cycles with an additional 7- or 8-substituted benzylidene group.

First, we studied the optimal synthesis conditions for the reaction between C_5-C_6 cycloalkanones (**1a**,**b**), benzaldehyde (**2a**) and pyridinium salt (**3**). The impact of various reaction conditions on the reaction outcome, including amount of pyridinium salt, time of reaction, and solvents are summarized in Table 1. The reaction proceeded with low yield in PEG-400 using 1.2 eq. of pyridinium salt (entries 1, 8) with the formation of cross-conjugated dienone (**5a**, entry 1) as by-product. Using a larger amount of N-(2-hydroxyphenacyl) pyridinium iodide **3** in the reaction increased on the yield of the



Scheme 2. Gram-scale multi-component Kröhnke reaction in PEG-400.

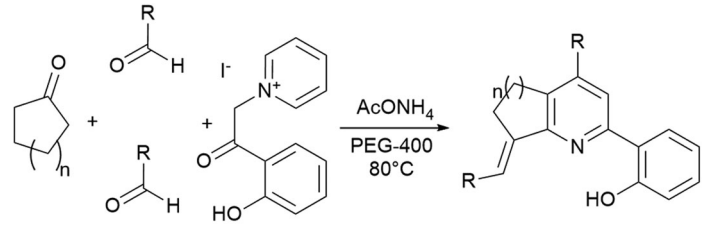
product and reduces reaction time (entries 2–4, 9–11). We have found that the optimal reagent ratio (**1–2a–3**) is 1:2:1.5 (entries 4, 11) and temperature 80 °C. A further increase in the amount of pyridinium salt did not lead to an increase in yield. When carrying out the synthesis in PEG-400, the reaction proceeded smoothly with good yields, while for other solvents the transformation takes much more time and the final product yields are smaller (entries 5–7, 12–14). The reaction was monitored via TLC with the mixture of hexane-chloroform-ethyl acetate at 3:1:1 ratio used as an eluent. At the end of the reaction, water is added to the reaction mixture; the product is filtered off and recrystallized from ethanol-chloroform mixture (with 3:1 ratio).

For the quantitative generalization of multicomponent Kröhnke-type synthesis, the above optimized reaction conditions were applied for the reaction between 5 mmol cycloalkanone **1**, 10 mmol benzaldehyde **2a**, and 7,5-mmol N-(2-hydroxyphenacyl) pyridinium iodide **3**. Reaction finished after 15 min for cyclopentanone **1a** and 20 min for cyclohexanone **1b** with good yield (**4a**, 73%; **4g** 66%) (Scheme 2).

After optimization, a series of annulated 2-(2-hydroxyphenyl)pyridines by C_5-C_6 cycles (**4a–k**) was synthesized with moderate to good yields with reaction time 8 and 50 min. It should be noted that during the reaction in PEG-400, aromatic aldehydes containing electron-donating groups, formed annulated 2-(2-hydroxyphenyl) pyridines with good yield. When the same aromatic aldehyde was used as a substrate, the yields of the products for cyclopentanone were higher and the reaction time was shorter than compared to cyclohexanone (Table 2). Isolated products by reactions between the pyridinium salt **3**, cyclopentanone **1a** and 4-N,N-dimethylaminobenzaldehyde **2g** (reaction time 30 min) and between cyclohexanone **1b** and 4-chlorobenzaldehyde **2b** (reaction time 10 min) either were not characterized due to their low solubility in organic solvents. The same is true for the reaction between pyridinium salt, cycloalkanones, 4- and 3-nitro substituted benzaldehydes.

Many works[21] with various C-nucleophiles and N-nucleophiles have been presented for cross-conjugated dienones, but, as far as we know, the use of these substrates in the Kröhnke reaction has never been described. We performed a counter synthesis of annulated pyridines from cross-conjugated dienones (**5a–f**, **6a–e**) (Table 3). When using this synthetic route, the reaction time increased, the yield of the product decreased, and it was necessary to use a larger amount of pyridinium salt and ammonium acetate, a

Table 2. One-pot synthesis of annulated pyridines.



