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АКАДЕМИЯСЫ» РҚБ

«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ  
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ» РҚБ

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РОО «НАЦИОНАЛЬНОЙ  
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## INCLUSION OF AZULENE INTO THE BACKBONE OF CONJUGATED OLIGOMERS: IMPROVEMENT OF PROTON SENSITIVITY AND ELECTRONIC ABSORPTION

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**Abstract.** The non-alternant aromatic hydrocarbon azulene is gaining attention as a modular building block for organic  $\pi$ -conjugated materials due to its dipolar structure and unique electronic and optical properties.

In this study, new pyrrole-azulene  $\pi$ -conjugated co-oligomers—3,6-Bis(5-(1-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **5** and 3,6-Bis(5-(2-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **7** — were synthesized by reacting 2-(1-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolanes and 2-(2-azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolanes with 3,6-bis(5-bromo-2-thienyl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione via Suzuki–Miyaura cross-coupling in the presence of a palladium catalyst. The chemical structure and

purity of the synthesized co-oligomers **5** and **7** were confirmed using NMR ( $^1\text{H}$  and  $^{13}\text{C}$ ), IR spectroscopy, mass spectrometry, and elemental analysis. It was established that the resulting regioisomeric oligomers **5** and **7** are stable compounds and fully soluble in organic solvents such as dichloromethane, chloroform, toluene, chlorobenzene, and tetrahydrofuran at room temperature. This molecular design leverages both the dipolar nature of azulene and its capability for protonation-deprotonation reactions through the electron-donating five-membered ring. The photophysical properties of the pyrrole-azulene  $\pi$ -conjugated co-oligomers were characterized by UV-Vis and fluorescence spectroscopy. It was shown that, under protonation with trifluoroacetic acid, the co-oligomers exhibit significant changes in their absorption spectra in the UV-visible region, with increased intensity and a red shift of absorption maxima up to 830 nm, indicating a significant reduction in the HOMO-LUMO energy gap due to extended conjugation and enhanced  $\pi$ -electron delocalization. These results demonstrate that the incorporation of azulene units into heterocyclic conjugated oligomer structures holds great potential for developing new proton-sensitive organic functional materials.

**Key words:** azulene,  $\pi$ -conjugated azulenes, azulene oligomers, cross-coupling, electron absorption, proton sensitivity.

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## **ҚОСАРЛАНҒАН ОЛИГОМЕРЛЕР НЕГІЗІНЕ АЗУЛЕНДІ ЕНГІЗУ: ПРОТОНҒА СЕЗІМТАЛДЫҚ ПЕН ЭЛЕКТРОНДЫҚ СІЦІРУДІ ЖАҚСARTY**

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**Аннотация.** Азулен – альтернантты емес ароматты көмірсутек, оның ерекше дипольді химиялық құрылымы, сондай-ақ ерекше электрондық және оптикалық қасиеттері жаңа органикалық  $\pi$ -конъюгацияланған функционалдық материалдарды құруға арналған ықтимал модульдік құрылыс материалы ретінде көп назар аудартады.

