Short Note

8-(2-Methoxyphenyl)-6-methyl-2-(1-methyl-1Hbenzo[d]imidazol-2-yl)quinoline

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8-(2-Methoxyphenyl)-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline

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Abstract: For very first time, we report the synthesis of 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **1**. This was achieved in several steps, including usage of Suzuki reaction for functionalization of 2-(1H-benzo[d]imidazol-2-yl)quinoline moiety. The new compound exhibits blue fluorescence. Its structure was confirmed with 1D- and 2D-NMR spectroscopy, IR-spectroscopy, high-resolution mass spectrometry and X-ray analysis.

Keywords: quinoline; benzimidazole; fluorescence; single crystal diffraction; Suzuki reaction

1. Introduction

Heterocyclic systems with 2-(*1H*-benzo[d]imidazol-2-yl)quinoline moiety can be intriguing for various fields of chemical research and application. This statement is based on several reasons - first, a great amount of quinoline derivatives exhibit biological activity and are used [1] as antimalarial, antiviral, antibacterial and anticancer pharmaceuticals. Second, benzimidazole derivatives are well known [2,3] for their analgesic, antiproliferative, antidiabetic, anticancer, anti-inflammatory and antioxidant activity. Third, 2-(*1H*-benzo[d]imidazol-2-yl)quinolines were studied [4,5] for cytotoxic, antimicrobial, insecticidal and herbicidal activity. Beyond their biological activity, 2-(1*H*-benzo[d]imidazol-2-yl)quinolines have been used as ligands [6–8], coordinated to transition metals.

Benzoimidazolylquinolines could be prepared by following several main synthetic strategies: from 1) quinaldines and benzene-1,2-diamine in the presence of mild oxidant (elemental suphur [4,9] or I_2/DMSO [5,10]), 2) carbaldehyde [11] or carboxylic acid [6,7] and benzene-1,2-diamine, and 3) trichalomethylquinoline and benzene-1,2-diamine [12]. A similar way could be applied for the synthesis of N-substituted in the benzimidazole system members of this class of compounds [8].

Herein, we report the synthesis of a new fluorescent compound, 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **1**. This is the first benzoimidazolylquinoline representative with aromatic substituent in the quinoline system. Its structure and optical properties were also studied.

2. Results and Discussion

2.1. Synthesis

The synthesis of the new compound, 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **1**, was achieved in several steps (Schemes 1–3). First, 2-bromo-4-methylaniline **2** [13] was cyclized with but-2-enal (crotonaldehyde) to 8-bromo-2,6-dimethylquinoline **3** (Scheme 1). The crude product was purified via its $ZnCl_2$-complex [14], which is

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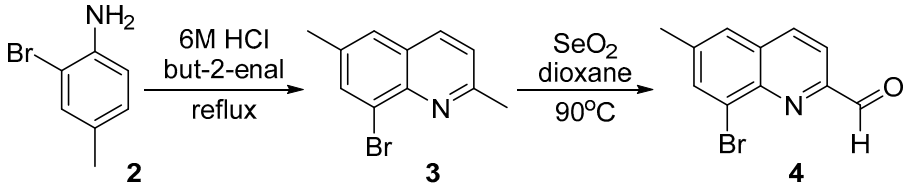
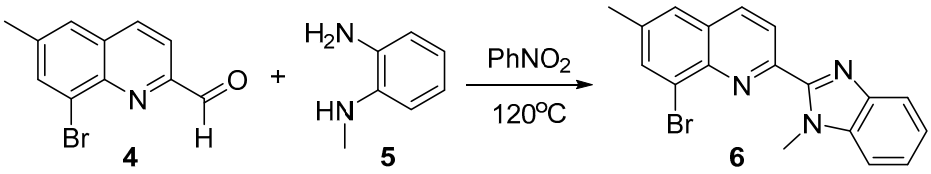
insoluble in water and most organic solvents. This quinoline was reported previously [15], however a detailed procedure was not described. Later the isolated in pure form 8-bromo-2,6-dimethylquinoline **3** was oxidized with selenium dioxide to 8-bromo-6-methylquinoline-2-carbaldehyde **4**, using procedure [16], previously applied for the 8-bromo-2-methylquinoline structure. It seems that these conditions are mild enough to oxidize the methyl group in 2-nd position, but to avoid the oxidation of the same group at 6-th position. Oxidation of the last one is possible, but at harsh reaction conditions [17]. The obtained aldehyde was previously noted in the literature [18], however again the synthetic procedure and compound's physical properties were not reported.



