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Short Note

4,4-Difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalen-2-yl)-4-bora-3a,4a-diaza-*s*-indacene

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Abstract: The title compound, 4,4-difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(2-naphthalen-2-yl)-4-bora-3a,4a-diaza-*s*-indacene (**1**), was synthesized for the first time in a 62% yield by the P_2O_5-promoted condensation of 2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]-ethan-1-ol (**2**) with 3-phenyl-5-(1*H*-pyrrol-2-yl)isoxazole (**3**) followed by the oxidation of dipyrromethane **4** and the complexation of dipyrromethene thus formed with BF_3. The product fluoresces in a long wave region (651–662 nm) with a high quantum yield (0.49–0.61).

Keywords: fluorophore BODIPY; 2,2,2-trifluoro-1-(1*H*-pyrrol-2-yl)ethan-1-ol; 3-phenyl-5-(1*H*-pyrrol-2-yl)isoxazole; condensation

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1. Introduction

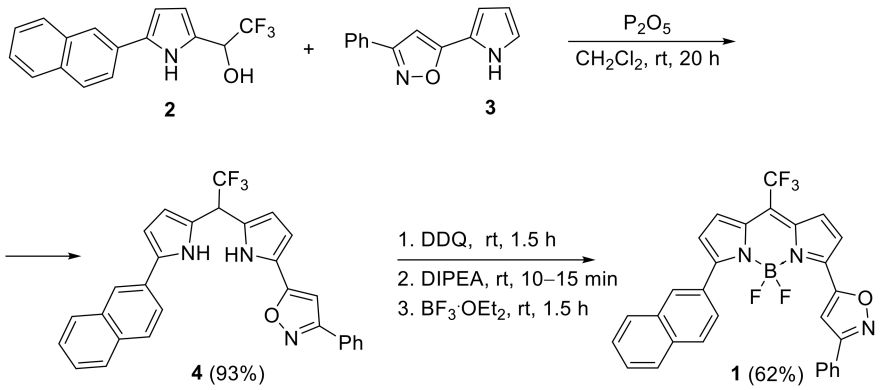
Bright fluorescent dyes with a 4,4-difluoro-4-bora-3a,4a-diaza-*s*-indacene core, known as BODIPY dyes, are of extreme importance for materials science and have gained much attention over the last few years [1,2]. The unique combination of photo- and thermal stability, high quantum yield and easy absorption/emission wavelength tuning from UV–vis to near IR region make BODIPY derivatives suitable for application as chemosensors for organic and inorganic targets, OLEDs, nonlinear optics, dye lasers, perovskite- and dye-sensitized solar cell, semiconductors [3], etc. The low cytotoxicity, stability in physiological media and absorption/emission in NIR region allows wide bioapplications of BODIPY fluorophores as in vitro or in vivo probes, fluorescent labels of proteins, lipids, DNA, staining agents for lifetime cell observations or for biochemical investigations, e.g., in antibacterial or anticancer developments [4,5].

The nature of the BODIPY core unit provides HOMO–LUMO electron transitions under external irradiation and eventually fluorescent properties of a dye molecule. However, specific wavelength values of absorption and emission are determined by the structure of functional substituents in the pyrrole counterparts and at a methylene spacer. One of the most common ways to modulate the properties of the BODIPY fluorophore is to extend the conjugation chain by the introduction of various aryl or heterocyclic substituents at the 3- and 5-positions of the BODIPY core [6,7]. Additionally, it is known that the replacement of the usual alkyl- or aromatic substituents in the *meso*-position of the BODIPY core by strong electron-withdrawing fluorinated ones results in a deep bathochromic shift to the red region, the radiation in which has better tissue penetration and less photodamage [8–12]. The introduction of isoxazole scaffolds [13,14], which exhibit a wide spectrum of targets and broad biological activities, to the molecule can provide additional benefits for fluorophore.

