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Synthesis of 2-(2-Methoxyphenyl)-4-methyl-6-nitroquinoline and 2-(2-Methoxyphenyl)-4-methylquinolin-6-amine

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Abstract

A new compound, 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline, was obtained via Suzuki reaction. An unexpected side product, 2-(2-methoxyphenyl)-4-methylquinolin-6-amine, was isolated. The structures of the novel compounds were confirmed by ^{1}H, ^{13}C and 2D-NMR. Their optical properties were also studied.

Keywords: heterocyclic compounds; nitroquinoline; 2-phenyllepidine; pH-sensitive fluorescence; Suzuki reaction

1. Introduction

In recent decades heterocyclic compounds bearing 2-phenylquinoline moiety attract attention due to their various applications. This system is used in coordination chemistry as a ligand in copper [1], platinum [2] and especially iridium complexes, which are usually fluorescent and demonstrate neuroimaging potential [3], enzyme inhibition activity [4], intracellular nitric oxide sensing ability [5], long-lifetime, deep-red emission with high color purity [6], luminescent biosensor potential [7], and anticancer activity [8]. The 2-phenylquinoline moiety is found also in fluorescent N,O-bidentate difluoroboron complexes [9] and in covalent organic frameworks (COFs) [10]. The bioactivity of the molecules bearing 2-phenylquinoline backbone is also of great importance: they possess antifungal [11] and anti-inflammatory properties [12], anticoronavirus [13] and anticancer activity [14,15], as well as gastroprotective properties [16].

Herein, we present the synthetic protocol for obtaining 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1**, a new compound and the first reported 2-phenylquinoline derivative, substituted with a NO_2-group in the sixth position of the quinoline system and with a CH_3O-group in the second position of the phenyl substituent. It should be noted that the methyl substituent in the fourth position could be synthetically useful—4-methylquinolines can serve as a starting material for monomethine cyanine dyes after N-alkylation of the quinoline nitrogen atom [17,18]. During the synthesis of the title compound, the formation of an interesting side product was observed, which was recognized as 2-(2-methoxyphenyl)-4-methylquinolin-6-amine **2**. It demonstrates fluorescence, which is sensitive to the acidity of the solution. The optical properties of both compounds were studied.

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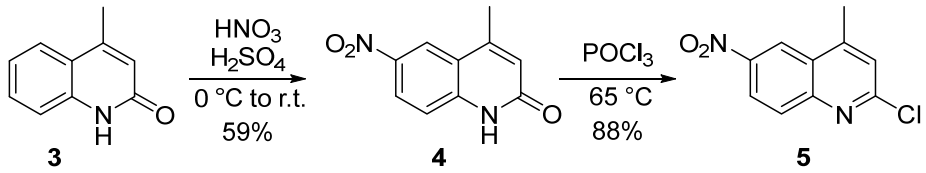
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2. Results and Discussion

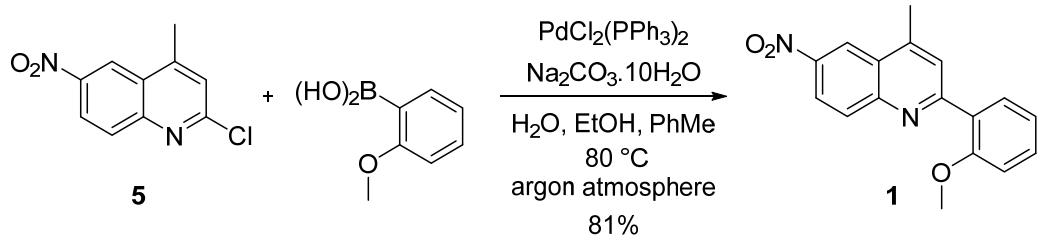
*2.1. Synthesis of 2-(2-Methoxyphenyl)-4-methyl-6-nitroquinoline **1***

The synthesis of the title compound, 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1**, included several steps and started with the preparation of 2-chloro-4-methyl-6-nitroquinoline **5** (Scheme 1). Firstly, 4-methylquinolin-2-one **3** [19] was nitrated and later the resulting 4-methyl-6-nitroquinolin-2-one **4** was treated with $POCl_3$ [19] in order to obtain 2-chloro-4-methyl-6-nitroquinoline **5**.



