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Synthesis of Unsymmetrical Thioflavylium Dyes from Julolidine Derivatives and Polyfluorinated Triphenyldihydropyrazoles

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Abstract—Unsymmetrical polymethine dyes have been synthesized from 6-*tert*-butyl-4-methylthioflavylium perchlorates and nitrogen-containing heterocyclic aldehydes derived from julolidine and polyfluorinated triphenyl-4,5-dihydro-1*H*-pyrazoles. Spectral characteristics of the obtained compounds have been studied.

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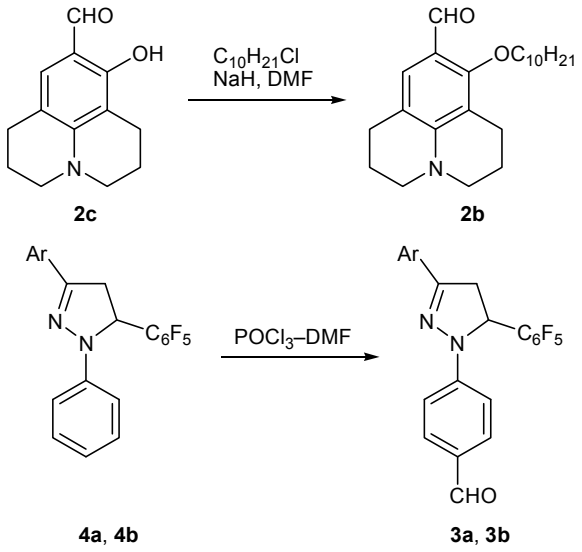
Nonlinear optical devices utilize highly polar unsymmetrical chromophores characterized by second-order nonlinear polarizability [1]. Unsymmetrical polymethine dyes demonstrate high nonlinear optical properties. To fabricate an electro-optic material, a high concentration of a dye in a polymeric matrix should be attained. Wu et al. [2] synthesized a highly soluble dye containing an alkoxy-substituted julolidine ring, which showed a large optical nonlinearity and allowed a concentration of up to 40 wt % to be achieved in the host–guest polymer system. In order to obtain high optical nonlinearity at the semiconductor laser wavelength (λ 1550 nm) for optical fiber data transmission in the field of telecommunications, the dye absorption maximum should be close to the doubled laser frequency, i.e., the dye should absorb in the region λ 700–800 nm [3]. Taking the above stated into account, it seemed reasonable to synthesize highly soluble unsymmetrical polymethine dyes with the long-wave electronic absorption maximum in the region λ 700–800 nm.

Red shift of the absorption maximum of polymethine dyes is determined by both polymethine chain length and heteroatom nature in the heteroaromatic moiety. Extension of the polymethine chain is accompanied by reduction of the thermal and chemical stability and increase of the number of conformations of the dye molecule. Thiopyrylium dyes containing only one double bond in the polymethine chain displayed a red shift of the absorption maximum as compared to their pyrylium and pyridinium analogs [4].

We tried to synthesize polar styryl type thioflavylium dyes possessing one or two *tert*-butyl groups in the acceptor thioflavylium fragment (to improve the solubility) and a cycloaliphatic julolidine fragment or polyfluorinated triphenyldihydropyrazole residue in the donor fragment. We previously synthesized a series of polymethine dyes on the basis of 2- and 4-methylthioflavylium perchlorates with one or two *tert*-butyl groups in their molecules [5]. These symmetrical and unsymmetrical (styryls) dyes showed good film-forming properties and improved solubility in both organic solvents and polymeric matrix; many of them displayed third-order optical nonlinearity [4, 6].