Жұмыста 2-(1-Азуленил)-4,4,5,5-тетраметил-1,3,2-диоксбораландар мен 2-(2-Азуленил)-4,4,5,5-тетраметил-1,3,2-диоксбораландар Сузуки-Мияура кросс-байланыстыру реакциясында палладий катализаторының қатысуымен 3,6-Бис(5-бромо-2-тиенил)-2,5-бис(2-этилгексил)пирроло[3,4-с]пиррол-1,4-дионмен әрекеттесіп, келесі жаңа пирроло-азуленді  $\pi$ -конъюгацияланған сополимерлер синтезделді: 3,6-Бис(5-(1-азуленил)тиофен-2-ил)-2,5-ди(2-этилгексил)пирроло[3,4-с]пиррол-1,4-дион **5** және 3,6-Бис(5-(2-азуленил)тиофен-2-ил)-2,5-ди(2-этилгексил)пирроло[3,4-с]пиррол-1,4-дион **7**. Синтезделген **5** және **7** сополимерлердің химиялық құрылымы мен тазалығы ЯМР ( $^1\text{H}$  және  $^{13}\text{C}$ ), ИҚ спектроскопиясы, масс-спектрометрия және элементтік талдау әдістерімен дәлелденді. Алынған региоизомерлі сополимерлердің тұрақты қосылыстар екені және дихлорметан, хлороформ, толуол, хлорбензол, тетрагидрофуран сияқты органикалық еріткіштерде бөлме температурасында толық еритіні анықталды. Мұндай молекулалық конструкция азуленнің дипольдік табиғатын да, оның электрондонорлы бес мүшелі сақина бойында протондандыру-протондансыздандыру реакцияларына түсу қабілетін де пайдаланатыны көрсетілген. Алынған пирроло-азуленді  $\pi$ -конъюгацияланған сополимерлердің фотофизикалық қасиеттері Ультракүлгін-көрінетін (UV-Vis) және флуоресценция спектроскопиясы әдістерімен сипатталды. Үшфторсірке қышқылы арқылы протондалған жағдайда сополимерлердің Ультракүлгін-көрінетін (UV-Vis) аймағында жұтылу спектрлерінің айтарлықтай өзгерістері байқалды, бұл жұтылу максимумдарының 830 нм-ге дейін қызыл ығысуы және интенсивтілігінің артуымен сипатталды. Бұл нәтижелер  $\pi$ -электрондардың делокализациясының ұлғаюы мен конъюгацияның кеңеюіне байланысты ең жоғарғы бос емес молекулалық орбиталь – ең төменгі бос молекулалық орбиталь (HOMO-LUMO) молекулалық орбитальдарының энергетикалық алшақтығының төмендегенін көрсетті. Алынған нәтижелер азуленді блоктарды гетероциклді конъюгацияланған олигомерлердің құрылымына енгізудің жаңа протондық органикалық функционалдық материалдарды жасауға үлкен әлеуеті бар екенін дәлелдейді.

**Түйін сөздер:** азулен,  $\pi$ -конъюгацияланған азулендер, азулен олигомерлері, кросс-байланыстыру, электрондарды жұту, протондарға сезімталдық.

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## **ВВЕДЕНИЕ АЗУЛЕНА В ОСНОВУ СОПРЯЖЕННЫХ ОЛИГОМЕРОВ: УЛУЧШЕНИЕ ПРОТОННОЙ ЧУВСТВИТЕЛЬНОСТИ И ЭЛЕКТРОННОГО ПОГЛОЩЕНИЯ**

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**Аннотация.** Неальтернантный ароматический углеводород азулен как потенциальный модульный строительный блок для создания новых органических  $\pi$ -сопряженных функциональных материалов привлекает все больше внимания благодаря своей уникальной дипольной химической структуре, а также особым электронным и оптическим свойствам.

В данной работе взаимодействием 2-(1-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolanes и 2-(2-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolanes с 3,6-Bis(5-bromo-2-thienyl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione в условиях кросс-сочетания Сузуки–Мияуры в присутствии палладиевого катализатора были синтезированы новые пирроло-азуленовые  $\pi$ -сопряженные со-олигомеры: 3,6-Bis(5-(1-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **5** и 3,6-Bis(5-(2-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **7**. Химическое строение и чистота синтезированных со-олигомеров **5** и **7** были доказаны ЯМР (ЯМР <sup>1</sup>H и <sup>13</sup>C), ИК, масс-спектрометрией и элементным анализом. Установлено, что полученные региоизомерные со-олигомеры **5** и **7** являются стабильными соединениями и полностью растворимы в органических растворителях, таких как дихлорметан, хлороформ, толуол, хлорбензол, тетрагидрофуран при комнатной температуре. Показано, что



такая молекулярная конструкция использует как дипольную природу азулена, так и его способность к реакциям протонирования – депротонирования по электронодонорному пятичленному циклу. Фотофизические свойства полученных пирроло-азуленовых  $\pi$ -сопряженных со-олигомеров охарактеризованы УФ-Vis и флуоресцентной спектроскопией. Показано, что, полученные со-олигомеры в условиях протонирования трифторуксусной кислотой демонстрируют значительные изменения спектров поглощения в УФ-видимой области с повышением интенсивности и красным смещением максимумов поглощения до 830 нм, указывающее на существенные уменьшения энергетической щели молекулярных орбиталей HOMO-LUMO вследствие расширения сопряжения и роста делокализации  $\pi$ -электронов. Полученные результаты демонстрируют, что введение азуленовых звеньев в структуру гетероциклических сопряженных олигомеров имеют большой потенциал для разработки новых протонных органических функциональных материалов.

