[2-(2-Nitrophenyl)oxiran-1-yl](aryl(methyl))ketones in the synthesis of 3-hydroxyquinolin-4(1*H*)-ones and 2-arylquinolines

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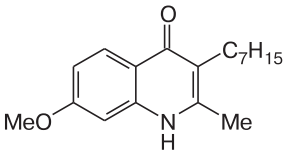
The applicability of [2-(2-nitrophenyl)oxiran-1-yl](aryl(methyl))ketones in the synthesis of 3-hydroxyquinolin-4-ones and 2-arylquinolines was studied.

Key words: 3-hydroxyquinolin-4(1*H*)-ones, 4-bromo-3-hydroxyquinolines, 3-hydroxyquinolines, 2-arylquinolines, [2-(2-nitrophenyl)oxiran-1-yl](aryl(methyl))ketones.

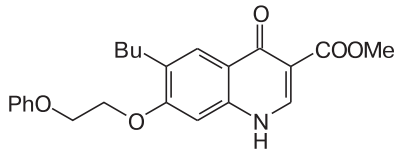
Quinoline and quinolinone derivatives are of interest owing to a wide range of their pharmacological activities.[1—7] Thus, these compounds are well known for their antimicrobial activity.[1] Quinolin-4-one-based compounds are widely used as antimalarial agents[3] (Fig. 1). Due to the danger of malaria that causes some million deaths in tropical countries annually and rapidly developing resistance of the causative agents of this disease to the medications used, the quinolin-4-one derivatives are of significant interest as antimalarial drug candidates.

In the present work, we describe the application of [2-(2-nitrophenyl)oxiran-1-yl](aryl(methyl)ketones in the synthesis of 3-hydroxyquinolin-4(1*H*)-ones and 2-arylquinolines.

3-Hydroxyquinolin-4(1*H*)-one-based systems and, in particular, 2-heptyl-3-hydroxy-4(1*H*)-quinolone (*Pseudomonas* quinolone signal (PQS)) act as intercellular signaling molecules in quorum sensing of *Pseudomonas aeruginosa*.[7] Despite the biological importance of PQS, to date only few approaches to PQS and the related systems are known.



Endochin



ICI 56,780

Fig. 1. Quinolin-4-one-based antimalarial agents.

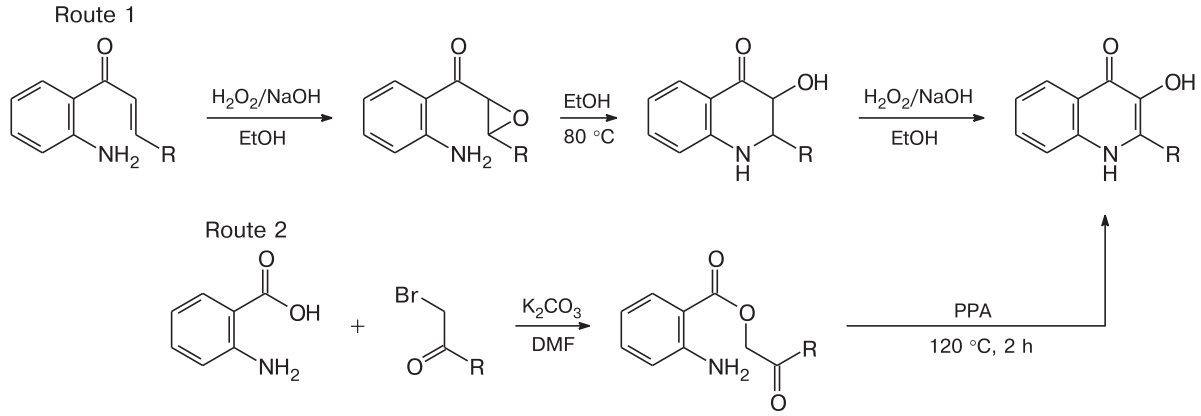
2-Substituted 3-hydroxyquinolin-4-ones are mainly synthesized by two methods. The first method is based on the Algar—Flynn—Oyamada reaction[7—9] and utilizes 2′-aminochalcones instead of 2′-oxychalcones traditionally used in this reaction (Scheme 1, route 1). The second method involves the synthesis of acetonyl and phenyl antranylates followed by their intramolecular cyclization in polyphosphoric acid (PPA) at 120 °C for 1—2 h (Scheme 1, route 2). It is of note that the second procedure was found to be more convenient for the synthesis of 3-hydroxyquinolin-4-ones.[9—14]

The described herein procedure involves acidic hydrolysis of 4-bromo-3-hydroxyquinolines **3** that can be accessed by bromination of 3-hydroxyquinolines **2** (Scheme 2). Synthesis of compounds **2** by reductive cyclization of [2-(2-nitrophenyl)oxiran-1-yl](aryl(methyl))ketones **1** was reported by us earlier[15] (see Scheme 2).