Scheme 1. Synthesis of 2-chloro-4-methyl-6-nitroquinoline **5**.

The purified 2-chloro-4-methyl-6-nitroquinoline **5** was reacted with (2-methoxyphenyl) boronic acid in the presence of excess amount of sodium carbonate and 3 mol % of $PdCl_2(PPh_3)_2$ following standard Suzuki reaction protocol (Scheme 2).



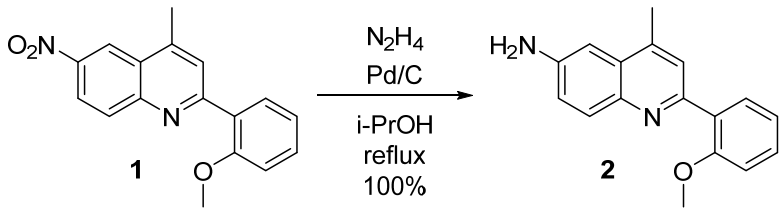
Scheme 2. Synthesis of 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1**.

The target compound **1** was obtained, purified and isolated in high yield in the form of pale-yellow needles.

During TLC monitoring of the reaction progress, the formation of a small quantity (18.5 mg or 7%) of a side product was observed. This compound demonstrates intense yellow fluorescence. Surprisingly, during the reaction work-up, not yellow, but blue fluorescence was observed in solution.

It was assumed that the observed fluorescence could be a result of the presence of small amounts of 2-(2-methoxyphenyl)-4-methylquinolin-6-amine **2**, a consequence of the NO_2-group reduction in compound **1**.

To confirm this assumption, 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1** was reacted with hydrazine in the presence of palladium on carbon (Scheme 3). Both products—from the reduction and the side product of the Suzuki coupling reaction—were compared by their chromatographic properties and NMR-spectra. That unequivocally proved the above-mentioned assumption.



Scheme 3. Synthesis of 2-(2-methoxyphenyl)-4-methylquinolin-6-amine **2**.

We can suppose three plausible mechanisms describing the formation of the amino compound **2**. First, the palladium catalyst can react with ethanol in the presence of a base, leading to the formation of acetaldehyde and a palladium hydride complex. This complex can react with a second ethanol molecule, releasing more acetaldehyde and even hydrogen. Similar dehydrogenation of alcohols to aldehydes in the presence of palladium complex and phosphorus ligand at 100 °C is previously reported [20]. The Pd-H-complex is a strong reductor and can transform the NO_2- into NH_2-group. On the other hand, the released hydrogen can be adsorbed by the fine palladium particles, which are a result of $PdCl_2(PPh_3)_2$ decomposition, and thus the activated hydrogen can reduce the nitro compound **1** to the side product **2**.

According to our second theory, ethanol is dehydrogenated by the palladium particles, produced by decomposition of $PdCl_2(PPh_3)_2$. The produced hydrogen and the palladium particles act as a catalytic system for NO_2-group reduction. The dehydrogenation of alcohols on a palladium surface [21] or on palladium nanoparticles in the presence of a base [22] is possible at elevated temperatures.

The third possible explanation of the observation of a side product is the direct reduction [23] of the NO_2-group by PPh_3 in the presence of boronic acid and transition metal complex. Probably, this P(III) compound dissociates from the palladium atom in the catalyst and is oxidized by the NO_2-group to triphenylphosphine oxide.

2.2. NMR Studies

The purity and the structure of compounds **1**, **2** and **5** were confirmed by NMR-spectroscopy. Every signal, observed in their ^{1}H- and ^{13}C-spectra, was assigned to a specific atom, using DEPT-135, COSY, HSQC and HMBC techniques. See Supplementary Materials for copies of the NMR spectra.

