

Short Note

# 3-(Phenylethynyl)-7H-benzo[de]anthracen-7-one

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**Abstract:** The present work describes the facile synthesis of 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one via a Sonogashira coupling reaction. The structure of the synthesized benzanthrone derivative is characterized by <sup>1</sup>H- and <sup>13</sup>C-NMR spectroscopy and high-resolution mass spectrometry. The photophysical properties of the title compound are investigated by means of UV-Vis and fluorescence spectroscopy in various organic solvents.

**Keywords:** benzanthrone; fluorescence; Sonogashira coupling

## 1. Introduction

Amongst anthraquinoid dyes, 7H-benzo[de]anthracen-7-one (benzanthrone) derivatives have gained wide recognition lately due to their excellent luminescent properties, such as photostability, sizable Stokes shifts, and noticeable solvatochromism, as well as their tunable fluorescence emission (from green to red) that is dependent on the nature of the substituents of the benzanthrone molecule and solvent properties [1–3] which enable these organic chromophores for use in a vast number of applications, both for scientific and technological needs. Namely, these compounds have reasonable potential to be used: as lipophilic fluorescent probes for parasitic trematodes and nematodes; for the diagnostics of several plant species' callus embryos by confocal laser scanning microscopy imaging [4–7]; for the selective detection of amyloid fibrils of the enzyme lysozyme [8,9]; as luminophore dyes in liquid crystal displays and polymeric materials [10–12]; for the production of organic thin films; and as probes for chromium(III) cations and the pH of solutions [13–15].

Currently, amid many organic chromophores,  $\pi$ -conjugated luminescent molecules have engrossed much attention owing to their high-fluorescence quantum yields, satisfactory robustness, and biocompatibility, as well as capability to emit light in solutions, nanoaggregates and thin films, thus qualifying these compounds for use in sensing and imaging applications [16]. Several theoretical and practical studies have shown that upon the direct attachment of phenylacetylene moieties to luminescent molecules such as pyrene [17–19], carbazole [20], anthraquinone [21], naphthalimide, and quinolythiazole [22,23], photophysical parameters can be adjusted, enhancing fluorescent properties. Furthermore, research indicates that fluorescence yields, the size of Stokes shifts, desired absorption, and emission maxima can be modulated by the introduction of electron-withdrawing and electron-donating groups onto the phenyl rings of phenylacetylene moieties as well as the length of  $\pi$ -conjugation [24].

Besides being excellent fluorophores, phenylacetylene derivatives are useful precursors to a wide range of other compounds and materials [25–27].

With all the above mentioned in mind, we have decided to provide our knowledge on the as-yet unreported phenylacetylene derivative of benzanthrone. Herein, we report the synthesis and photophysical properties of the newly obtained compound.



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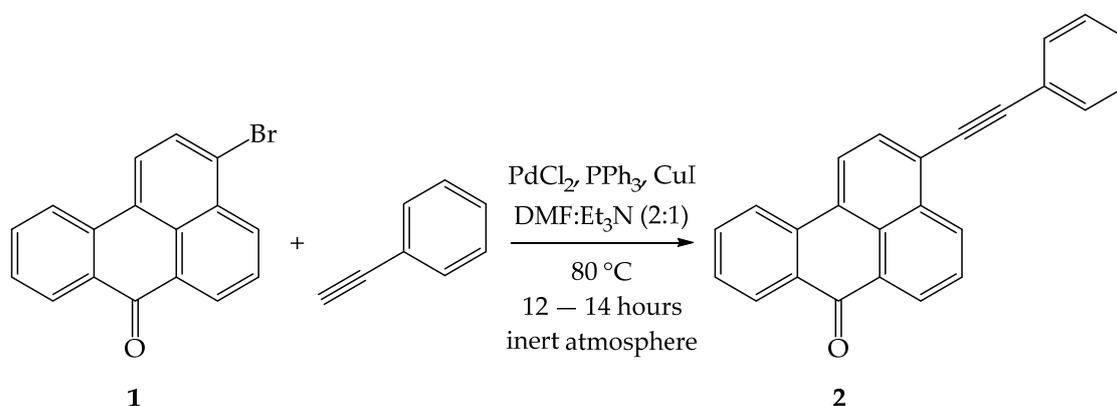


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## 2. Results and Discussion

### 2.1. Synthesis

As far as we know, in the scope of palladium-catalyzed reactions, only aryl cyanation and Buchwald–Hartwig amination reactions have been used to obtain new benzanthrone derivatives [13,28], while the Sonogashira coupling, which is employed for the synthesis of 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one (**2**) (Scheme 1), has not yet been utilized.