Scheme 1. Synthesis of 8-bromo-2,6-dimethylquinoline **3** and 8-bromo-6-methylquinoline-2-carbaldehyde **4**.

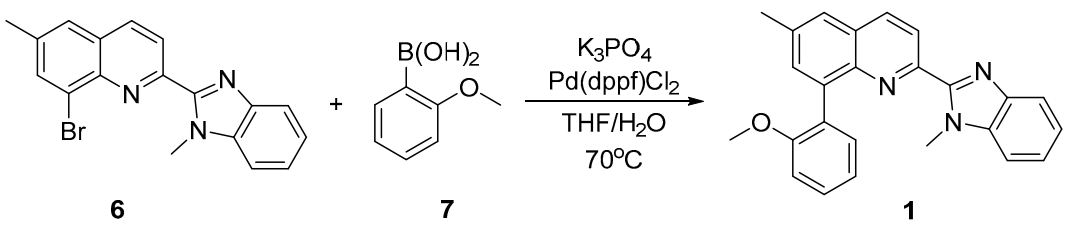
In the subsequent step (Scheme 2), 8-bromo-6-methylquinoline-2-carbaldehyde **4** was reacted with N^1-methylbenzene-1,2-diamine **5** at high temperature in dry nitrobenzene media [19], serving at the same time as a solvent and as a mild oxidant reagent.



Scheme 2. Synthesis of 8-bromo-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **6**.

The resulting 8-bromo-6-methyl-2-(1-methyl-*1H*-benzo[d]imidazol-2-yl)quinoline **6** has not been published in the literature before. Moreover, it is the first heterocyclic system with 2-(1H-benzo[d]imidazol-2-yl)quinoline backbone, bearing bromine atom in the phenyl ring of the quinoline moiety. This structure is of a great importance because such type of halogens could be substituted with different organic groups by transition metal catalyzed reaction as we managed to do later in this research.

In the final step (Scheme 3), 8-bromo-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **6** was used as a substrate in $Pd(dppf)Cl_2$-catalyzed Suzuki reaction [20]. As its coupling partner 2-methoxyphenylboronic acid **7** was used.



Scheme 3. Synthesis of 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-*1H*-benzo[d]imidazol-2-yl)quinoline **1**.

After the performed Suzuki reaction (Scheme 3), 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-*1H*-benzo[d]imidazol-2-yl)quinoline **1** was isolated in pure form. It is a new compound and the first

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reported 8-phenyl substituted 2-(*1H*-benzo[d]imidazol-2-yl)quinoline. Its successful synthesis proves that the bromine atom in position 8-th is reactive in Suzuki reaction. On the other hand, this result demonstrates that $Pd(dppf)Cl_2$ is suitable catalyst for functionalization of benzoimidazolylquinoline system with moderately sterically hindered boronic acids as 2-methoxyphenylboronic acid.

2.2. NMR Studies

All the structures of the obtained compounds were analyzed with NMR spectroscopy, ^{1}H, ^{13}C, COSY, HSQC and HMBC experiments were performed. See Supplementary Materials for copies of the NMR-spectra.

In the proton spectrum of 8-bromo-2,6-dimethylquinoline **3** two signals attributed to the two CH_3-groups and four signals for the aromatic protons were observed (Figures S10 and S11). The signal of the CH_3-group in the 2-nd position was observed in lower field, compared to the signal of the other methyl substituent.

In the proton spectrum of 8-bromo-6-methylquinoline-2-carbaldehyde **4** characteristic signal at 10.20 ppm was observed (Figures S17 and S18), related to the aldehyde group. The same substituent is responsible for the low fielded signal in the ^{13}C-spectrum, observed at 193.46 ppm (Figures S19 and S20). In the proton spectrum (Figures S24 and S25) of 8-bromo-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **6** signals for two methyl groups could be seen, respectively at 2.45 ppm for the CH_3-group from the quinoline and at 4.51 ppm for the CH_3-group from the benzoimidazolyl moiety. The most strongly shifted in low field are the signals in the ^{13}C-spectrum of the compound correspond to the carbon atom in the second positions in the quinoline and benzoimidazolyl systems (Figures S26 and S27). A prove for the successful arylation by the Suzuki reaction could be seen from the proton spectra of 8-(2-methoxyphenyl)-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **1** (Figures S1 and S2) and 8-bromo-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline **6** (Figures S24 and S25). By comparing the data, in the spectra of **1** additional signals for the alkyl protons and the aromatic system were determined.

