Mendeleev Communications

Mendeleev Commun., 2025, **35**, 457–460

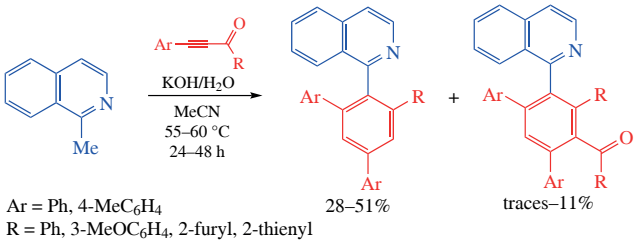
One-pot assembly of functionalized (*m*-terphenyl-4′-yl)isoquinolines from 1-methylisoquinoline and electrophilic acylacetylenes

Kseniya V. Belyaeva, Lina P. Nikitina, Veronika S. Saliy, Andrei V. Afonin, Igor A. Ushakov, Alexander V. Vashchenko and Boris A. Trofimov*

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, 664033 Irkutsk, Russian Federation. E-mail: boris_trofimov@irioch.irk.ru

DOI: 10.71267/mencom.7695

The relative reactivity of deprotonated CH_3 group and nitrogen atom of 1-methylisoquinoline towards electrophilic triple bond crucially depends on the acetylene structure and the reaction conditions. 1-Acyl-2-arylacetylenes react with 1-methylisoquinoline in a 2 : 1 molar ratio (34 mol% $KOH \cdot 0.5 H_2O$, H_2O/MeCN, 55–60 °C, 24–48 h) to give 1-[5′-(het)aryl-*m*-terphenyl-4′-yl]isoquinolines in 28–51% yields and 1-[6′-acyl-5′-(het)aryl-*m*-terphenyl-4′-yl]isoquinolines in trace to 11% yields. The minor product (proved by X-ray) results from 1,3-shift of the acyl group in the carbanionic intermediate.



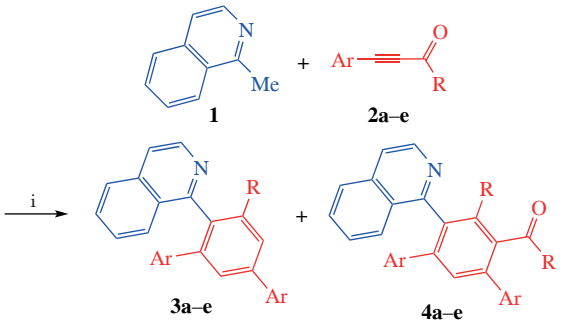
Keywords: alkyne, functionalization, isoquinoline, *m*-terphenyl, zwitteriones.

Functionalized isoquinolines are the pharmacophoric structural elements of natural alkaloids, biologically active compounds and drugs.[1–5] 2-(Isoquinolin-1-yl)benzoic acids are successfully cyclized to furnish the azabenzanthrone derivatives, the potential precursors for the synthesis of menisporphine alkaloids and daurioxoisoaporphines.[6] 1-Phenylisoquinoline is utilized as a component of photosensitizer complexes for photocatalytic CO_2 reduction.[7] Axially chiral 1-arylisoquinoline derivatives are valuable ligands and catalysts for various asymmetric catalytic reactions (see Online Supplementary Materials, Figure S1).[8]

No wonder that the search for new families of functionalized isoquinolines is in the focus of modern organic synthesis. In this context, the use of 1,3(4)-dipole (zwitterionic) intermediates generated from pyridinoids and electron-deficient acetylenes was shown to be attractive.[9–13] In the case of 2-methyl substituted pyridinoids, the C–H bond is capable of being deprotonated and often competes as a nucleophile with the C=N fragment. For example, the methyl group located α-position to the positively charged nitrogen atom in zwitterionic intermediates of 2-methylquinolines[14–16] can be involved in the reactions with electrophilic alkynes such as 1-cyano-2-phenylacetylene,[14] 1-acyl-2-arylacetylenes[15] and bis(het)arylated ethynyl ketones[16] (see Online Supplementary Materials, Scheme S1). In these previous works we showed that two molecules of electrophilic alkynes could react with 2-methylquinoline (KOH, MeCN) engaging the C–H bonds to deliver 2-[3-cyano-2-phenylallyl(allylidene)]quinolines for 1-cyano-2-phenylacetylene[14] and 2-(5′-aryl-*m*-terphenyl-4′-yl)quinolines for 1-acyl-2-arylacetylenes.[15] The results on this unique functionalization of the quinoline core require further investigations to establish whether this reaction is general enough.