**Ключевые слова:** азулен,  $\pi$ -сопряженные азулены, олигомеры азулена, кросс-сочетание, поглощение электронов, чувствительность к протонам.

**Introduction.** The development of a new strategy for the synthesis of functional materials is of considerable interest in terms of improving electronic devices such as molecular switches, field effect transistors (OFETs), light emitting diodes (OLEDs), solar cells (OSCs), nonlinear optical (NLO) and others (Wang, et.al.,2004; Dong, et.al., 2016; Ou, et.al.,2019; Kurotobi, et.al., 2006; Cristian, et.al., 2004; Yamaguchi, et.al., 2016; Nishimura, et.al., 2015). In this direction, research focuses on the application of various building blocks that can fine-tune the electronic structure of the material and lead to improved efficiency and productivity of devices (Foggi, et.al., 2003; Mrozek, et.al., 2001; Ito, et.al., 2003; Salman, et.al., 2005).

To date, the most effective molecules as a building block are non-alternative aromatic hydrocarbons, for example, azulenes. The choice of azulene **1** was predetermined by its unusual polarized structure, which can be considered as a tropylium cation condensed with a cyclopentadienyl anion (Figure 1). Azulene, in contrast to its isomer naphthalene, is colored dark blue due to the reduced aromaticity in conjunction with the large dipole moment of the order of 1.08 D (Chiu, et.al., 2014). Along with this, its low ionization energy and increased electron affinity provide tuning photophysical properties similar to fullerene compounds (Xia, et.al., 2014).

Here we report the synthesis of novel pyrrole-azulene  $\pi$ -conjugated co-oligomers **5** and **7**, via Suzuki – Miyaura cross-coupling reaction using Pd(PPh<sub>3</sub>)<sub>4</sub> as catalyst. This molecular design takes advantage of both the dipole nature of azulene and its ability to undergo protonation-deprotonation reactions along the electron-donor five-membered cycle.

**Methods and Materials.** NMR spectra of <sup>1</sup>H and <sup>13</sup>C were recorded on a Jeol ECA-500 MHz spectrometer in CDCl<sub>3</sub> at room temperature using tetramethylsilane (TMS) as an internal standard. The NMR spectrometer operating frequencies were 500,15 MHz (<sup>1</sup>H) and 100,61 MHz (<sup>13</sup>C). IR spectra were recorded on a Fourier spectrometer Avatar-360 in KBr tablets. Mass spectra (EI, MALDI-FT) were taken on a Thermo

Fisher Scientific LTQ FT Ultra mass spectrometer. Absorption spectra were recorded on a Shimadzu UV-1800 spectrophotometer.

Commercially available reagents and solvents: azulene, Bis(pinacolato)diboron, 3,6-Bis(5-bromo-2-thienyl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione, Pd(PPh<sub>3</sub>)<sub>4</sub>, [IrCl(cod)]<sub>2</sub>, Ag<sub>2</sub>CO<sub>3</sub>, THF, toluene, DMF and others were used without further purification. Bromoazulene **2** and 2-(2-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **6** were synthesized according to the literature methods.

*2-(1-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3)*. To the mixture of 1-bromoazulene **2** (54 mg, 0.3 mmol) and Bis(pinacolato)diboron (83 mg, 0.33 mmol) in THF (5 mL) was added Pd(PPh<sub>3</sub>)<sub>4</sub> (12 mg, 0.01 mmol) and Ag<sub>2</sub>CO<sub>3</sub> (275 mg, 1 mmol) under argon. The mixture was stirred at 75 °C for 18 hours and then cooled to room temperature. Water (20 mL) was added and then extracted with dichloromethane (3 × 20 mL). The combined organic extracts were dried over MgSO<sub>4</sub>, evaporated in vacuo and finally purified by column chromatography on silica gel using hexane: EtOAc (4:1) to give a violet crystal (66 mg, 88% yield). M.p. 56-57°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500,15 MHz): δ 1.43 (s, 12 H), 7.30 (t, J=9.7 Hz, 1 H), 7.40 (t, J = 9.9 Hz, 1 H), 7.43 (d, J= 3.7 Hz, 1 H), 7.68 (t, J= 9.9 Hz, 1 H), 8.36 (d, J=3.7 Hz, 1 H), 8.42 (d, J = 9.5 Hz, 1 H), 9.20 (d, J = 9.7 Hz, 1 H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100,61 MHz): δ 24.96, 82.89, 119.03, 124.64, 125.13, 136.42, 137.39, 138.24, 144.62, 145.59, 147.07. HRMS (EI): m/z [M]<sup>+</sup> calcd for C<sub>16</sub>H<sub>19</sub>BO<sub>2</sub> 254.1927; found, 254.1909.