2.3. Single Crystal Diffraction Studies

Suitable for single crystal x-ray diffraction measurements crystals were obtained by crystallization in acetonitrile. The titled compound **1** crystallizes in the triclinic *P-1* space group with one molecule in the asymmetric. The molecular structure of the compound is shown in Figure 1 and the crystal data is summarized in Table 1. The CCDC deposition file can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033).

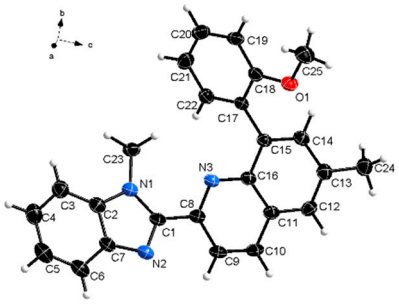

Figure 1. Molecular structure of the titled compound. Thermal ellipsoids are at 50% level.

Table 1. Experimental details of the single crystal diffraction.

Crystal data	
CCDC numbers	2374257
Empirical formula	$C_{25}H_{21}N_3O$

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Molecular weight, (g/mol)	379.45
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	133(2)
a, b, c (Å)	7.0467(5), 10.8556(7), 13.5823(9)
α, β, γ (°)	79.469(6), 79.307(6), 71.684(6)
V, (Å^3)	960.56(12)
Z	2
Radiation type	Mo Kα
μ, (mm^{-1})	0.082
Crystal size (mm)	0.200 x 0.080 x 0.060
Data collection	
Diffractometer	Rigaku XtaLAB Synergy
Absorption correction	Spherical Harmonics implemented in SCALE3 ABSPACK scaling algorithm
T_{min}, T_{max}	0.8728, 1.0000
No. of measured, independent and observed [I > 2σ(I)] reflections	4365, 4365, 3704
$(\sin\theta/\lambda)_{max}$, (Å^{-1})	0.625
Refinement	
R[F^2 > 2σ(F^2)], wR(F^2), S	0.0501, 0.1409, 1.066
No. of reflections	4365
No. of restrains	0
No. of parameters	266
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.169, -0.240

The crystal packing is shown in Figure 2 where weak intermolecular $\pi - \pi$ stacking is observed between the two adjacent molecules in the unit cell (summarized in Table 2).

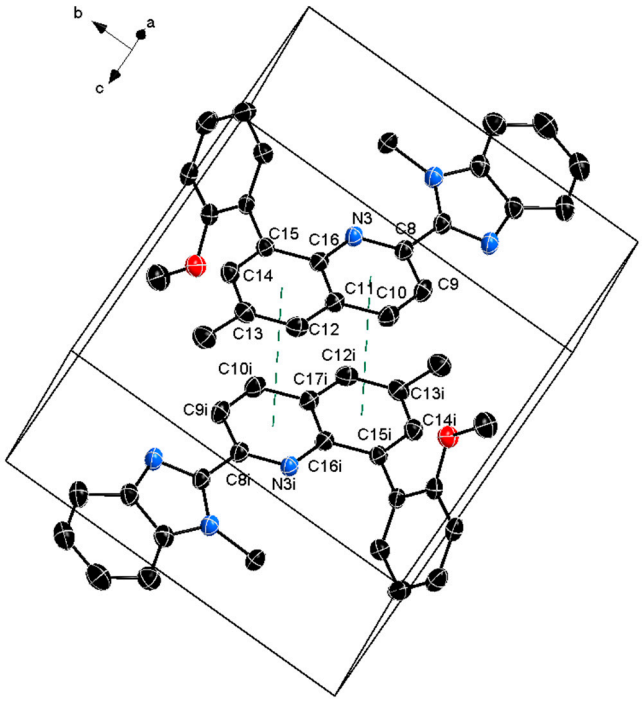

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Figure 2. Unit cell packing of the titled compound **1** with the π – π interactions showed with dashed green line. The thermal ellipsoids are at 50% and the hydrogen atoms are omitted for clarity.