We commenced the current study with the reaction of 1-methylisoquinoline **1** and 1-benzoyl-2-phenylacetylene **2a** as the model reactants (Scheme 1). The starting conditions were those found for a similar reaction with isomeric 2-methylquinoline,[15] *i.e.*, **1**/**2a** molar ratio of 1 : 1, 34 mol% $KOH \cdot 0.5 H_2O$, 5 equiv. H_2O, MeCN (0.25 ml), 24 h (Table 1, entry 1). The product turned out to be 1-(5′-phenyl-*m*-terphenyl-4′-yl)isoquinoline **3a** isolated in 22% yield. The reaction progress was monitored by IR spectroscopy to follow intensity of the absorption band of the C≡C bond of starting acylacetylene **2a** at 2199 cm^{-1}.

In the absence of water and alkali at room temperature (66 h) or when heated at 55–60 °C (MeCN, 24 h) or 100 °C (toluene, 8 h), the reaction did not occur (see Table 1, entries 2, 3). When the reaction was carried out in the presence of 34 mol%



3a–e/4a–e yields (%)

a	Ar = R = Ph	33/11
b	Ar = Ph, R = 3-$MeOC_6H_4$	39/traces
c	Ar = 4-MeC_6H_4, R = Ph	51/4
d	Ar = Ph, R = 2-furyl	32/7
e	Ar = Ph, R = 2-thienyl	28/10

Scheme 1 *Reagents and conditions*: i, **1**/**2** = 1 : 1 (mol/mol), 34 mol% $KOH \cdot 0.5 H_2O$, 5 equiv. H_2O, MeCN, 55–60 °C, 24 h (for **2a**,**d**,**e**) or 48 h (for **2b**,**c**). For optimization, see Table 1.

Table 1 Reaction of 1-methylisoquinoline **1** with 1-benzoyl-2-phenylacetylene **2a** under different conditions.[a]

Entry	Base (mol%)	H_2O (equiv.)	*T*/°C	*t*/h	Conversion (%)		Yield (%)	
					1	**2a**	**3a**	**4a**
1	KOH[a] (34)	5	55–60	24	30	99	22	traces
2	None	None	20–25/55–60	66/24	No reaction			
3[b]	None	None	100	8	No reaction			
4	KOH (34)	None	55–60	168	58	85	0	0
5[c]	KOH (34)	5	55–60	24	44	99	33	11
6[c,d]	KOH (34)	5	55–60	48	39	99	33	8
7[c]	KOH (17)	5	55–60	24	39	54	10	0
8[c]	KOH (100)	5	55–60	24	100	100	26	6
9	NaOH (37)	5	55–60	24	51	67	21	traces
10	DABCO (37)	5	55–60	24	35	36	12	0
11[b,e]	KOH (34)	5	55–60	48	44	99	0	0

[a] **1** (0.5 mmol), **2a** (0.5 mmol), MeCN (0.25 ml); KOH was used as hemihydrate $KOH \cdot 0.5H_2O$. [b] In toluene. [c] In 0.5 ml of MeCN. [d] Molar ratio **1/2a** was 1 : 2. [e] No reaction in THF.