In the ^{1}H-NMR spectrum of **1**, two intense singlets were observed, which correspond to the methyl and methoxy group in the quinoline and in the phenyl fragment, respectively. The CH_3O-signal was downshifted in comparison with the CH_3-signal because of the electronegativity of the oxygen atom. If compared, the spectra of 2-chloro-4-methyl-6-nitroquinoline **5** and 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1** show that only the signal of the proton in position 3 in the quinoline system is influenced by the introduction of the 2-CH_3OPh substituent—it was downshifted from 7.34 ppm to 7.83 ppm. The ^{13}C-NMR spectrum of **1** shows the same phenomenon that occurs for the carbon atom in position 2.

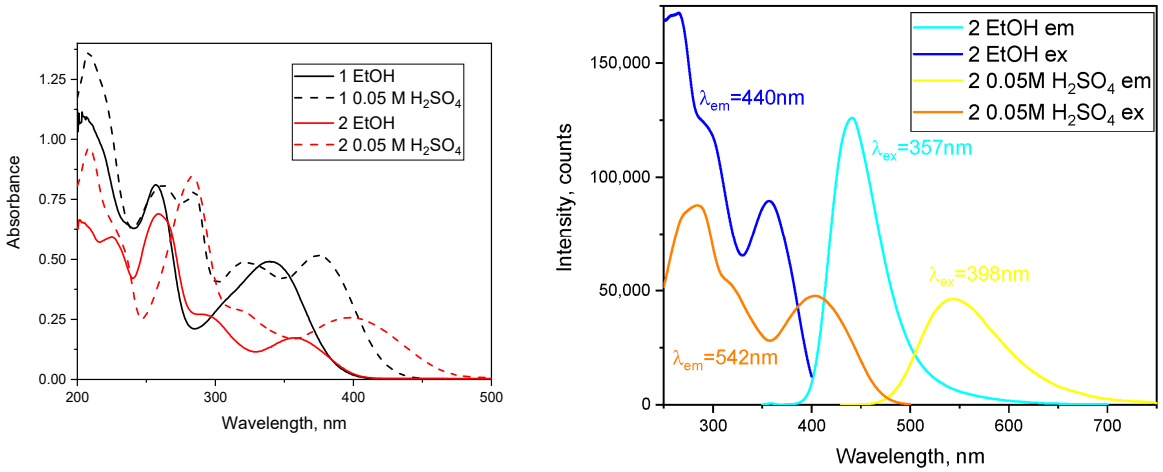
In the ^{1}H-NMR spectrum of 2-(2-methoxyphenyl)-4-methylquinolin-6-amine **2**, a broad singlet was observed due to the presence of an NH_2-group in position 6. In a result of the lack of the electron-withdrawing NO_2-group, the signals of the protons in the third, and especially in the fifth and in the seventh positions of the quinoline system were shifted to upper field.

The reduction of the NO_2-group affected most strongly the detected signal of the carbon atom in position 5: its signal was shifted from 120.8 ppm in the spectrum of nitro compound **1** to 104.06 ppm in the spectrum of amine **2**. In the same way, but not that significantly, the chemical shifts for carbon atoms in positions 2, 4 and 8a were affected.

2.3. Optical Studies

The UV-VIS spectra of the two synthesized compounds, recorded in EtOH and 0.05 M H_2SO_4 media, are shown in Figure 1. In the UV spectrum of compound **1** in EtOH, absorption maxima are observed at 205, 257 and 338 nm, which could be attributed to the characteristic π-π* electronic transitions of benzene and quinoline chromophores. In acidic media (0.05 M H_2SO_4), a bathochromic shift is observed due to the protonation of the quinoline nitrogen atom, as four maxima at 262, 284, 321 and 374 nm are visible. Such a

shift is expected and is observed in other quinoline derivatives [24,25]. Upon protonation, a positive charge occurs, which modifies the electronic structure, resulting in lowering the energy gap between the molecular orbitals involved in the transition. In addition, the protonation increases the polarity of the molecule, leading to stabilization of the excited state to a greater extent compared to the ground state.