Table 2. Observed π – π interactions where *Cg2* and *Cg4* are the centroids of the C11/C12 – C16 and N3 – C16, respectively. *Cg2i* and *Cg4i* are their respective counterparts generated by symmetry code (*i*) 1-x, 1-y, 1-z.

π – π interaction	Cg – Cg, Å	α, °	Slippage, Å
Cg2 – *Cg4i*	3.8224(14)	0.58(11)	1.639
Cg2i – *Cg4*	3.8224(14)	0.58(11)	1.606

Crystal structure is additionally stabilized by intermolecular C – H ···π interactions all with C – π distance less than 4 Å indicating relatively strong interactions. These interactions are summarized in Table 3 and shown in Figure S31.

Table 3. *The i*ntermolecular C – H ···π interactions.

Interaction	H···π, Å	C··· π, Å	C – H··· π, °
C23 – H23B···*Cg3ii*	2.62	3.513(3)	152
C24 – H24A···*Cg5iii*	2.94	3.723(3)	138

2.4. Optical Properties

The recorded UV-Vis absorption spectra of the titled compound **1** in four different solvents are shown in Figure 3. The compound shows broad absorption peak in the range of 240 – 260 nm which can be attributed to π → π* transition due to the conjugated π-system. The broad emission band in the range of 270 – 320 nm is attributed to the presence of a lone pair (n → π*) of heteroatoms in the conjugated system (in this case nitrogen atoms). The last observed region of interest is between 325 – 375 nm involving three distinctive emission bands which are due to the extended conjugation system. Interestingly, these bands show solvatochromic effect and they are bathochromically shifted in respect to lowering the polarity of the solvent. Furthermore, the FWHM has lower values for the less polar solvents which is well expressed in pentane (the non-polar solvent of the four used).

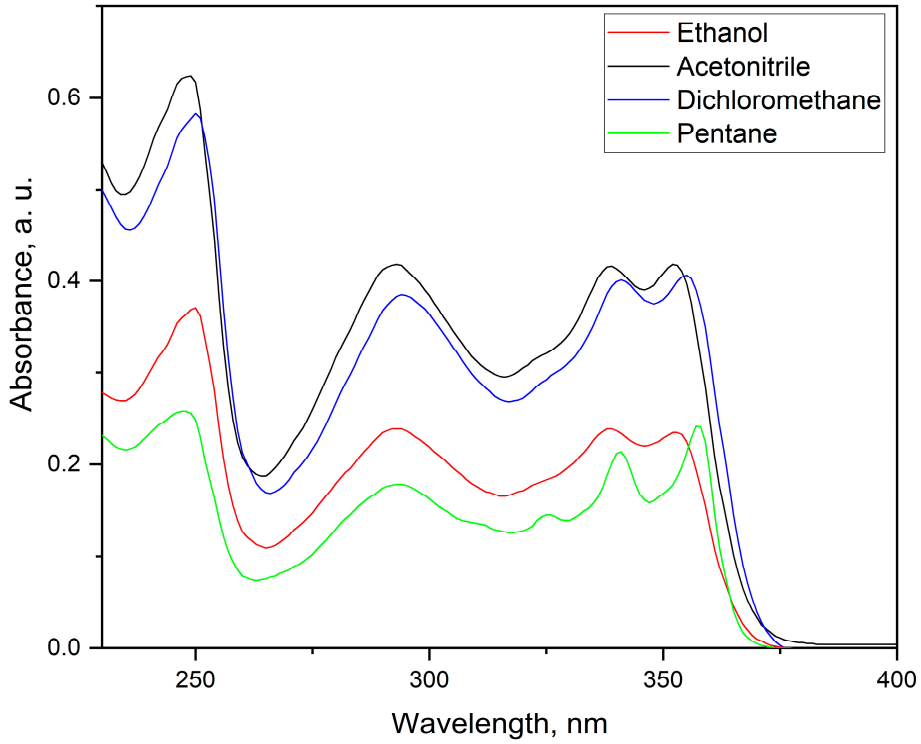

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Figure 3. UV-Vis absorption spectra of titled compound **1** in different solvents.

The compound exhibits well defined fluorescence properties in the UV-blue region having a relatively *large* Stokes shift (λ_A - λ_F = 57 nm) when irradiated at λ_{ex} = 250 nm (Figure 4). Different solvents had no effect on the fluorescence properties of the compound.

