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Unusual iodination of arylsulfonylallenes

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List of contents

I. Experimental section. Experimental procedures. Characterization of compounds

The NMR spectra of solutions of compounds in $CDCl_3$ or acetone-d_6 were recorded at 400, and 100 MHz for ^1H and ^{13}C NMR spectra respectively at 25°C with a Bruker AVANCE III 400 spectrometer. The solvent residual signals $CDCl_3$ (δ 7.26 ppm) or acetone-d_6 (δ 2.05 ppm) for ^1H NMR spectra and the carbon signal of $CDCl_3$ (δ 77.0 ppm) or acetone-d_6 (δ 206.3 ppm) for ^{13}C NMR spectra were used as references. IR spectra of compounds were taken in KBr at a IR Affinity-1 machine. HRMS was carried out in ESI mode at a Bruker Micro-TOF mass spectrometer. The preparative reactions were monitored by thin-layer chromatography carried out on silica gel plates using UV light for detection. Preparative TLC was performed on silica gel 5-40 μm.

X-ray diffraction study. A suitable crystal was selected and studied on Agilent Technologies (Oxford Diffraction) «Supernova». The crystal was kept at 100(2) K during data collection. Using Olex2[S1] the structure was solved with the ShelXS[S2] structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization. CCDC 1580907– (**2a**), CCDC 1843278– (**3b**), CCDC 1843308– (**5**), contain the supplementary crystallographic data, which can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.htmL or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) + 44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk.

Allenes **1**were synthesized according to the literature procedure.[S3] Spectral characteristics ofthe following compounds correspond to the literature data:3-methyl-1-(4-chlorophenylsulfonyl)buta-1,2-diene (**1b**),[S4] 3-methyl-1-(4-methylphenylsulfonyl)buta-1,2-diene (**1c**),[S5] 2-(4-methylphenylsulfonyl)ethenylidenecyclohexane (**1d**),[S5] 1-(phenylsulfonyl)propa-1,2-diene (**1i**),[S6] 1-(4-methylphenylsulfonyl)propa-1,2-diene (**1j**),[S6] 1-(phenylsulfonyl)propan-2-one (**1a**).[S7]

3-Methyl-1-(phenylsulfonyl)buta-1,2-diene (1a). Yield 2.37 g (71%). Colorless oil.**^{1}H NMR** (400 MHz, $CDCl_3$) δ, ppm: 7.86 – 7.79 (m, 2H), 7.50 – 7.44 (m, 3H), 6.00-6.05 (m, 1H), 1.69 (d, *J* = 2.8 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ, ppm: 203.7, 141.3, 133.2, 129.1, 127.4, 107.0, 98.8, 19.3; **IR** (KBr), cm^{-1}: 1306 (S=O), 1969 (C=C=C); **HRMS(ESI)**: m/z calcd for $C_{11}H_{12}NaO_2S$ $[M+Na]^+$ 231.0450 , found 231.0447.

1-Bromo-3-methyl-1-(phenylsulfonyl)buta-1,2-diene (1e). Yield 3.15 g (80%). Colorless solid, mp 77-78 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ, ppm: 7.98 – 7.95 (m, 2H), 7.70 – 7.66 (m, 1H), 7.61 – 7.56 (m, 2H), 1.92 (s, 6H).; **^{13}C NMR** (100 MHz, $CDCl_3$) δ, ppm: 201.0, 138.3, 133.8, 129.0, 128.8, 114.7, 89.6, 19.9; **IR** (KBr) cm^{-1}: 1321 (S=O), 1956(C=C=C); **HRMS (ESI)**: m/z calcd for $C_{11}H_{11}BrNaO_2S$ $[M+Na]^+$ 308.9555 , found 308.9560.

3-Methyl-1-(4-methylphenylsulfonyl)-1-phenylbuta-1,2-diene (1f). Yield 2.54 g (70%). Greenish solid, mp 66-68°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.75-7.70 (m, 2H), 7.50-7.47 (m, 4H), 7.33-7.28 (m, 3H), 2.42 (s, 3H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, acetone-d6) δ 202.8, 144.0, 137.8, 129.4, 129.4, 128.6, 128.3, 127.9, 127.9, 106.7, 21.1, 19.3. **HRMS (ESI)**: m/z calcd for $C_{18}H_{18}NaO_2S$ $[M+Na]^+$ 321.0925 , found 321.0935.

3,4,4-Trimethyl-1-(4-methylphenylsulfonyl)-1-phenylpenta-1,2-diene (1g). Yield 1.56 g (81%). Yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.03 (d, *J* = 7.8 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.48 – 7.40 (m, 2H), 7.36 – 7.29 (m, 3H), 2.42 (s, 3H), 1.81 (s, 3H), 1.08 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ: 201.4, 144.0, 137.8, 137.8, 129.3, 129.3, 128.6, 128.6, 128.4, 128.3, 119.8, 35.1, 28.5, 21.5, 14.5. **HRMS (ESI)**: m/z calcd for $C_{21}H_{24}NaO_2S$ $[M+Na]^+$ 363.1395, found 363.1396.

2,7-Dimethyl-4,5-bis(4-methylphenylsulfonyl)octa-2,3,5,6-tetraene (**1h**). Yield 2.12 g (87%). Colorless solid, mp 55°C.**^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.62 – 7.59 (m, 2H), 7.28 – 7.25 (m, 2H), 2.45 (s, 3H), 1.77 (s, 6H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ: 204.7, 144.0, 137.5, 129.3, 127.8, 124.9, 109.5, 19.4, 9.3. **HRMS (ESI)**: m/z calcd for $C_{24}H_{26}Na_2O_4S_2$ $[M+2Na]^+$ 488.1068 , found 488.1063.

General procedure for synthesis of diiodoalkenes 2a-d from allenes 1a-c,g. A mixture of allene **1** (0.144 mmol) and 36.4 mg (0.144 mmol) of iodine in $CHCl_3$ (10 mL) was stirred in glass autoclave for 30 min at 80 or 130°C as indicated in Table 1. After cooling, the solvent was distilled off under reduced pressure (without heating) to give pure product.

1,2-Diiodo-3-methyl-1-(phenylsulfonyl)but-2-ene (2a). Yield 66.5 mg (98%). Black solid, melting with destruction after 60°C. Structure confirmed by single crystal X-ray analysis.**^{1}H NMR** (400 MHz, $CHCl_3$) δ: 7.96 – 7.91 (m, 2H), 7.71 – 7.65 (m, 1H), 7.57 – 7.52 (m, 2H), 6.24 (s, 1H), 1.84 (s, 3H), 1.39 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$)δ: 148.0, 134.5, 129.7, 129.0, 127.4, 95.0, 49.1, 33.4, 20.8; **IR** (KBr), cm^{-}

[1]: 568 (C-I), 598 (C-I), 1307 (S=O), 1581 (C=C**)**; **HRMS (ESI)**: m/z calcd for $C_{11}H_{12}I_2NaO_2S$ $[M+Na]^+$ 484.8539, found 484.8544.

1,2-Diiodo-3-methyl-1-(4-chlorophenylsulfonyl)but-2-ene (2b). Yield 74.4 mg (98%). Black solid, melting with decomposition after 45°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 1H), 7.54 (d, *J* = 8.6 Hz, 2H), 6.26 (s, 1H), 1.91 (3H), 1.52 (3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.2, 141.4, 137.7, 131.3, 129.3, 94.6, 48.6, 33.5, 20.9. **HRMS (ESI)**: m/z calcd for $C_{11}H_{11}ClI_2NaO_2S$ $[M+Na]^+$ 518.8155, found 518.8165.

1,2-Diiodo-3-methyl-1-(4-methylphenylsulfonyl)but-2-ene (2c). Yield 68.4 mg (98%). Black solid, melting with destruction after 45ºC. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.3 Hz, 2H), 6.21 (s, 1H), 2.45 (s, 3H), 1.86 (s, 3H), 1.42 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.6, 138.6, 129.8, 129.6, 127.5, 95.1, 49.0, 33.4, 21.7, 20.7. **HRMS (ESI)**: m/z calcd for $C_{12}H_{12}I_2NaO_2S$ $[M+Na]^+$ 496.8545, found 496.8551.

[1,2-Diiodo-2-(4-methylphenylsulfonyl)ethylidene]cyclohexane (2d). Yield 45.3 mg (61%). Colorless solid, melting with destruction after 70ºC. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.2 Hz, 1H), 7.52 (s, 1H), 7.36 (d, *J* = 8.4 Hz, 1H), 2.47 (s, 1H), 1.76 – 1.45 (m, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.5, 135.7, 129.7, 128.3, 128.3, 36.4, 24.9, 21.6, 21.6; **HRMS (ESI)**: m/z calcd for $C_{15}H_{18}I_2NaO_2S$ $[M+Na]^+$ 538.9015, found 538.9020.

General procedure for synthesis of iodoalkenes 3a-c from allenes 1d,e,f,h. A mixture of allene **1** (0.144 mmol) and 72.8 mg (0.288 mmol) of iodine in $CHCl_3$ (10 mL) was stirred in glass autoclave at 40°C for 30 min. After cooling, the solvent was distilled off under reduced pressure (without heating) to give pure product.

(1*Z*)-1-Bromo-2-iodo-3-methyl-1-(phenylsulfonyl)buta-1,3-diene (3a). Yield 57.1 mg (93%) Red solid, melting with decomposition after 70ºC. **^{1}H NMR** (400 MHz, acetone-d6) δ 7.82 – 7.72 (m, 2H), 7.77 – 7.55 (m, 3H), 5.66 (s, 1H), 5.60 – 5.49 (m, 1H), 2.08 (s, 3H); **^{13}C NMR** (101 MHz, acetone-d_6) δ 140.9, 138.5, 134.2, 130.8, 129.4, 128.8, 126.8, 120.3, 20.7; **HRMS (ESI)**: m/z calcd for $C_{11}H_{10}BrINaO_2S$ $[M+Na]^+$ 434.8527, found 432.8528.

(1*Z*)-2-Iodo-3-methyl-1-(4-methylphenylsulfonyl)-1-phenylbuta-1,3-diene. From mixture with *E*-**3b.** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.16 (m, 9H), 5.41 (1H), 5.30 (1H), 2.41 (3H), 2.16 (3H). ^{13}C NMR (101 MHz, $CDCl_3$) selected signals δ 171.59, 146.97, 143.99, 139.71, 117.26, 36.39, 21.55, 20.85.

(1*E*)-2-Iodo-3-methyl-1-(4-methylphenylsulfonyl)-1-phenylbuta-1,3-diene (3b). Yield 33.7 mg (55%). Red solid, melting with destruction after 60ºC. Structure confirmed by single crystal X-ray analysis.**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.2 Hz, 2H), 7.39 – 7.30 (m, 5H), 7.19 (d, *J* = 8.2 Hz, 1H), 5.22 (s, 1H), 5.04 (s, 1H), 2.41 (s, 3H), 1.71 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.1, 144.4, 136.9, 131.6, 130.9, 130.4, 129.2, 129.1, 129.0, 128.8, 128.2, 115.2, 23.4, 21.5; **HRMS (ESI)**: m/z calcd for $C_{18}H_{17}INaO_2S$ $[M+Na]^+$ 446.9892, found 446.9899.

(3*E*,5*E*)-3,6-Diiodo-2,7-dimethyl-4,5-bis(4-methylphenylsulfonyl)octa-1,3,5,7-tetraene (3c). Yield 88.9 mg (70%). Solidifying yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.3 Hz, 4H), 7.20 (d, *J* =

8.0 Hz, 4H), 5.22 (s, 2H), 5.05 – 5.04 (m, 2H), 2.41 (s, 6H), 2.16 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.4, 136.9, 130.9, 130.4, 129.2, 128.8, 128.1, 115.2, 23.4, 21.5. **HRMS (ESI)**: m/z calcd for $C_{24}H_{24}I_2NaO_4S_2$ $[M+Na]^+$ 716,9103, found 716,9113.

Procedure for synthesis of mixture of compounds 4 and 5 from allene 1f. A mixture of allene **1f** (47.0 mg, 0.144 mmol) and 44.8 mg (0.288 mmol) of bromine in $CHCl_3$ (10 mL) was stirred at 30ºC in high pressure glass tube for 30 min. After cooling, the solvent was distilled off under reduced pressure (without heating) to give inseparable mixture of compounds **4** and **5**. **(1*E*)-2-Bromo-3-tert-butyl-3-methylene-1-(4-methylphenylsulfonyl)-1-phenylbuta-1,3-diene** (**4**). Yield 40%. Yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 – 7.32 (m, 5H), 7.18 – 7.10 (m, 4H), 5.40 (s, 1H), 5.38 (s, 1H), 2.40 (s, 3H), 1.46 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ (selected signals) 136.6, 135.5, 129.0, 128.8, 128.6, 115.8, 31.0, 21.6 **HRMS(ESI)**: m/z calcd for $C_{21}H_{23}BrNaO_2S$ $[M+Na]^+$ 441.0500, found 441.0511. **(1*E*,3*Z*)-2,4-Dibromo3-tert-butyl -1-(4-methylphenylsulfonyl)-phenylbuta-1,3-diene** (**5**). Solidifying yellow oil. Structure confirmed by single crystal X-ray analysis. Yield 45%. Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.30 (m, 5H), 7.19 – 7.08 (m, 4H), 6.47 (s, 1H), 2.39 (s, 3H), 1.49 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ (101 MHz, $CDCl_3$) δ 156.2, 144.1, 135.1, 129.2, 129.2, 129.0, 128.8, 128.8, 128.8, 128.7, 128.7, 107.2, 35.5, 31.2, 21.4 **HRMS(ESI)**: m/z calcd for $C_{21}H_{22}Br_2NaO2S$ $[M+Na]^+$ 518.9605, found 518.9609.

II. Copies of NMR spectra

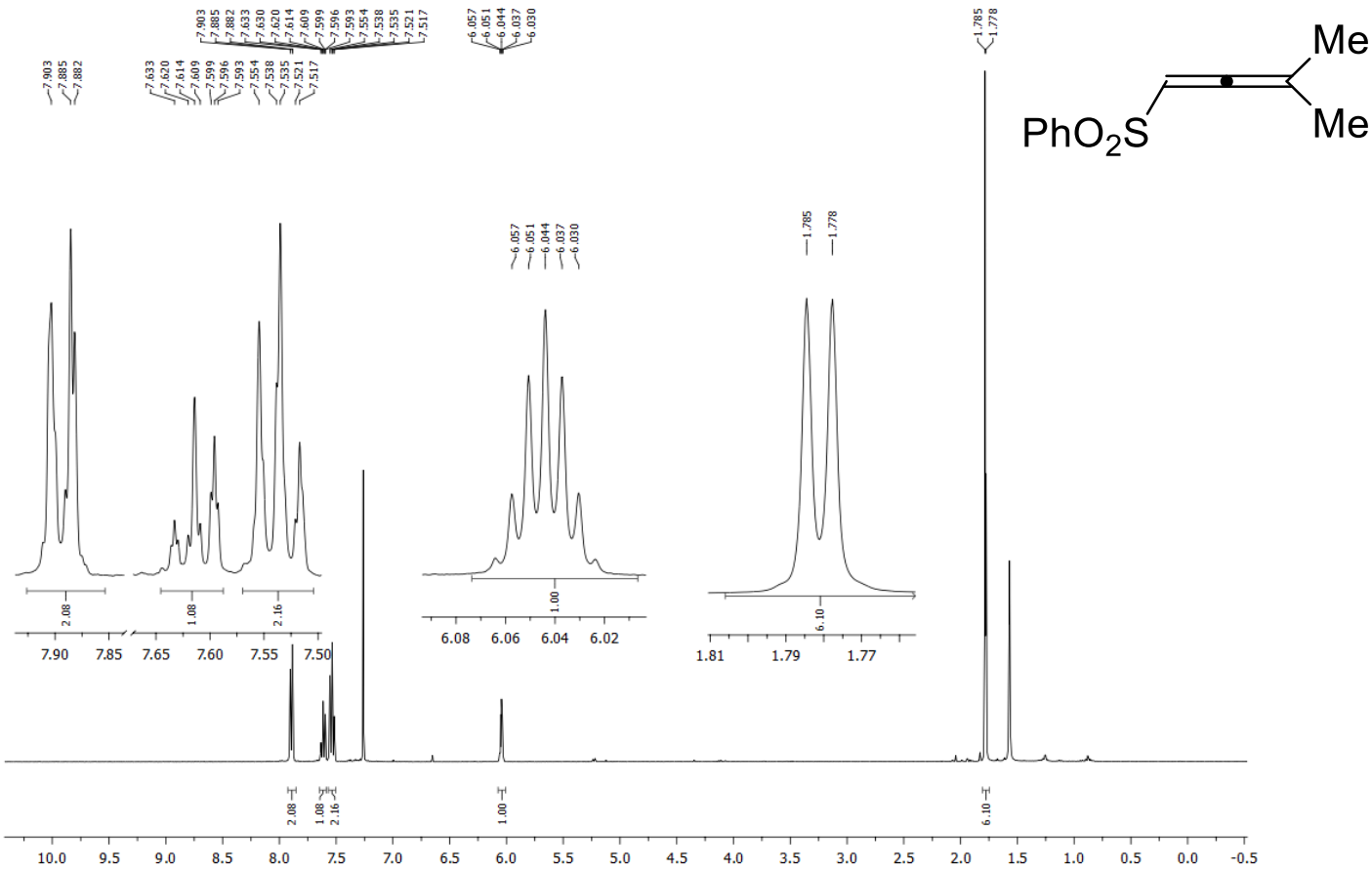


Fig. S1. ^{1}H NMR spectrum of compound **1a** (400 MHz, $CDCl_3$).

