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- Authors: Jonas Köhling, Volodymyr Kozel, Vladislav Jovanov, Romana Pajkert, Sergey N. Tverdomed, Oleg Gridenco, Malte Fugel, Simon Grabowsky, Gerd-Volker Röschenthaler, and Veit Wagner

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Synthesis and characterization of oxazaborinin phosphonate for blue OLED emitter applications

Jonas Köhling,[a] Volodymyr Kozel,[b] Vladislav Jovanov, [a] Romana Pajkert,[b] Sergey N. Tverdomed, [b] Oleg Gridenco,[c] Malte Fugel,[d] Simon Grabowsky,[d] Gerd-Volker Röschenthaler[b] and Veit Wagner *[a]

Abstract: Blue-light emitting material based on a boron complex containing heteroaromatic phosphonate ligand is synthesized and characterized. The Phospho-Fries rearrangement is used in the synthesis route of the ligand as a convenient method of introducing phosphonate groups into phenols. Structural, thermal and photophysical properties of the resulting oxazaborinin phosphonate compound have been characterized. DFT geometry optimizations are studied as well as the spatial position and symmetry of the HOMO and LUMO. Good thermal stability up to 250 °C enables vacuum deposition methods next to solution processing. Combining the work function with the optical band gap from UV-Vis measurement shows that the band alignment is possible with standard contact materials. Photoluminescence reveals an emission peak at 428 nm, which is suitable for the blue light-emitter.

Introduction

Since the breakthrough made by C. W. Tang et al.^[1] in 1987, organic light-emitting diodes (OLEDs) have been seen as one of the most promising technologies for display and solid state lighting applications. In the last two decades, a number of materials have been developed and improved by both academic and industrial research to fulfil the requirements of these applications.^[2–12] Intense effort has been devoted to developing materials and device structures for OLEDs in order to obtain higher efficiencies. Among the primary-colour luminescent materials for OLEDs, the blue

[a]	J. Köhling, Dr. V. Jovanov, Prof. Dr. V. Wagner
	Department of Physics and Earth Sciences
	Jacobs University gGmbH
	Campus Ring 1, 28759 Bremen, Germany
	E-mail: v.wagner@jacobs-university.de
[b]	Dr. V. Kozel, Dr. R. Pajkert, Dr. S. N. Tverdomed,
	Prof. Dr. G-V. Röschenthaler
	Department of Life Sciences and Chemistry
	Jacobs University gGmbH
	Campus Ring 1, 28759 Bremen, Germany
[C]	O. Gridenco
	Semiconductor Optics, Institute of Solid State Physics
	University of Bremen

 28359 Bremen, Germany
[d] M. Fugel, Prof. Dr. S. Grabowsky Institute of Inorganic Chemistry and Crystallography University of Bremen
28359 Bremen, Germany

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emitters generally show noticeably inferior а electroluminescence (EL) performance with regard to efficiencies, lifespan, colour quality, and charge-carrier injection/transport. $^{[4,10-12]}$ According to the standards of the full-colour display industry, blue emitting materials should emit a saturated blue or deep-blue colour to meet the demand of high quality displays. This indicates that the blue emitters should possess a very wide band-gap, making it difficult to design their molecular structure. Thus, the development of efficient blue emitters with improved electroluminescent (EL) performances is attractive and challenging.

Among the EL materials, four-coordinated organoboron complexes have attracted much attention due to their intense luminescence, high carrier mobility, chemical and thermal stability as well as facile synthetic accessibility.^[6,13–17] Importantly the application of boron instead of transition metals (e.g. iridium, copper etc.) is environmentally and economically preferable. The structures of the boron-bridged molecules are π -conjugated and can easily be tuned for desirable properties by modifying the chelating ligand or substituent on boron.^[6] Although, boron complexes

N,N-chelating ligands (e.g. BODIPYs) are well investigated and widely used as fluorescent dyes, their less investigated N,O-analogues are becoming increasingly important because of their stronger emission in solid state.[18-23] Compared to widely used N,O-ligands, such as 8pyridylphenolates hydroxyquinolines, have less а conjugated system which results in blue emission (hypsochromic effect).^[24] Indeed, only few blue emitters of four-coordinate boron with pyridylphenolate ligands have been reported in the literature^[25,26] and additional investigations are needed.