2. Results

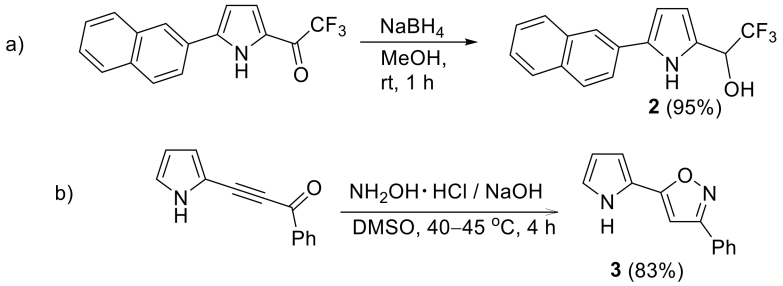
We have shown that 4,4-difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalen-2-yl)-4-bora-3a,4a-diaza-*s*-indacene (**1**), fluorescing in a long wave region (662 nm) with a high quantum yield (0.61), can be obtained according to our previously developed methodology [9] by the P_2O_5-promoted condensation of 2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]ethan-1-ol (**2**) with 3-phenyl-5-(1*H*-pyrrol-2-yl)isoxazole

(**3**) (CH_2Cl_2, rt, 20 h) followed by the oxidation of dipyrromethane **4** and the complexation of dipyrromethene thus formed with BF_3 in the presence of diisopropylethylamine at room temperature for 1.5 h (Scheme 1). The last two stages are realized as a one-pot procedure to furnish the target fluorophore BODIPY **1** in a 62% yield.



Scheme 1. Synthesis of 4,4-difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalene-2-yl)-4-bora-3a,4a-diaza-*s*-indacene (**1**).

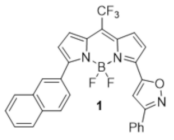
2,2,2-Trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]ethan-1-ol (**2**) and 3-phenyl-5-(1*H*-pyrrol-2-yl)isoxazole (**3**), required for the synthesis of fluorophore **1**, were obtained, by the reduction of easily available [15] 5-(naphthalen-2-yl)-2,2,2-trifuoroacetylpyrrole with $NaBH_4$ (Scheme 2a) (methanol, rt, 1 h) and the reaction of 2-benzoylethynylpyrrole with NH_2OH (Scheme 2b), respectively [16].



Scheme 2. Synthesis of precursors of 4,4-difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalen-2-yl)-4-bora-3a,4a-diaza-s-indacene (**1**): (**a**) synthesis of 2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]ethan-1-ol (**2**); (**b**) synthesis of 3-phenyl-5-(1*H*-pyrrol-2-yl)isoxazole (**3**).

The compound **1** in *n*-hexane, CH_2Cl_2, THF, and ethylacetate fluoresces with high quantum yields (Φ_F 0.49–0.70); it has a nanosecond fluorescence life-time (τ), typical for BODIPY molecules. Alternatively, this fluorophore exhibits only weak fluorescence in MeCN, whereas fluorescence life-time in this solvent was very short. The spectroscopic and photophysical characteristics of fluorophore **1**, including the positions of the maxima of the absorption (λ_{abs}) and fluorescence (λ_{fl}) bands, fluorescence quantum yields (Φ_F), and life-time (τ) in different solvents are shown in Table 1. Normalized absorption and fluorescence spectra of compound **1** in different solvents are given in the SM (Figure S11).

Table 1. Spectroscopic and photophysical characteristics of compound **1** in different solvents.

BODIPY	Solvent	λ_{abs}., nm	λ_{fl}., nm	Φ_F	τ/ns
	*n*-hexane	614	651	0.70	6.2
	CH_2Cl_2	600	662	0.61	5.4
	THF	610	662	0.49	4.5
	Ethylacetate	601	651	0.50	4.8
	MeCN	592	659	0.033	0.6

The structure and composition of synthesized compound **1** were confirmed by ^{1}H, ^{13}C, ^{19}F, ^{11}B NMR, and IR spectroscopy (see Supplementary Materials). Elemental analysis established the chemical formula of compound **1**.