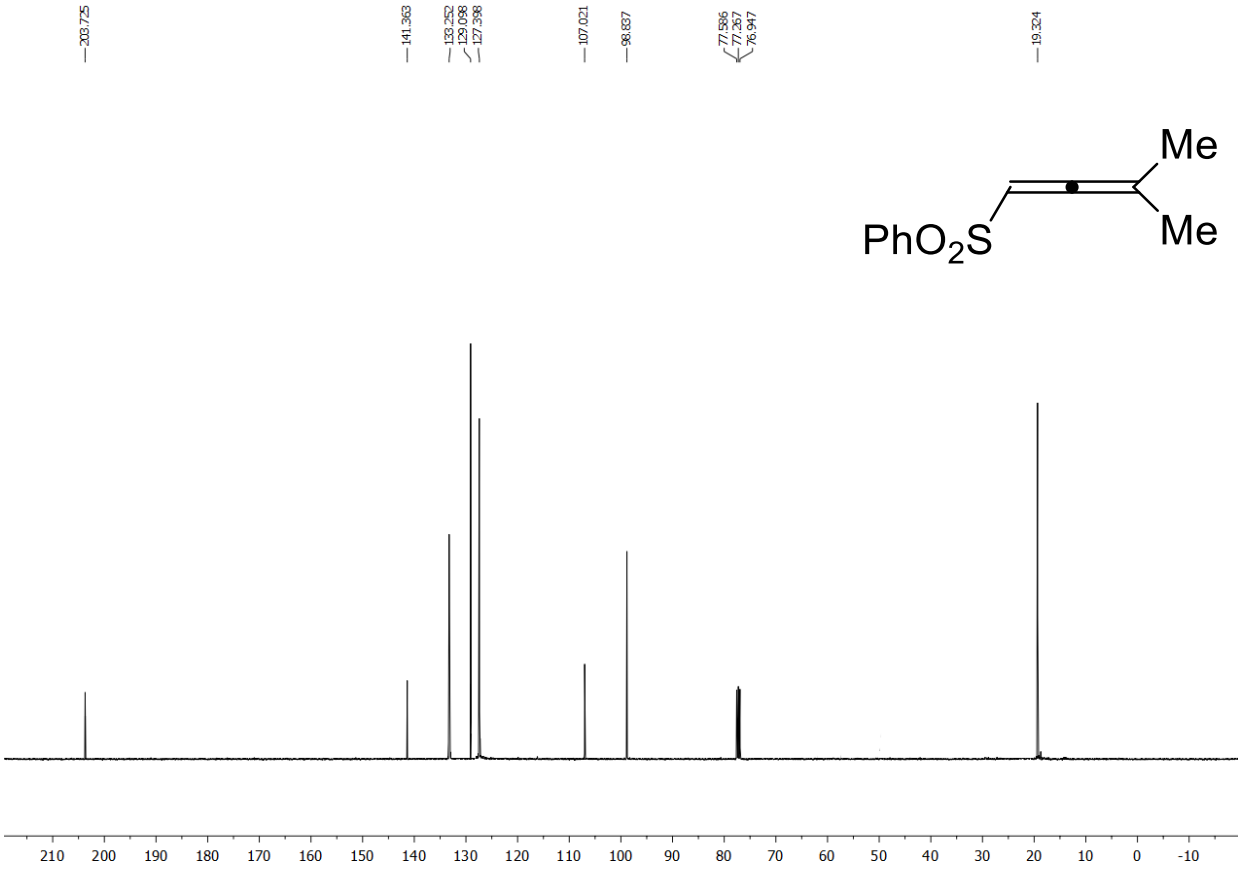


Fig. S2. ^{13}C NMR spectrum of compound **1a** (100 MHz, $CDCl_3$).

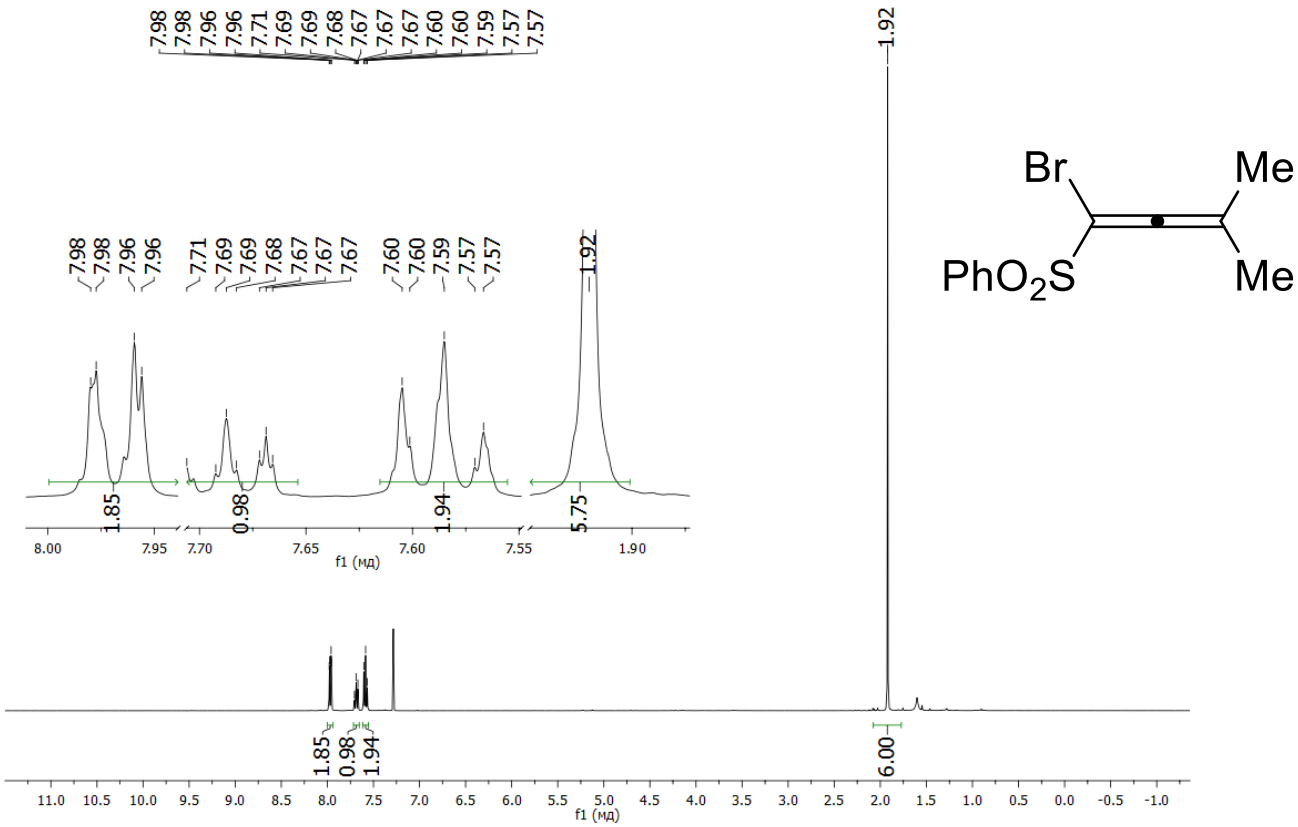


Fig. S3. ^{1}H NMR spectrum of compound **1e** (400 MHz, $CDCl_3$).

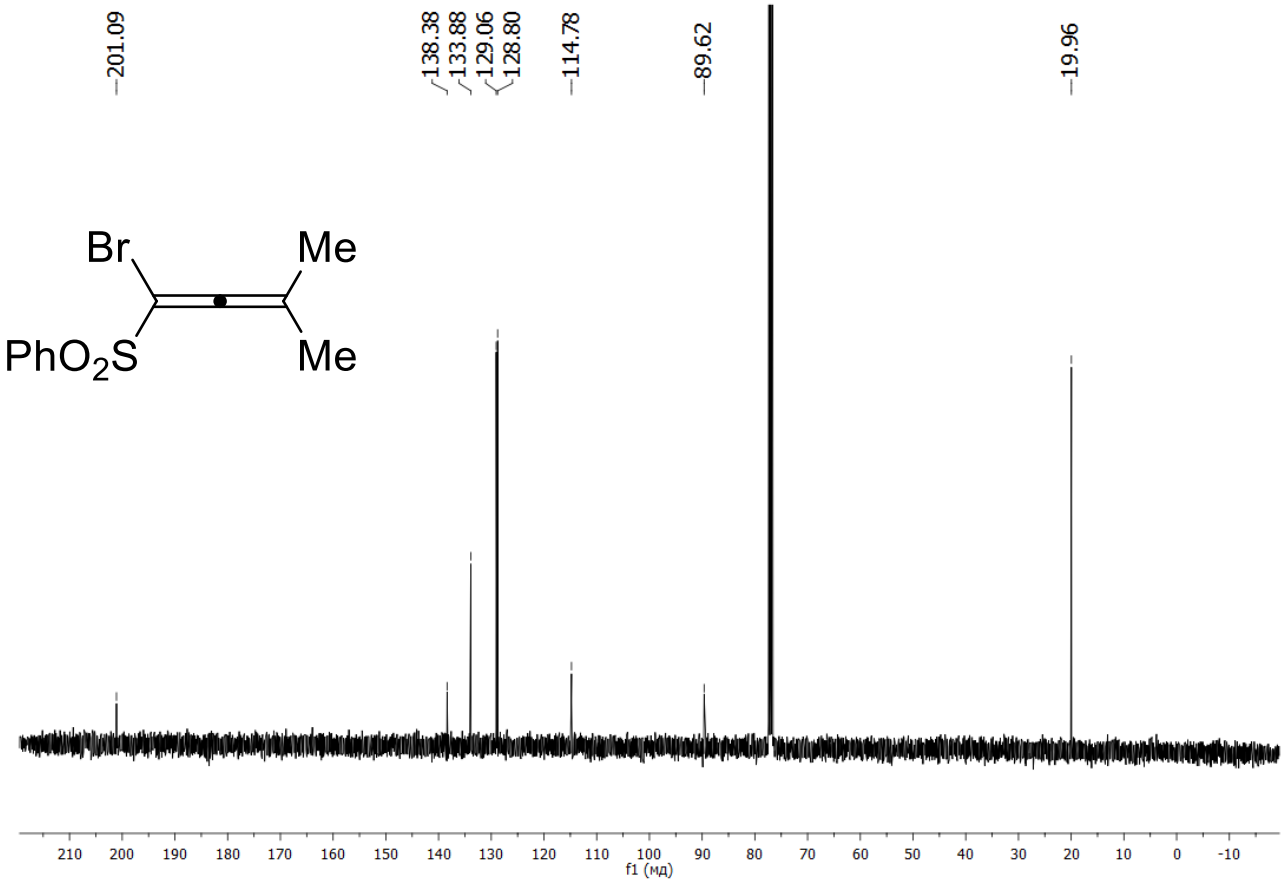


Fig. S4. ^{13}C NMR spectrum of compound **1e** (100 MHz, $CDCl_3$).

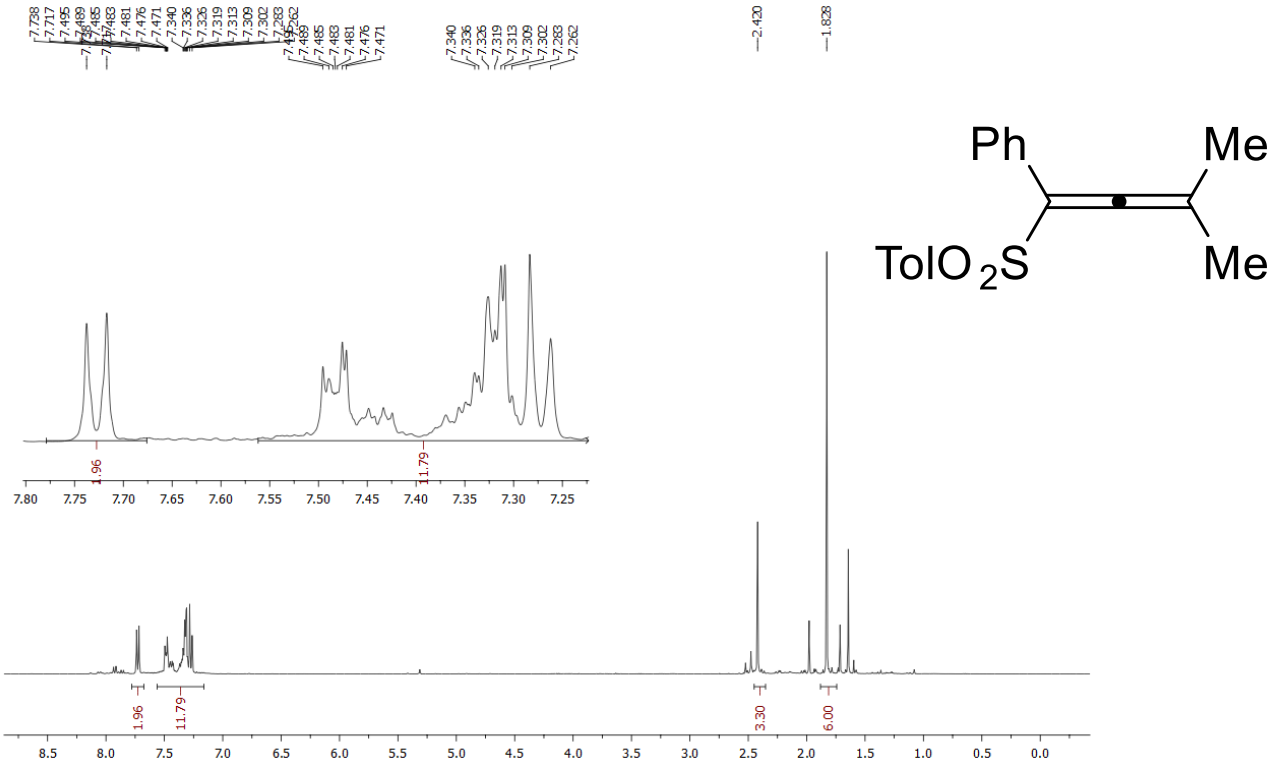


Fig. S5. ^{1}H NMR spectrum of compound **1f** (400 MHz, $CDCl_3$).