$KOH \cdot 0.5\,H_2O$ at 55–60 °C, the conversion of reactants **1** and **2a** increased to 58 and 85%, respectively, but the target product **3a** was not detected (entry 4). Obviously, in the absence of water product **3a** is not formed. Further, in order to reduce the consumption of acetylene **2a** for side processes of oligomerization, the reaction mixture was twice diluted with acetonitrile (0.5 instead of 0.25 ml of MeCN), the yield of product **3a** being increased to 33% (entry 5). At the reactant **1/2a** ratio of 1 : 2, the yield of **3a** remained the same (entry 6). Surprisingly, all our further attempts to improve the reaction outcome appeared to be unsuccessful. At a lower loading of potassium hydroxide (17 mol%), the yield of terphenyl derivative **3a** dropped to 10% (entry 7) while with 100 mol% KOH the **3a** yield was as modest as 26% (entry 8). In the presence of other bases (NaOH and DABCO), the reaction proceeded less efficiently (yields 21 and 12%, respectively, entries 9, 10). Replacement of acetonitrile by tetrahydrofuran or toluene (55–60 °C) negatively affected synthesis of the target product **3a** (entry 11). In most cases, along with compound **3a**, 1-(6′-benzoyl-5′-phenyl-*m*-terphenyl-4′-yl)isoquinoline **4a** was formed in trace to 11% yields. The maximum yield of product **4a** (11%) was observed under the most favorable conditions for the synthesis of **3a** (entry 5). An intriguing feature of the reaction is that despite of 1 : 1 reactant ratio, two molecules of acetylene **2a** reacted with one molecule of isoquinoline **1** leaving the second molecule of isoquinoline intact. Moreover, at 1 : 2 reactants **1/2a** ratio, the yield of the target product **3a** was not improved. This means that after the formation of intermediate 1 : 1 adduct, the next steps, *i.e.* addition of the second molecule of acetylene and cyclization of open-chain intermediate proceed much faster than the first one.

Scheme 1 also shows that the reaction under study can be extended over other 1-acyl-2-arylacetylenes **2b–e** including those having furyl or thienyl substituents. The yields of the major products **3** spanned 28–51%, while those of the minor ones **4** ranged from trace to 11%. The complexity of this cascade process and the limited list of acetylenes examined did not allow making clear-cut conclusions on the yield/structure dependence. Meanwhile, the highest yield of product **3c** (51%), synthesized from the least electrophilic 1-benzoyl-2-*p*-tolylacetylene **1c**, may hint that electron-donating substituents facilitate better the cyclization than oligomerization of the intermediate open-chain bis-adduct.

The structures of all the products were proved by ^1H, ^{13}C, 2D NOESY NMR spectroscopy results, which were in good agreement with data obtained for arylterphenyl derivatives of quinoline[15] and oxazinoisoquinolines.[17] The unexpected location of acyl substituents in the terphenyl part in minor products **4** was established by X-ray crystal analysis of product **4d** (Figure 1).† As seen from Figure 1, furoyl substituent is located *ortho* to the 2-furyl substituent that points to 1,3-transfer of the acyl substituent which should normally be situated between the phenyl groups.

Acylacetylenes such as 1-acetyl-2-phenylacetylene, benzoylacetylene, 1,2-dibenzoylacetylene or 1-benzoyl-2-(5-phenyl-1-vinylpyrrol-2-yl)acetylene did not give the target products of 1-methylisoquinoline functionalization. Details of their reactivity are discussed in Online Supplementary Materials.

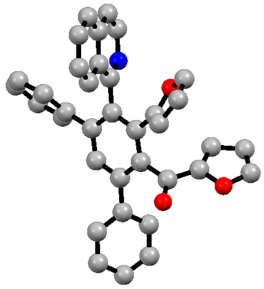
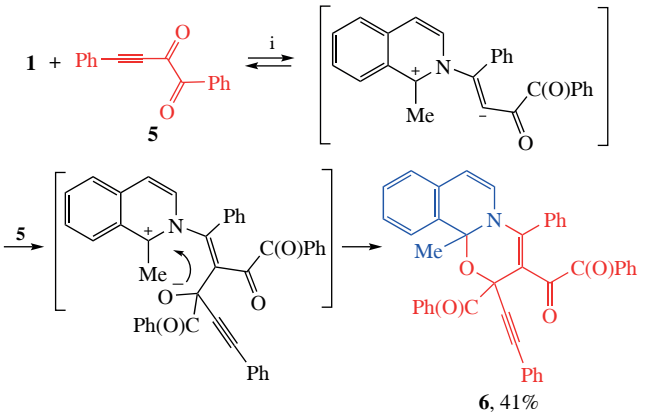


Figure 1 X-ray structure of product **4d**. Thermal ellipsoids set at 50% probability. For clarity, hydrogen atoms and molecule of solvent are omitted.