(**a**) (**b**)

Figure 1. (**a**) UV-VIS of 2-(2-methoxyphenyl)-4-methyl-6-nitroquinoline **1** (3.2×10^{-5} M) and 2-(2-methoxyphenyl)-4-methylquinolin-6-amine **2** (3.4×10^{-5} M); and (**b**) luminescence and excitation spectra of 2 in EtOH and 0.05 M H_2SO_4. The excitation and emission wavelengths are shown on the spectra.

The reduction of the nitro group to an amino group results in a bathochromic shift in the UV-VIS spectrum compared to compound **1**, with absorption maxima at 213, 259, 357 nm and a shoulder at 322 nm (Figure 1). Upon protonation, similarly to compound **1**, a further shift to longer wavelengths is observed, giving rise to absorption bands at 210, 283 and 398 nm.

The molar absorptivities in EtOH of **1** are—24,265 $M^{-1}.cm^{-1}$ at 257 nm and 14,690 $M^{-1}.cm^{-1}$ at 338 nm, respectively (Table 1 and Figure S22). For compound **2**, the absorptivities in the same solvent are 20,241 $M^{-1}.cm^{-1}$ at 259 nm and 5168 $M^{-1}.cm^{-1}$ at the longest wavelength maximum, 357 nm.

Table 1. Summary of the photophysical data of compounds **1** and **2**.

Solvent	λ_{abs}, nm [1] Compounds 1/2	λ_{em}, nm [2] Compound 2	Stokes Shift, cm^{-1} Compound 2	Quantum Yield Compound 2
EtOH	338/357	440	5284	72.9%
0.05 M H_2SO_4	374/398	542	6675	34.3%

[1] The longest wavelength maximum. [2] Excitation at the longest wavelength maximum in the UV-VIS spectrum in the corresponding solvent.

The fluorescence spectrum of compound **2** in EtOH shows an emission maximum at 440 nm, corresponding to blue fluorescence (Figure 1). In 0.05 M H_2SO_4, a significant bathochromic shift of approximately 100 nm to 542 nm is observed and the compound exhibits yellow fluorescence. Also, the emission intensity and efficiency are reduced in acidic conditions. The excitation spectra suggest a few possible channels corresponding well to the absorption maxima of the samples. The quantum yields for compound **2** are 72.9% and 34.3% for EtOH and 0.05 M H_2SO_4 solutions, respectively. The concentration of compound **2** for the luminescence measurements is $5 \cdot 10^{-5}$ M.

3. Materials and Methods

UV-Vis spectra were carried out on a Shimadzu UV-1800 spectrophotometer (Kyoto, Japan). The luminescence spectra, excitation spectra and quantum yields were measured on Perkin Elmer FL8500 (Shelton, CT, USA). For the emission and excitation spectra, a single cell holder was used, and for the QY measurements—an integrating sphere coated with Spectralon® (North Sutton, NH, USA) [26]. Excitation and emissions slits were set to 5 nm, and the scan speed was 240 nm/min for all measurements. The emission spectra and QY were measured with excitation wavelength equal to the longest wavelength absorption maxima of each sample—357 nm and 398 nm for ethanolic solution and H_2SO_4 solution, respectively. De Mello's method was used to measure and calculate the quantum yields [27].

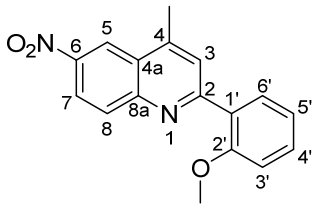
The IR spectra were recorded with a Shimadzu FTIR-8400S spectrophotometer (Kyoto, Japan). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 500 spectrometer (Rheinstetten, Germany) (at 500 MHz for ^{1}H and 126 MHz for ^{13}C, respectively). Chemical shifts are given in ppm. In the ^{13}C-NMR spectra, "^{4}C" denotes carbon atoms that are quaternary and not bonded to a hydrogen atom. The NMR spectra of compounds **1**, **2** and **5** can be found in the Supplementary Materials. Reactions were monitored by TLC on silica gel 60 F_{254}. Melting points were determined on an SRS MPA120 EZ-Melt apparatus (Sunnyvale, CA, USA) and are used without correction.