Thus, we have synthesized 4-difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalen-2-yl)-4-bora-3a,4a-diaza-*s*-indacene (**1**), a new BODIPY fluorophore with an extended conjugation chain by the introduction of naphthyl and 3-phenylisoxazolyl substituents, which fluoresces intensively in a long wavelength region. The introduction of a pharmacologically valuable isoxazole scaffold to molecule of fluorophore **1** significantly expands its application area.

3. Materials and Methods

General. NMR spectra were recorded on a Bruker DPX-400 spectrometer (Bruker, Billerica, MA, USA) (400.13 MHz for ^{1}H, 100.6 MHz for ^{13}C, 376.5 MHz for ^{19}F, 40.5 MHz for ^{15}N and 128.4 MHz for ^{11}B) in $CDCl_3$ or DMSO-d_6. The internal standards were the residual solvent signals 7.27 ppm for ^{1}H and 77.1 ppm for ^{13}C ($CDCl_3$), 2.50 ppm for ^{1}H and 39.5 ppm for ^{13}C (DMSO-d_6). The ^{19}F chemical shifts were referenced to $CFCl_3$. Coupling constants (*J*) were measured from one-dimensional spectra, and multiplicities were abbreviated as follows: s (singlet), br. s (broad singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). The assignment of signals in the ^{1}H NMR spectra was made using COSY and NOESY experiments. Resonance signals of carbon atoms were assigned based on ^{1}H-^{13}C HSQC and ^{1}H-^{13}C HMBC experiments. The values of the δ ^{15}N were measured through the 2D ^{1}H-^{15}N HMBC experiment. The ^{15}N chemical shifts were referenced to CH_3NO_2.

IR spectra were recorded on a two-beam Bruker Vertex 70 spectrometer (Bruker, Billerica, MA, USA), in a film. UV–vis absorption spectra were recorded on a Hitachi U-3010 (Hitachi High-Tech, Japan) spectrophotometer. Fluorescence spectra were recorded on a Hitachi F-4500 (Hitachi High-Technologies, Tokyo, Japan) fluorescence spectrophotometer. Elemental analyses (C, H, N) were performed on an EA FLASH 1112 Series (CHN Analyzer) instrument (Thermo Finnigan, Rodano, Italy). Fluorine content was determined on a SPECOL 11 (Carl Zeiss Jena, Jena, Germany) spectrophotometer. Melting points (uncorrected) were measured using a Stuart Scientific melting point SMP3 apparatus.

4,4-Difluoro-3-(3-phenylisoxazol-5-yl)-8-trifluoromethyl-5-(naphthalen-2-yl)-4-bora-3a,4a-diaza-s-indacene (**1**). The mixture of 3-phenyl-5-(5-{2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]ethyl}-1*H*-pyrrol-2-yl)-isoxazole (**4**) (1.934 g, 4.0 mmol) and DDQ (0.908 g, 4.0 mmol) in CH_2Cl_2 (70 mL) was stirred at room temperature for 1.5 h. DIPEA (5.170 g, 40.0 mmol) was added and the mixture was stirred for 15 min, then $BF_3.OEt_2$ (6.813 g, 48.0 mmol) was added dropwise. The mixture was stirred for 1.5 h, then ~2/3 of solvent was removed under a vacuum and the obtained residue was purified by column chromatography (SiO_2, eluent *n*-hexane/CH_2Cl_2, gradient 1:0 → 0:1) to afford compound **1** (1.313 g, 62%) as dark blue crystals, mp 211–212 °C. IR spectrum (film) ν, cm^{-1}: 1625, 1566, 1494, 1444, 1411, 1394, 1345, 1300, 1277, 1248, 1225, 1140, 1140, 1111, 1088, 1052. ^{1}H NMR (400.13 MHz, $CDCl_3$) δ: 8.57 (s, 1H, naphthyl), 8.12–8.09 (m, 1H, naphthyl), 8.02–8.00 (m, 2H, naphthyl), 7.96–7.94 (m, 1H, naphthyl), 7.87–7.85 (m, 2H, Ho, Ph), 7.64–7.59 (m, 3H, H-7, H-4 oxazole, naphthyl), 7.56 (s, 1H, naphthyl), 7.47–7.45 (m, 4H, H-1, Hm, p, Ph), 7.29 (d, *J* = 4.5 Hz, 1H, H-2), 7.03 (d, *J* = 4.5 Hz, 1H, H-6). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 165.8, 163.7, 161.4, 142.9, 136.1, 134.6, 133.2, 132.9, 132.5, 131.3 (t, *J* = 4.38 Hz), 130.2, 129.5, 129.0 (2C), 128.8, 128.7, 128.6, 128.5, 128.4, 127.9, 127.1 (2C), 127.0, 126.2 (t, *J* = 4.72 Hz), 125.7, 123.9, 122.5 (q, *J* = 276.0 Hz, CF_3), 121.1, 105.5 (t, *J* = 9.2 Hz). ^{15}N NMR (40.5 MHz, $CDCl_3$) δ: −200.1 (N-3a), −192.6 (N-4a), −7.9 (NO). ^{19}F NMR (376.5 MHz, $CDCl_3$) δ: −137.1 (m, BF_2), −54.9 (CF_3). ^{11}B NMR (128.4 MHz, $CDCl_3$) δ: 1.1 (t, *J* = 30.9 Hz, BF_2). Found, %: C, 65.61; H, 3.42; F, 18.05; N, 7.68. $C_{29}H_{17}BF_5N_3O$ (529.28). Calcd, %: C, 65.81; H, 3.24; B, 2.04; F, 17.95; N, 7.94; O, 3.02.