An introduction of phosphonate functional groups has recently shown improved electron injection/transporting capability and excellent adhesion of emitters on cathode material.^[27] The high electronegativity of oxygen makes the P=O group highly polar and electron withdrawing, which, with its tetrahedral geometrical structure, is advantageous for its application as host and EL material in OLEDs. The electron withdrawing properties of the phosphonate group can make the connected core electron deficient, improving its electron transport properties.^[28] HOMO and LUMO are also shifted due to the electron withdrawing character of the P=O bond.^[29] The tetrahedral geometry is also beneficial to the amorphous morphology of the organic materials because it is difficult to stack molecules containing P=O

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regularly. Thus, the introduction of this group into pyridylphenol ligands of boron complexes could make it a favourable technique for property tuning in order to obtain superior blue emitters for OLEDs. Therefore, the aim of our project is the introduction of a phosponate group into a pyridylphenol ligand for the synthesis of boron complexes which are promising as efficient blue emitting materials.

The Phospho-Fries-rearrangement is a convenient method of introduction of phosphonate groups into phenols^[30] which previously has not been applied for heteroaromatic compounds. Hence, in this work we present the first example of application of Phospho-Fries-rearrangement for the synthesis of 2-hydroxyphenylpyridine phosphonate ligands for the N,O-difluoroboronic complex, a potential EL material. Furthermore, we have fully characterized the resulting compound including computational calculations of the molecule and experimental measurements to determine structural, thermal and photophysical properties. Finally, possible device architectures are also discussed.

Results and Discussion

Synthesis route

The classical Phospho-Fries-rearrangement involves the conversion of an aryl phosphate to an orthohydroxyarylphosphonate.^[27-29] The transposition of the phosphoryl moiety is initiated by formation of an aryl anion adjacent to the phosphate substituent.[31] LDA being a strong and non-nucleophilic base is used to generate an aromatic anion to avoid substitution at the phosphonate group.^[32] We have revealed that 2-hydroxyphenylpyridine (1) reacts with diethyl chlorophosphate in presence of triethylamine as a base to form diethyl 2-(pyridine-2yl)phenyl phosphate (2) in 50% yield (Scheme 1). The reaction was carried out in dry dichloromethane in presence of catalytic amount of DMAP, the product was purified by column chromatography and its structure has been established by NMR. The obtained phosphate 2 was then treated with LDA to form the typical product of a Phospho-Fries-rearrangement (Scheme 1). The rearrangement was performed in THF at -78 °C using various amounts of LDA. When 1.1 equiv. of a base reacted with phosphate 2, the conversion of 2 achieved 70% and a mixture of orthohydroxy phosphonate 3 and starting material 2 was obtained in a 7:3 ratio, respectively (Tab. 1). By increasing the amount of a base up to 1.5 equiv., the ortho-lithiation and 1,3-O→C migration proceeded smoothly giving rise to the desired phosphonate 3 as a sole product (Tab. 1). Treatment of 2 with 2 equiv. of LDA led also to the rearranged product 3, however, partial decomposition of 3 occurred, plausibly due to the C-P cleavage of the phosphonate 3 (Tab. 1).

Table 1 Optimization of the LDA amount used for Phospho-Fries rearrangement in THF from -78 $^\circ\text{C}$ to rt.

Amount of LDA (equiv.)	Conversion of 2 into 3 (%) ^a
1.1	70
1.5	100

[a] Determined by ³¹P{¹H} NMR

[b] The presence of a phosphate group at δ_P = 0.5 ppm was detected

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Moreover, it was found that the order of addition of reagents had an important influence on the reaction outcome. When a solution of compound **2** in anhydrous THF was added dropwise into pre-generated LDA (1.5 equiv.) in THF at -78 °C, full conversion of **2** into **3** was achieved. On the other hand, the 'reverse' order of addition of reagents (LDA was added to the solution of phosphate **2** in THF at -78 °C) led only to a partial rearrangement of **2** and decomposition of **3** even when large excess of LDA (2 equiv.) was applied.