In the present work we have synthesized polar styryl chromophores by condensation of 6-*tert*-butyl-4-methylthioflavylium perchlorates **1a** and **1b** with aldehydes derived from julolidine and polyfluorinated triaryldihydropyrazoles. The aldehyde components based on cyclic amines were selected taking into account higher quantum yields of the corresponding chromophores [7, 8]. Polyfluorinated triaryldihydropyrazoles were described by us previously [9]; they showed intense fluorescence and enhanced photostability compared to nonfluorinated analogs [cf. 10]. The presence of pentafluorophenyl groups in their molecules provides the possibility for subsequent function-

Scheme 1.



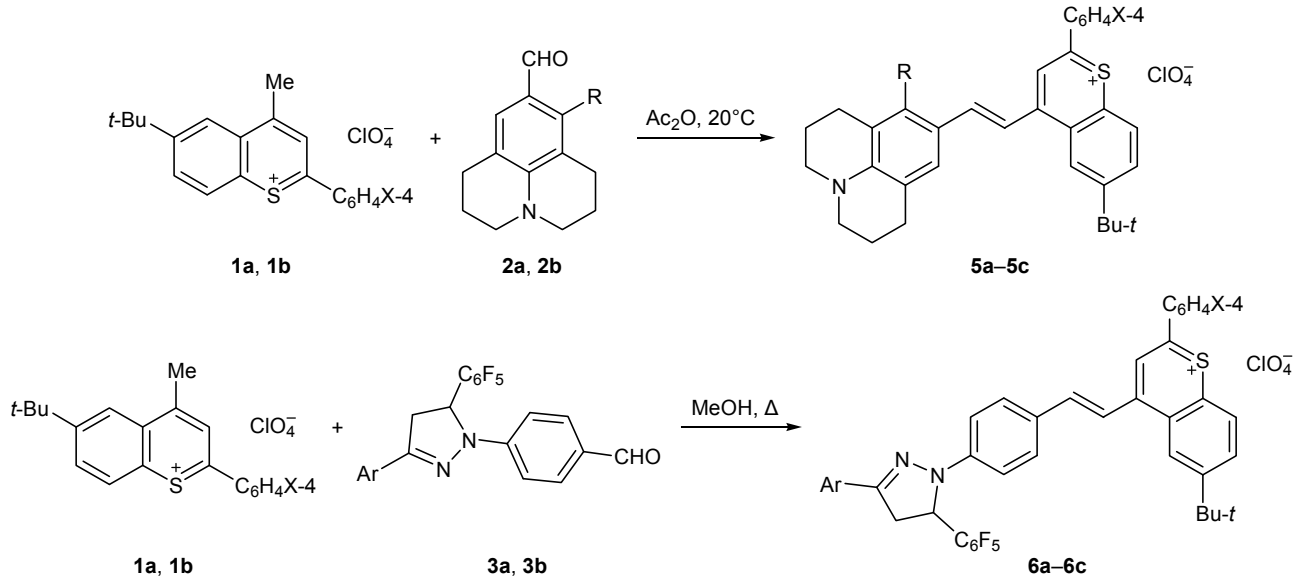
Ar = Ph (**a**), C_6F_5 (**b**).

alization of chromophores and self-assembly via stacking interactions with aromatic polymers and oligomers [11]. These considerations substantiated the use of formyl derivatives of polyfluorinated triaryldihydropyrazoles as heterocyclic analogs of *N*,*N*-dialkylaminobenzaldehydes.

Thus, the carbonyl components were 9-formyljulolidines **2a**–**2c** and 4-[3,5-bis(pentafluorophenyl)- or 5-(pentafluorophenyl-3-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl]benzaldehydes **3a** and **3b**. 2,3,6,7-Tetrahydro-1*H*,5*H*-pyrido[3,2,1-*ij*]quinoline-9-carbaldehyde **2a** was synthesized by the Vilsmeier–Haack formylation of commercial julolidine [12]. Aldehyde **2b** was prepared by alkylation of 8-hydroxy-9-formyljulolidine **2c** with 1-chlorodecane in the presence of sodium hydride by analogy with [13]. Aldehydes **3a** and **3b** were prepared from 1-phenyldihydropyrazoles **4a** and **4b** according to Vilsmeier [14, 15], but using a larger excess of phosphoryl chloride (Scheme 1).