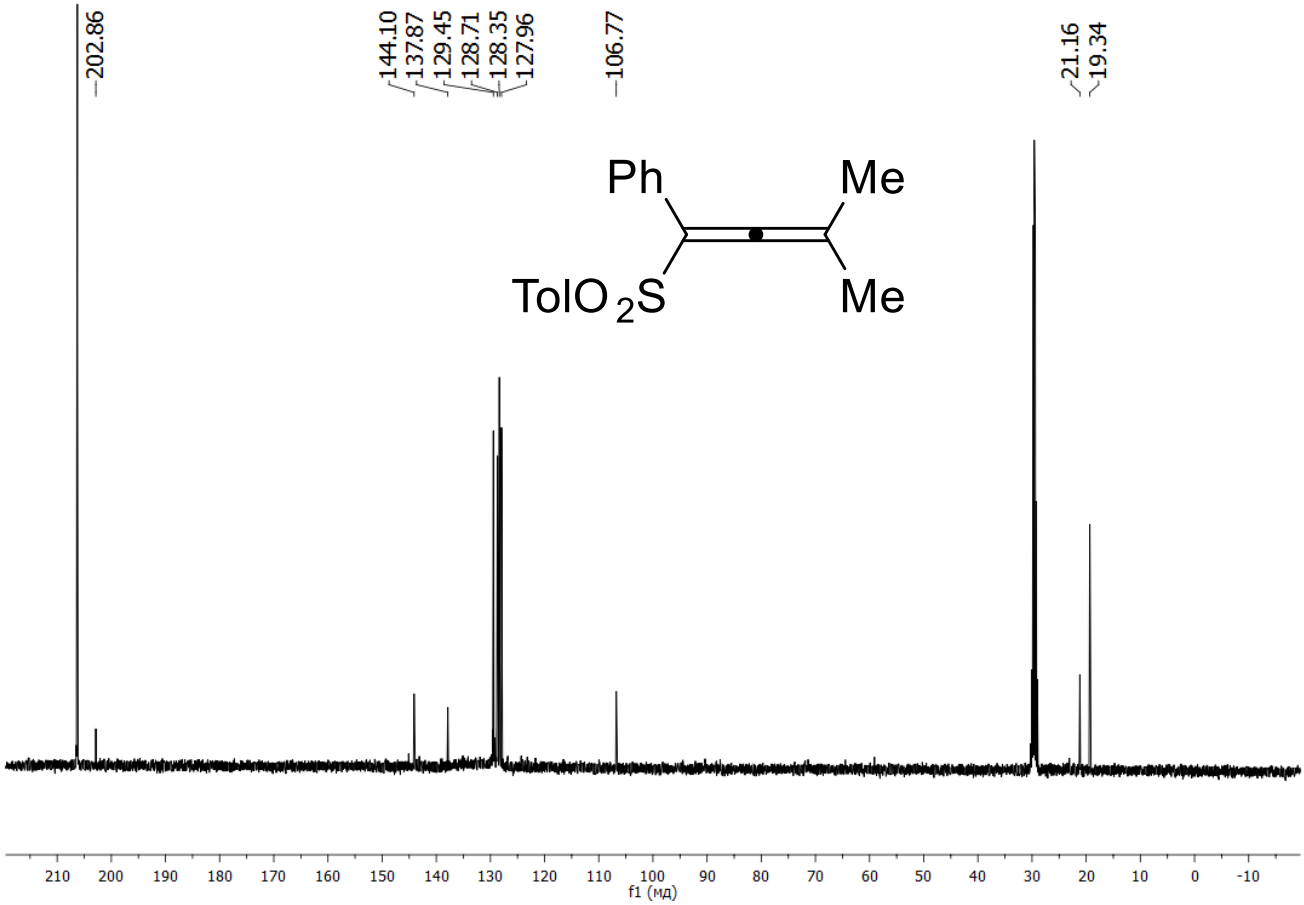


Fig. S6. ^{13}C NMR spectrum of compound **1f** (100 MHz, acetone-d_6).

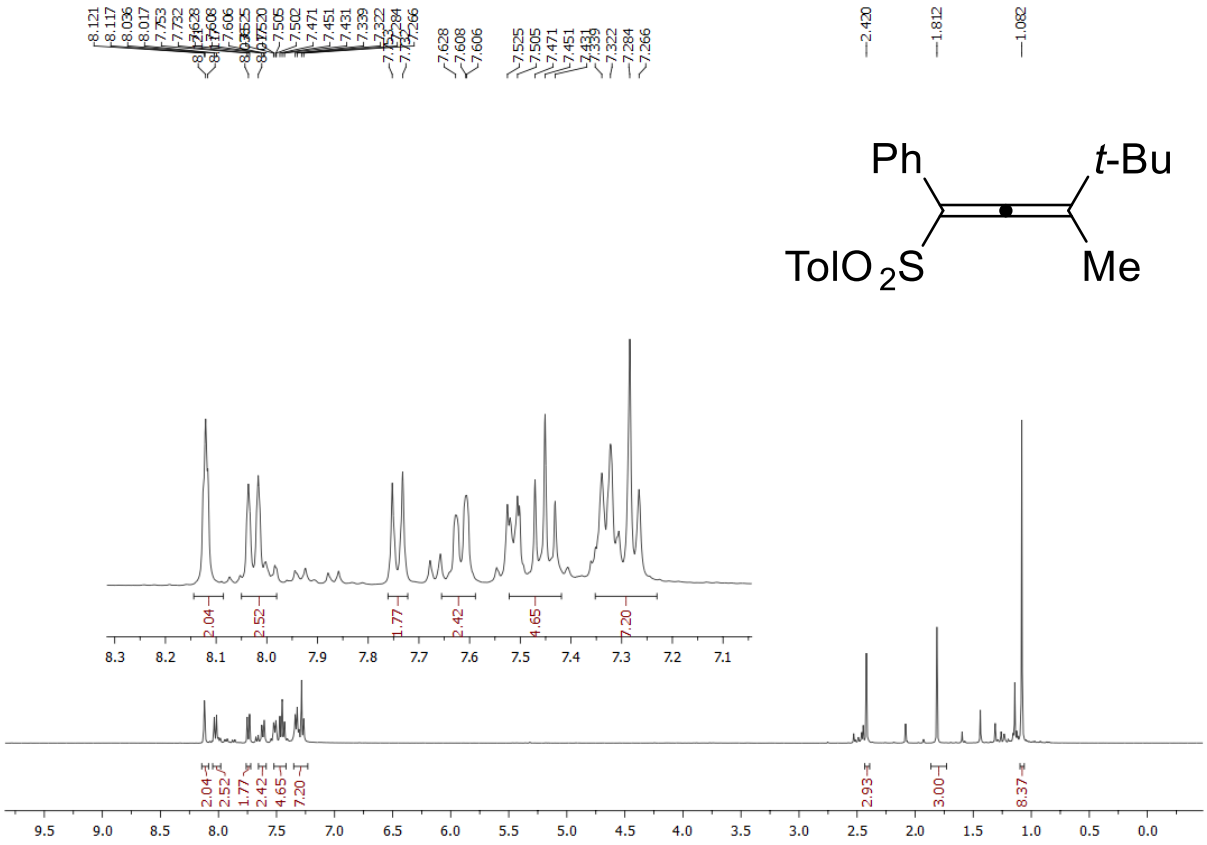


Fig. S7 .[1]H NMR spectrum of compound **1h** (400 MHz, $CDCl_3$).

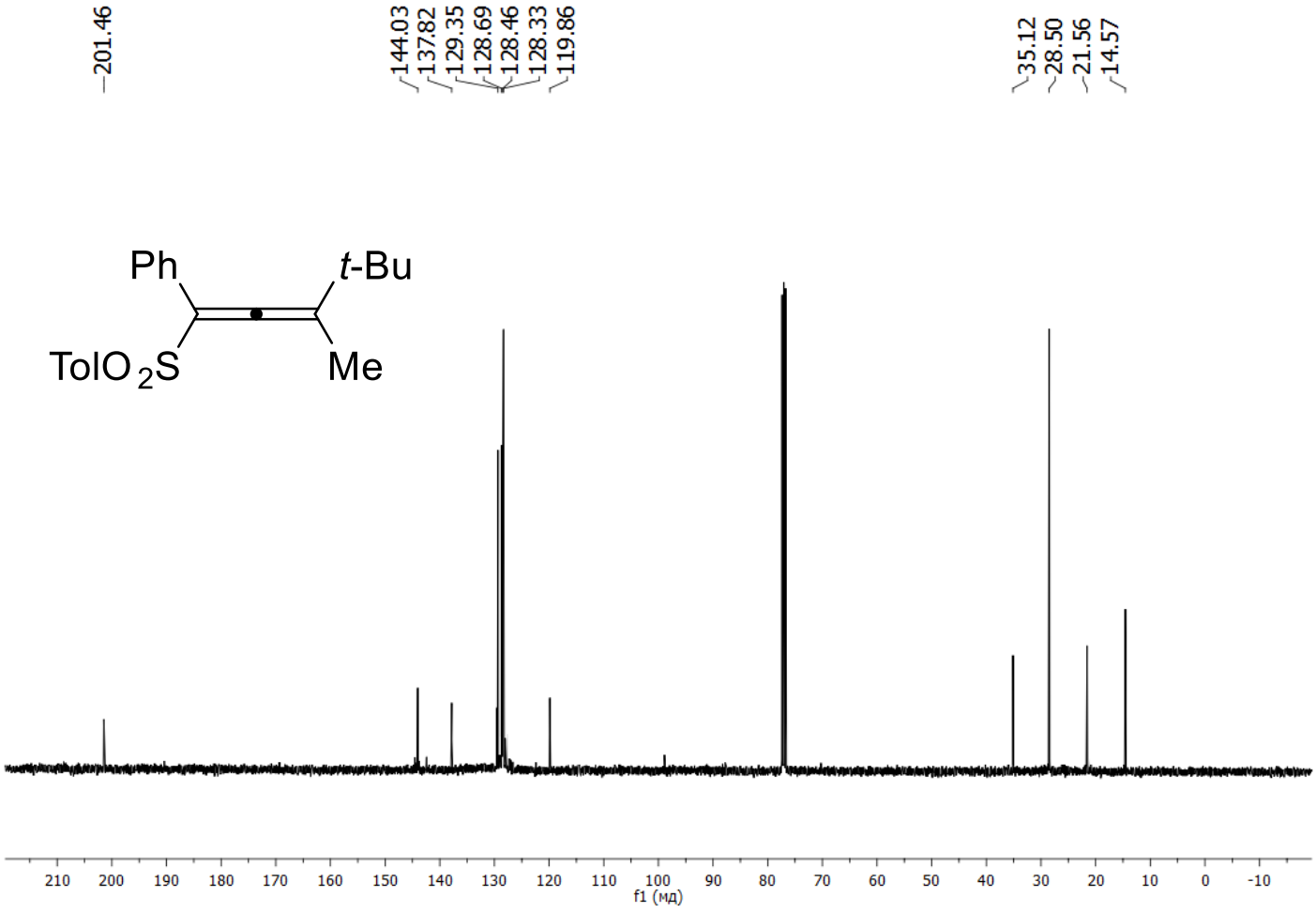


Fig. S8. [13]C NMR spectrum of compound **1h** (100 MHz, acetone-d6).

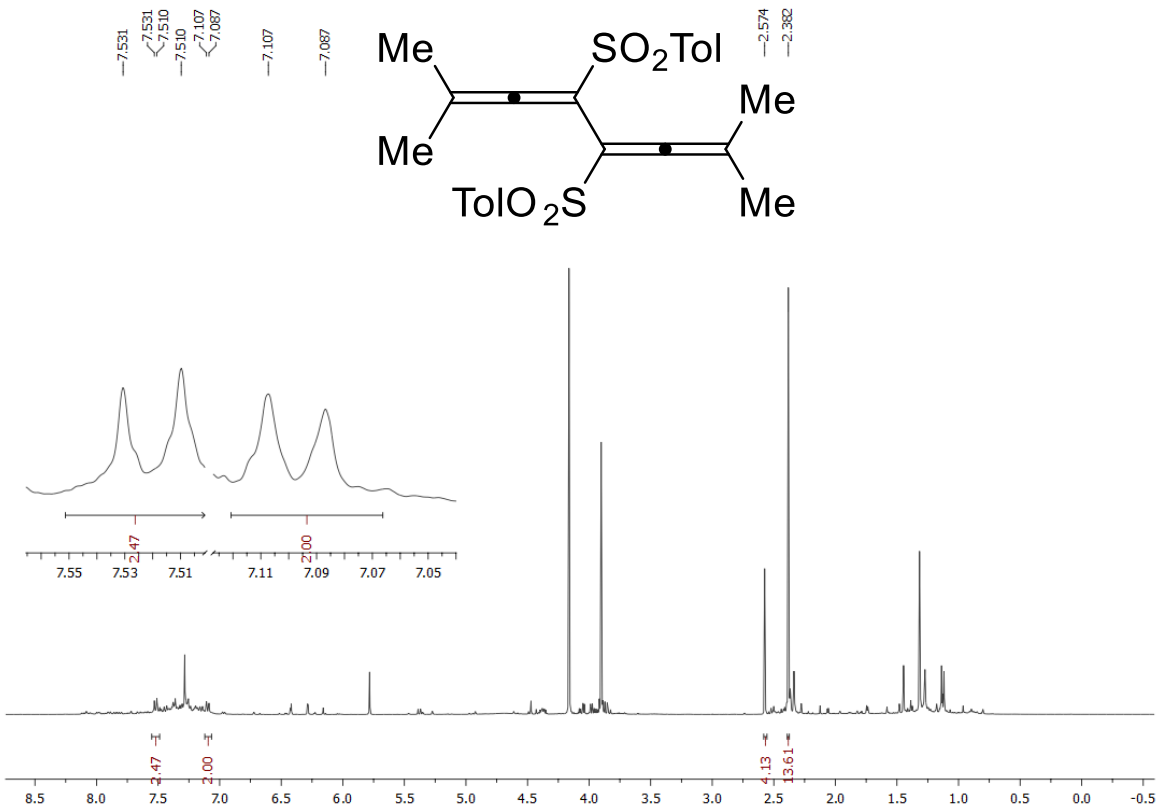


Fig. S9. ^{1}H NMR spectrum of compound **1g** (400 MHz, $CDCl_3$).

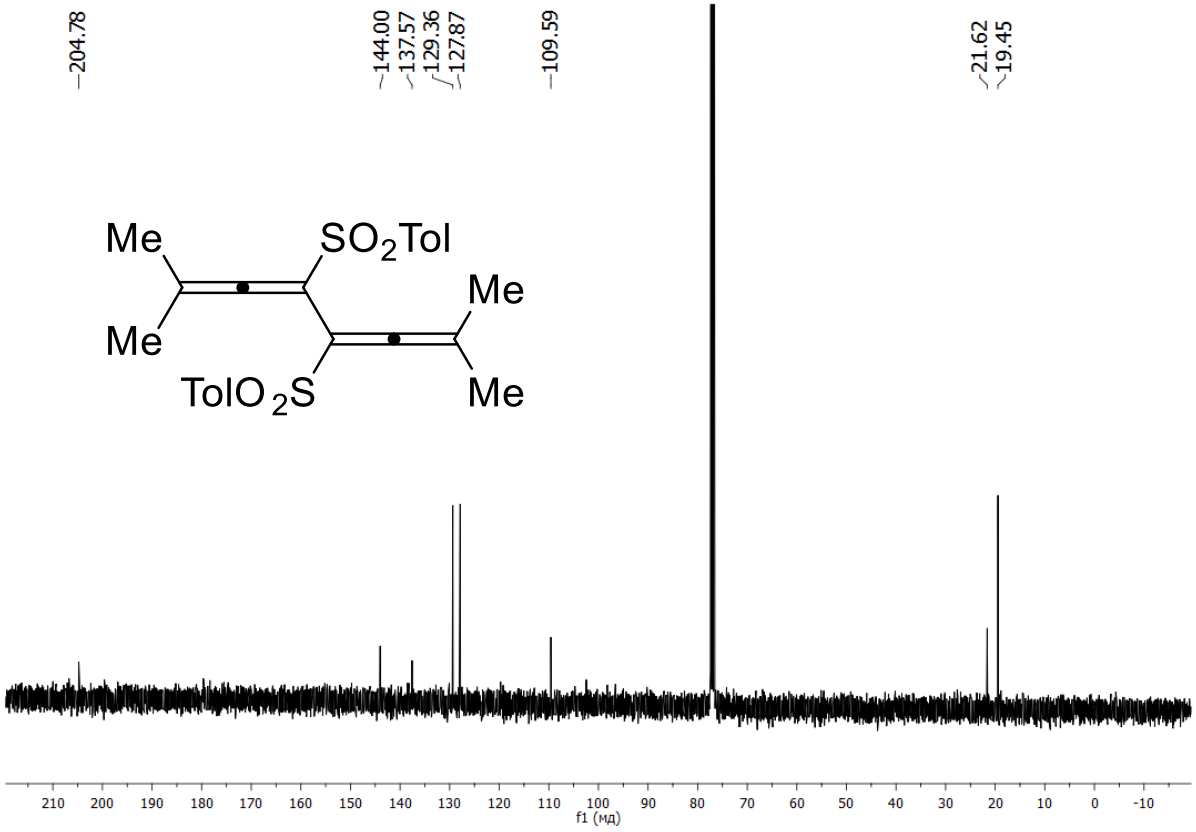


Fig. S10.^{13}C NMR spectrum of compound **1g** (100 MHz, $CDCl_3$).