2-Methoxyphenylboronic acid was purchased from Fluorochem (Cork, Ireland). All solvents (ethanol, toluene, dichloromethane, hexanes, isopropanol, ethyl acetate and chloroform) and inorganics ($PdCl_2(PPh_3)_2$, $Na_2CO_3.10H_2O$, Na_2SO_4, N_2H_4 and palladium on carbon) were purchased from local suppliers and were used without further purification.

*3.1. Synthesis of 2-(2-Methoxyphenyl)-4-methyl-6-nitroquinoline **1***

In a Schenk tube, equipped with a stirring bar, 2-chloro-4-methyl-6-nitroquinoline (0.223 g, 1 mmol), (2-methoxyphenyl)boronic acid (0.182 g, 1.2 mmol), $Na_2CO_3.10H_2O$ (1.145 g, 4 mmol), $PdCl_2(PPh_3)_2$ (0.023 g, 0.03 mmol), water (2 mL), ethanol (1 mL) and toluene (2 mL) were placed. The vessel was purged with argon and heated at 80 °C while stirring for 36 h. After cooling to room temperature, the mixture was extracted with dichloromethane. The combined organic layers were dried with Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by flash column chromatography on silica (dichloromethane:hexanes = 2:1). Yield: 0.238 g (81%) of yellow needles, mp 194.5–195.5 °C, R_f (silica) = 0.21 (hexanes-dichloromethane, 1:2)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.88 (d, *J* = 2.5 Hz, 1H, ***H5***), 8.36 (dd, *J* = 9.2, 2.5 Hz, 1H, ***H7***), 8.16 (d, *J* = 9.2 Hz, 1H, ***H8***), 7.83 (s, 1H, ***H3***), 7.79 (dd, *J* = 7.6, 1.7 Hz, 1H, ***H6′***), 7.38 (td, *J* = 8.5, 1.7 Hz, 1H, ***H4′***), 7.06 (td, *J* = 7.4, 0.5 Hz, 1H, ***H5′***), 6.98 (d, *J* = 8.3 Hz, 1H, ***H3′***), 3.82 (s, 3H, CH_3O), 2.74 (d, *J* = 0.5 Hz, 3H, CH_3). ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.44 (***C2***), 157.36 (***C2′***), 150.54 (***^{4}C8a***), 145.26 (***C4***), 144.96 (***C6***-NO_2), 131.78 (***C8***), 131.54 (***C6′***), 131.21 (***C4′***), 128.71 (***C1′***), 126.17 (***^{4}C4a***), 125.70 (***C3***), 122.44 (***C7***), 121.37 (***C5′***), 120.80 (***C5***), 111.52 (***C3′***), 55.70 (CH_3O), 18.97 (CH_3).

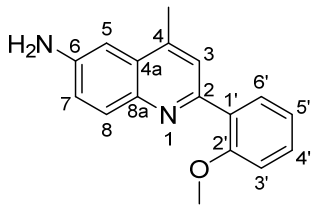


Anal. calcd. %, C, 69.38; H, 4.79; N, 9.52; O, 16.31, found % 69.43; H, 4.71; N, 9.99; O, 16.21.