*3,6-Bis(5-(1-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione (5)*. To the mixture of 3,6-Bis(5-bromo-2-thienyl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **4** (68 mg, 0.1 mmol) and 2-(1-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3** (56 mg, 0.22 mmol) in toluene (5 mL) and DMF (0.5 mL) were added Pd(PPh<sub>3</sub>)<sub>4</sub> (12 mg, 0.01 mmol) and Ag<sub>2</sub>CO<sub>3</sub> (181 mg, 0.66 mmol) under argon. The mixture was stirred at 85°C for 18 hours and then cooled to room temperature. Water (20 mL) was added and then extracted with dichloromethane (3 × 20 mL). The combined organic extracts were dried over MgSO<sub>4</sub>, evaporated in vacuo and finally purified by column chromatography on silica gel using hexane: EtOAc (2:3) to give a dark blue solid (46 mg, 60% yield). IR ν/cm-1: 1610 cm-1 (C=O). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500,15 MHz): 0.72–1.10 (m, 30H), 4.25 (br, m, 4H), 6.50 (br, m, 4H), 7.05–7.07 (m, 2H), 7.62–7.83 (m, 7H), 8.0 (m, 2H), 8.21–8.25 (m, 1H), 8.53–8.51 (m, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100,61 MHz): δ 10.7, 13.9, 23.1, 23.6, 28.6, 30.4, 39.4, 46.6, 106.5, 109.9, 118.5, 119.1, 123.1, 124.9, 125.2, 132.4, 134.7, 135.9, 136.2, 137.8, 139.1, 143.4, 143.7, 155.4, 161.3. HRMS (EI): m/z [M]<sup>+</sup> calcd for C<sub>50</sub>H<sub>52</sub>N<sub>2</sub>O<sub>2</sub>S<sub>2</sub> 776.3725; found, 776.3705

*3,6-Bis(5-(2-azulenyl)thiophen-2-yl)-2,5-di(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione (7)*. To the mixture of 3,6-Bis(5-bromo-2-thienyl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4-dione **4** (68 mg, 0.1 mmol) and 2-(2-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **6** (56 mg, 0.22 mmol) in toluene (5 mL) and DMF (0.5 mL) were added Pd(PPh<sub>3</sub>)<sub>4</sub> (12 mg, 0.01 mmol) and Ag<sub>2</sub>CO<sub>3</sub> (181 mg, 0.66 mmol) under argon. The mixture was stirred at 85 °C for 18 hours and then cooled to room temperature. Water (20 mL) was added and then extracted with dichloromethane (3 × 20 mL). The combined organic extracts were dried over MgSO<sub>4</sub>, evaporated in vacuo and finally purified by column chromatography on silica gel using hexane: EtOAc (2:3)

to give a dark blue solid (61 mg, 80% yield). IR (KBr): 1610  $\text{cm}^{-1}$  (C=O).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500,15 MHz): 0.72 (m, 6H), 1.08 (m, 24H), 4.26 (m, 4H), 7.00 (m, 2H), 7.40–7.43 (m, 2H), 7.59–7.68 (m, 2H), 7.76 (m, 2H), 7.86–7.90 (m, 2H), 8.12 (m, 2H), 8.34 (m, 2H), 8.27 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100,61 MHz):  $\delta$  10.6, 14.1, 23.3, 23.6, 28.5, 30.4, 39.4, 46.8, 107.7, 113.0, 113.6, 123.4, 124.5, 132.7, 136.6, 137.0, 137.1, 141.3, 145.0, 155.3, 161.2. HRMS (EI):  $m/z$   $[\text{M}]^+$  calcd for  $\text{C}_{50}\text{H}_{52}\text{N}_2\text{O}_2\text{S}_2$  776.3725; found, 776.3707.

**Results and discussion.** Synthetic routes leading to pyrrole-azulene conjugated co-oligomers **5** and **7** are shown in Schemes 1 and 2. As can be seen from Scheme 1, bromoazulene **2** was synthesized according to the literature methodology (Merghatuly, et.al., 2023). The borylation reaction of **2** with Bis(pinacolato)diboron in THF in the presence of  $\text{Pd}(\text{PPh}_3)_4$  then leads to the key compound 2-(1-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3** in high 88% yield.