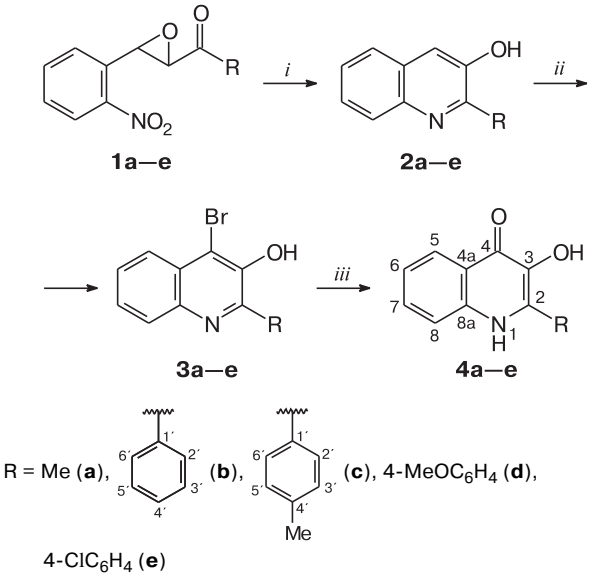
Structure of compound **4d** was evaluated using 1D and 2D NMR experiments (COSY, HSQC (^{1}H—^{13}C), HMBC (^{1}H—^{13}C, ^{1}H—^{15}N), and HSQC (^{1}H—^{15}N)). The presence of the methoxy group in compound **4d**, makes it easy starting from its signal (δ_H 3.85 and δ_C 55.3) to successively attribute all the signals of the aryl substituent using HMBC NMR experiments. In the NMR spectra, it is possible to unambiguously identify the doublet of the H(8) quinoline proton showing a cross-peak with the N(1) atom ($\delta_{^{15}N}$ 125.8) and the doublet of the H(5) proton that couples with the C(5) atom (δ_H 8.14 and δ_C 124.3). At the same time, the ^{1}H—^{13}C HMBC spectrum exhibits the cross-peak

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 1020—1024, May, 2019.

Scheme 1



Scheme 2

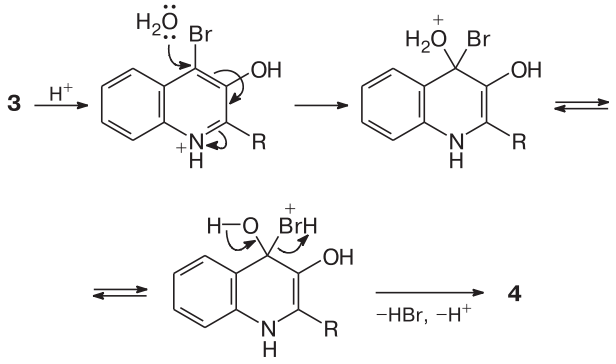


Reagents and conditions: *i*. $Na_2S_2O_4$ (75 mol.%), dioxane, water, reflux, 3 h; *ii*. Br_2, MeOH, reflux, 3 h; *iii*. AcOH, water, reflux, 24 h.

between the H(5) atom and the carbonyl carbon (δ_C 169.3) thus confirming location the latter at the position 4 of the quinoline ring. Finally, the fragments are combined together by the cross-peak between the NH proton and the C(3) atom (δ_C 137.4). It is notable that the chemical shift of the C(3) atom is characteristic of this atom bearing the OH group. The similarity of the ^{1}H NMR spectra of all the synthesized compounds **4** was considered a good reason to assign to compounds **4a**—**c**,**e** the structures similar to that of **4d**.