Styryl dyes **5a**–**5c** were synthesized by condensation of perchlorates **1a** and **1b** with aldehydes **2a** and **2b** in acetic anhydride at room temperature, and dyes **6a**–**6c** were obtained by heating salts **1a** and **1b** with aldehydes **3a** and **3b** in boiling methanol (Scheme 2). The structures of **5a**–**5c** and **6a**–**6c** were confirmed by ^1H and ^{19}F NMR spectra and elemental analyses. Figures 1 and 2 show the electronic absorption spectra of **5a**–**5c** and **6a**–**6c**. The long-wave absorption maxima of **5a**–**5c** (Fig. 1) are located in the desired region (λ ~800 nm), and their position weakly depends on the number of *tert*-butyl groups in the thioflavylium fragment and on the presence of alkoxy group in the julolidine moiety. Dyes **6a**–**6c** (Fig. 2) absorb at shorter wavelengths (λ_{max} 690–740 nm), and compound **6a** with one perfluorophenyl group in the pyrazole fragment is characterized by the longest-wavelength maximum. This is consistent with the electron-withdrawing effect of the dihydropyrazole residue containing one or two perfluorophenyl groups.

Scheme 2.



2, R = H (**a**), $OC_{10}H_{21}$ (**b**); **3**, Ar = Ph (**a**), C_6F_5 (**b**); **5**, R = H, X = *t*-Bu (**a**); R = $OC_{10}H_{21}$, X = H (**b**), *t*-Bu (**c**); **6**, Ar = Ph, X = H (**a**); Ar = C_6F_5, X = H (**b**), *t*-Bu (**c**).

EXPERIMENTAL

The analytical and spectral studies were performed at the Joint Chemical Service Center, Siberian Branch, Russian Academy of Sciences. The NMR spectra were recorded on Bruker AC-300 [300.13 (^{1}H), 282.37 MHz (^{19}F)] and AV-400 spectrometers [400.13 MHz (^{1}H)]. The proton chemical shifts were measured relative to the residual proton signals of the deuterated solvents ($CHCl_3$, δ 7.24 ppm; DMSO-d_5, δ 2.50 ppm; CHD_2CN, δ 1.96 ppm). The ^{19}F chemical shifts were measured relative to hexafluorobenzene used as internal standard. The electronic absorption spectra were recorded on a Hewlett Packard 8453 spectrophotometer. The products were purified by chromatography on aluminum oxide (Brockmann activity grade II) or silica gel (50–100 μm).

6-*tert*-Butyl-4-methylthioflavylium perchlorates **1a** and **1b** were synthesized according to the procedure described in [5].

8-(Decyloxy)-2,3,6,7-tetrahydro-1*H*,5*H*-pyrido-[3,2,1-*ij*]quinoline-9-carbaldehyde (2b). A solution of 0.18 g (7.5 mmol) of sodium hydride (0.3 g of a 60% suspension in mineral oil) in 2 mL of DMF was added dropwise under argon to a solution of 0.7 g (3.2 mmol) of compound **2c** in 5 mL of anhydrous DMF, and 1.5 mL (1.3 g, 7.3 mmol) of 1-chlorodecane was then added in one portion. The mixture was stirred for 5 h at 100°C, cooled to room temperature, poured onto ice, and extracted with methylene chloride. The extract was washed with water and dried over $MgSO_4$, the solvent was removed under reduced pressure, and the residue was subjected to column chromatography on Al_2O_3. A wide dark yellow fraction was collected using benzene as eluent. Removal of the solvent from the eluate gave 1.16 g of a brown oily material which (according to the ^{1}H NMR data) was a solution of aldehyde **2b** in an aliphatic hydrocarbon resulting from dimerization of 1-chlorodecane. We failed to isolate pure aldehyde **2b**, and it was identified only by ^{1}H NMR and used without purification. Yield 0.5 g (43%, ^{1}H NMR). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.85 t (3H, CH_3, J = 6.2 Hz), 1.15–1.49 m (12H, CH_2), 1.68–1.82 m (4H, CH_2), 1.84–1.94 m (4H, CH_2), 2.63–2.73 m (4H, CH_2), 3.20–3.26 m (4H, CH_2), 3.81 t (2H, OCH_2, J = 6.6 Hz), 7.30 s (1H, H_{arom}), 9.97 s (1H, CHO).