Despite of possible α -lithiation of the pyridine ring, product 3 was obtained with high regioselectivity, and the products of functionalization in the pyridine ring were not registered even in trace amount. The reaction was performed at -78 °C in dry THF. The hydroxyphenyl phosphonate 3 was obtained in 71% yield and fully characterized by NMR. It is known that 2-(pyridin-2-yl)phenol reacts with boron trifluoride etherate to give a corresponding difluoroboron complex.^[24] On other hand, phosphonic units are known to be reactive towards fluorinating agents forming fluorophosphates.^[33] Indeed, we have found that compound 3 reacts with BF3 · Et2O upon formation of the difluoroborate complex 4 without affecting the diethyl phosphonate function. The reaction was carried out in dry benzene in presence of triethylamine, and product 4 was obtained in 53% yield after column chromatography purification. The complex was fully characterized by ¹H NMR (Fig 1.). The proton resonances were assigned by H,H-COSY, H,C-HMQC and H,C-HMBC spectra (ESI Fig. S1 - Fig. S6) and are in agreement with the DFT calculations. Characteristic signals were found for ¹⁹F at δ F = -147.57, and ¹¹B at δ B = -0.24 ppm.



Scheme 1. Synthetic route to diethyl (6,6-difluoro-6H-6λ4,7λ4-benzo[e]pyrido[1,2-c][1,3,2]oxazaborinin-4-yl)phosphonate.



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Figure 1. ¹H NMR spectra of compound 4 measured in CDCl₃.

Further, the crystal structure of a single crystal of 4 was determined by XRD. It crystallizes in the space group P2(1)/c in a monoclinic crystal system with the lattice parameters b = 8.7134(13) Å, c = 15.997(3) Å,a = 11.8968(18) Å, β = 104.962(6)°, V = 1602.05(40) Å and Z = 4 (Full information in ESI Tab. S1). Selected bond lengths of the oxazaborinin part are compared with an overall good agreement to the DFT calculation in Tab. 2.

Table 2 Selected bond lengths of compound 4 calculated by DFT (B3LYP/6-31G*) and measured with XRD.

Bond	DFT [Å]	XRD [Å]
$B - F_1$	1.3723	1.3836 (22)
$B - F_2$	1.3723	1.3778 (19)
$B - O_1$	1.4557	1.4399 (22)
$B - N_1$	1.6240	1.5999 (22)
P – C ₁	1.8166	1.7915 (15)

XRD studies of similar structures show that the basic oxazaborinin molecule class tends to form π - π stacking in the state,[34] solid which can lead to fluorescence quenching.^[13,14,19,20,35] An approach of introducing bulky side substituents is used to overcome this problem. Indeed, the X-ray crystal analysis of compound 4 reveals that the alignment of diethylphosphonate groups between layers reduces the formation of the π - π stacking between phenyl or pyridine rings (Fig. 2) compared to a basic oxazaborinin molecule class.^[34]





Figure 2. A part of a crystal-packing pattern of compound 4 that shows no stacking between adjacent interlayered crystals reviewed along the y-axis (a)

and along the x-axis (b).

HOMO - 1

Figure 3. A part of a crystal-packing pattern of compound 4 that shows no stacking between adjacent interlayered crystals reviewed along the y-axis (a) and along the x-axis (b).