3-Phenyl-5-(5-{2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1H-pyrrol-2-yl]-ethyl}-1H-pyrrol-2-yl)isoxazole (**4**). The mixture of 2,2,2-trifluoro-1-[5-(naphthalen-2-yl)-1*H*-pyrrol-2-yl]ethan-1-ol (**2**) (1.456 g,

5.0 mmol), 3-phenyl-5-(1*H*-pyrrol-2-yl)ioxazole (**3**) (1.051 g, 5.0 mmol) and P_2O_5 (0.780 g, 5.5 mmol) in CH_2Cl_2 (50 mL) was stirred at room temperature for 20 h. Then, the mixture was diluted with a saturated solution of $NaHCO_3$ (50 mL). The organic layer was separated, and an aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were washed with water (3 × 30 mL) and dried over $CaCl_2$. After removing the solvent, 2.248 g (93%) of compound **4** was obtained as a dark rose solid, mp 104–106 °C. IR spectrum (film) ν, cm^{-1}: 3422, 3274, 3058, 3012, 2926, 1699, 1684, 1627, 1559, 1511, 1456, 1400, 1256, 1190, 1162, 1108, 1042. ^{1}H NMR (400.13 MHz, DMSO-d_6) δ: 12.10 (br. s, 1H, NH), 11.60 (br. s, 1H, NH), 8.12 (s, 1H, naphthyl), 7.91–7.81 (m, 6H, Ph, naphthyl), 7.54–7.48 (m, 4H, Ph, naphthyl), 7.45–7.42 (m, 1H, Hp, Ph), 7.12 (s, 1H, H-4, oxazole), 6.73–6.72 (m, 1H, H-4′), 6.68–6.66 (m, 1H, H-3), 6.40–6.38 (m, 1H, H-3′), 6.31–6.30 (m, 1H, H-4), 5.19 (q, *J* = 9.6 Hz, 1H, CH). ^{13}C NMR (100.6 MHz, DMSO-d_6) δ: 164.0, 162.1, 133.4, 131.6, 131.5, 130.2, 129.9, 129.1 (2C), 128.6, 128.2, 128.0, 127.6, 127.4, 126.5, 126.47 (2C), 125.21, 125.20, 125.15 (q, *J* = 279.5 Hz, CF_3), 123.1, 120.5, 119.8, 110.1, 110.06, 109.7, 107.0, 95.2, 41.9 (q, *J* = 29.7 Hz, CH). ^{19}F NMR (376.5 MHz, $CDCl_3$) δ −67.9 (m, *J* = 8.58 Hz, CF_3). Found, %: C, 72.22; H, 4.09; F, 11.92; N, 8.52. $C_{29}H_{20}F_3N_3O$ (483.49). Calcd, %: C, 72.04; H, 4.17; F, 11.79; N, 8.69; O, 3.31.