3.2. Synthesis of 2-(2-Methoxyphenyl)-4-methylquinolin-6-amine **2**

2-(2-Methoxyphenyl)-4-methyl-6-nitroquinoline (0.380 g, 1.3 mmol), 0.038 g Pd/C (10% Pd), 25 mL isopropanol and 1 mL 80% aqueous N_2H_4 were placed in a round-bottom flask, equipped with a stirring bar and a reflux condenser. The dark reaction mixture was stirred under reflux for 2 h, and after cooling to room temperature, the mixture was filtered through celite. The volatiles were removed under reduced pressure, and the crude product was purified by column chromatography on silica (chloroform:ethylacetate). A quantitative yield of brown-red oil was obtained, R_f (silica) = 0.20 (chloroform-ethyl acetate, 3:1). The side product **2** was obtained as a dark oil in 7% yield (18.5 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.9 Hz, 1H, ***H8***), 7.83 (dd, *J* = 7.5, 1.7 Hz, 1H, ***H6′***), 7.60 (s, 1H, ***H3***), 7.39 (td, *J* = 8.3, 1.7 Hz, 1H, ***H4′***), 7.12 (td, *J* = 7.4, 0.5 Hz, 1H, ***H5′***), 7.04 (dd, *J* = 8.9, 2.5 Hz, 1H, ***H7***), 7.00 (d, *J* = 8.2 Hz, 1H, ***H3′***), 6.95 (d, *J* = 2.4 Hz, 1H, ***H5***), 4.02 (broad s, 2H, ***NH₂***), 3.81 (s, 3H, ***CH₃O***), 2.53 (s, 3H, ***CH₃***). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.14 (***C2′***), 153.03 (***C2***), 144.77 (***C6***-NH_2), 142.92 (***C8a***), 140.82 (***C4***), 131.32 (***C6′***), 131.06 (***C8***), 130.23 (***C1′***), 129.78 (***C4′***), 128.55 (***C4a***), 124.09 (***C3***), 121.15 (***C5′***), 121.01 (***C7***), 111.49 (***C3′***), 104.06 (***C5***), 55.68 (***CH₃O***), 18.97 (***CH₃***).

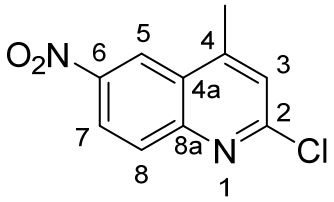


Anal. calcd. %, C, 77.25; H, 6.10; N, 10.60; O, 6.05, found % 77.17; H, 6.16; N, 10.58; O, 6.15.

3.3. Synthesis of 2-Chloro-4-methyl-6-nitroquinoline **5**

Compound **5** was prepared according to a previously reported procedure [19].

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.86 (d, *J* = 2.5 Hz, 1H, ***H5***), 8.43 (dd, *J* = 9.2, 2.5 Hz, 1H, ***H7***), 8.07 (d, *J* = 9.2 Hz, 1H, ***H8***), 7.34 (d, *J* = 0.6 Hz, 1H, ***H3***), 2.73 (d, *J* = 0.9 Hz, 3H, ***CH₃***). ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.44 (***C2***-*Cl*), 149.97 (***^{4}C8a***), 149.66 (***C4***-CH_3), 145.51 (***C6***-NO_2), 130.91 (***C8***), 126.22 (***^{4}C4a***), 124.48 (***C3***), 123.87 (***C7***), 120.84 (***C5***), 18.70 (***C***H_3).



4. Conclusions

A new 2-phenylquinoline, substituted with nitro, methyl and methoxy groups, was obtained. The respective amino compound was isolated as a side product and also obtained by using directed synthesis. The structures and the purity of these two new compounds were confirmed by 1D- and 2D-NMR-spectroscopy and elemental analysis. The new 2-(2-methoxyphenyl)-4-methylquinolin-6-amine demonstrates blue fluorescence in neutral media and yellow fluorescence in acidic solution.

Supplementary Materials: Figures S1–S21: ^{1}H, ^{13}C, DEPT-135, HSQC, HMBC-NMR spectra of compounds **1** and **2**, and ^{1}H, ^{13}C and DEPT-135 spectra of compound **5**. Figure S22: Calibration curves of compounds **1** and **2**. Figure S23: IR spectra of compounds **1** and **2**.

Author Contributions: Conceptualization, R.L.; methodology, R.L., I.Z.K., D.S. and M.I.; validation, I.Z.K., M.I., D.S. and R.L.; investigation, I.Z.K., M.I., D.S. and R.L.; data curation, R.L. and I.Z.K.; writing—original draft preparation, R.L. and I.Z.K.; writing—review and editing, I.Z.K., D.S. and M.I.; visualization, R.L., D.S. and I.Z.K.; supervision, R.L.; project administration, R.L.; funding acquisition, R.L. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Additional research data can be obtained from the corresponding author.

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Conflicts of Interest: The authors declare no conflicts of interest.

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