Formylation of polyfluorinated triaryldihydropyrazoles 4a and 4b (*general procedure*). A solution of 0.8 mL (8.8 mmol) of phosphoryl chloride in 2 mL of DMF was added dropwise under stirring in an argon atmosphere to a suspension of 1.2 mmol of compound **4a** or **4b** [9] in 5 mL of anhydrous DMF. The mixture was stirred for 4 h at 100–105°C, cooled, poured into water, and neutralized to pH 7 with a saturated solution of sodium carbonate. The precipitate was filtered off, washed with water, and dried.

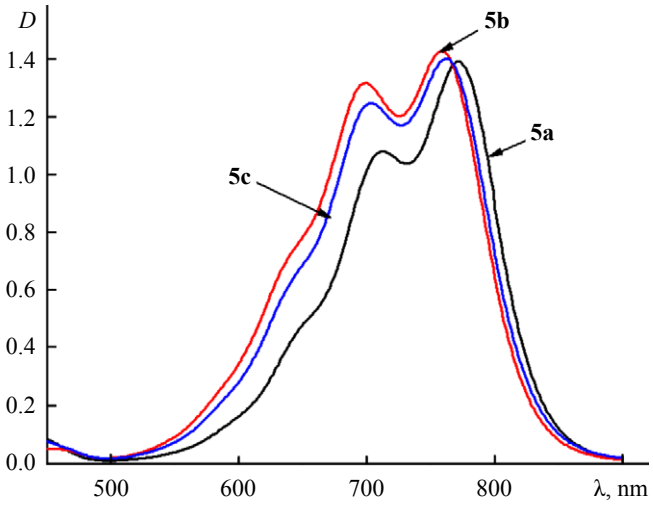


Fig. 1. Electronic absorption spectra of perchlorates **5a**–**5c** (chloroform, $c = 1 \times 10^{-4}$ M, d = 0.2 cm).

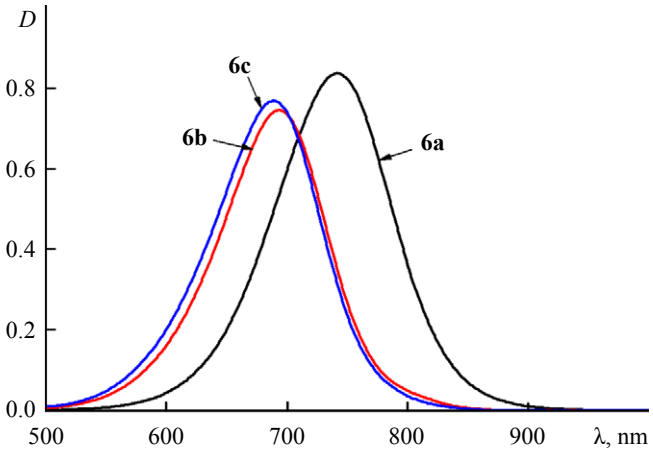


Fig. 2. Electronic absorption spectra of perchlorates **6a**–**6c** (chloroform, $c = 1 \times 10^{-5}$ M, d = 1 cm).