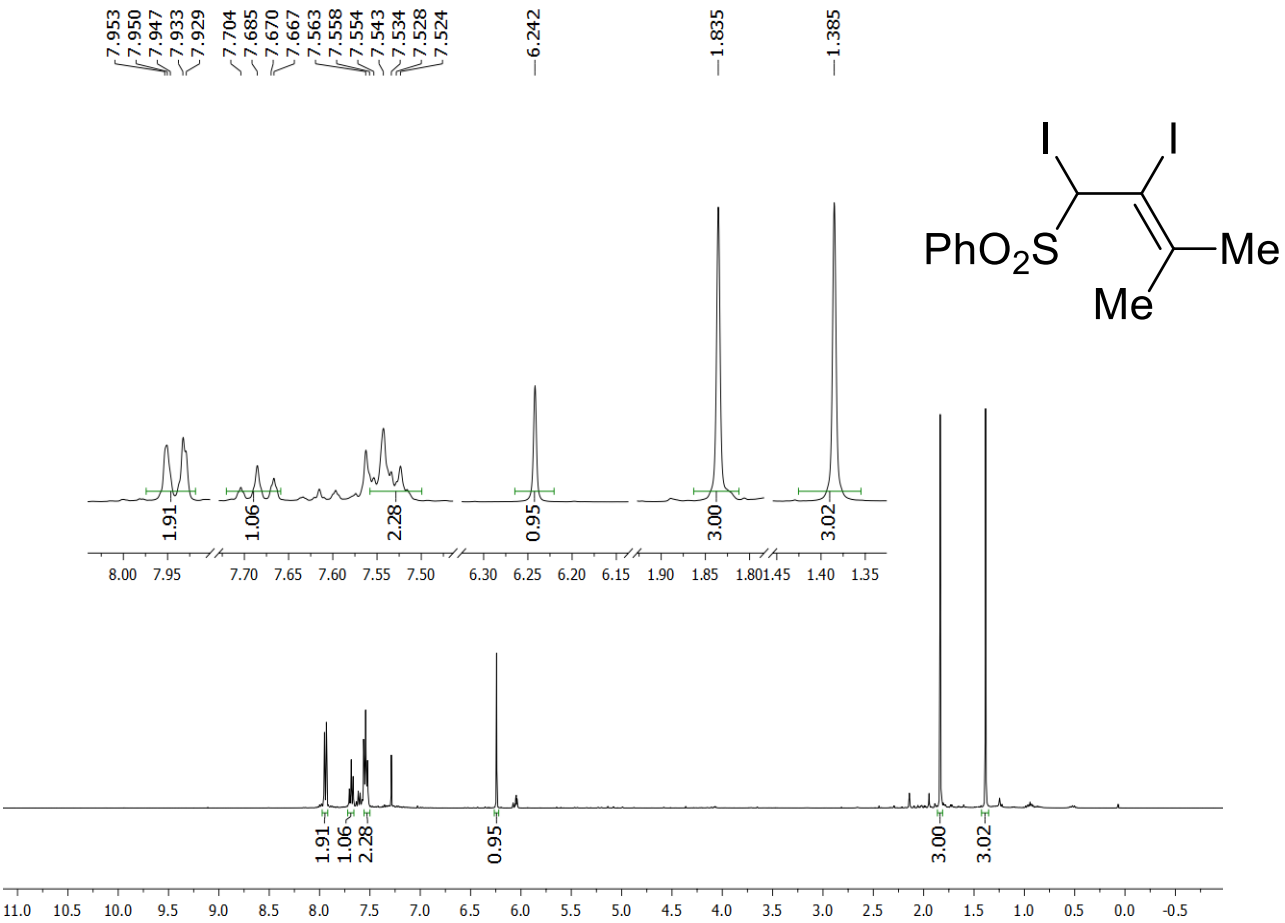


Fig. S11. ^{1}H NMR spectrum of compound **2a** (400 MHz, $CDCl_3$).

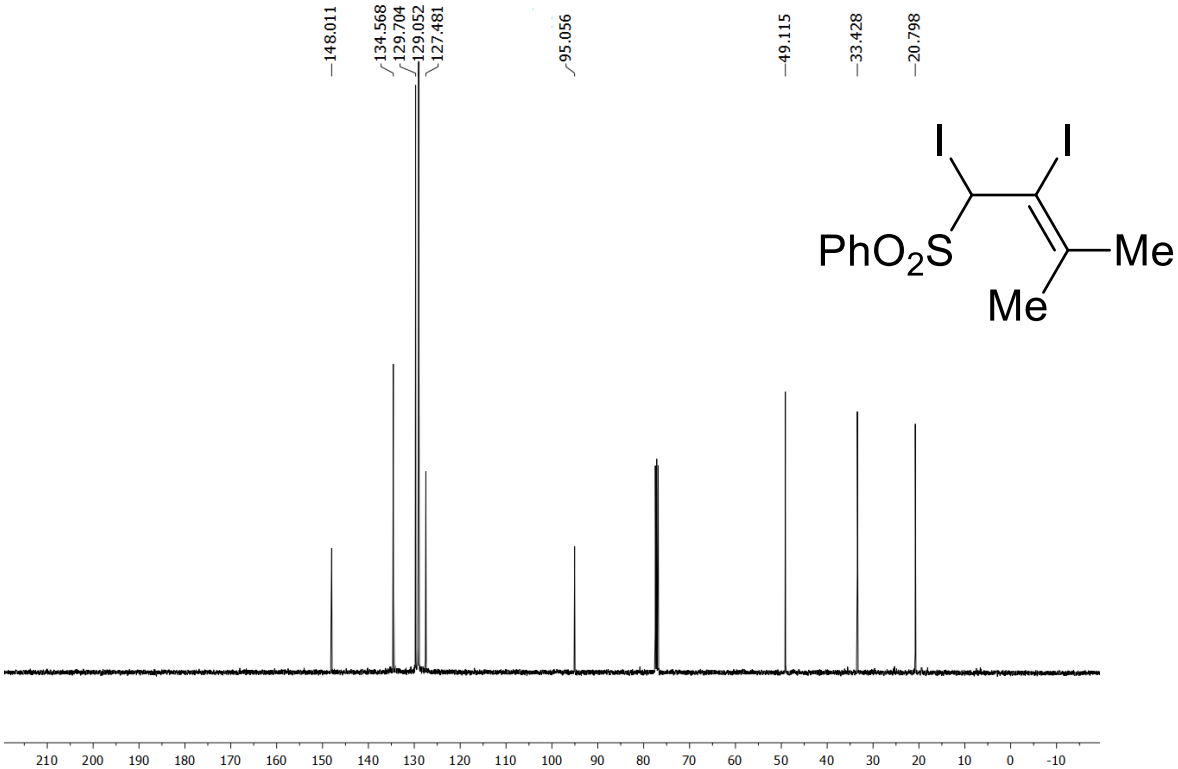


Fig. S12.^{13}C NMR spectrum of compound **2a** (100 MHz, $CDCl_3$).

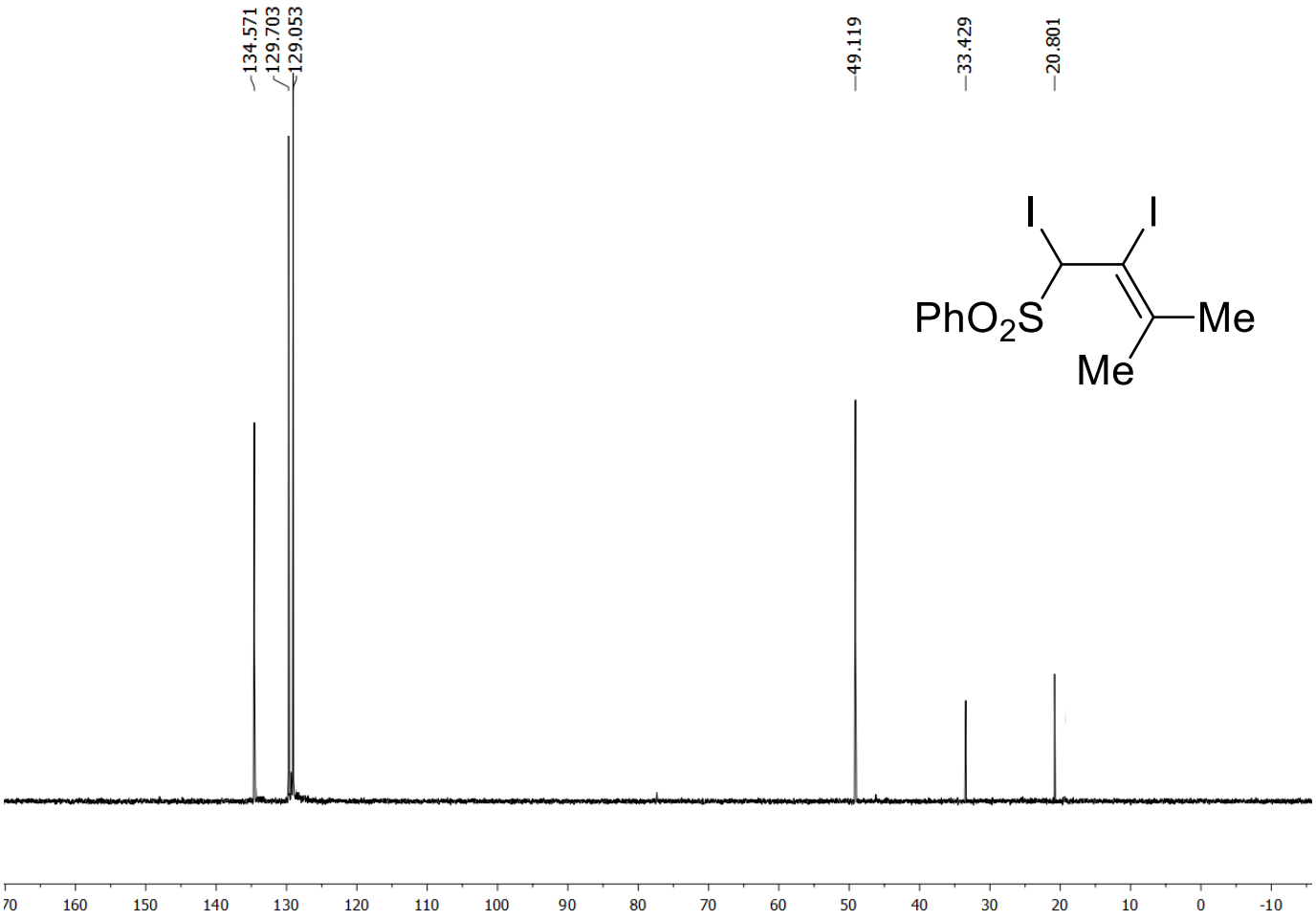


Fig. S13. DEPT NMR spectrum of compound **2a** (100 MHz, $CDCl_3$).

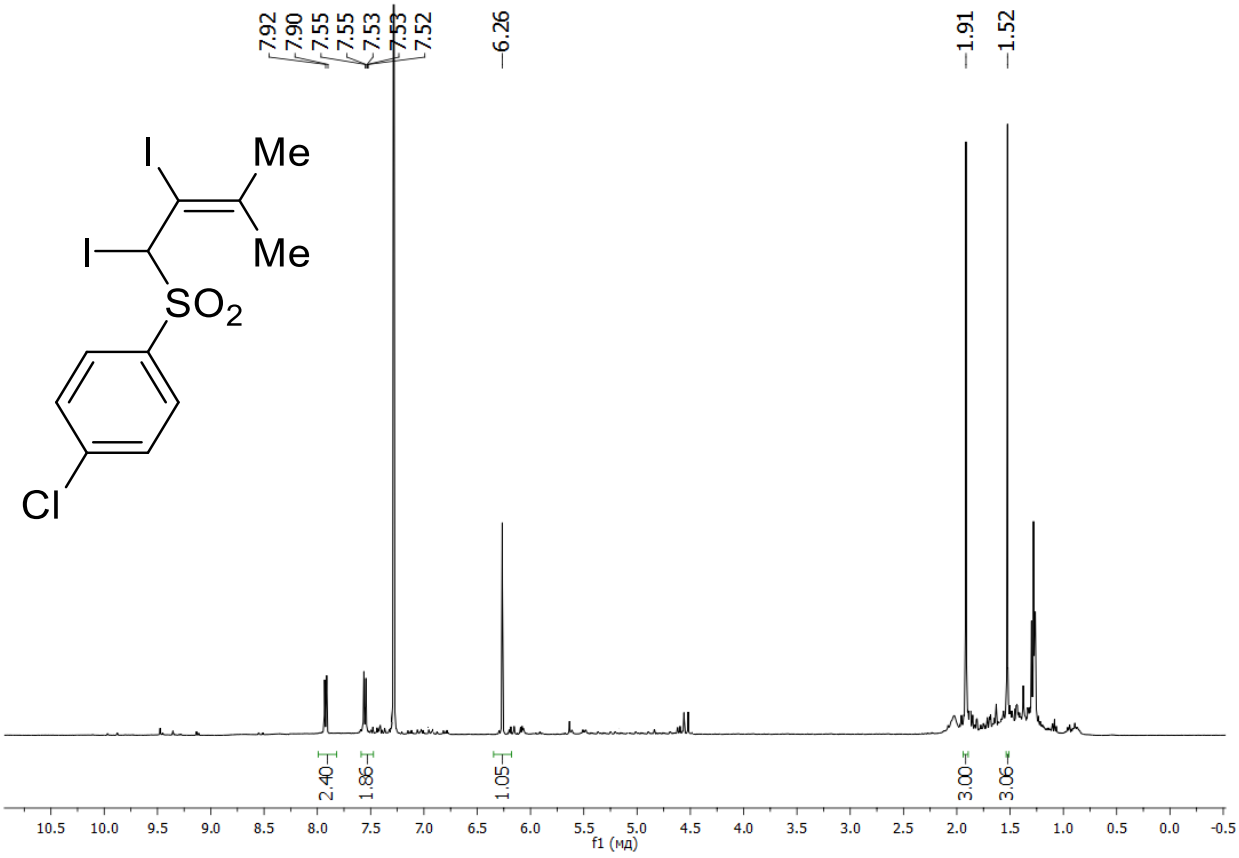


Fig. S14. ^1H NMR spectrum of compound **2b** (400 MHz, $CDCl_3$).

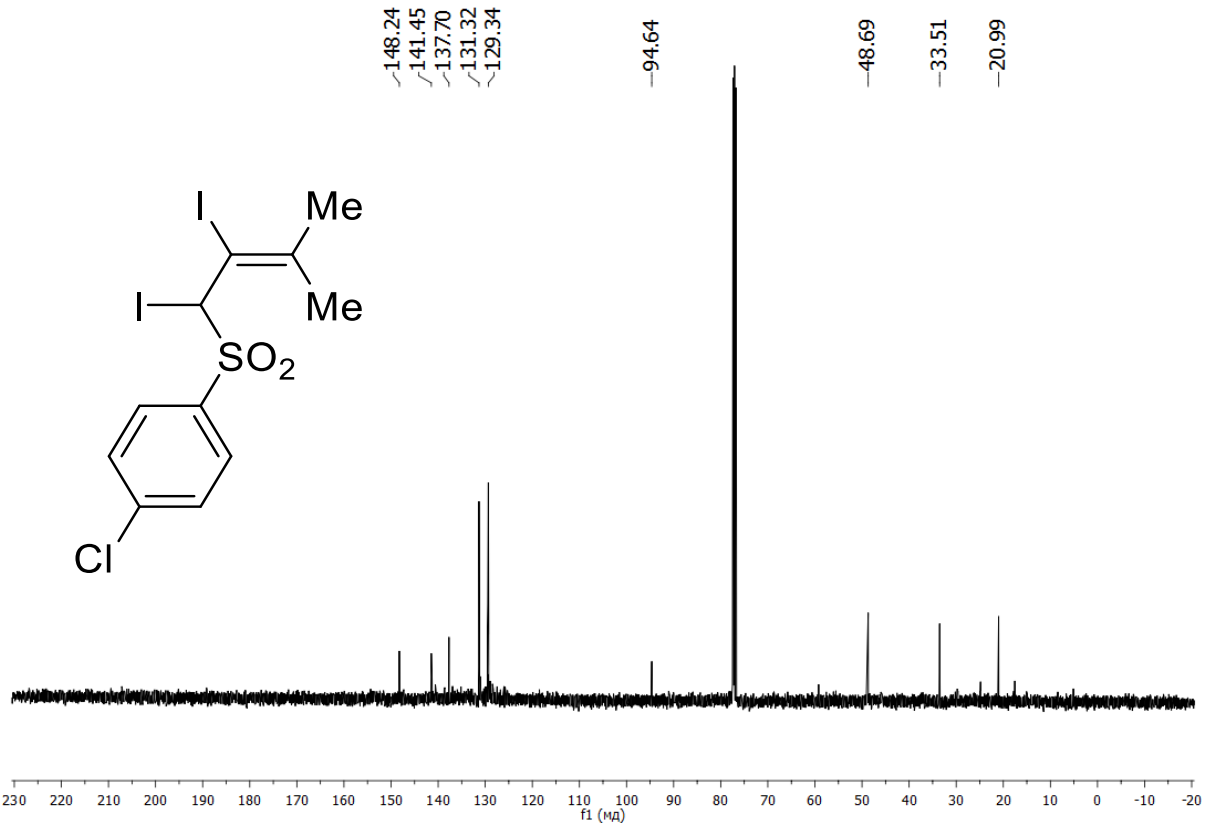


Fig. S15. ^{13}C NMR spectrum of compound **2b** (101 MHz, $CDCl_3$).