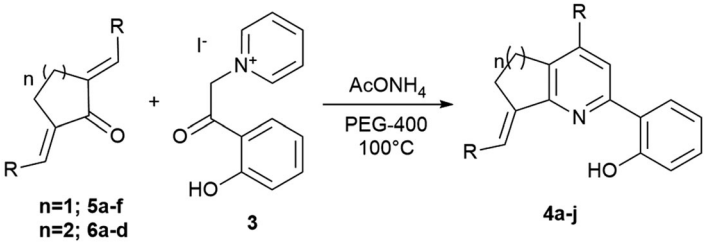
1a: n=1
1b: n=2

2a-g **3** **4a-j**

Entry	Substrate	Product	R	Time (min)	Yield[a] %
1	**1a** + **2a**	**4a**	Ph	8	76
2	**1a** + **2b**	**4b**	4-Cl-C_6H_4	10	69
3	**1a** + **2c**	**4c**	4-MeO-C_6H_4	12	68
4	**1a** + **2d**	**4d**	3,4-$(MeO)_2$-C_6H_3	18	71
5	**1a** + **2e**	**4e**	3,4,5-$(MeO)_3$-C_6H_2	25	64
6	**1a** + **2f**	**4f**	4-OH-3-MeO-C_6H_3	30	61
7	**1b** + **2a**	**4g**	Ph	10	71
8	**1b** + **2c**	**4h**	4-MeO-C_6H_4	15	59
9	**1b** + **2d**	**4i**	3,4-$(MeO)_2$-C_6H_3	25	66
10	**1b** + **2f**	**4j**	4-OH-3-MeO-C_6H_4	50	57

Reaction conditions: cycloalkanone **1** (1 mmol), substituted benzaldehyde **2** (2 mmol), N-(2-hydroxyphenacyl) pyridinium iodide **3** (1,5 mmol), ammonium acetate (**3g**) in 1 ml PEG-400 at 80 °C. [a]Isolated yield after recrystallization from EtOH-$CHCl_3$ mixture (with 3:1 ratio).

Table 3. Counter synthesis of annulated pyridines.

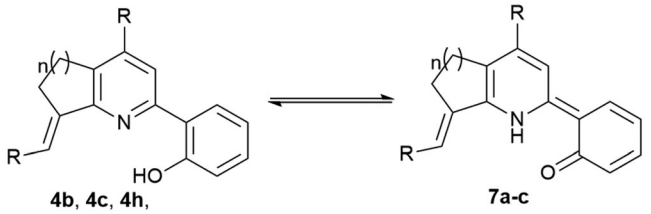


n=1; 5a-f
n=2; 6a-d

3 **4a-j**

Entry	Substrate + **3** (eq.)	Product	R	Time (min)	Yield[a] %
1	**5a,** 2.5	**4a**	Ph	40	46
2	**5b,** 2.5	**4b**	4-Cl-C_6H_4	45	51
3	**5c,** 3.0	**4c**	4-MeO-C_6H_4	90	41
4	**5d,** 3.0	**4d**	3,4-$(MeO)_2$-C_6H_3	120	34
5	**5e,** 3.5	**4e**	3,4,5-$(MeO)_3$-C_6H_2	160	30
6	**5f,** 3.5	**4f**	4-OH-3-MeO-C_6H_3	180	29
7	**6a,** 2.0	**4g**	Ph	30	54
8	**6b,** 2.5	**4h**	4-MeO-C_6H_4	45	47
9	**6c,** 2.5	**4i**	3,4-$(MeO)_2$-C_6H_3	70	42
10	**6d,** 3.0	**4j**	4-OH-3-MeO-C_6H_4	120	43

Reaction conditions: cross-conjugated dienone **5** or **6** (1 mmol), N-(2-hydroxyphenacyl) pyridinium iodide **3**, ammonium acetate (**5g**) in 3 ml PEG-400 at 100 °C. [a]Isolated yield after recrystallization from EtOH-$CHCl_3$ mixture (with 3:1 ratio).