4-[5-(Pentafluorophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl]benzaldehyde (3a). Yield 90%, mp 215–218°C (purified by alumina column chromatography with benzene as eluent). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.36 d.d, 3.94 d.d, and 5.81 d.d (1H each, $CHCH_2$, *ABX*, J = 17.8, 13.1, 5.6 Hz); 7.10 d (2H, H_{arom}, J = 8.6 Hz), 7.39–7.47 m (3H, H_{arom}), 7.70–7.78 m (4H, H_{arom}), 9.76 s (1H, CHO). ^{19}F NMR spectrum, δ_F, ppm: 1.74 (2F), 9.27 (1F), 19.42 (2F). Found, %: C 63.44; H 2.99; F 22.85; N 6.72. $C_{22}H_{13}F_5N_2O$. Calculated, %: C 63.46; H 3.15; F 22.81; N 6.73.

4-[3,5-Bis(pentafluorophenyl)-4,5-dihydro-1*H*-pyrazol-1-yl]benzaldehyde (3b). Yield 95%, mp 204–206°C [sublimed at 195°C (2 mm)]. ^{1}H NMR spectrum ($CHCl_3$), δ, ppm: 3.41 d.d, 4.02 d.d, and 5.86 d.d (1H each, $CHCH_2$, *ABX*, J = 18.2, 13.3, 5.5 Hz); 7.11 d and 7.74 d (2H, H_{arom}, *AB*, J = 8.8 Hz), 9.80 s (1H, CHO). ^{19}F NMR spectrum ($CHCl_3$), δ_F, ppm: 0.56 (2F), 2.15 (2F), 9.58 (1F), 10.06 (1F), 19.51 (2F), 23.32 (2F). Found, %: C 52.45; H 1.58; F 36.99; N 5.32. $C_{22}H_8F_{10}N_2O$. Calculated, %: C 52.19; H 1.59; F 37.52; N 5.53.

Dyes 5a–5c (*general procedure*). A mixture of equimolar amounts (0.5 mmol) of perchlorate **1a** or **1b** and aldehyde **2a** or **2b** in 2 mL of acetic anhydride was stirred for 3–4 h at 20°C. The mixture immediately turned bright green. The product was precipitated with diethyl ether, filtered off, washed with water, dried in air, and purified by column chromatography on Al_2O_3 using chloroform–acetonitrile (5 : 1) as eluent.

6-*tert*-Butyl-2-(4-*tert*-butylphenyl)-4-[(*E*)-2-(2,3,6,7-tetrahydro-1*H*,5*H*-pyrido[3,2,1-*ij*]quinolin-9-yl)prop-2-en-1-yl]-1λ^4-benzothiopyran-1-ylium perchlorate (5a). Yield 61%, mp 217–220°C (decomp.). Electronic absorption spectrum ($CHCl_3$), λ_{max}, nm (logε): 713 (4.73), 771 (4.84). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.35 s (9H, *t*-Bu), 1.45 s (9H, *t*-Bu), 1.92–2.01 m (4H, CH_2), 2.77–2.84 m (4H, CH_2), 3.59–3.66 m (4H, CH_2), 7.64 d (2H, H_{arom}, J = 8.4 Hz), 7.86–7.96 m (6H, 5H, H_{arom}, CH=), 8.42 s (1H, H_{arom}), 8.47 s (1H, H_{arom}), 8.56 d (1H, CH=, J = 14.0 Hz). Found, %: C 70.53; H 6.61; Cl 5.80; N 2.27; S 5.06. $C_{37}H_{42}ClNO_4S$. Calculated, %: C 70.29; H 6.70; Cl 5.61; N 2.22; S 5.07.