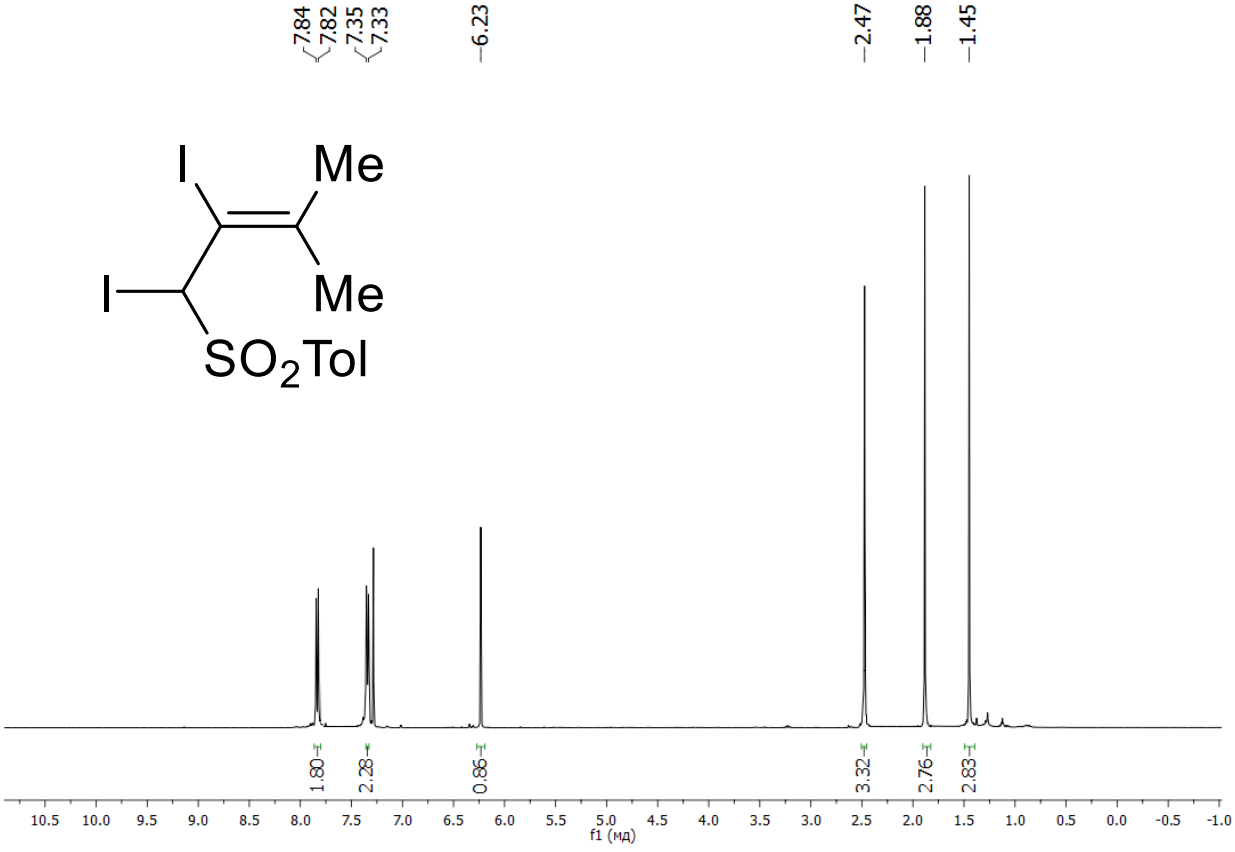


Fig. S16. ^{1}H NMR spectrum of compound **2c** (400 MHz, $CDCl_3$).

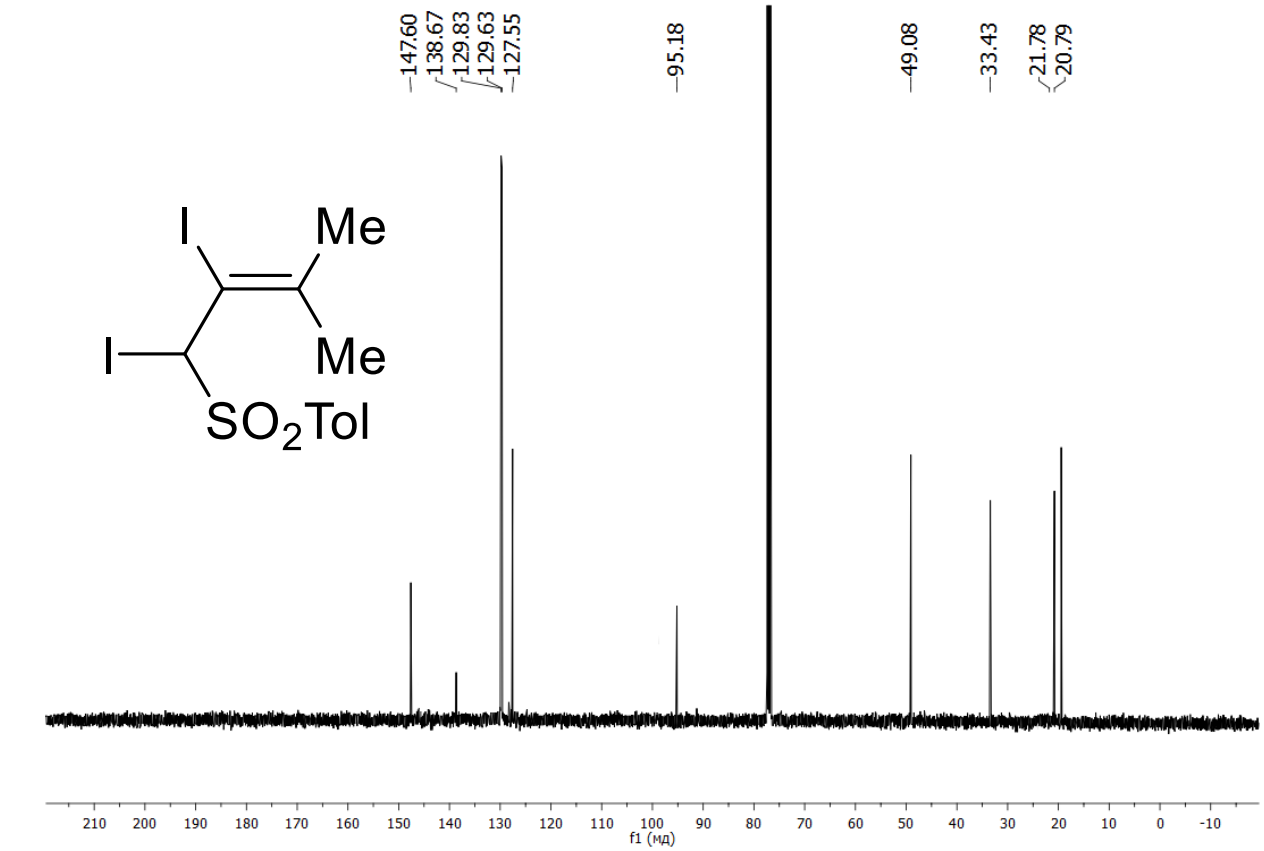


Fig. S17. ^{13}C NMR spectrum of compound **2c** (100 MHz, $CDCl_3$).

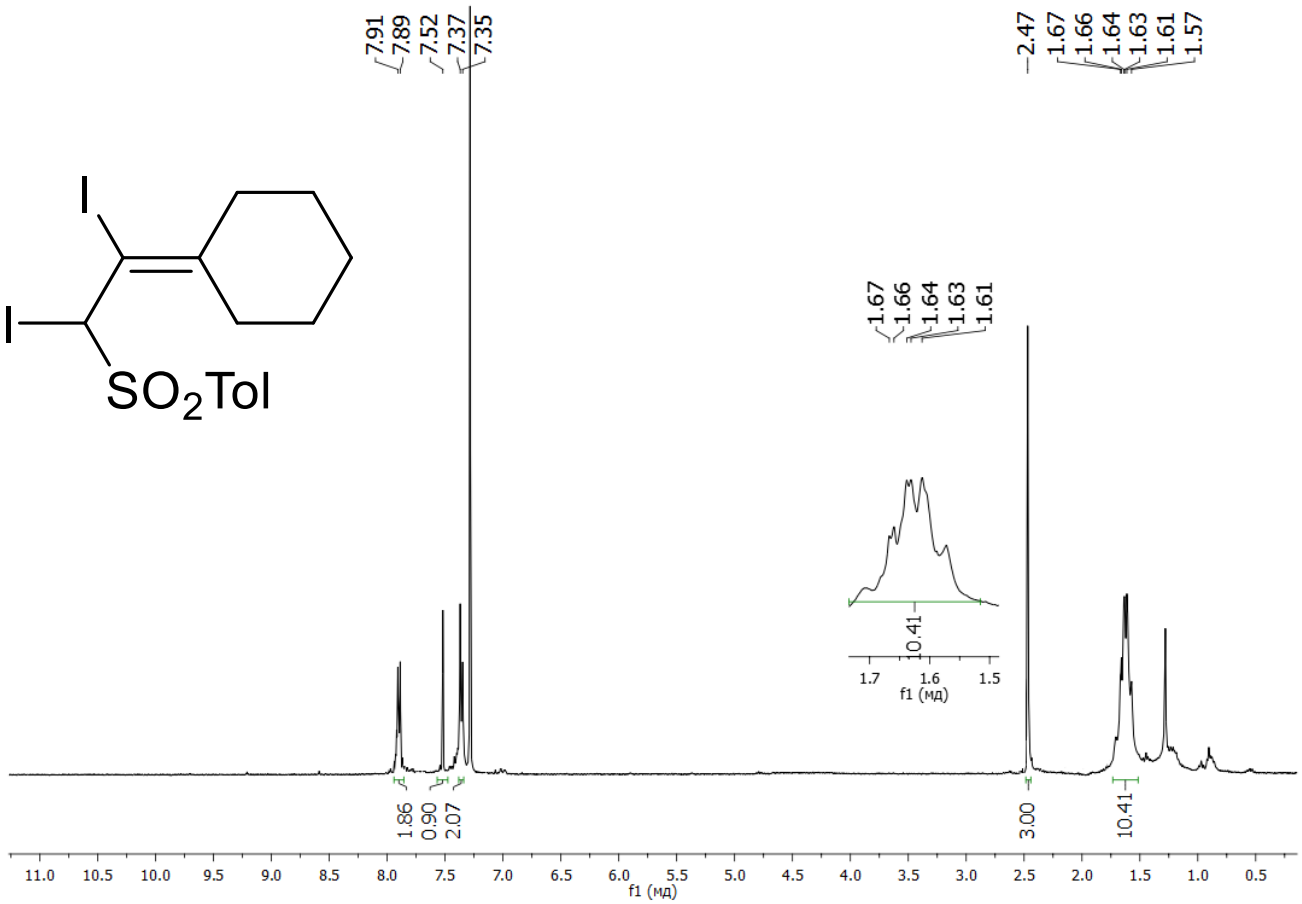


Fig. S18.^{1}H NMR spectrum of compound **2d** (400 MHz, $CDCl_3$).

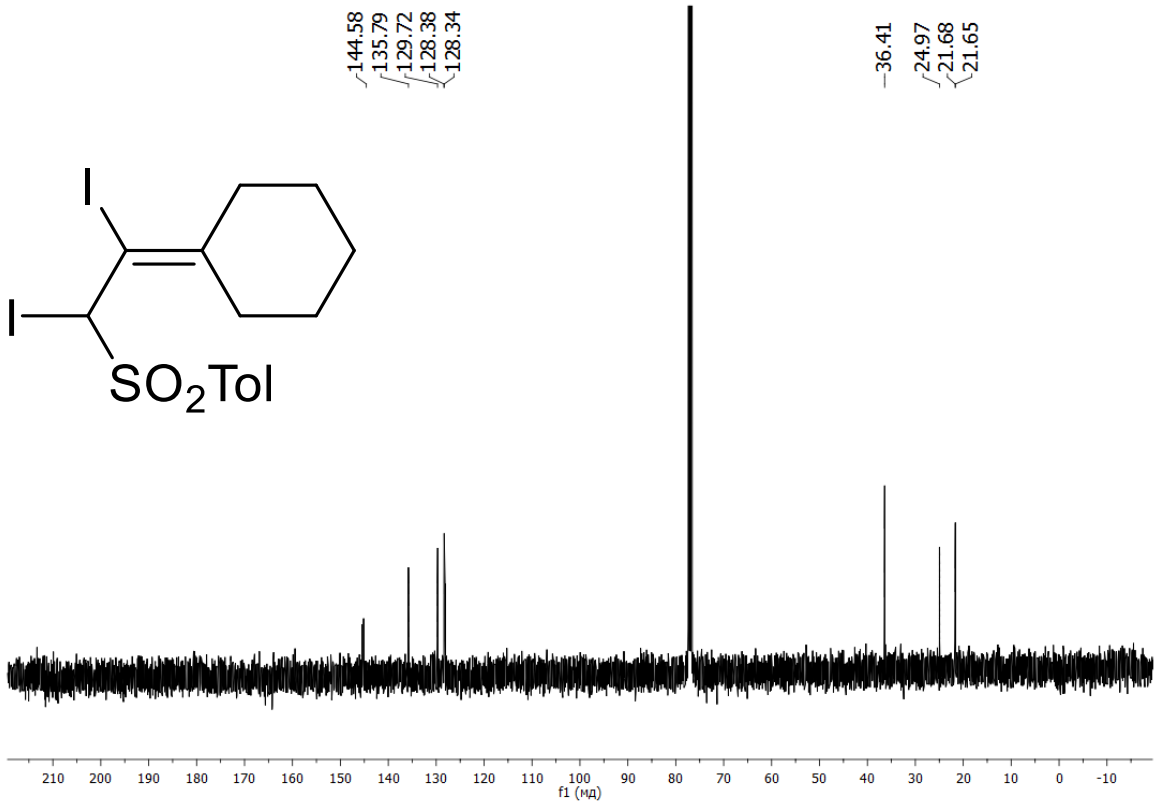


Fig. S19. ^{13}C NMR spectrum of compound **2d** (100 MHz, $CDCl_3$).