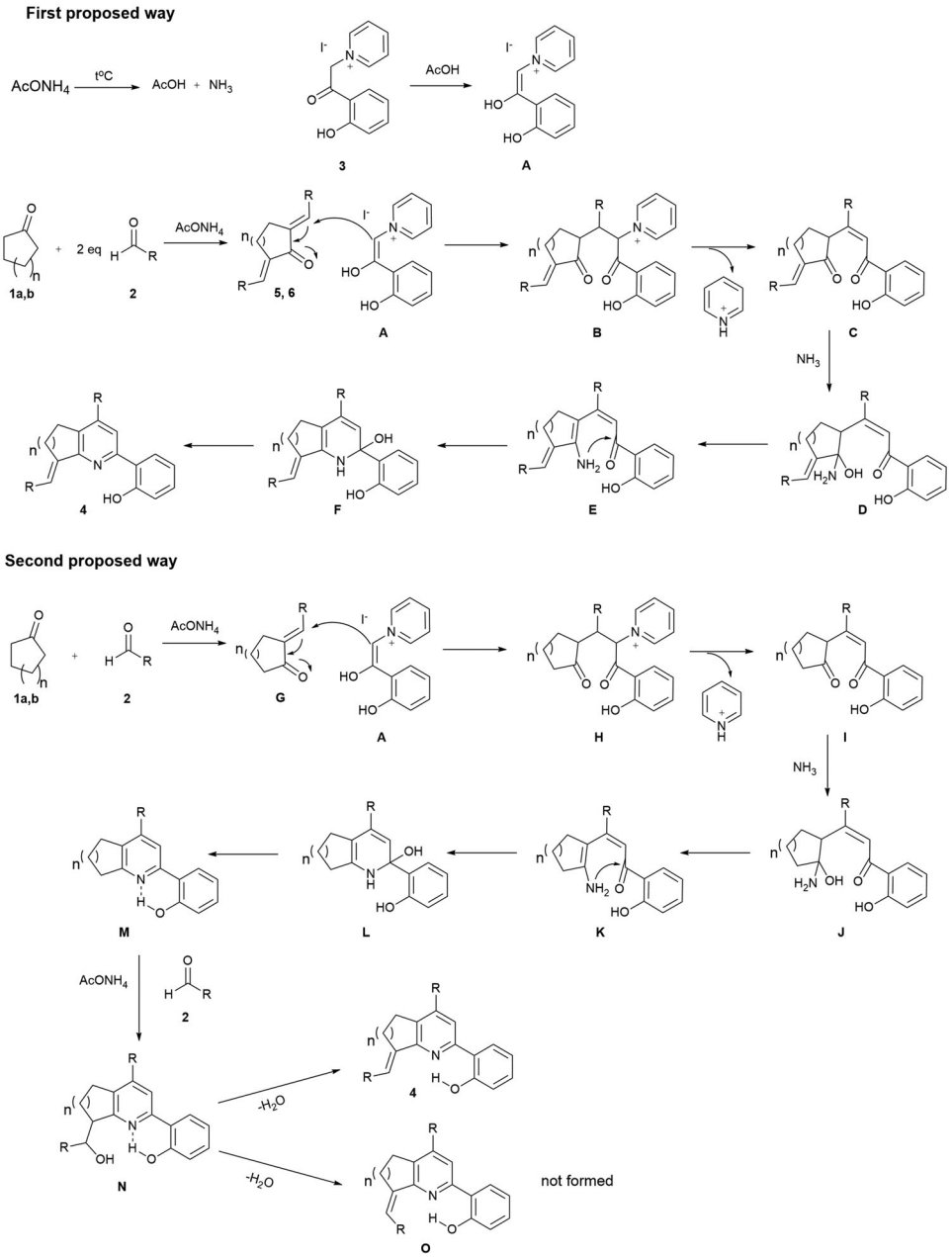
4b, **7a**: R=4-Cl-C_6H_4 n=1; **4c**, **7b**: R=4-MeO-C_6H_4 n=1;
4h, **7c**: R=4-MeO-C_6H_4 n=2;

Scheme 3. Enol-imino keto-amino tautomerism.

larger volume of PEG-400 and higher reaction temperature. Since cross-conjugated dienones had an *E* configuration, the synthesized annulated pyridines also had an *E* configuration. Using this path led to the same products as in the multi-component version. This means that the *E*-isomer also formed in the one-pot Kröhnke type reaction.

The composition of the synthesized compounds was established on the basis of elemental analysis data, the structures were further characterized by IR, ^{1}H and ^{13}C NMR spectroscopy. As the solubility of compound **4e** is very low, only its ^{1}H NMR spectrum was obtained. The characteristic ^{1}H chemical shift of the phenol proton of products **4** indicates the intramolecular hydrogen bond. This proton resonates as a single peak because of the difficulty of exchanging a phenolic proton with a deuterated solvent.[23] In addition, the value of the chemical shift of this proton is influenced by the substituents in the aromatic ring of the benzylidene group and the aryl group in position 4 of the pyridine ring. In the ^{13}C NMR spectrum of products **4b**, **4c**, **4h** a carbonyl carbon signal is detected in solution, but carbonyl absorption was absent in the IR spectra in the KBr matrix. For these compounds, the signal is detected at 176.55–176.32 ppm in $CDCl_3$, also in work[22] carbonyl carbon resonated at 174.8 and 171.1 ppm in $CDCl_3$ for gossypol imine derivatives. These signals may serve as indicators of enol-imino keto-amine tautomerism of these compounds and the existence of tautomers **7a–c** (Scheme 3) in solution. For compounds **4c** and **4h**, UV-VIS spectra were recorded in acetonitrile and it were recorded absorption of the carbonyl group with a maximum at 403–430 nm (**4h**) and 440–480 nm (**4c**) region, which is consistent with the paper on the tautomerism of 2-(2-hydroxyaryl)azines.[23]