**Scheme 1.** Synthesis of 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one (**2**).

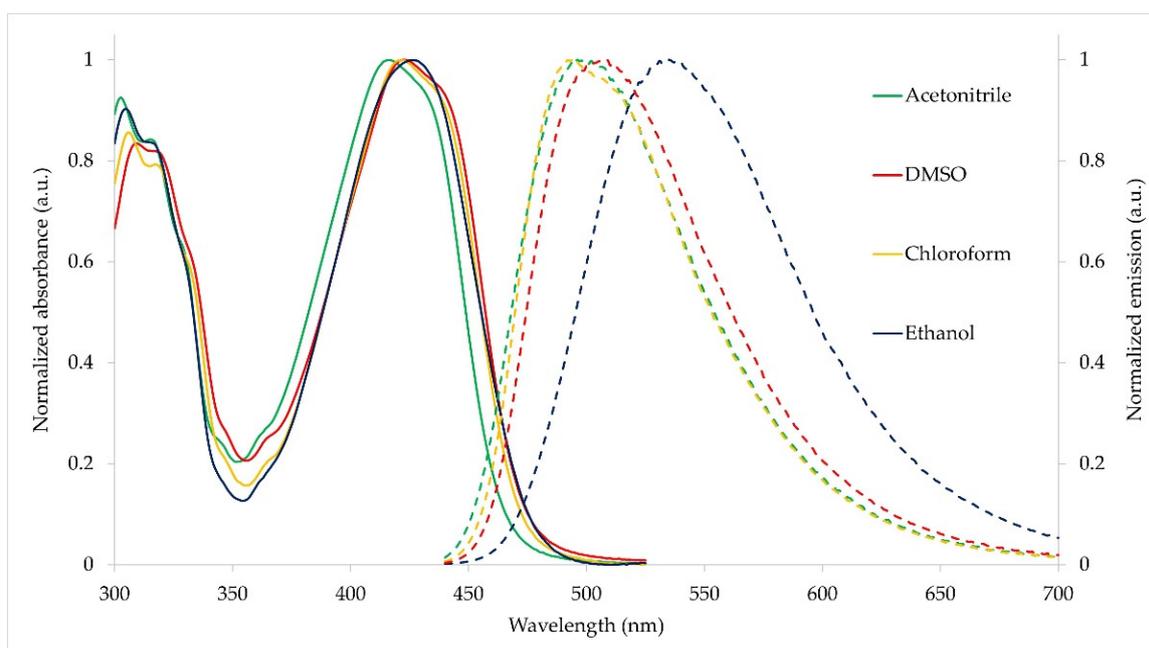
The title compound **2** was synthesized under Sonogashira coupling reaction conditions using 10 mol % PdCl<sub>2</sub> as the pre-catalyst with 20 mol % PPh<sub>3</sub> as the ligating species and 10 mol % CuI as the co-catalyst in *N,N*-dimethylformamide (DMF) at 80 °C with triethylamine as a base [29]. The structure of the obtained compound **2** was confirmed by <sup>1</sup>H-NMR spectroscopy (see Supplementary Materials), with the corresponding doublets (d), triplets (t), and multiplets (m) of aromatic protons (δ 8.62–7.33 ppm) typical for the phenyl group and benzanthrone residue. In the APT NMR spectrum, the appropriate peak of the benzanthrone carbonyl group carbon at 183.5 ppm, peaks of acetylene carbon atoms at 87.1 and 97.0 ppm, and peaks of aromatic carbon atoms (122.9–135.6 ppm) were found and the obtained data are in good correlation with the previously reported NMR studies of other benzanthrone derivatives [30–32]. The obtained infrared spectrum shows a carbon–hydrogen (C–H) stretching frequency band at 3057 cm<sup>−1</sup>, a peak of benzanthrone carbonyl group (C=O) vibrations at 1654 cm<sup>−1</sup>, and a band of carbon–carbon triple-bond (C≡C) vibrations at 2201 cm<sup>−1</sup>, characteristic for alkynes (see Supplementary Materials).

### 2.2. Photophysical Properties

Basic absorption and fluorescence characteristics are presented in Table 1. The photophysical properties of compound **2** were examined in organic solvents of varying polarity (Figure 1).

**Table 1.** Photophysical parameters of 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one in various solvents (at a concentration of 1 × 10<sup>−5</sup> mol L<sup>−1</sup>).

Solvent	Dielectric Constant	Absorption λ <sub>abs</sub> , nm	Fluorescence λ <sub>em</sub> , nm	Quantum Yield
Chloroform	4.89	422	492	0.38
Ethanol	24.55	426	535	0.68
Acetonitrile	35.94	416	496	0.22
Dimethyl sulfoxide	46.45	423	507	0.32



**Figure 1.** The UV-Vis absorption and fluorescence emission spectra of 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one in various organic solvents.

The mechanisms responsible for the fluorescence phenomena (such as internal charge transfer) have been thoroughly described in earlier publications [1,4]. The absorption spectra of the obtained benzanthrone phenylacetylene derivative **2** exhibited a broad band around 416–426 nm. In solution, substance **2** is fluorescent from 492 nm (chloroform) to 535 nm (ethanol), and hence the bathochromic shift reaches 43 nm. The quantum yields depending on solvent varied from 22% in acetonitrile to 68% in ethanol. The highest quantum yield is attained in ethanol most likely due to the protic nature of the solvent, which is possibly because the stabilization of the excited states prevents non-radiative decay. The polarity effect of the medium on fluorescence was pronounced to a greater extent than on the absorption of compound **2**, attaining a highest Stokes shift value of 109 nm ( $4782\text{ cm}^{-1}$ ) in ethanol.