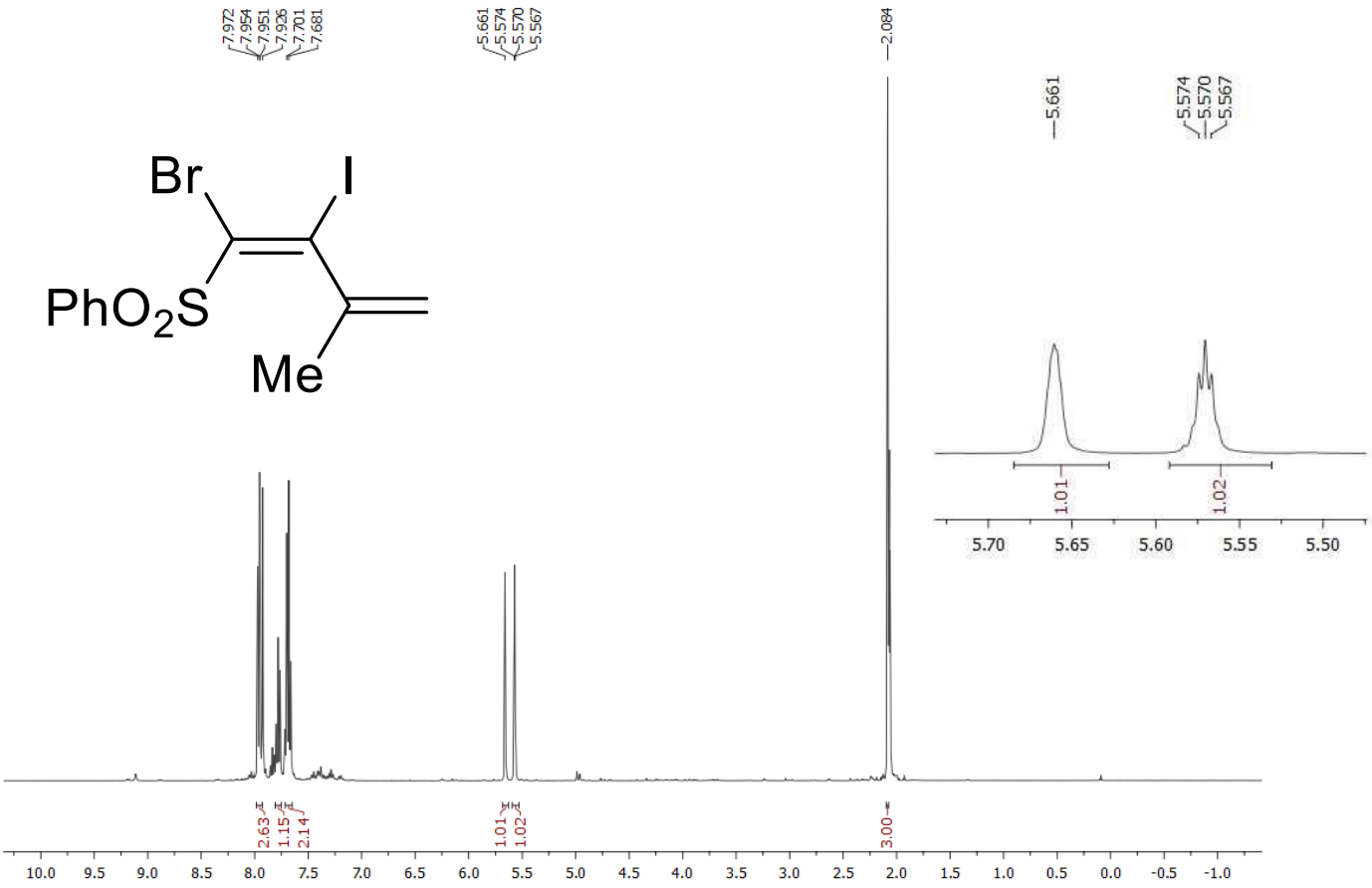


Fig. S20. ^{1}H NMR spectrum of compound *Z*-**3a** (400 MHz, acetone-d6).

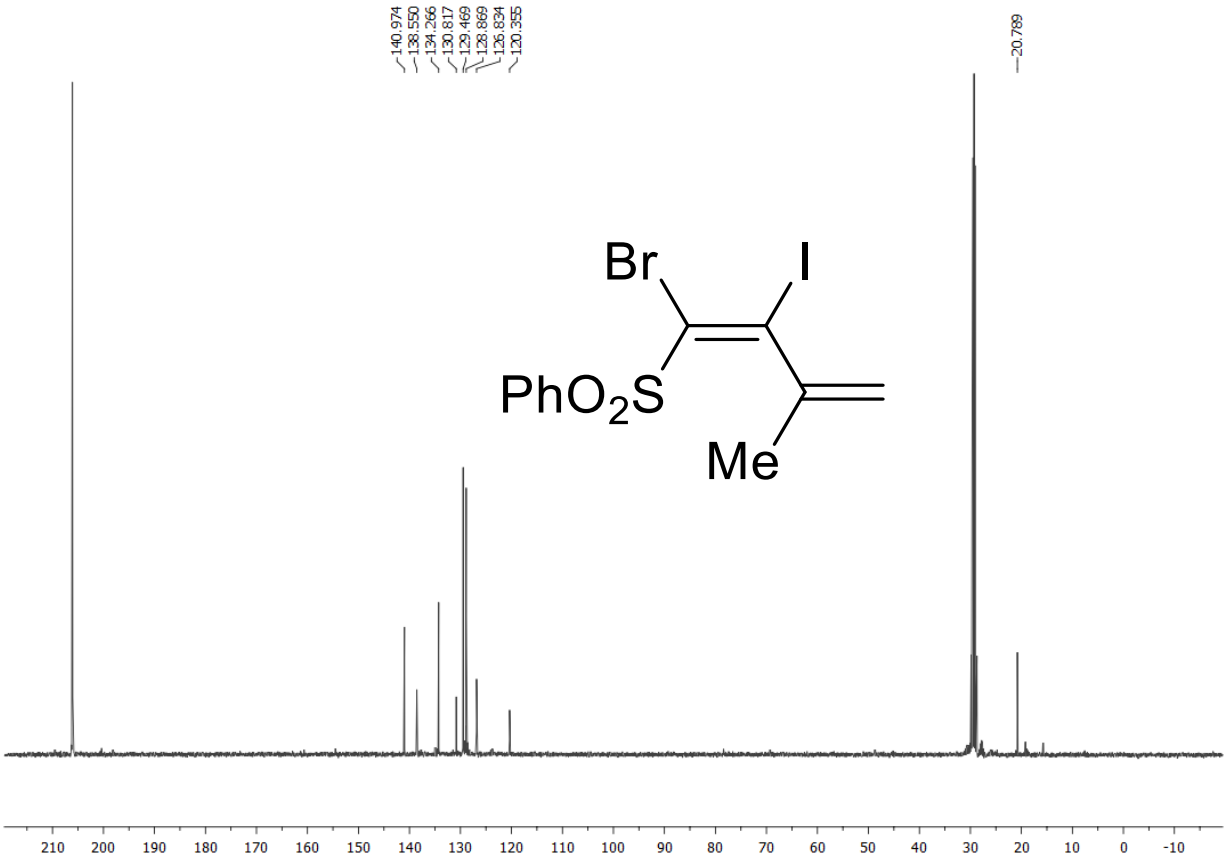


Fig. S21.[13]C NMR spectrum of compound *Z*-**3a** (100 MHz, acetone-d_6).

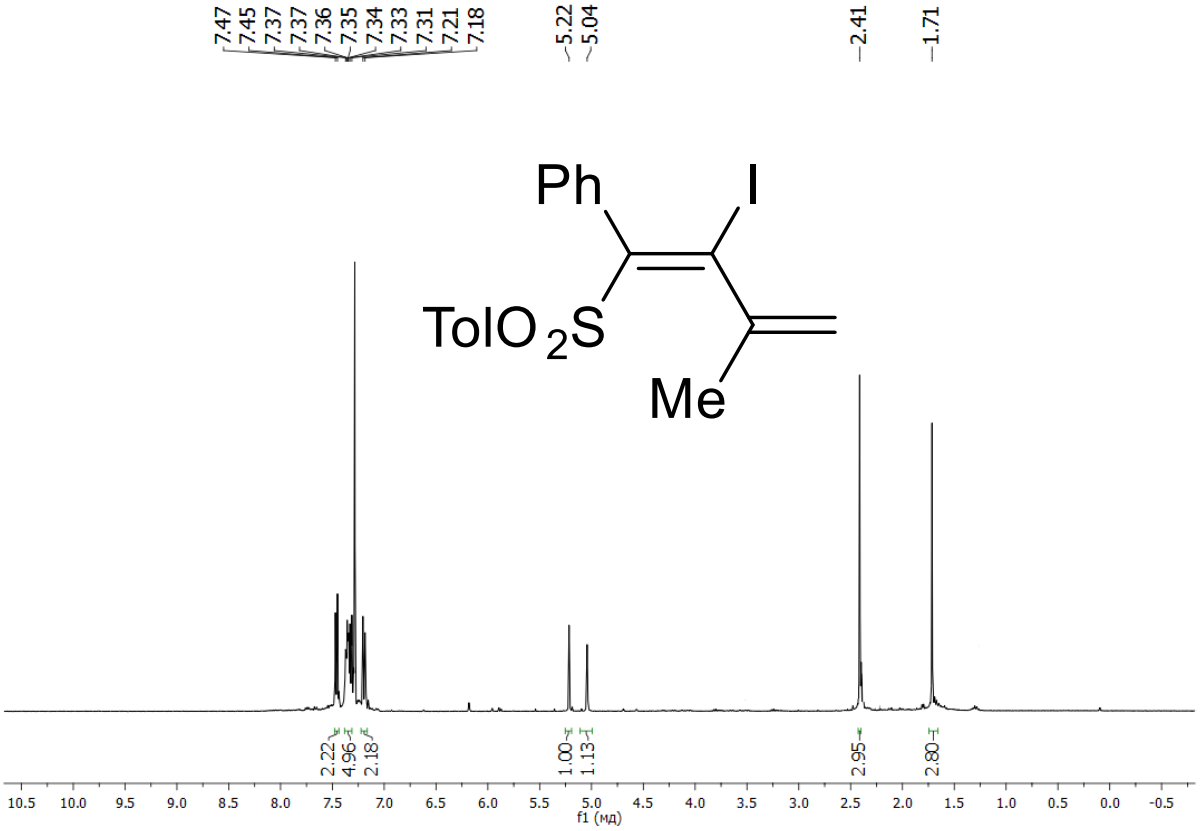


Fig. S22. [1]H NMR spectrum of compound *E*-**3b** (400 MHz,$CDCl_3$).

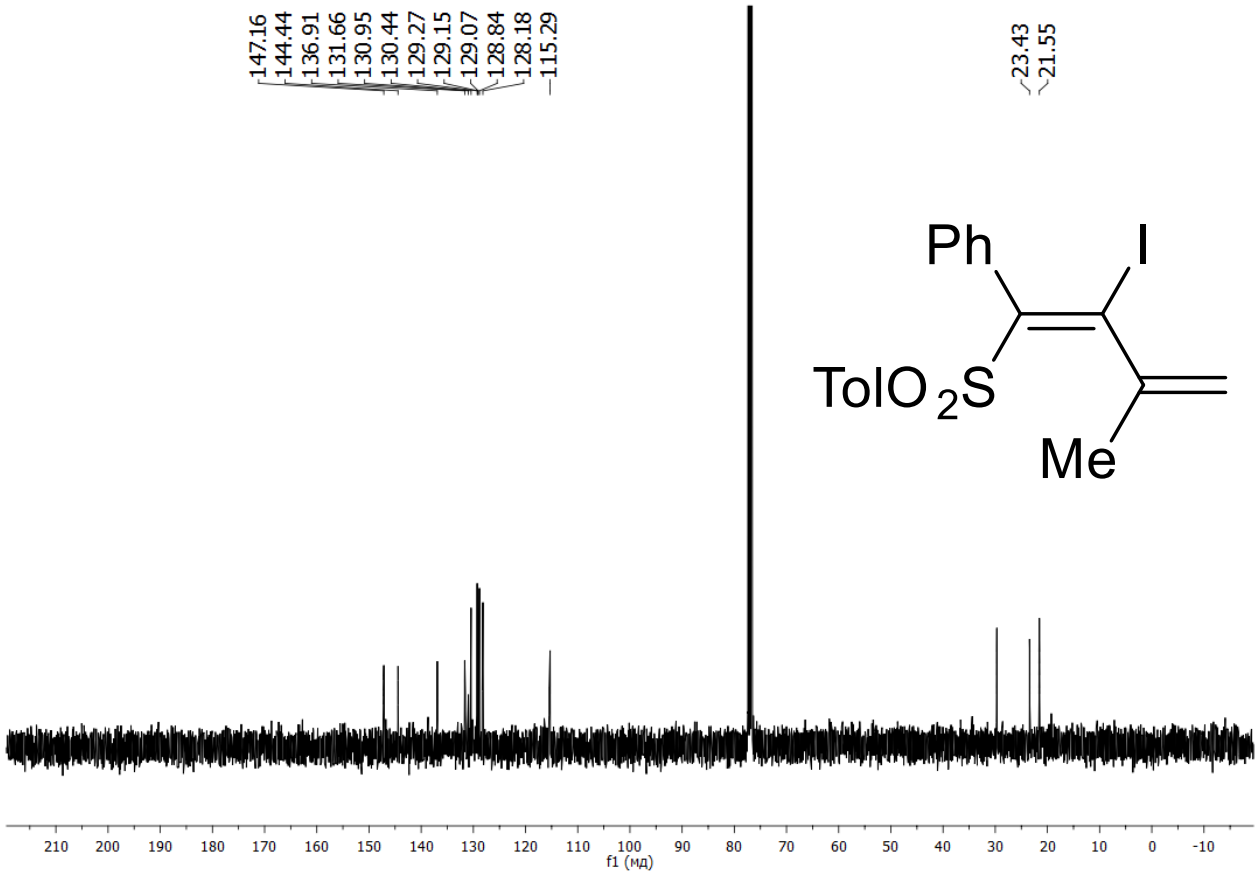


Fig. S23. ^{13}C NMR spectrum of compound *E*-**3b** (100 MHz, $CDCl_3$).

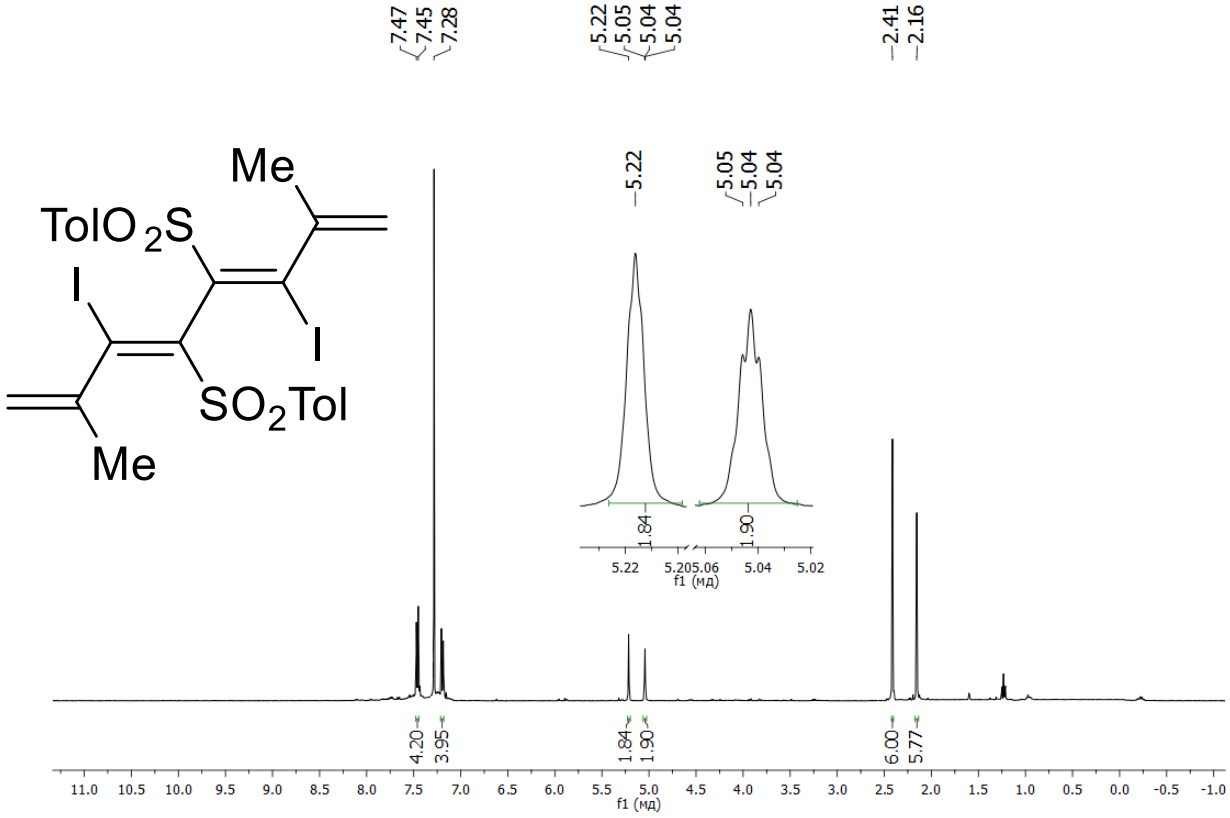


Fig. S24. ^{1}H NMR spectrum of compound *E*,*E*-**3c** (400 MHz, $CDCl_3$).