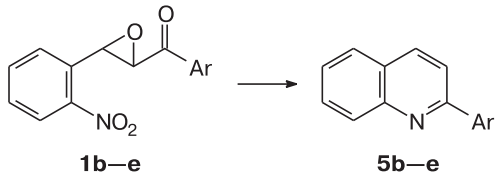
The mechanism providing 3-hydroxyquinolin-4(1*H*)-ones **4** *via* acidic hydrolysis of 4-bromo-3-hydroxyquinolines **3** is shown in Scheme 3.

Scheme 3



When we repeated the synthesis of quinolin-3-ols **2** (see Scheme 2) for their use in further transformations, we found that even at insignificant changes in the reaction conditions quinolines **5b**—**e** are formed along with the target compounds **2b**—**e** (Scheme 4). The only exception was compound **1a** bearing methyl group at the position 2 that produces no quinoline **5a** under the conditions used. It should be emphasized that no compounds **5b**—**e** are formed at the prolonged reflux of quinolines **2b**—**e** with sodium dithionite in water—dioxane.

Scheme 4



Ar = Ph (**b**), 4-MeC_6H_4 (**c**), 4-$MeOC_6H_4$ (**d**), 4-ClC_6H_4 (**e**)

Reagents and conditions: $Na_2S_2O_4$ (97 mol.%), dioxane, water, reflux, 12 h.

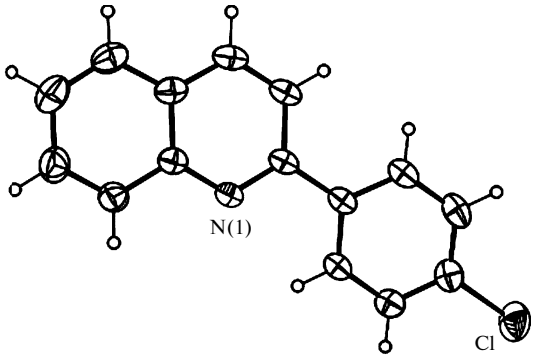


Fig. 2. ORTEP representation of the X-ray crystal structure of 2-(4-chlorophenyl)quinoline (**5e**) (thermal ellipsoids are drawn at 30% probability level).

Structure of compound **5b** was established by NMR experiments. Structure of compound **5e** was studied by X-ray diffraction analysis (Fig. 2). The single crystal X-ray diffraction experiments reveal that in the crystal of **5e** the planes of chlorophenyl and quinoline ring system are slightly twisted with respect to each other with a dihedral angle of 10.20(7)°.

A plausible mechanism of transformation of oxiranes **1** to compounds **5** is shown in Scheme 5. This transformation can be initiated by either reduction of the nitro group or the Meinwald rearrangement[16,17] followed by the nitro group reduction. The Meinwald rearrangement of compounds **1** and **A** can occur *via* the migration of both 1-positioned and 2-positioned hydrogen atoms of the oxirane ring (see Scheme 5). In the first case, 1,2-diketones (**B** and **C**) are formed; the second case gives rise to 1,3-diketones. The formation of hydroxyquinolines **2** (see Scheme 2) can be rationalized only if the first step of the reaction gives rise to 1,2-diketones.[15] For quinolines **5** to be formed, the direction of the oxirane ring opening is insignificant, but we prefer the first possibility and explain the formation of compounds **5** along with hydroxyquinolines **2** by the change in the reaction conditions rather than the competitive reactions of the oxirane ring opening. We are in favor of this opinion due to the absence of even trace amounts of quinoline **5a** in the reaction mixture obtained by the reaction of [2-(2-nitrophenyl)oxiran-1-yl]methylketone (**1a**) with sodium dithionite and subsequent aqueous workup. We believe that the replacement of the Me group by the aryl substituent should not exert such a strong effect on the formation of 1,3-diketones if the first reaction step is the oxirane ring opening.

The synthesis of 2-arylquinolines is often based on the modified Friedländer reaction[18—24] that proceeds mainly in the presence of metal catalyst. Due to the growing demand for the quinoline derivatives in pharmaceutical industry, there is a continuous search for new synthetic approaches to 2-arylquinolines.[25—32] Therefore, the developed by us synthesis of 2-arylquinolines is of interest because of the availability of the starting materials, easiness of both the synthetic procedure and isolation of the target product. The moderate yields of the target quinolines **5** are compensated by the simultaneous formation of other pharmacologically valuable compounds, 3-hydroxy-2-arylquinolines **2**.