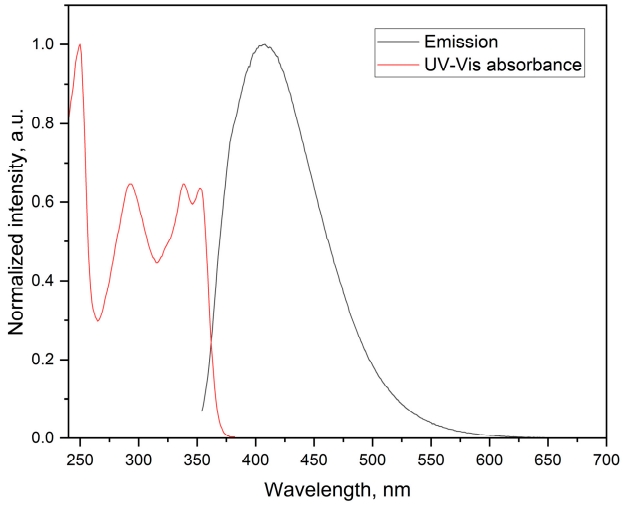

Figure 4. Fluorescence spectrum of the titled compound.

4. Materials and Methods

Melting points were determined on an SRS MPA120 EZ-Melt apparatus.

^{1}H and ^{13}C NMR spec-139 tra were recorded on a Bruker AVNEO 400 spectrometer (at 400 MHz for 1H and 100.6 140 MHz for 13C respectively. Chemical shifts are given in ppm. The NMR-spectra of all synthesized compounds could be found in the **Supplementary Materials**. Liquid chromatography mass spectrometry analysis (LC-HRAM) was carried out on Q Exactive® hybrid quadrupole-Orbitrap® mass spectrometer (ThermoScientific Co, Waltham, MA, USA) equipped with a HESI® (heated electrospray ionization) module, TurboFlow® Ultra High Performance Liquid Chromatography (UHPLC) system (ThermoScientific Co, Waltham, MA, USA) and HTC PAL® autosampler (CTC Analytics, Zwingen, Switzerland). The chromatographic separations of the analyzed compounds were achieved on Nucleoshell C18 (100 × 2.1 mm, 2.7 µm) analytical column (Macherey-Nagel, Düren, Germany). Full-scan mass spectra over the *m*/*z* range 100–600 were acquired in positive ion mode at resolution settings of 140,000. The used mass spectrometer operating parameters were: spray voltage—4.0 kV; capillary temperature—320 °C; probe heater temperature—300 °C; sheath gas flow rate 40 units; auxiliary gas flow 12 units; sweep gas 2 units (units refer to arbitrary values set by the Q Exactive Tune software); and S-Lens RF level of 50.00. Nitrogen was used for sample nebulization and collision gas in the HCD cell. All derivatives were quantified using 5 ppm mass tolerance filters to their theoretically calculated m/z values.

The data set for single crystal x-ray diffraction analysis was collected using a Rigaku XtaLAB Synergy-S diffractometer with a microfocus sealed tube and a HyPix-6000HE Hybrid Photon Counting (HPC) detector. Monochromated MoKa radiation (l = 0.71073 Å) was used. Data was collected at 130(2) K and corrected for absorption effects using the multi-scan method. The structure was solved by direct methods using SHELXT [21] and was refined by full matrix least squares calculations on F2 (SHELXL2019 [22]) in the graphical user interface Shelxle [23].

All non-H-atoms were located in the electron density maps and refined anisotropically. C-bound H atoms were placed in positions of optimized geometry and treated as riding atoms. Their isotropic displacement parameters were coupled to the corresponding carrier atoms by a factor of 1.2 (CH) or 1.5 (CH_3). The structure was refined as a two-component non-merohedral twin. Both components were found in the reciprocal lattice viewer of Crysalis-Pro (Rigaku). The structure was solved using

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the hklf4 file, whereas for the final refinement the hkfl5 was used (BASF 0.250(1). Component 2 was rotated by 179.8° around [0.85 0.42 0.32] in the reciprocal lattice.

The UV-VIS spectra of the titled compound **1** in different solvents (ethanol, pentane, acetonitrile, dichloromethane all of spectroscopic grade obtained by Sigma-Aldrich Chemie GmbH, Taufkirchen, Germany) were measured using Evolution 300 UV/VIS spectrometer (Thermo Scientific, Dreieich, Germany).