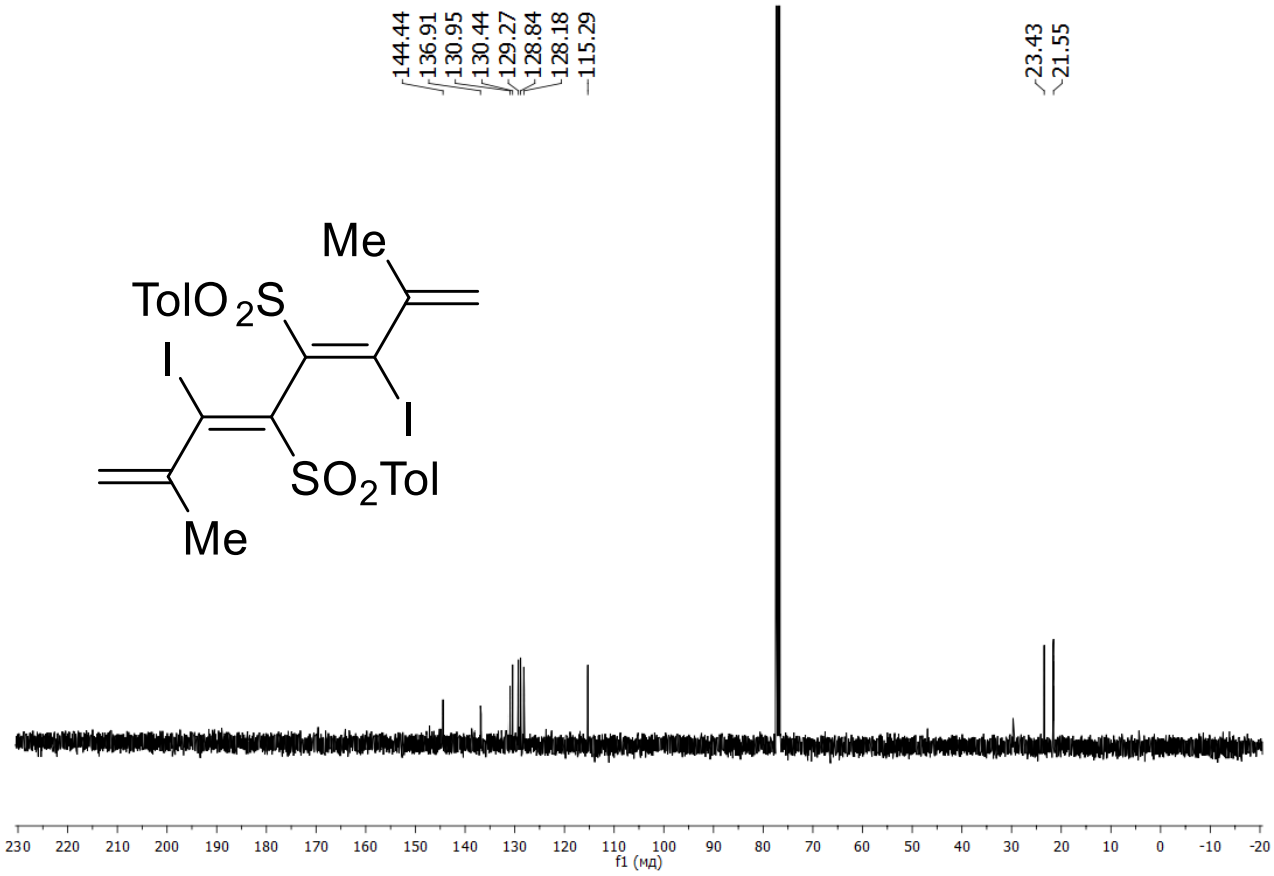
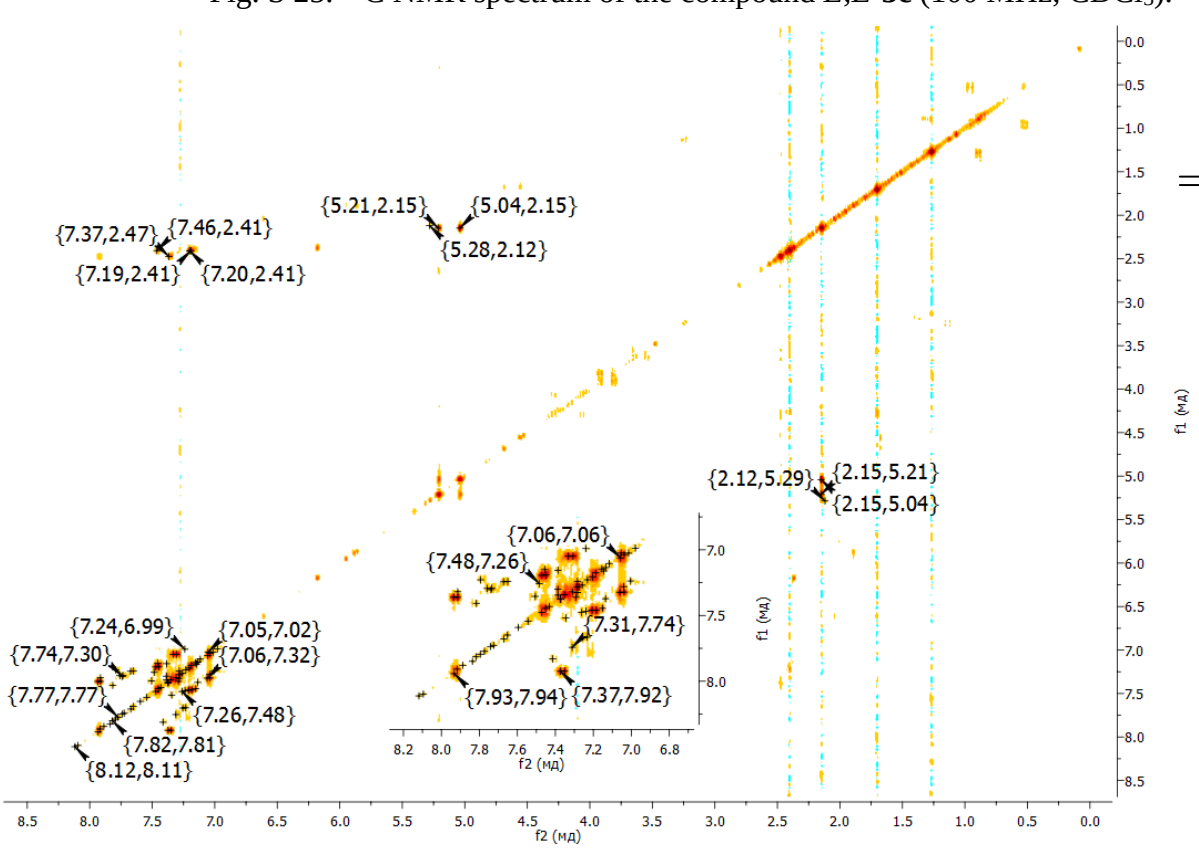


Fig. S 25. ^{13}C NMR spectrum of the compound *E*,*E*-**3c** (100 MHz, $CDCl_3$).



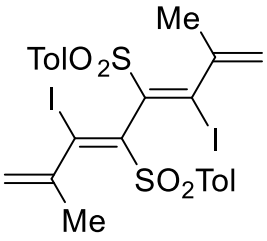


Fig. S26. COSY NMR spectrum of compound *E*,*E*-**3c** (100 MHz, $CDCl_3$).

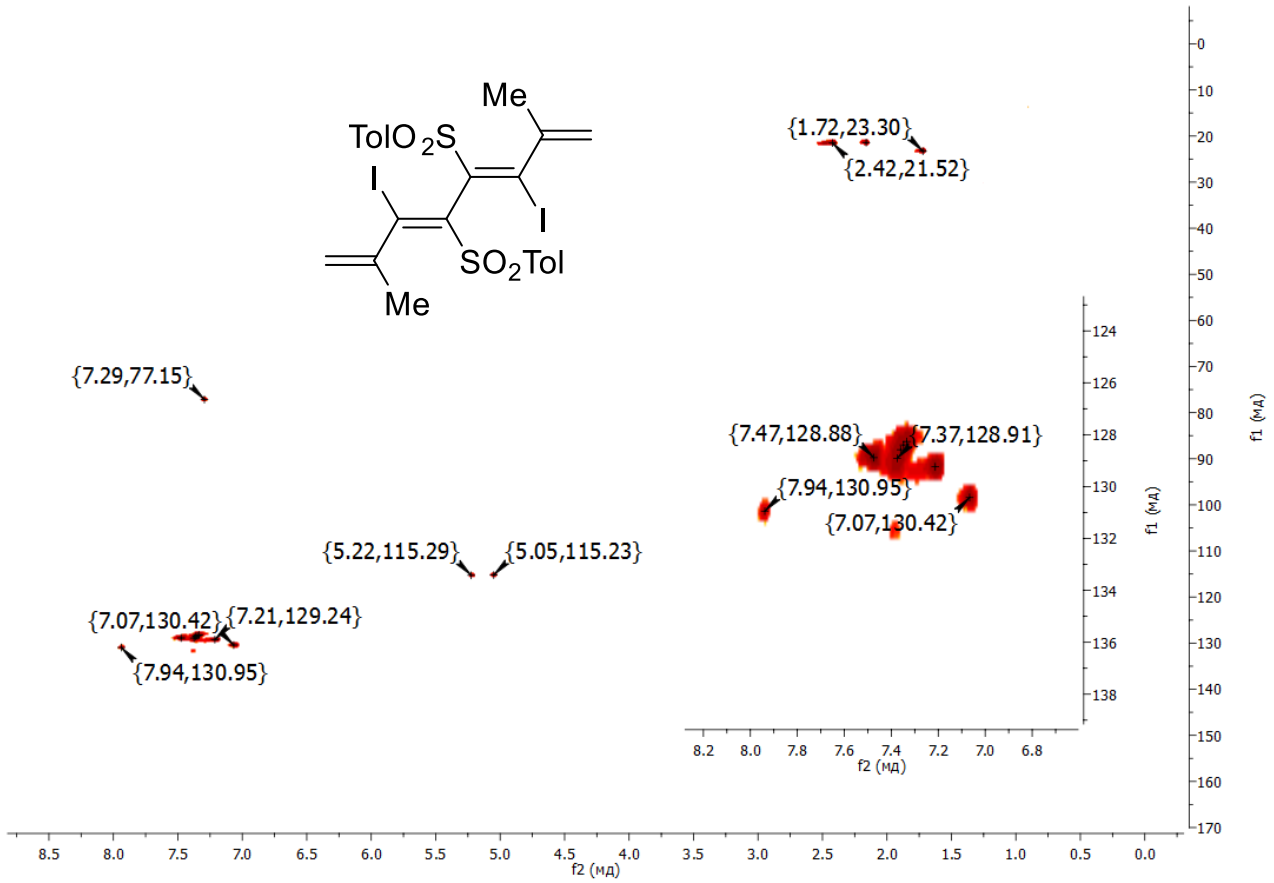


Fig. S27. HSQC NMR spectrum of compound *E*,*E*-**3c** (100 MHz, $CDCl_3$).

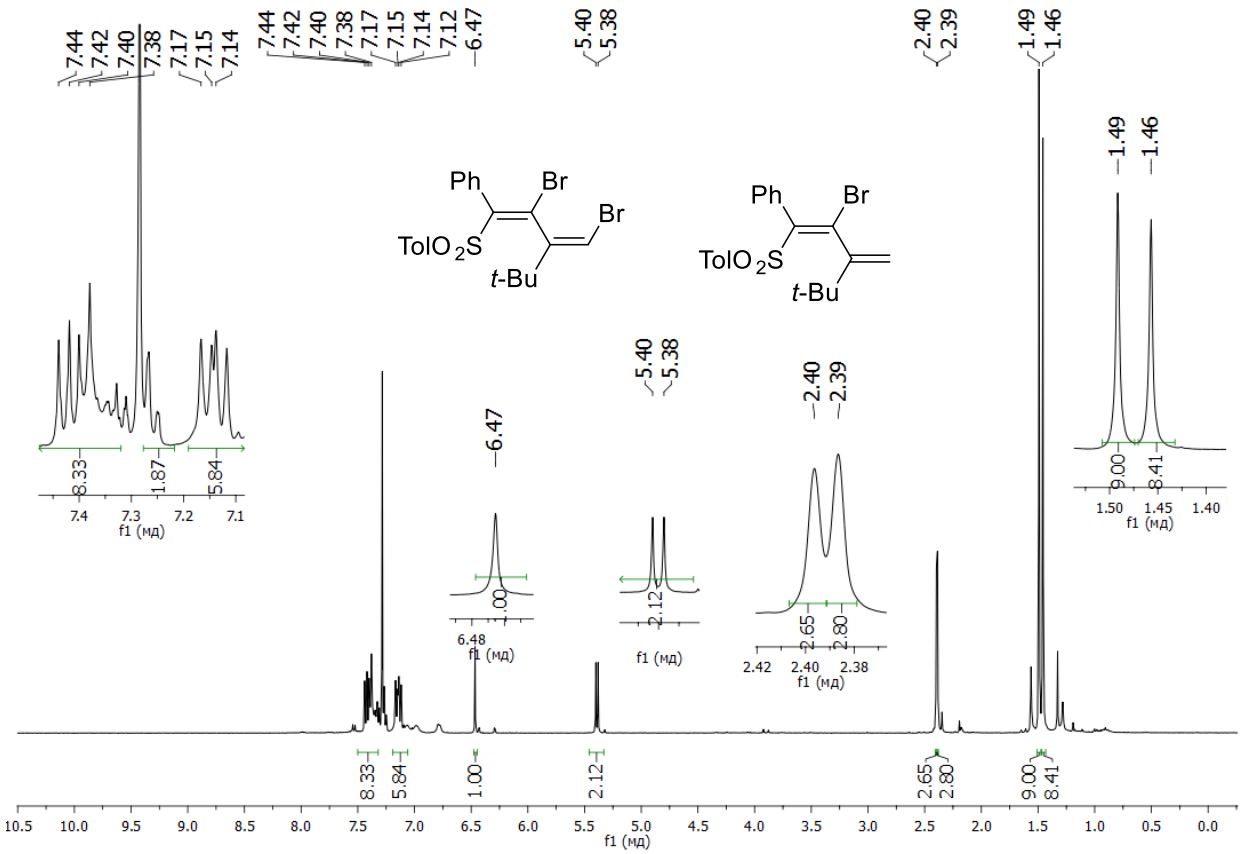


Fig. S28. ^1H NMR spectrum of the mixture of compounds **4** and **5** (400 MHz,$CDCl_3$).

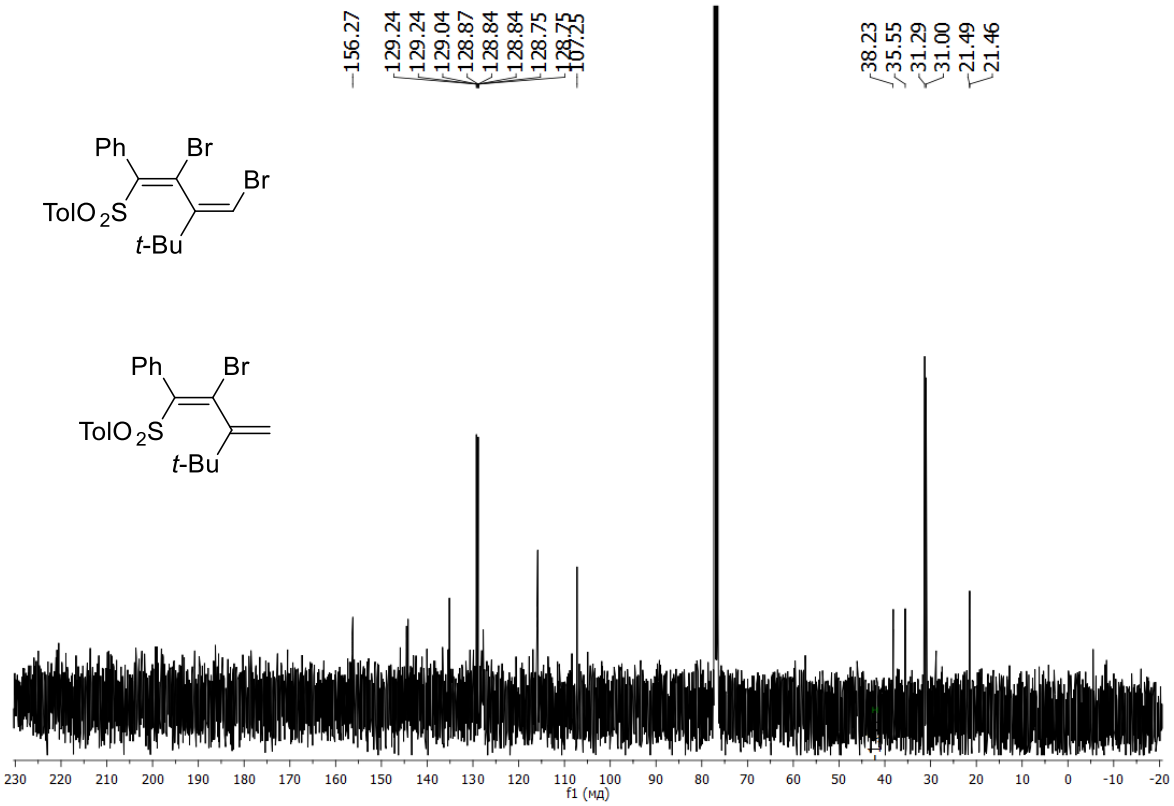


Fig. S29. ^{13}C NMR spectrum of mixture of compounds **4** and **5** (100 MHz, $CDCl_3$).