### 3. Materials and Methods

#### 3.1. Materials and Basic Measurements

All of the reagents and solvents were obtained commercially and used without any additional purification. The progress of the reaction and the assessment of the purity of the synthesized compound was performed by TLC on MERCK Silica gel F254 plates in dichloromethane (DCM) as an eluent and visualized under UV light. Column chromatography was carried out on silica gel (60 Å, 40–63 µm, UPAG-AG).

MP70 Melting Point System apparatus was used for the determination of the melting point, and was uncorrected.  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra were recorded on a Bruker Avance 500 MHz (Bruker Corporation, Billerica, MA, USA) in  $\text{CDCl}_3$  at ambient temperature, using solvent peaks as the internal reference. Chemical shift ( $\delta$ ) values are reported in ppm. The high-resolution mass spectrum was recorded on an Agilent 1290 Infinity series UPLC connected to an Agilent 6230 TOF mass spectrometer. The IR spectrum was recorded on a Thermo Scientific Nicolet i550 Spectrometer (ATR accessory; no. of scans: 64; resolution: 4; data spacing:  $0.482\text{ cm}^{-1}$ ).

UV-Vis absorption spectra were recorded with a PerkinElmer Lambda 35 spectrometer. Emission spectra and quantum yields for solutions were recorded using a QuantaMaster 40 steady-state spectrofluorometer (Photon Technology International, Inc. (Birmingham,

NJ, USA)) equipped with a 6-inch integrating sphere by LabSphere, utilizing the software package provided by the manufacturer.

### 3.2. Synthesis and Characterization

#### 3-(Phenylethynyl)-7H-benzo[de]anthracen-7-one (2).

3-Bromobenzanthrone (309 mg, 1 mmol), PdCl<sub>2</sub> (18 mg, 0.1 mmol), PPh<sub>3</sub> (53 mg, 0.2 mmol) and CuI (19 mg, 0.1 mmol) were placed in a 20 mL screw-cap vial under an argon atmosphere followed by the addition of DMF (10 mL) and Et<sub>3</sub>N (5 mL). The reaction mixture was heated to 80 °C and kept at this temperature for 12–14 h (TLC control). When conversion was completed, the reaction mixture was then poured into a mixture of aqueous ammonia sol. 27% (10 mL), water (20 mL), and ice. The resulting precipitate was well mixed for 30 min, filtered, and dried in a desiccator over anhydrous CaCl<sub>2</sub>. Then, it was purified by means of column chromatography (eluent: DCM) to obtain a yellow compound in 74% yield with m.p. of 150–151 °C. R<sub>f</sub> = 0.68 (DCM). IR, cm<sup>-1</sup>: 3057 (C–H), 2201 (C≡C), 1654 (C=O), 1597, 1574, 1509, 1480, 1443, 1385, 1312, 1275, 1204, 1169, 1128, 1082, 1042, 999, 956, 916, 837, 777, 744, 687, 656, 624, 600, 571, 526, 474. HRMS (ESI): *m/z* calculated for [C<sub>25</sub>H<sub>14</sub>O + H<sup>+</sup>] 331.1117, found 331.1115.

<sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>) δ 8.65–8.58 (m, 2H), 8.34 (dd, *J* = 7.9, 1.5 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 8.08 (d, *J* = 8.1 Hz, 1H), 7.69 (t, *J* = 7.3 Hz, 2H), 7.61–7.54 (m, 3H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.37–7.28 (m, 3H).

<sup>13</sup>C-NMR (126 MHz, CDCl<sub>3</sub>) δ 183.5 (C, C=O), 135.6 (C), 133.4 (CH), 133.3 (CH), 132.7 (C), 131.8 (CH), 130.8 (C), 130.6 (CH), 130.1 (CH), 128.9 (CH), 128.6 (C), 128.6 (CH), 128.5 (CH), 128.1 (CH), 127.7 (C), 127.1 (CH), 127.0 (C), 123.4 (CH), 123.3 (C), 123.2 (CH), 122.9 (C), 97.0 (C≡C), 87.1 (C≡C).

## 4. Conclusions

As a result, 3-(phenylethynyl)-7H-benzo[de]anthracen-7-one was obtained in good yield via a Sonogashira coupling reaction. The structure of the acquired compound was confirmed by means of <sup>1</sup>H-NMR, APT, and IR spectroscopy and HRMS. UV-Vis and fluorescence spectroscopy in various organic solvents was used to investigate the photophysical properties of the title compound. The study revealed that the substance is fluorescent in different solutions, attaining the highest quantum yield of 68% and the highest Stokes shift of 109 nm in ethanol. Such notable luminescent properties allow this compound to be further studied for the visualization of biological objects and use in other technological applications.

**Supplementary Materials:** The following are available online. Figure S1. <sup>1</sup>H-NMR spectrum of compound 2; Figure S2. <sup>1</sup>H-NMR spectrum of compound 2 (aromatic region); Figure S3. APT spectrum of compound 2; Figure S4. APT spectrum of compound 2 (120–140 ppm); Figure S5. HRMS (ESI) data for compound 2. Figure S6. IR spectrum of compound 2.

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