† *Crystal data for* **4d** · 2$CHCl_3$. $C_{36}H_{23}NO_3 \cdot 2CHCl_3$ (M = 756.32). The determination of the unit cell and the data collection were performed on a Bruker D8 VENTURE PHOTON 100 CMOS diffractometer with CuKα radiation (λ = 1.54178) at 293.2(2) K using the ω–φ scan technique. Approximate dimensions 0.38×0.28×0.13 mm were used for the X-ray crystallographic analysis. The X-ray intensity data were measured. The integration of the data using a triclinic unit cell with $P\bar{1}$ space group yielded a total of 65192 reflections to a maximum θ angle of 67.0°, of which 6355 were independent (completeness = 98.8%, R_{int} = 4.83%, R_{sig} = 2.36%) and 4761 were greater than $2\sigma(F^2)$. The final cell constants are a = 11.2135(3), b = 12.3672(3) and c = 13.7438(3) Å, α = 107.445(1), β = 98.040(1) and γ = 90.728(1)°, Z = 2, V = 1797.43(8) Å^3. Data were corrected for absorption effects using the multi-scan method (SADABS).[18] The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.259 and 0.541. The structure was solved using the Bruker SHELXTL Software Package[19] and refined using Olex2[20] package. All H atoms were treated by mixed method.

The final anisotropic full-matrix least-squares refinement on F^2 with 489 variables converged at R_1 = 5.30%, for the observed data and wR_2 = 15.76% for all data. The goodness-of-fit was 1.05. The largest peak in the final difference electron density synthesis was 0.29 e$^-$ Å^{-3} and the largest hole was –0.42 e$^-$ Å^{-3}. On the basis of the final model, the calculated density was 1.397 g cm^{-3} and F(000), 772 e$^-$.

CCDC 2389926 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* https://www.ccdc.cam.ac.uk.



Scheme 2 *Reagents and conditions*: i, MeCN, 5 °C, 24 h.

An exotic electrophilic acetylene, 1,4-diphenylbut-3-yne-1,2-dione **5**, was found to be so reactive that it was capable of assembling with 1-methylisoquinoline **1** at 5 °C in the absence of bases (MeCN, 24 h) to provide 1 : 2 adduct **6** with oxazino[2,3-*a*]isoquinoline core in 41% yield (Scheme 2). Apparently, in this case exclusively C=N nucleophilic site of 1-methylisoquinoline was engaged into the transformation.

Presumably the formation of 1-(5′-aryl-*m*-terphenyl-4′-yl)-isoquinolines **3** and 1-(6′-acyl-5′-aryl-*m*-terphenyl-4′-yl)isoquinolines **4** (Scheme 3, route *A*) involves the hydroxide-mediated deprotonation of the methyl group, and the thus formed carbanion adds to the triple bond of acetylene **2** to deliver carbanionic intermediate **A**. The latter attacks the second molecule of acylacetylene **2**, generating the carbanionic intermediate **B**, which is further neutralized by a proton of water to the open-chained diene **C**. The latter undergoes the ring closure involving the deprotonated CH_2 group and the carbonyl function of the acyl group. Dehydration of the intermediate **D** provides substituted triarylphenyl derivative **E**, which would eliminate potassium carboxylate under the action of KOH, leading to the final products **3**.

The rearrangement products, 1-(6′-acyl-5′-aryl-*m*-terphenyl-4′-yl)isoquinolines **4**, are the result of 1,3-acyl transfer on the stage of intermediate **B** followed by quenching of the emerging carbanion with a proton of water, ring-closure and dehydration (see Scheme 3, route *B*). The driving force of this process is a lower proton concentration due to the higher medium basicity. In such a case, the transferring acyl cation would partially replace the medium proton.