DFT calculations at a B3LYP/6-31g* level were carried out to get insights into electronic and structural properties at a molecular level. The relaxed structure and the spatial resolution of the last two highest occupied (HOMO-1 and HOMO) and first two lowest unoccupied (LUMO and LUMO+1) molecular orbitals are shown in Fig 3. Overall the molecule shows a flat structure, with a small twist at the heteroatoms oxygen and boron in the backbone. It is expressed by the dihedral angle of $\theta_{COBN} = -45.2^{\circ}$. The induced stress twists the pyridine ring with respect to the phenyl ring by θ_{CCCC} = -13.6°. The energy levels of the molecular orbitals are calculated to be -6.891 eV, -6.295 eV, -2.266 eV and -1.702 eV, respectively. The LUMO molecular orbitals are located mainly in the 2-phenyl-pyridine ring of the backbone. The LUMO is predominately located in the pyridine part and also distributes slightly over the oxazaborinine. The LUMO+1 is located only over the 2-phenyl-pyridine. Both, LUMO and LUMO+1 do not spatially distribute towards the phosphonate group. The occupied molecular orbitals are also mainly located at the 2phenyl-pyridine ring of the backbone. However, the HOMO and HOMO-1 are predominantly located at the phenyl ring and are less pronounced at the pyridine part. They differ from the spatial point of view from each other that the HOMO is more distinct at the oxygen of the oxazaborinine than at the phosphonate group, whereas the opposite holds true for the HOMO-1. However, spatial positon and symmetry of molecular orbitals provide sufficient overlap which promotes transitions.

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Figure 4. TGA (blue) and DSC (red) measurement of compound 4 under nitrogen.

Thermal properties

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are used to investigate the thermal properties of compound **4**. The weight loss below T = 100 °C is assigned to desorbing water. The material is stable until the decomposition temperature of $T_D = 251$ °C. Until this temperature the DSC curve is in the endothermic region and shows a melting temperature in the region between T = 144 – 147 °C. At temperatures higher than T = 251 °C, the curves indicate that the material is first evaporating and then thermally decomposing. The results confirm that the material possesses good thermal properties for deposition under high vacuum utilizing organic molecular beam deposition (OMBD).



Photophysical properties

In order to determine suitability of the material as blue emitter UV-VIS and photoluminescence measurements were conducted. For this purpose, we prepared a solution in ACN and deposited thin films on glass substrates by evaporation and solution processing.

Fig. 5 shows the results of UV-Vis and PL measurements of compound **4**. UV-Vis spectra are measured in solution (ACN), for bladed and evaporated thin films. Solution and solid state absorptions are very similar except for a small broadening that occurs in the solid state. This indicates that intermolecular interactions are minor and give only a negligible contribution to



the absorption spectra. All measurements show absorption band maxima at $\lambda = 332$ nm, $\lambda = 299$ nm, $\lambda = 260$ nm and $\lambda = 216$ nm, which are expected to be π - π^* transitions within the aromatic backbone of the molecule and n- π^* transitions of free electron pairs. The optical band gap is estimated to be E_G = 366 nm (3.39 eV) as the onset of the absorption. The absorption spectra for the evaporated film is multiplied by 6 to be directly comparable with other results.

Basic oxazaborinin molecules exhibit promising fluorescence quantum yields for OLED-emitters.^[20,34] The PL spectra of the bladed thin film of compound **4** is given in Fig 5. and shows two emission maxima at $\lambda = 428$ nm and $\lambda = 500$ nm. The shoulder features indicate underlying emissions which give only a small contribution and cause the broadening of the spectra. The onset of the PL is estimated to be at $\lambda = 401$ nm (3.09 eV) which is red-shifted by 35 nm with respect to the absorption of the material. This is caused by thermalization and different geometries between the ground state and the excited state, the so called Stokes shift.

Figure 6 He Ia UPS spectra after sputter-cleaning OMBD compound 4 (20 nm) on top of gold (V_{bias} = 10 V).



To determine the energy levels of molecular orbitals of compound **4** and find potential contact materials UPS measurements were conducted.