The fluorescence spectrum was recorded using Varian Cary Eclipse spectrometer (Agilent, Santa Clara, California, USA) equipped with a 150 W Xe flash lamp as an excitation source in acetonitrile solution.

The FT-IR analysis were carried out on a FT-IR Nicolet 6700-Thermo Scientific in KBr pellets.

N^1-methylbenzene-1,2-diamine and 2-methoxyphenylboronic acid were purchased from Fluorochem and were used without further purification. All the other solvents and reagents were purchased from local suppliers. Nitrobenzene was dried by storing for one week over CaH_2 and used without distillation.

Synthesis of 8-Bromo-2,6-dimethylquinoline ***3***

In a 100 mL round-bottom flask 2-bromo-4-methylaniline (22.300 g, 0.12 mol, 1 eqv) and 60 mL 6M HCl were mixed. The mixture was heated to reflux. Crotonaldehyde (9.250 g, 0.13 mol, 1.1 eqv) was added dropwise (for 25 minutes) to the boiling solution. The heating continued for 3.5 hours and the reaction mixture was cooled to room temperature. The solution was washed with diethyl ether (80 mL). The crude reaction mixture was transferred to a beaker and to it was added solution of $ZnCl_2$ (16.360 g, 0.12 mol, 1 eqv) in minimal amount of water. The formed suspension was stirred for 30 min at room temperature, 30 min in an ice bath and then filtered. The yellow precipitate was transferred to a beaker with 50 mL ice water, stirred and filtered again. Zinc-quinoline complex was washed in the same manner, using 50 mL cold 3M HCl, 50 mL cold water, 100 mL isopropanol and finally 100 mL diethyl ether. The pale yellow crystalline solid has dried (18.1 g) and transferred to a beaker with 25 mL conc. aqueous NH_3 and 50 mL ethyl acetate. The resulting emulsion was stirred for 5 min and the aqueous layer was extracted with dichloromethane. Combined organic layers were dried with Na_2SO_4 and the solvent was removed under reduced pressure, leaving pale yellow crystalline solid (11.667 g, 41%), m.p.= 92°-92.5°C (methanol), lit., [15] 96.0-97.0 °C (benzene).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, J = 8.4 Hz, 1H, ***H4***), 7.88 (d, J = 1.7 Hz, 1H, ***H7***), 7.50 (s, 1H, ***H5***), 7.30 (d, J = 8.4 Hz, 1H, ***H3***), 2.81 (s, 3H, *C**H**$_3$-C2*), 2.50 (s, 3H, *C**H**$_3$-C6*). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.47 (***^{4}C2***), 143.41 (***^{4}C8a***), 136.09 (***^{4}C6***), 135.94 (***C4***), 135.00 (***C7***), 127.65 (***^{4}C4a***), 126.52 (***C5***), 123.71 (***^{4}C8***), 122.83 (***C3***), 25.62 (*C**H**$_3$-C6*), 21.10 (*C**H**$_3$-C2*). HRMS (ESI) m/z calculated for $[M+H]^+$ 236.00704, found 236.00693 (ppm: 0.47).

Synthesis of 8-Bromo-6-methylquinoline-2-carbaldehyde ***4***

In air, selenium dioxide (3.055 g, 27.5 mmol, 1.3 eqv) was dissolved in 110 mL dioxane. The resulting transparent colorless solution was heated at 90°C for 30 minutes. To the mixture 8-bromo-2,6-dimethylquinoline (5.000 g, 21.2 mmol, 1 eqv) was added and almost immediately the solution turned brown-red and dark precipitate was formed. The resulting suspension was heated at 90°C for 3.5 hours more, while monitored with TLC using alumina plates and mobile phase hexanes:dichloromethane=2:1 (on silica the starting quinoline and the respective carbaldehyde are indistinguishable). The reaction mixture was cooled to room temperature, filtered through celite pad and the volatiles were removed under reduced pressure. To the pale brown residue dichloromethane (150 mL) was added and the insoluble particles were filtered. The clear solution was transferred to a separatory funnel with 50mL saturated aqueous Na_2CO_3 solution. The water layer was extracted with dichloromethane and the combined organic extracts were dried with Na_2SO_4. After removal of the solvent, the crude product was purified by using column flash chromatography, mobile phase hexanes:dichloromethane from 3:1 to 1:1. The aldehyde was isolated as yellow crystals (4.75 g, 90%), m.p.=141°-142°C (dichloromethane). Rf=0.55 (hexanes:CH_2Cl_2=1:1) on silica.