In the published work[11] on annulated pyridines it was not reported which isomer was isolated. Some cyclopenta[b]pyridines in the ^{13}C NMR spectrum showed 4 signals in the region 29.4–27.3, which may be evidence of the presence *Z* and *E* isomers. Based on these data and our results, we propose reaction mechanisms for multicomponent version, which are depicted in Scheme 4. First possible way is based on cross-conjugated intermediate. Cycloalkanone **1** and aromatic aldehyde **2** undergo aldol condensation to form *E*-cross-conjugated dienone **5**, **6**, assisted by ammonium acetate.[24] Pyridinium salt **3** enolizes to form enol **A,** catalyzed by acetic acid, followed Michael reaction with cross-conjugated dienone to form 1,5-diketone **B**. Pyridinium cation is then eliminated to form unsaturated 1,5-diketone **C**. Nucleophilic addition ammonia to diketone **C** followed dehydration via **D** generates enamine **E**. Enamine cyclizes with the carbonyl to generate hydroxy-intermediate **F**. Dehydration of intermediate **F** generates the target



Scheme 4. Proposed mechanisms of multi-component reaction.

product **4**. In the second proposed way, cycloalkanone **1** and aromatic aldehyde **2** undergo aldol condensation to form monoenone **G**, which then reacts with enol **A** by Michael reaction to form diketone **H**. Pyridinium cation is then eliminated to form unsaturated 1,5-diketone **I**. Nucleophilic addition ammonia to diketone **I** followed dehydration via **J** generates enamine **K**, which cyclizes with the carbonyl to generate hydroxy-intermediate **L**. Dehydration of intermediate **L** generates the annulated

pyridines **M**. Ammonium acetate and intramolecular hydrogen bond in annulated pyridine **M** promote aldol reaction with the aromatic aldehyde **2** to form aldol **N**, which dehydrated to form product **4**. Intramolecular hydrogen bond in aldol **N** between the phenolic proton and the nitrogen atom of the pyridine ring sterically blocks the dehydration and formation of the *Z*-isomer **O** and therefore only *E*-isomers are formed. When using cross-conjugated dienones **5**, **6**, the possible mechanism should be considered along the first way. Comparing the results of the classical and multi-component versions of the Kröhnke reaction for the synthesis of annulated 2-(2-hydroxyphenyl)pyridines **4** and based on published data, we believe that the multi-component version of this transformation proceeds through the second proposed pathway.

In conclusion, we have developed new conditions for Kröhnke reaction for multi-component and classical version in PEG-400 between readily available cycloalkanones, aromatic aldehydes or the corresponding cross-conjugated dienones and N-(2-hydroxyphenacyl)pyridinium iodide in the presence of ammonium acetate resulting in a fast and efficient synthesis of the previously unknown 2-(2-hydroxyphenyl)-4-arylpyridines annulated by C_5-C_6 cycles and containing a substituted benzylidene group. According to the ^{13}C NMR and UV/VIS data, the existence of two tautomeric forms of some compounds was shown. The resulting compounds can be considered as potential N-, O-bidentate ligands or ESIPT fluorophores.

Experimental section

Method A. Multi-component version of Kröhnke reaction for products 4; general procedure

A mixture of cycloalkanone **1** (1 mmol, 1 eq.), aromatic aldehyde **2** (2 mmol, 2 eq.), N-(2-hydroxyphenacyl) pyridinium iodide **3** (1.5 mmol, 1.5 eq.) ammonium acetate (**3g**) and 1 ml of PEG-400 was stirred at 80 °C for same time. The reaction was monitored by thin-layer chromatography. After the reaction is finished, ice cold water was added to the reaction mixture (50 ml) and stirred for 15 min. The resulting solid product **4** was filtered, rinsed with ice cold water (50 ml) and recrystallized from EtOH-$CHCl_3$ mixture (at 3:1 ratio).

Method B. Classical version of Kröhnke reaction for products 4; general procedure

A mixture of cross-conjugated dienone **5** or **6** (1 mmol, 1 eq.), N-(2-hydroxyphenacyl) pyridinium iodide **3** (2.0–3.5 mmol, 2–3.5 eq.) ammonium acetate (**5g**) and 3 ml of PEG-400 was stirred at 100 °C for same time. The reaction was monitored by thin-layer chromatography. After the reaction is finished, ice cold water was added to the reaction mixture (50 ml) and stirred for 15 min. The resulting solid product **4** was filtered, rinsed with ice cold water (50 ml) and recrystallized from EtOH-$CHCl_3$ mixture (at 3:1 ratio).

Full experimental details including all information, ^1H and ^{13}C NMR spectra, elemental analysis and IR spectra data have been provided in supporting information as a separate file.

Disclosure statement

No potential conflict of interest was reported by the authors.

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