Further, the Suzuki-Miyaura cross-coupling reaction between pyrrole-dione **4** and **3** in toluene in the presence of  $\text{Pd}(\text{PPh}_3)_4$  catalyst gives the final co-oligomer **5** in good 60% yield. Another key molecule 2-(2-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **6** (Scheme 2) was prepared by direct  $\text{C}_2$ -H borylation of azulene according to the literature methodology (Kurotobi, et.al.,2003).

Similarly, the Suzuki-Miyaura reaction between **4** and 2-(2-Azulenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **6** results in the co-oligomer **7** product with high yield of 60%. The obtained regioisomeric co-oligomers **5** and **7** are completely soluble in organic solvents such as dichloromethane, chloroform, toluene, tetrahydrofuran at room temperature.

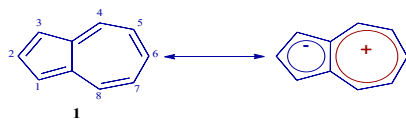
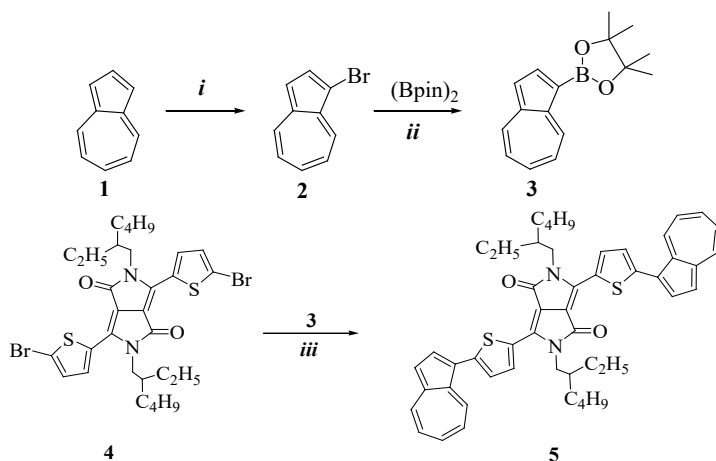
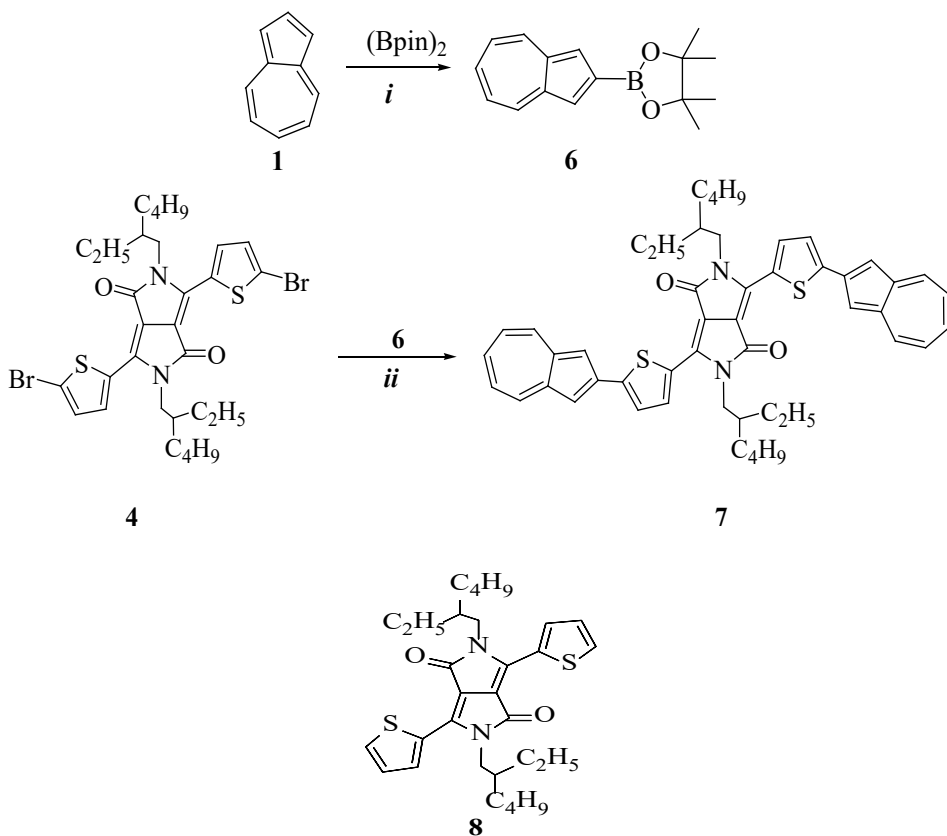


Figure 1. Polarized resonance structure of azulene **1**.