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^{1}H NMR (500 MHz, $CDCl_3$) δ 10.20 (s, 1H, *C**HO***), 8.13 (d, J = 8.4 Hz, 1H, ***H4***), 7.96 (d, J = 8.4 Hz, 1H, ***H3***), 7.93 (d, J = 1.6 Hz, 1H, ***H7***), 7.55 (s, 1H, ***H5***), 2.49 (s, 3H, *C**H**$_3$*). ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.46 (*C**HO***), 152.46 (*^{4}**C2***), 143.62 (*^{4}**C8a***), 140.34 (*^{4}**C6***), 137.28 (***C4***), 136.35 (***C7***), 131.30 (*^{4}**C4a***), 126.65 (***C5***), 125.65 (*^{4}**C8***), 118.16 (***C3***), 21.55 (*C**H**$_3$*). HRMS (ESI) m/z calculated for $[M+H]^+$ 249.98650, found 249.98619 (ppm: 1.24).

*Synthesis of 8-Bromo-6-methyl-2-(1-methyl-1H-benzo[d]imidazol-2-yl)quinoline **6***

In air, 8-bromo-6-methylquinoline-2-carbaldehyde (0.310 g, 1.24 mmol. 1 eqv) and N^1-methylbenzene-1,2-diamine (0.151 g, 1.24 mmol. 1 eqv) were dissolved in 5 mL dry nitrobenzene. The solution was heated at 120°C for 22 hours, cooled to room temperature and directly introduced in silica loaded flash chromatography column. The product was isolated in the form of brown solid (0.383 g, 88%), m.p.= 194°-195°C (ethyl acetate). Rf=0.50 (CH_2Cl_2:ethyl acetate=8:1) on silica.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.6 Hz, 1H, ***H3**-quinoline*), 8.07 (d, *J* = 8.6 Hz, 1H, ***H4**-quinoline*), 7.84 (d, *J* = 1.5 Hz, 1H, ***H7**-quinoline*), 7.79 (d, *J* = 7.8 Hz, 1H, ***H4**-benzoimidazolyl*), 7.47 (s, *J* = 8.5 Hz, 1H, ***H5**-quinoline*), 7.40 (d, *J* = 8.0 Hz, 1H, ***H7**-benzoimidazolyl*), 7.29 (t, *J* = 7.2 Hz, 1H, ***H6**-benzoimidazolyl*), 7.25 (t, *J* = 7.8 Hz, 1H, ***H5**-benzoimidazolyl*), 4.51 (s, 3H, *C**H**$_3$-benzoimidazolyl*), 2.45 (s, 3H, *C**H**$_3$-quinoline*). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.30 (*^{4}**C2**-quinoline*), 149.45 (*^{4}**C2**-benzoimidazolyl*), 142.92 (*^{4}**C8a**-quinoline*), 142.62 (*^{4}**C3a**-benzoimidazolyl*), 138.03 (***C6**-quinoline*), 137.69 (*^{4}**C7a**-benzoimidazolyl*), 136.39 (***C4**-quinoline*), 135.41 (***C7**-quinoline*), 128.89 (*^{4}**C4a**-quinoline*), 126.49 (***C5**-quinoline*), 124.98 (*^{4}**C8**-quinoline*), 123.78 (***C6**-benzoimidazolyl*), 122.74 (***C5**-benzoimidazolyl*), 122.37 (***C3**-quinoline*), 120.25 (***C4**-benzoimidazolyl*), 110.11 (***C7**-benzoimidazolyl*), 33.85 (*C**H**$_3$-benzoimidazolyl*), 21.38 (*C**H**$_3$-quinoline*). HRMS (ESI) m/z calculated for $[M+H]^+$ 352.04530, found 352.04438 (ppm: 2.61).