Fig. 6 shows the He I α UPS spectra of compound **4** using gold as a reference. The ionization energy for this molecule is equal to the energy of the HOMO level, which is measured to be $E_{HOMO} = -5.288 \text{ eV}$, while the Fermi energy is $E_F = -4.153 \text{ eV}$. By adding the optical band gap energy to the energy of the HOMO level allows us to estimate the energy for the LUMO as $E_{LUMO} = -2.038 \text{ eV}$, relative to the vacuum level. With the Fermi energy

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located closer to the HOMO level, the material can be characterized as p-type. Based on the determined absolute energy levels of the molecular orbitals, materials like ITO and Liq/AI are suitable contact materials as shown in Fig. 6. Further alignment between the contact materials and compound **4** can be achieved by additional hole transport layers (HTL) and electron transport layers (ETL), which are not shown in Fig. 7.



Figure 7 Energy level diagram for compound 4 with suitable contact materials. Possible charge transport layers are not displayed.

Conclusions

In this study, we present the design, synthesis and characterization of the luminescent organic semiconductor diethyl (6,6-difluoro-6H-6l4,7l4-benzo[e]pyrido[1,2-c][1,3,2]oxaza-

borinin-4-I)phosphonate. The molecular structure was confirmed with NMR and XRD. Thermal stability was examined by TGA. Selected structural properties were compared to DFT calculations. Also, the DFT calculations were used to visualize the spatial distribution of the last two occupied and first two unoccupied molecular orbitals. HOMO and LUMO are mainly located in the conjugated aromatic backbone, which includes the oxazaborinin structure. The compound shows good thermal stability and thin layers can be deposited under high vacuum by thermal evaporation. UV-Vis and photoluminescence measurements were carried out for thin films as well as dissolved material to determine photophysical properties such as the wide optical band gap (366 nm, 3.39 eV). Also the material exhibits two emission maxima in the blue range of visible light around 428 nm and 500 nm in the blue region of visible light. The exact energy levels ($E_{HOMO} = -5.288 \text{ eV}$, $E_F = -$ 4.153 eV and as ELUMO = -2.038 eV) are determined by UV photoelectron spectroscopy and suitable contact materials for device fabrication are given. The reported data show that this class of molecule has a strong potential for applications in OLED devices.

Experimental Section

General information

All chemicals and solvents were purchased from Sigma-Aldrich and used without further purification.

Sample preparation and characterization

NMR and XRD. ¹H NMR (400 MHz), ¹¹B NMR (128 MHz), ¹³C NMR (100 MHz), ¹⁹F NMR (376 MHz) and ³¹P NMR (162 MHz) were recorded on JEOL ECX 400 MHz spectrometer. ¹H, ¹¹B, ¹³C, ¹⁹F and ³¹P NMR chemical shifts (δ) are reported in ppm from tetramethylsilane, BF₃·OEt₂, CFCl₃, and H₃PO₄, using the residual solvent resonance as an internal reference. Coupling constants (J) are given in Hz. XRD of a single crystal was measured with a Bruker D8 Venture diffractometer fitted with a IµS microfocus radiation source using MoKα radiation and a Photon-100 CMOS detector. The data collection was performed at a temperature of 100 K.

TGA and DSC. TGA and DSC measurements were carried out with a Thermal Analysis Q 600 under nitrogen with a heating ramp of 5 °C/min to determine the melting point and thermal properties of the material.

Sample preparation. Quartz substrates were cleaned before deposition by 2 min ultrasonication in acetone (tech. grade), followed by 10 min ultrasonication in a mixture of acetone and isopropanol (1:1, VLSI grade) and finally rinsed with deionized water (18.3 M Ω) for 1 min. The substrates were dried first with nitrogen and then on a hotplate at 120 °C before 10 min UV/ozone treatment. For UPS measurements a 200 nm thick gold layer was sputtered on top of the substrate (Quorum QT150T). Thin films with a thickness of 200 nm were deposited by doctor blading (Erichsen COATMASTER 510) 40 µL of a 20 mg/mL solution of the compound in chlorobenzene. The blade was operated at a speed of 50 mm/s with 2 mm height. Afterwards the samples were dried at 75 °C for 3 min. The crystallization occurred over night in a glovebox environment (MBraun, nitrogen, $O_2 < 2$ ppm, $H_2O < 2$ ppm). Thin films with a thickness of 20 nm were deposited by organic molecular beam deposition OMBD in UHV at 170 °C with a rate less than 1 nm/min and monitored with a quartz scale (intellimetrics, IL 150) utilizing the density obtained from XRD.