6-*tert*-Butyl-4-{(*E*)-2-[8-(decyloxy)-2,3,6,7-tetrahydro-1*H*,5*H*-pyrido[3,2,1-*ij*]quinolin-9-yl]prop-2-en-1-yl}-2-phenyl-1λ^4-benzothiopyran-1-ylium perchlorate (5b). Yield 58%, mp 166–169°C (decomp.). Electronic absorption spectrum ($CHCl_3$), λ_{max}, nm (logε): 699 (4.82), 758 (4.85). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.85 t (3H, CH_3, J = 6.5 Hz), 1.15–1.29 m (14H, CH_2), 1.44 s (9H, *t*-Bu), 1.77–1.86 m (2H, CH_2), 1.97–2.08 m (4H, CH_2), 2.76–2.89 m (4H, CH_2), 3.61–3.67 m (4H, CH_2), 3.86 t (2H, OCH_2, J = 6.6 Hz), 7.51–7.57 m (4H, H_{arom}), 7.67–7.78 m (5H, H_{arom}, CH=), 8.07 br.s (1H, H_{arom}), 8.28 d (1H, H_{arom}, J = 1.5 Hz), 8.32 d (1H, CH=, J = 14.2 Hz). Found, %: C 70.81; H 7.55; Cl 4.60; N 1.92; S 4.16. $C_{43}H_{54}ClNO_5S$. Calculated, %: C 70.51; H 7.43; Cl 4.84; N 1.91; S 4.38.

6-*tert*-Butyl-2-(4-*tert*-butylphenyl)-4-{(*E*)-2-[8-(decyloxy)-2,3,6,7-tetrahydro-1*H*,5*H*-pyrido[3,2,1-*ij*]quinolin-9-yl]prop-2-en-1-yl}-1λ^4-benzothiopyran-1-ylium perchlorate (5c). Yield 56%, mp 101–104°C. Electronic absorption spectrum ($CHCl_3$), λ_{max}, nm (logε): 704 (4.79), 762 (4.84). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.84 t (3H, CH_3, J = 6.5 Hz), 1.20 br.s (14H, CH_2), 1.37 s (9H, *t*-Bu), 1.47 s (9H, *t*-Bu), 1.80–1.88 m (2H, CH_2), 1.99–2.12 m (4H, CH_2), 2.78–2.92 m (4H, CH_2), 3.64–3.70 m (4H, CH_2), 3.88 t (2H, OCH_2, J = 6.6 Hz), 7.53–7.58 m (4H, H_{arom}), 7.69–7.76 m (4H, H_{arom}, CH=), 8.12 s (1H, H_{arom}), 8.29 d (1H, H_{arom}, J = 1.5 Hz), 8.36 d (1H, CH=, J = 13.8 Hz). Found, %: C 71.59; H 7.99; Cl 4.88; N 1.89; S 3.75. $C_{47}H_{62}ClNO_5S$. Calculated, %: C 71.59; H 7.93; Cl 4.50; N 1.78; S 4.07.

Dyes 6a–6c (*general procedure*). A mixture of equimolar amounts (0.2 mmol) of perchlorate **1a** or **1b** and aldehyde **3a** or **3b** in 2 mL of methanol was heated for 30 min under reflux. The mixture was cooled, and the precipitate was filtered off and washed with diethyl ether. The product was additionally purified by silica gel column chromatography using methylene chloride–acetonitrile (10 : 1) as eluent.

6-*tert*-Butyl-4-[(*E*)-2-{4-[(5*S*)-5-(pentafluorophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl]-phenyl}prop-2-en-1-yl]-2-phenyl-1λ^4-benzothiopyran-1-ylium perchlorate (6a). Yield 61%, mp 277–279°C. Electronic absorption spectrum ($CHCl_3$): λ_{max} 742 nm (logε 4.92). ^{1}H NMR spectrum (CD_3CN), δ, ppm: 1.55 s (9H, *t*-Bu); 3.54 d.d, 4.06 d.d, and 5.94 d.d (1H each, $CHCH_2$, *ABX*, J = 18.2, 12.5, 5.1 Hz); 7.16 d (2H, H_{arom}, J = 8.8 Hz), 7.48–7.54 m (3H, H_{arom}), 7.74–8.25 m (12H, H_{arom}, CH=), 8.45 d (1H, CH=, J = 15.0 Hz), 8.72 s (1H, H_{arom}), 8.81 s (1H, H_{arom}). ^{19}F NMR spectrum (CD_3CN), δ, ppm: 1.46 (2F), 8.67 (1F), 20.45 (2F). Found, %: C 64.20; H 4.08; Cl 4.50; F 11.94; N 3.56; S 4.10. $C_{42}H_{32}ClF_5N_2O_4S$. Calculated, %: C 63.75; H 4.08; Cl 4.48; F 12.01; N 3.54; S 4.05.