Scheme 1. Reagents and conditions: (i) NBS, DCM, rt; (ii)  $\text{Pd}(\text{PPh}_3)_4$ ,  $\text{Ag}_2\text{CO}_3$ , THF, 75°C; (iii)  $\text{Pd}(\text{PPh}_3)_4$ ,  $\text{Ag}_2\text{CO}_3$ , toluene, DMF, 60°C.



Scheme 2. Reagents and conditions: (i)  $[\text{IrCl}(\text{cod})_2]$ , 2,2'-bpy, cyclohexane, reflux; (ii)  $\text{Pd}(\text{PPh}_3)_4$ ,  $\text{Ag}_2\text{CO}_3$ , toluene, DMF, 60°C

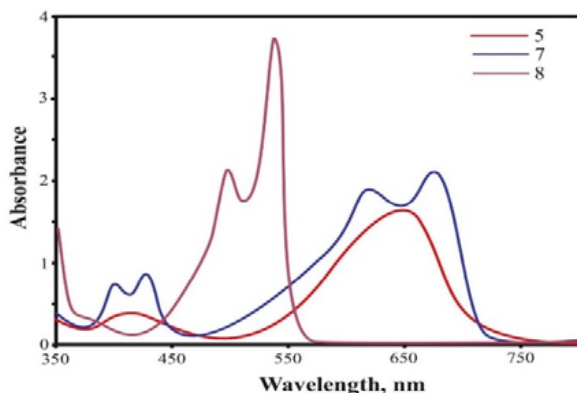
The absorption spectra of co-oligomers **5** and **7** in UV-Vis range in dichloromethane (DCM) at room temperature are presented in Figure 2 and the corresponding data are summarized in Table 1. To evaluate the efficacy of  $\pi$ -conjugation through a thienyl spacer, the electron spectra of the co-oligomers were compared with the baseline pyrrolopyrrole-dione **8** (Table 1, Figure 2). As can be seen, **5** and **7** show significant redshifts in the visible region at 110 and 133 nm relative to compound **8**, indicating the decrease in the energy gap of the frontal molecular orbitals of HOMO-LUMO due to the expansion of the  $\pi$ -conjugation and the growth of delocalization of  $\pi$ -electrons.

It should be noted that regioisomeric co-oligomers **5** and **7** show significant absorbances in the long wavelength part of the spectrum in the range 560-720 nm, which are characteristic of materials of solar cells and near IR dyes (Kurotobi, et.al., 2006; Cristian, et.al.,2004; Yamaguchi, et.al., 2016). In addition, there is one broad band in the spectrum and the hypsochromic shift at 23 nm of the absorption maximum of **5** compared to **7**. This can occur as the result of the decrease in the energy gap HOMO - LUMO with the  $\text{C}_2$  replacement of the five-membered azulene cycle **7**, showing the discrete vibration structure (Figure 2).

Table 1 - UV-Vis absorption data for **5**, **7** and **8** in DCM ( $1 \times 10^{-5}$  M) at rt

| Co-oligomers | Absorption   |                                   |   |                      |                                   |
|--------------|--------------|-----------------------------------|---|----------------------|-----------------------------------|
|              | Unprotonated |                                   |   | Protonated           |                                   |
|              | Solvent      | $\lambda_{\text{abs}}, \text{nm}$ | $\epsilon, \text{M}^{-1}\text{cm}^{-1}$ | Solvent <sup>a</sup> | $\lambda_{\text{abs}}, \text{nm}$ |
| <b>5</b>     | DCM          | 416                               | 4000                                    | TFA/DCM (2:8)        | 420                               |
|              |              | 601                               | 15200                                   |                      | 586                               |
|              |              | 650                               | 17000                                   |                      | 628                               |
| <b>7</b>     | DCM          | 431                               | 8800                                    | TFA/DCM (2:8)        | 433                               |
|              |              | 620                               | 19000                                   |                      | 622                               |
|              |              | 673                               | 21000                                   |                      | 798                               |
| <b>8</b>     | DCM          | 355                               | 14600                                   | TFA/DCM(2:8)         | 352                               |
|              |              | 500                               | 21000                                   |                      | 498                               |
|              |              | 540                               | 37500                                   |                      | 538                               |

<sup>a</sup> DCM/TFA = volume to volume

Figure 2. UV-vis absorption spectra of **5**, **7** and **8** in DCM at rt.