UPS. UV photoelectron spectroscopy was carried out in UHV (10^{-8} mbar) using a Specs PHOIBOS 100 hemispherical energy analyzer and He I α radiation (E = 21.2 eV). A bias of V = 10 V was applied while measurement. Before UPS measurement the surfaces were cleaned by argon sputtering.

UV-VIS and PL. UV-VIS measurements were carried out with a Perkin Elmer Lambda 12. For PL measurements, a microscope setup with a 36 x objective, numerical aperture 0.5 into which a 325 nm cw (He-Cd) laser was coupled, was used to collect the PL and recorded using a single-grating, thermos-electric cooled AvaSpec-ULS2048LTEC spectrometer.

DFT calculations

First, the geometry of a single molecule in vacuum was optimized by density functional theory (DFT) with B3LYP^[36–39] (Becke three parameter Lee-Yang-Parr) exchange-correlation functional and the Gaussian 09 code.^[40] 6-31G^{*[41]} was used as a basis set for all atoms (H, B, C, N, O, F and P). Afterward, the frequency calculations were carried out, and it was ensured that no imaginary vibrations were calculated. Avogadro software^[42] was used to visualize the computational structure and the molecular orbitals for the following states HOMO-1, HOMO, LUMO, and LUMO+1. Finally, the NMR shielding tensors and magnetic susceptibilities were calculated using the Gauge-Independent Atomic Orbital method (GIAO) approximation. All NMR shielding tensors were referenced to the tetramethylsilane (TMS) calculated spectra utilizing the beforehand mentioned parameters. For the NMR calculations solvent

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effects were taken into account with the Polarizable Continuum Model (PCM).

Synthesis

Synthesis of 2-(pyridin-2-yl)-phenol (1). 2-(Pyridin-2-yl)-phenol was synthesized according to the reported literature.^[43]

A solution of 2-phenylpyridine (4.37 g, 28 mmol), PdCl₂ (0.5 g, 2.8 mmol), and 35% aqueous H₂O₂ (13.7 g, 1.5 mmol) in 4-methyl-2-pentanone (10 mL) was stirred for 12 h at 100 °C. Then, the reaction mixture was filtered through a celite pad and washed with EtOAc. The solution was treated with a 0.5 M aqueous Na₂S₂O₃ solution, and then the organic layer was dried over MgSO₄ and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel (petroleum ether / DCM, 3 : 7) and the product **2** was obtained with a yield of 78% (3.76 g). The analytical data are in a good agreement with those from the publication.

Synthesis of diethyl 2-(pyridine-2-yl)phenyl phosphate (2). To a solution of DMAP (488 mg, 4.4 mmol) TEA (1.07 g, 4.4 mmol) and 2-(pyridin-2-yl)-phenol (1) (680 mg, 4.0 mmol) in 20 mL dry DCM diethyl chlorophosphate (690 mg, 4.4 mmol) was added drop-wise. Then, the solution was stirred for 24 h at room temperature. Afterwards, the reaction mixture was treated twice with saturated NaHCO₃ (2 x 30 mL) and brine (1 x 20 mL) before drying in vacuo. The crude product was purified through column chromatography on silica gel (eluent: EtOAc). The final product was obtained as colorless oil with a yield of 50% (1.23 g).