*Synthesis of 8-(2-Methoxyphenyl)-6-methyl-2-(1-methyl-1H-benzo[d]imidazol-2-yl)quinoline **1***

In inert atmosphere, 8-bromo-6-methyl-2-(1-methyl-1*H*-benzo[d]imidazol-2-yl)quinoline (0.176 g, 0.5 mmol, 1 eqv), 2-methoxyphenylboronic acid (0.091 g, 0.6 mmol, 1.2 eqv), $K_3PO_4.7H_2O$ (0.508 g, 1.5 mmol, 3 eqv) and Pd(dppf)Cl_2 (0.0183 g, 25 μmol, 5 mol %) were suspended in mixture of 5 mL THF and 2 ml water. The resulting biphasic system was stirred at 70°C for 19 hours, cooled to room temperature and diluted with 50 mL ethyl acetate. The ethyl acetate solution was dried with Na_2SO_4 and the volatiles were removed under reduced pressure. The dark residue was purified using flash column chromatography on silica, eluting with mobile phase hexanes:ethyl acetate from 8:1 to 2:1. The resulting compound was recrystallized from ethyl acetate. The desired product was isolated in pure form in the form of pale-yellow solid (0.120 g, 63%), m.p.= 193°-195°C (ethyl acetate). Rf=0.44 (hexanes:ethyl acetate=2:1) on silica.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (d, J = 8.6 Hz, 1H, ***H3**-quinoline*), 8.12 (d, J = 8.6 Hz, 1H, ***H4**-quinoline*), 7.77 – 7.71 (m, 1H, ***H4**-N-benzoimidazolyl*), 7.55 (s, 1H, ***H5**-quinoline*), 7.48 (d, J = 1.8 Hz, 1H, ***H7**-quinoline*), 7.34 (td, J = 8.3, 1.7 Hz, 1H, ***H4**-2-methoxyphenyl*), 7.26 (dd, J = 7.4, 1.7 Hz, 1H, ***H6**-2-methoxyphenyl*), 7.25 – 7.17 (m, 3H, ***H5**,**H6** and **H7**-N-benzoimidazolyl*), 6.99 (td, J = 7.4, 0.8 Hz, 1H, ***H5**-2-methoxyphenyl*), 6.94 (d, J = 8.3 Hz, 1H, ***H3**-2-methoxyphenyl*), 3.75 (s, 3H, *C**H**$_3$-N-benzoimidazolyl*), 3.57 (s, 3H, *C**H**$_3$-2-methoxyphenyl*), 2.51 (s, 3H, *C**H**$_3$-quinoline*). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.39 (*^{4}**C2**-2-methoxyphenyl*), 150.24 (*^{4}**C2**-benzoimidazolyl*), 148.96 (*^{4}**C2**-quinoline*), 144.58 (*^{4}**C8a**-quinoline*), 142.58 (*^{4}**C7a**-benzoimidazolyl*), 138.54 (*^{4}**C1**-2-methoxyphenyl*), 137.50 (*^{4}**C3a**-benzoimidazolyl*), 136.91 (*^{4}**C6**-quinoline*), 135.99 (***C4**-quinoline*), 132.96 (***C7**-quinoline*), 131.77 (***C6**-2-methoxyphenyl*), 129.56 (*^{4}**C8**-quinoline*), 128.78 (***C4**-2-methoxyphenyl*), 127.79 (*^{4}**C4a**-quinoline*), 126.32 (***C5**-quinoline*), 123.32 (***C6**-benzoimidazolyl*), 122.46 (***C5**-benzoimidazolyl*), 121.32 (***C3**-quinoline*), 120.22 (***C5**-2-methoxyphenyl*), 120.07 (***C4**-benzoimidazolyl*), 110.59 (***C3**-2-methoxyphenyl*), 109.80 (***C7**-benzoimidazolyl*), 55.59 (*C**H**$_3$-2-methoxyphenyl*), 32.56 (*C**H**$_3$-N-benzoimidazolyl*), 21.80 (*C**H**$_3$-quinoline*). HRMS (ESI) m/z calculated for $[M+H]^+$ 380.17633, found 380.17574 (ppm: 1.55). FT-IR (KBr/cm^{-1}): 2933m, 1911w, 1728w, 1598s, 1474w, 1435s, 1393w, 1238s, 1183w, 924w, 874s, 740s (Figure S32).

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5. Conclusions

The single crystal diffraction analysis shows the presence of weak π – π stacking between the two adjacent molecules in the same unit cell and relatively strong C-H···π interaction between the molecules in neighboring unit cells. The material is a candidate for the construction a blue emitting organic light emiting diod due to his fluorescent properties.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org, Figures S1–S32. ^{1}H, ^{13}C, COSY, HSQC, HMBC NMR spectra of all compounds, UV, fluorescent and IR spectra of compound **1**.

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