Under the conditions of protonation of co-oligomers **5** and **7** with trifluoroacetic acid (when adding 5%, 10%, 15% and 20% TFA) in DCM (Figure 3, Table 1), the significant changes are observed in the electron spectra with the slight (about 22 nm) shift in the absorption maximum at **5** compared to the very significant (about 198 nm) red shift at oligomer **7**. For comparison, starting compound **8** was subjected to the same acid treatment and its absorption spectra showed no change in absorption (Table 1). This observation indicates that protonation proceeds exclusively along the azulene part of molecules **5** and **7**, which gives the 6  $\pi$ -electron tropylium cation (Figure 1), and not along the nitrogen atoms of the pyrrolopyrrole-dione link.

Stepwise changes in absorption spectra in the visible region during protonation of TFA were also found.

When 20% TFA was added, oligomer **5** showed the decrease in absorbance at 650 nm and the appearance of new bands with maxima at 586 and 628 nm (Figure 3, a). At TFA concentrations of 15 and 20%, **7** shows the new absorption band at 830 nm, which gradually shifts to the blue region to 798 nm with increasing intensity and isosbestic point at 693 nm (Figure 3, b). In addition, color changes are observed in solutions of

co-oligomers **5** and **7** when different amounts of TFA are added (Figure 3). So, solution **5** goes from blue to dark blue, and **7** from blue to dark green.

It should be noted that neutralization of acidic solutions **5** and **7** with triethylamine leads to the reduction of the original UV-vis spectra of neutral states, which indicates a completely reversible protonation-deprotonation process.

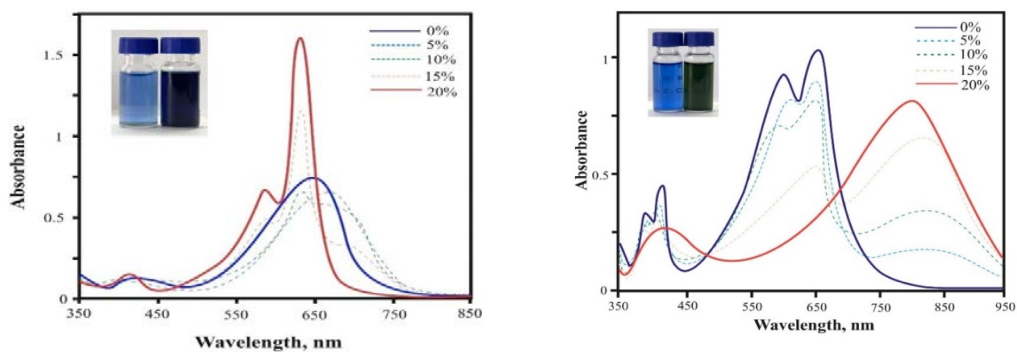


Figure 3. UV-vis spectra (a) **5** and (b) **7** under TFA protonation conditions in DCM.

**Conclusion.** Thus, new pyrrole-azulene  $\pi$ -conjugated co-oligomers **5** and **7** were synthesized by the Suzuki-Miyaura cross-coupling reaction with high yields. Co-oligomers show significant redshifts in the visible region relative to the parent molecule: in neutral states up to 673 nm, and in protonated states up to 830 nm, which indicates significant decreases in the energy gap of the frontal molecular orbitals of HOMO-LUMO, due to the expansion of conjugation and growth of delocalization of  $\pi$ -electrons. Solutions of the resulting co-oligomers in DCM were also found to significantly change color when different amounts of trifluoroacetic acid were added. The results show that the incorporation of azulene units into the heterocyclic conjugated oligomer structure has great potential for the development of novel proton functional materials. It should be noted that the obtained co-oligomers are analogues of previously synthesized fluorene-azulene  $\pi$ -conjugated copolymers: poly[2,7-(9,9-dioctylfluorene-2-azulenyl)-alt11-azulenyl-alt3-azulenyl-alt[1-azulenyl]], which are used in practice as fluorescent proton materials.

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