 ^{1}H NMR (400 MHz, CDCl₃) δ 8.70 (ddd, J = 4.6, 1.4, 1.4 Hz, 1H), 7.80 – 7.70 (m, 3H), 7.49 – 7.44 (td, J = 8.1, 1.1 Hz 1H), 7.40 – 7.33 (ddd, J = 8.1, 7.4, 1.8 Hz, 1H), 4.08 – 3.91 (m, 1H), 1.19 (tt, J = 7.1, 1.0 Hz, 2H). ^{13}C NMR (101 MHz, CDCl₃) δ 155.22 (d, J = 1.3 Hz), 149.59, 148.07, 136.07, 132.10 (d, J = 6.7 Hz), 131.48, 130.03 (d, J = 1.2 Hz), 125.45 (d, J = 1.0 Hz), 125.07, 122.33, 120.64 (d, J = 2.3 Hz), 64.61 (d, J = 6.2 Hz), 16.07 (dd, J = 6.8, 1.2 Hz). ^{31}P NMR (162 Hz, CDCl₃) δ -6.30 (s).

Synthesis of diethyl (2-hydroxy-3-(pyridin-2-yl)phenyl)phosphonate (3).

Diethyl pyridinyl phosphate (2) (620 mg, 2.0 mmol) was dissolved in anhydrous THF (20 mL) and added drop-wise to 1.5 equivalents of LDA (3.0 mmol) synthesized in-situ in anhydrous THF, while the temperature was maintained at -78 °C. Then, the reaction mixture was stirred for 2 h at -78 °C. Afterwards, the mixture was allowed to warm up to room temperature and then left overnight (12 h). 1 M HCl solution was added to the organic phase until the pH value turned acidic. The aqueous phase was extracted with DCM (3 x 20 mL) and concentrated in vacuo. The final product was obtained as dark brown oil with a yield of 71% (440 mg).

³¹P NMR (162 MHz, CDCl₃) δ 18.83 (s).

Synthesis of diethyl (6,6-difluoro-6H-6λ4,7λ4-benzo[e]pyrido[1,2c][1,3,2]oxazaborinin-4-yl)phosphonate (4).

To the solution of **3** (440 mg, 1.2 mmol) in benzene (10 mL) TEA (151 mg, 2.4 mmol) was added at room temperature. The solution was stirred for 20 min and then boron trifluoride etherate (0.46 mL, 3.7 mmol) was added. The mixture was stirred for 1 h at 50 °C. A yellow solid was gradually precipitated from the solution. After cooling to room temperature, the mixture was filtrated and the solid was washed several times with diethyl ether. The insoluble part was purified by column chromatography (DCM / methanol, 20 : 1) giving **4** as colorless solid with a yield of 53% (270 mg).

M.p. 144-147 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.70 (d, J = 5.5 Hz, 1H), 8.23 (ddd, J =1.4 Hz, 7.3 Hz, 8.7 Hz, 1H), 8.13 (d, J =8.7 Hz, 1H), 8.09 (ddd, J = 1.8 Hz, 7.3 Hz, 14.2 Hz, 1H), 8.01 (d, J = 7.8 Hz, 1H), 7.63 (ddd, J = 0.9 Hz, 5.7 Hz, 7.3 Hz, 1H), 7.12 (ddd, J = 33.7 Hz, 7.8 Hz, 14.2 Hz, 1H), 4.23 (m, 4H), 1.36 (td, J = 3.4 Hz, 7.0 Hz, 6H), ¹³C NMR (101 MHz, CDCl₃) δ 16.42 (d, J = 6.7 Hz), 62.84 (d, J = 5.8 Hz), 116.86 (d, J = 10.5 Hz), 119.42 (s), 120.33 (d, J = 14.4 Hz), 120.96 (s),123.61 (s), 129.97 (d, J = 1.9 Hz), 140.60 (d, J = 6.7 Hz), 141.28 (s), 142.77 (s), 149.61 (s), 157.56 (d, J = 2.9 Hz), ¹¹B NMR (128 MHz, CDCl₃) δ -0.23 (s), ¹⁹F NMR (376 MHz, CDCl₃) δ -147.56 (d, J = 23.1 Hz), ³¹P NMR (162 MHz, CDCl₃) δ 15.48 (s)

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