4-[(*E*)-2-{4-[(5*S*)-3,5-Bis(pentafluorophenyl)-4,5-dihydro-1*H*-pyrazol-1-yl]phenyl}prop-2-en-1-yl]-6-*tert*-butyl-2-phenyl-1λ^4-benzothiopyran-1-ylium perchlorate (6b). Yield 76%, decomposition point 289–292°C. Electronic absorption spectrum ($CHCl_3$): λ_{max} 694 nm (logε 4.87). ^{1}H NMR spectrum (CD_3CN), δ, ppm: 1.54 s (9H, *t*-Bu); 3.56 d.d, 4.14 d.d, and 6.04 d.d (1H each, $CHCH_2$, *ABX*, J = 18.8, 13.0, 5.3 Hz); 7.21 d (2H, H_{arom}, J = 8.9 Hz), 7.69–7.83 m (4H, H_{arom}), 7.94 d (2H, H_{arom}, J = 8.9 Hz), 8.07–8.14 m (2H, H_{arom}), 8.23 and 8.47 (1H each, CH=CH, *AB*, J = 15.5 Hz), 8.37 d (1H, H_{arom}, J = 8.8 Hz), 8.81 d

(1H, H_{arom}, J = 1.6 Hz), 8.96 s (1H, H_{arom}). ^{19}F NMR spectrum (CD_3CN), δ_F, ppm: 0.64 (2F), 1.50 (2F), 8.89 (1F), 9.85 (1F), 20.50 (2F), 24.53 (2F). Found, %: C 57.35; H 3.10; Cl 4.10; F 21.94; N 3.24; S 3.66. $C_{42}H_{27}ClF_{10}N_2O_4S$. Calculated, %: C 57.25; H 3.09; Cl 4.02; F 21.56; N 3.18; S 3.64.

4-[(*E*)-2-{4-[(5*S*)-3,5-Bis(pentafluorophenyl)-4,5-dihydro-1*H*-pyrazol-1-yl]phenyl}prop-2-en-1-yl]-6-*tert*-butyl-2-(4-*tert*-butylphenyl)-1λ^4-benzothiopyran-1-ylium perchlorate (6c). Yield 68%, decomposition point 255–257°C. Electronic absorption spectrum ($CHCl_3$): λ_{max} 689 nm (log ε 4.88). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.36 s (9H, *t*-Bu), 1.50 s (9H, *t*-Bu); 3.41 d.d, 4.08 d.d, and 5.91 d.d (1H each, $CHCH_2$, *ABX*, J = 18.5, 13.1, 4.8 Hz); 7.15 d (2H, H_{arom}, J = 8.3 Hz), 7.69 d (2H, H_{arom}, J = 8.3 Hz), 7.93–8.25 m (5H, H_{arom}, CH=), 8.59 s (1H, H_{arom}), 8.67 d (1H, CH=, J = 15.2 Hz), 9.00 s (1H, H_{arom}). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: 0.67 (2F), 2.52 (2F), 9.97 (1F), 10.29 (1F), 19.56 (2F), 23.84 (2F). Found, %: C 58.76; H 3.79; Cl 3.78; F 20.19; N 3.01; S 3.75. $C_{46}H_{35}ClF_{10}N_2O_4S$. Calculated, %: C 58.94; H 3.76; Cl 3.78; F 20.27; N 2.99; S 3.42.

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