In summary, we succeeded in developing the simple synthesis of pharmacologically useful substances, *i.e.*, 2-substituted 3-hydroxyquinolin-4-ones and 2-arylquinolines, from readily available starting materials.

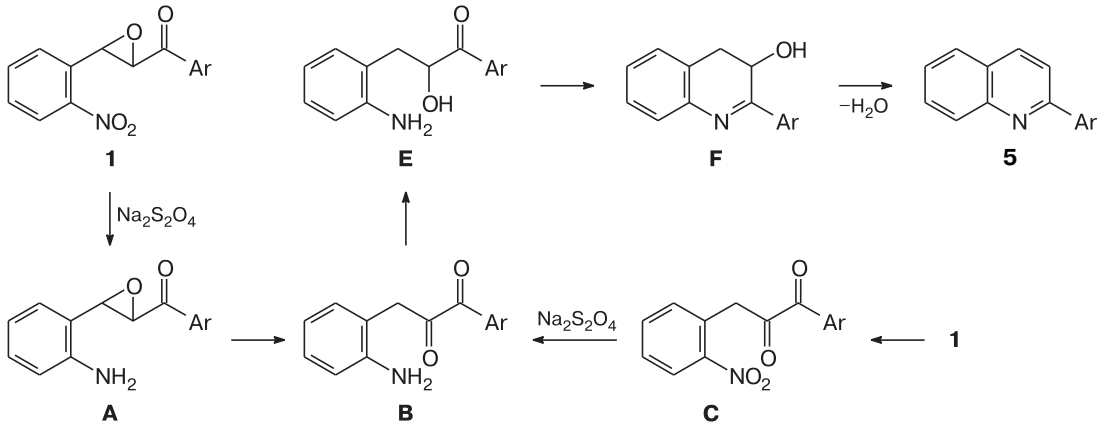
Experimental

Melting points were measured with a Stuart SMP-10 apparatus. IR spectra were recorded with a Bruker Vector-22 spectrometer in KBr pellets. ^{1}H, ^{13}C, and ^{15}N NMR spectra were run on a Bruker AVANCE III-500 instrument (working frequencies of 500.1 (^{1}H), 125.8 (^{13}C), and 50.7 (^{15}N) MHz) in DMSO-d_6.

Synthesis and physicochemical properties of compounds **1a—e**, **2a—e**, and **3a—e** were published by us earlier.[15]

Synthesis of 3-hydroxy-2-methyl(aryl)quinolin-4-ones 4a—e (general procedure). A solution of compound **3** (2 mmol) in

Scheme 5



a mixture of AcOH (50 mL) and water (0.1 mL, 5.5 mmol) was refluxed for 24 h and poured into water (150 mL). After 24 h, the precipitate formed was collected by filtration, washed with water, dried, and washed with ethanol.

3-Hydroxy-2-methylquinolin-4(1*H*)-one (4a). Yield 0.18 g (52%). M.p. 293—294 °C (*cf.* Ref. 11: 298—306 °C). Found (%): C, 68.71; H, 5.23; N, 8.31. $C_{10}H_9NO_2$. Calculated (%): C, 68.56; H, 5.18; N, 8.00. IR, ν/cm^{-1}: 3255, 2919, 2836 (NH, OH), 1639 (C=O), 1551, 1488 (C=C). ^{1}H NMR, δ: 2.40 (s, 3 H, Me); 7.22, 7.56 (both dd, 1 H each, H(6) and H(7), J = 7.5 Hz, J = 7.4 Hz); 7.55, 8.10 (both d, 1 H each, H(8) and H(5), J = 7.5 Hz, J = 7.4 Hz); 11.86 (br.s, NH).