The assembly of ethynyloxazino[2,3-*a*]isoquinoline **6** also likely passes through a 1,3(4)-dipole intermediate (see Scheme 2). But in this case, its carbanionic center attacks β-carbonyl group of the second molecule of diketo acetylene **5**, and the resulting oxygen-centered anionic intermediate would form the covalent bond with the positively charged position 1 of the isoquinoline part to close the oxazine ring. As seen, here only one nucleophilic site of 1-methylisoquinoline, C=N moiety, is chemoselectively involved into the reaction with two molecules of electrophilic acetylene **5** avoiding deprotonation of the CH_3 group. Generally, the results obtained clearly demonstrate that dual nucleophilicity of 1-methylisoquinoline can be differently and selectively ruled depending on the structure of acetylenic partner.

It should be mentioned that poly(het)arylbenzenes are used in the design of organic electroluminescent devices.[21–25] They are also utilized in the synthesis of dendrimers[26,27] and fullerene fragments.[28,29] For the synthesis of poly(het)arylbenzenes, different cross-couplings in the presence of transition metals[23,25] or acid-catalyzed cycloadditions[27] were applied. However, the approaches to the synthesis of azine-based analogs are just sporadic.[21–23,30]

In conclusion, 1-methylisoquinoline undergoes extraordinary functionalization with electrophilic acetylenes under very simple conditions (heating in MeCN with KOH or without it at 5 °C in MeCN) involving either deprotonated CH_3 group or nitrogen atom depending on the structure of acetylenic partner. In the former case, derivatives of 1-(5′-aryl-*m*-terphenyl-4′-yl)-isoquinolines are formed. Highly electrophilic 1,4-diphenylbut-3-yne-1,2-dione when reacted with 1-methylisoquinoline attacks exclusively the C=N site to afford oxazino[2,3-*a*]-isoquinoline derivative in 41% yield. Thus, a conceptually new approach to functionalization of isoquinolines has been outlined.

The spectral data were obtained with the equipment of the Baikal Analytical Center for collective use SB RAS. ESIHRMS-TOF spectra were performed by A. V. Kuzmin in Shared Research Facilities for Physical and Chemical Ultramicroanalysis LIN SB RAS.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.71267/mencom.7695.

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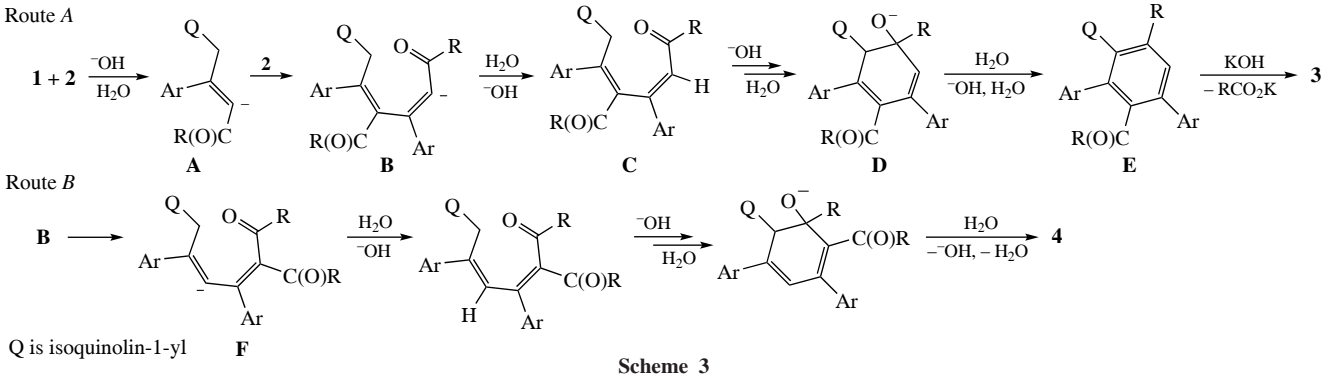

Scheme 3

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Received: 28th November 2024; Com. 24/7695