2,2,2-Trifluoro-1-[5-(naphthalen-2-yl)pyrrol-2-yl]ethan-1-ol (**2**). To the mixture of 2,2,2-trifluoro-1-[5-(naphthalen-2-yl)pyrrol-2-yl]ethanone (2.347 g, 8.2 mmol) and $NaHCO_3$ (1.447 g, 17.2 mmol, 2.1 equiv.) in MeOH (65 mL) $NaBH_4$ (0.620 g, 16.4 mmol) was added in portions with 10 min of intensive stirring (room temperature). The mixture was stirred for 50 min. The residue, after removing MeOH, was diluted with water (3 mL), extracted with diethyl ether (4 × 10 mL) and dried over Na_2SO_4. After removing the solvent, compound **2** was obtained as a beige solid (2.265 g, 95%), mp 132–134 °C. IR spectrum (film), ν, cm^{-1}: 3421, 3057, 3024, 2928, 1700, 1684, 1629, 1605,1509, 1504, 1457, 1405, 1385, 1362, 1269, 1210, 1174, 1124, 1042. ^{1}H NMR (400.13 MHz, $CDCl_3$) δ: 8.95 (br. s, 1H, NH), 7.89–7.81 (m, 4H, naphthyl), 7.68–7.66 (m, 1H, naphthyl), 7.52–7.44 (m, 2H, naphthyl), 6.62–6.61 (m, 1H, H-4), 6.61–6.40 (m, 1H, H-3), 5.18 (q, *J* = 6.6 Hz, $CHCF_3$), 3.12 (br. s, 1H, OH). ^{13}C NMR (100.6 MHz, DMSO-d_6) δ: 133.5, 131.7, 131.5, 130.0, 128.2, 127.9, 127.7, 127.6, 126.5, 124.8 (q, *J* = 282.3 Hz, CF_3), 125.3, 123.2, 120.7, 109.0, 106.9, 65.9 (q, *J* = 32.3 Hz, C-OH). ^{15}N NMR (40.5 MHz, $CDCl_3$) δ: −240.5 (NH). ^{19}F NMR (376.5 MHz, $CDCl_3$) δ: −78.3 (d, *J* = 6.6 Hz, CF_3). Found, %: C, 66.11; H, 4.01; F, 19.39; N, 4.99. $C_{16}H_{12}F_3NO$ (291.27). Calcd, %: C, 65.98; H, 4.15; F, 19.57; N, 4.81; O, 5.49.

3-Phenyl-5-(pyrrol-2-yl)isoxazole (**3**). A solution of $NH_2OH{\cdot}HCl$ (2.088 g, 30.0 mmol) in H_2O (5 mL) was added to a solution of NaOH (1.80 g, 45.0 mmol) in H_2O (5 mL). This was added to a solution of 2-benzoylethynylpyrrole (0.585 g, 3.0 mmol) in DMSO (50 mL) and the reaction mixture was stirred at 45–50 °C for 4 h. The reaction mixture was cooled to room temperature and poured into brine, residue filtered off, washed with water and dried, giving pure isoxazole **3** (0.524 g, 83%) as light yellow crystals, mp 123–125 °C. Spectral characteristics of isoxazole **3** correspond to the literature data [16].

Supplementary Materials: The followings can be downloaded online. Copies of ^{1}H NMR, ^{13}C, ^{19}F, ^{11}B NMR. Normalized absorption and fluorescence spectra of compound **1**.

Author Contributions: Conceptualization, B.A.T. and L.N.S.; methodology, E.F.S., O.V.P. and D.N.T., preparation of compounds, E.F.S., O.V.P. and D.N.T., formal analysis, I.A.U.; data curation, L.N.S.; writing—original draft preparation, L.N.S., O.V.P. and D.N.T.; writing—review and editing, O.V.P., E.F.S., I.A.U. and L.N.S.; supervision, Boris A. Trofimov. All authors have read and agreed to the published version of the manuscript.

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