3-Hydroxy-2-phenylquinolin-4(1*H*)-one (4b). Yield 0.25 g (53%). M.p. 270—271 °C (*cf.* Ref. 3: 265—275 °C; *cf.* Ref. 8: 260—262 °C; *cf.* Refs 10, 12: 278—281 °C; *cf.* Ref. 13: 275—278 °C). Found (%): C, 76.07; H, 4.32; N, 5.74. $C_{15}H_{11}NO_2$. Calculated (%): C, 75.94; H, 4.67; N, 5.90. IR, ν/cm^{-1}: 3240, 2925, 2854 (NH, OH), 1633 (C=O), 1547, 1485 (C=C). ^{1}H NMR, δ: 7.36, 7.72 (both dd, 1 H each, H(6) and H(7), J = 7.7 Hz, J = 7.4 Hz); 7.45—7.59 (m, 5 H, Ph); 7.84, 8.25 (both d, 1 H each, H(8) and H(5), J = 7.8 Hz, J = 7.4 Hz); 11.59 (br.s, NH).

3-Hydroxy-2-(4-methylphenyl)quinolin-4(1*H*)-one (4c). Yield 0.26 g (51%). M.p. 269—272 °C. Found (%): C, 76.23; H, 5.42; N, 5.31. $C_{16}H_{13}NO_2$. Calculated: C, 76.47; H, 5.22; N, 5.57. IR, ν/cm^{-1}: 3300, 2922, 2854 (NH, OH), 1634 (C=O), 1547, 1485 (C=C). ^{1}H NMR, δ: 2.39 (s, 3 H, Me); 7.26, 7.58 (both dd, 1 H each, H(6) and H(7), J = 8.0 Hz, J = 7.9 Hz); 7.38, 7.71 (both d, 2 H each, Ar, J = 8.7 Hz); 8.14 (d, 2 H, H(8) and H(5), J = 7.9 Hz); 11.50 (br.s, NH).

3-Hydroxy-2-(4-methoxyphenyl)quinolin-4(1*H*)-one (4d). Yield 0.29 g (55%). M.p. 273—274 °C. Found (%): C, 71.77; H, 4.77; N, 5.21. $C_{16}H_{13}NO_3$. Calculated (%): C, 71.89; H, 4.91; N, 5.24. IR, ν/cm^{-1}: 3266, 2955 (NH, OH), 1634 (C=O), 1547, 1487 (C=C). ^{1}H NMR, δ: 3.85 (s, 3 H, OMe); 7.13, 7.78 (both d, 2 H each, Ar, J = 8.7 Hz); 7.28, 7.58 (both dd, 1 H each, H(6) and H(7), J = 7.4 Hz, J = 7.2 Hz); 7.72, 8.14 (both d, 1 H each, H(8) and H(5), J = 7.4 Hz, J = 7.2 Hz); 11.51 (br.s, NH). ^{13}C{^{1}H} NMR, δ: 169.3 (C(4)), 160.0 (C(4′)), 137.8 (C(8a)), 137.4 (C(3)), 131.8 (C(2)), 130.6 (C(2′), C(6′)), 130.4 (C(7)), 124.3 (C(1′)), 124.3 (C(5)), 121.9 (C(6)), 121.5 (C(4a)), 118.4 (C(8)), 113.7 (C(3′), C(5′)), 55.3 (OCH_3). ^{15}N NMR, δ: 125.8.

2-(4-Chlorophenyl)-3-hydroxyquinolin-4(1*H*)-one (4e). Yield 0.32 g (58%). M.p. 289—291 °C (*cf.* Ref. 10: 291.5—294 °C). Found (%): C, 66.38; H, 3.52; Cl, 13.22; N, 5.28. $C_{15}H_{10}ClNO_2$. Calculated (%): C, 66.31; H, 3.71; Cl, 13.05; N, 5.16. IR, ν/cm^{-1}: 3276, 2958 (NH, OH), 1637 (C=O), 1548, 1492 (C=C). ^{1}H NMR, δ: 7.27, 7.60 (both dd, 1 H each, H(6) and H(7), J = 7.6 Hz, J = 7.5 Hz); 7.63, 7.84 (both dd, 2 H each, Ar, J = 8.4 Hz); 7.71, 8.14 (both d, 1 H each, H(8) and H(5), J = 7.6 Hz); 11.57 (br.s, NH).

Synthesis of 2-arylquinolines 5b—e (general procedure). To a solution of [2-(2-nitrophenyl)oxiran-1-yl]arylketone **1b—e** (3 mmol) in dioxane (200 mL), a solution of $Na_2S_2O_4$ (5.22 g, 90 mmol) in water (300 mL) was added. The reaction mixture was refluxed for 12 h and poured into water. After 12 h, the precipitate formed was collected by filtration, washed with water, and dried. According to ^{1}H NMR data, the obtained solid was a mixture of compounds **2** and **5** in a ~3 : 1 ratio. Compounds **2** and **5** were separated by silica gel column chromatography (silica gel 40 A, elution with $CHCl_3$).

2-Phenylquinoline (5b). Yield 0.13 g (21%). M.p. 82 °C (*cf.* Ref. 14: 82—83 °C; *cf.* Ref. 15: 85—87 °C; *cf.* Ref. 19: 80—82 °C; *cf.* Ref. 27: 81—82 °C). Found (%): C, 87.53; H, 5.48; N, 6.57. $C_{15}H_{11}N$. Calculated (%): C, 87.77; H, 5.40; N, 6.82. IR, ν/cm^{-1}: 1596, 1491 (C=C, C=N). ^{1}H NMR, δ: 7.53 (dd, 1 H, H(4′), J = 7.5 Hz, J = 7.5 Hz); 7.57 (dd, 2 H, H(3′) and H(5′), J = 7.6 Hz, J = 7.5 Hz); 7.63, 7.83 (both ddd, 1 H each, H(6) and H(7), J = 7.7 Hz, J = 7.7 Hz, J = 1.1 Hz); 8.03, 8.11 (both d, 1 H each, H(5) and H(8), J = 7.6 Hz); 8.14, 8.53 (both d, 1 H each, H(3) and H(4), J = 8.6 Hz); 8.22 (dd, 2 H, H(2′) and H(6′), J = 7.7 Hz, J = 1.1 Hz). ^{13}C{^{1}H} NMR, δ: 156.2 (C(2)), 147.3 (C(8a)), 138.7 (C(1′)), 137.4 (C(4)), 130.1 (C(7)), 129.7 (C(4′)), 129.1 (C(8)), 129.0 (C(3′), C(5′)), 127.9 (C(5)), 127.3 (C(2′), C(6′)), 127.1 (C(4a)), 126.6 (C(6)), 119.9 (C(3)). ^{15}N NMR, δ: 301.2. Product **2b** (0.46 g (69%)) was isolated along with compound **5b**.

2-(4-Tolyl)quinoline (5c). Yield 0.12 g (19%). M.p. 85 °C (*cf.* Ref. 14: 83—84 °C; *cf.* Ref. 15: 80—82 °C; *cf.* Ref. 19: 80—81 °C; *cf.* Ref. 27: 82—83 °C). Found (%): C, 87.33; H, 6.08; N, 6.63. $C_{16}H_{13}N$. Calculated (%): C, 87.64; H, 5.98; N, 6.39. IR, ν/cm^{-1}: 1596, 1498 (C=C, C=N). ^{1}H NMR, δ: 2.38 (s, 3 H, Me); 7.35, 8.17 (both d, 2 H each, Ar, J = 7.8 Hz); 7.58, 7.77 (both dd, 1 H each, H(6) and H(7), J = 7.9 Hz, J = 7.9 Hz); 7.97, 8.41 (both d, 1 H each, H(5) and H(8), J = 8.2 Hz); 8.06, 8.09 (both d, 1 H each, H(3) and H(4), J = 8.0 Hz). Product **2c** (0.49 g (69%)) was isolated along with compound **5c**.

2-(4-Methoxyphenyl)quinoline (5d). Yield 0.14 g (20%). M.p. 123—124 °C (*cf.* Ref. 14: 123—124 °C; *cf.* Ref. 15: 117—120 °C; *cf.* Ref. 27: 123—124 °C). Found (%): C, 81.39; H, 5.38; N, 6.03. $C_{16}H_{13}NO$. Calculated (%): C, 81.68; H, 5.57; N, 5.95. IR, ν/cm^{-1}: 1596, 1497 (C=C, C=N). ^{1}H NMR, δ: 3.81 (s, 3 H, OMe), 7.09, 8.16 (both d, 2 H each, Ar, J = 8.5 Hz); 7.56, 7.76 (both dd, 1 H each, H(6) and H(7), J = 7.9 Hz, J = 7.9 Hz); 7.94 (d, 1 H, H(5), J = 8.0 Hz), 8.04 (d, 2 H, H(3) and H(8), J = 8.0 Hz); 8.39 (d, 1 H, H(4), J = 8.0 Hz). Product **2d** (0.52 g (70%)) was isolated along with compound **5d**.

2-(4-Chlorophenyl)quinoline (5e). Yield 0.16 g (23%). M.p. 113 °C (*cf.* Ref. 14: 112—113 °C; *cf.* Ref. 15: 110—113 °C; *cf.* Ref. 27: 111—112 °C). Found (%): C, 75.35; H, 4.39; Cl, 15.00; N, 5.99. $C_{15}H_{10}ClN$. Calculated (%): C, 75.16; H, 4.21; Cl, 14.79; N, 5.84. IR, ν/cm^{-1}: 1595, 1487 (C=C, C=N). ^{1}H NMR, δ: 7.61, 8.31 (both d, 2 H each, Ar, J = 8.6 Hz); 7.61, 7.79 (both ddd, 1 H each, H(6) and H(7), J = 8.4 Hz, J = 7.4 Hz, J = 1.4 Hz); 8.01, 8.07 (both d, 1 H each, H(5) and H(8), J = 7.4 Hz, J = 8.4 Hz); 8.16, 8.47 (both d, 1 H each, H(3) and H(4), J = 8.6 Hz). Product **2e** (0.52 g (68%)) was isolated along with compound **5e**.

X-ray diffraction study of compound 5e. A single crystal of compound **5e** was grown by slow evaporation of its acetone solution at room temperature. The X-ray diffraction data were collected at 293(2) K with a Bruker KAPPA APEX II diffractometer equipped with a four circle CCD detector (graphite monochromator, λ(Mo-$K\alpha$) = 0.71073 Å, ω scan mode). APEX3 v2015.9-0 (Bruker AXS) program package was used for data collection and indexing, structure solution and refinement of the single cell parameters, semi-empirical correction accounting (using equivalent reflections based on the Laue group), accounting for the systematic errors, and determination of the space group of the crystal. The structure was solved by a direct method using SHELXT-2018/2 software[33] and refined by a full matrix least squares method on F^2 using SHELXL-2018/3 software.[34] All nonhydrogen atoms were refined anisotropically. Hydrogen atoms were positioned geometrically and refined using a riding model. All calculations were performed with WinGX-2018.3 program package.[35]

Compound **5e** ($C_{15}H_{10}ClN$, M = 239.69) crystallizes in the monoclinic $P2_1/c$ space group. The single cell parameters are as follows: a = 13.2578(11) Å, b = 14.8327(12) Å, c = 6.0085(5) Å, β = 92.120(4)°; V = 1180.76(17) Å^3; Z = 4, Z' = 1. Colorless prism (0.584×0.471×0.318 mm^3); d_{calc} = 1.348 g cm^{-3}, μ(Mo-$K\alpha$) = = 0.297 mm^{-1}; $F(000)$ = 496; T_{max} = 0.6463, T_{min} = 0.5618. The data collection range is 3.148° ⩽ θ ⩽ 25.249° (−15 ⩽ h ⩽ 15, −17 ⩽ k ⩽ 17, −6 ⩽ l ⩽ 7), the completeness of data collection to θ_{max} is 99.4%; 7687 reflections were collected including 2124 unique reflections; R_{int} = 0.0264, R_σ = 0.0266. Refinement of 154 parameters gives R_1 = 0.0434, wR_2 = 0.1109 for 1597 reflections with $I > 2\sigma(I)$ and R_1 = 0.0616, wR_2 = 0.1244 for all collected reflections, S = 1.038, residual electron density is ρ_{max}/ρ_{min} = 0.209/−0.297 e Å^{-3}.

The complete data on the structure of compound **5e** is deposited with Cambridge Crystallographic Data Center with CCDC 1891235 and available free of charge *via* http://www.ccdc.cam.ac.uk.

Physicochemical studies were performed at the Collective spectral and analytical Center for physicochemical studies of the structure, properties and composition of compounds and materials of FRC Kazan Scientific Center of the Russian Academy of Sciences.

This work was partially financially supported by the Russian Science Foundation (Project No. 18-13-00315).

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Received January 24, 2019;
accepted March 7, 2019