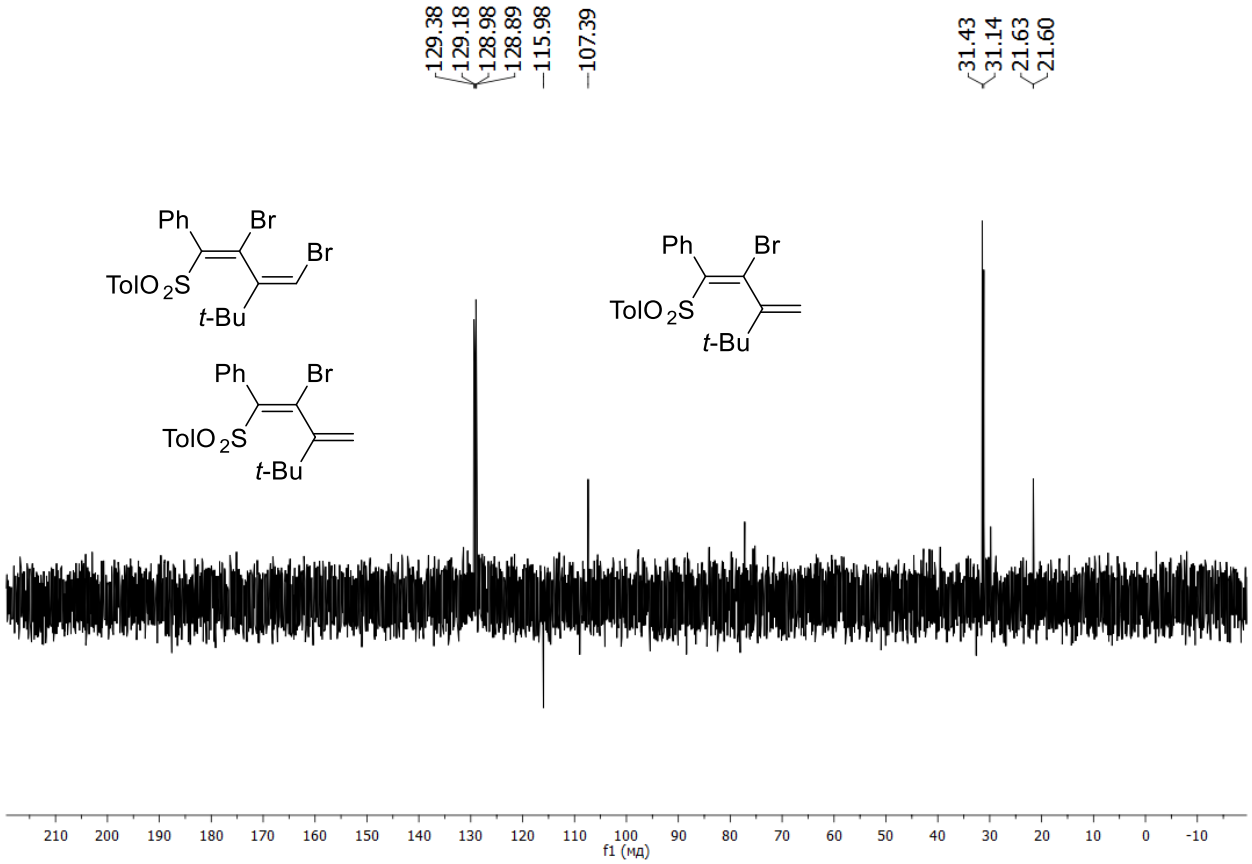


Fig. S30. DEPT NMR spectrum of the mixture of compounds **4** and **5** (100 MHz, $CDCl_3$).

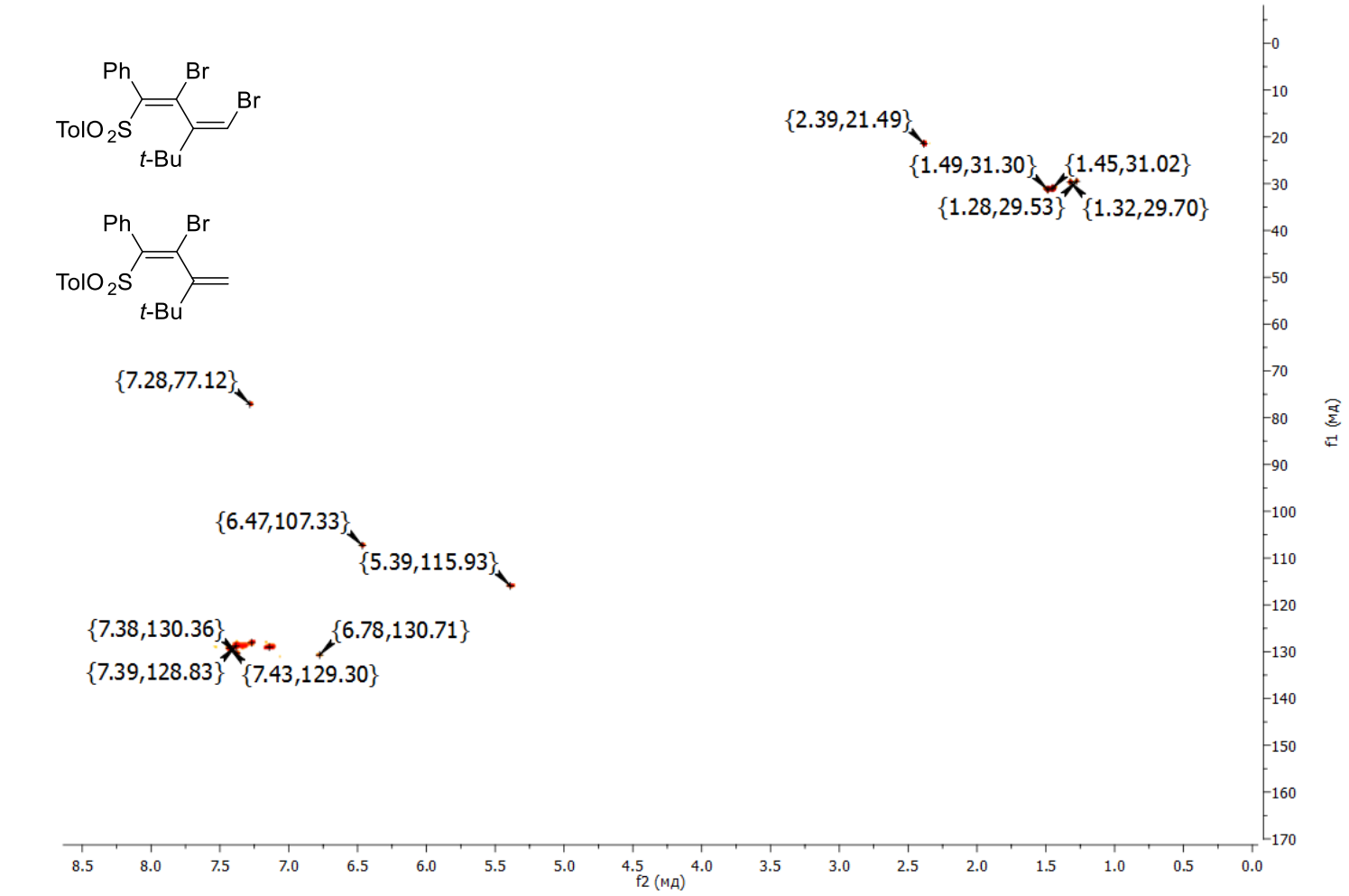


Fig. S31. HSQC NMR spectrum of the mixture of compounds **4** and **5** (100 MHz, $CDCl_3$).

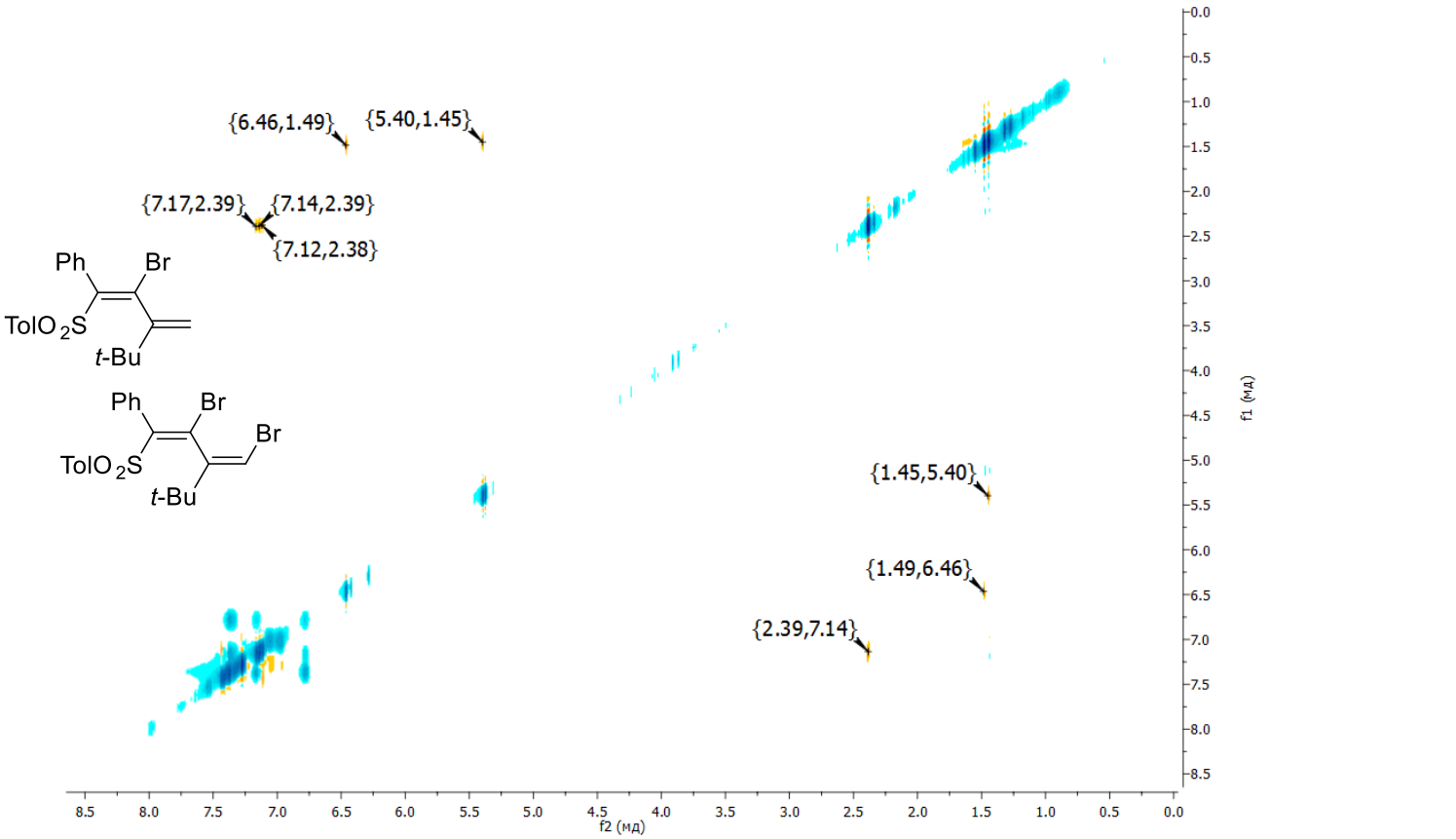


Fig. S32. NOESY NMR spectrum of the mixture of compounds **4** and **5** (100 MHz, $CDCl_3$).

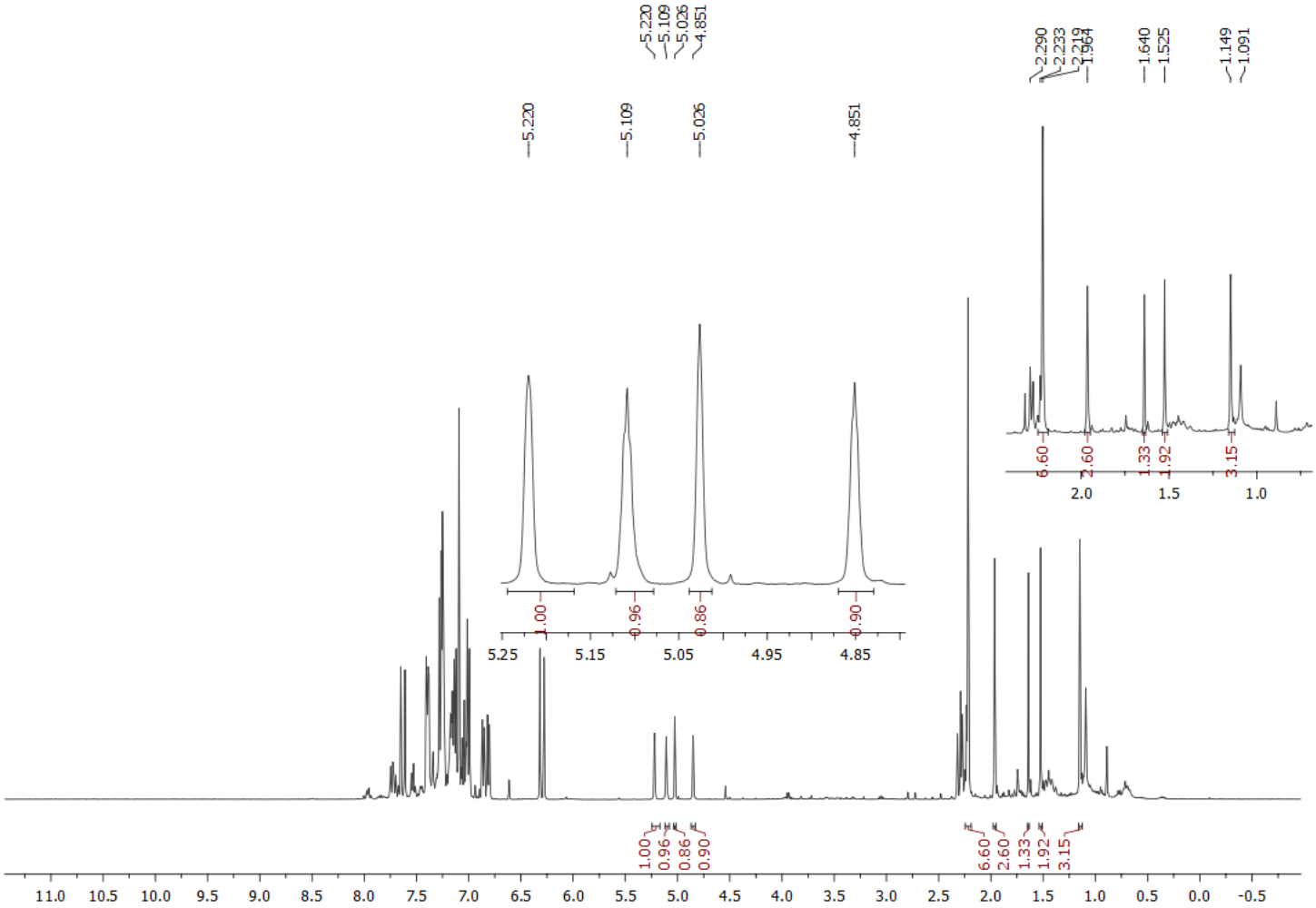


Fig. S33. ^{1}H NMR spectrum of the mixture of compounds *E*-**3b** and *Z*-**3b** (100 MHz, $CDCl_3$).

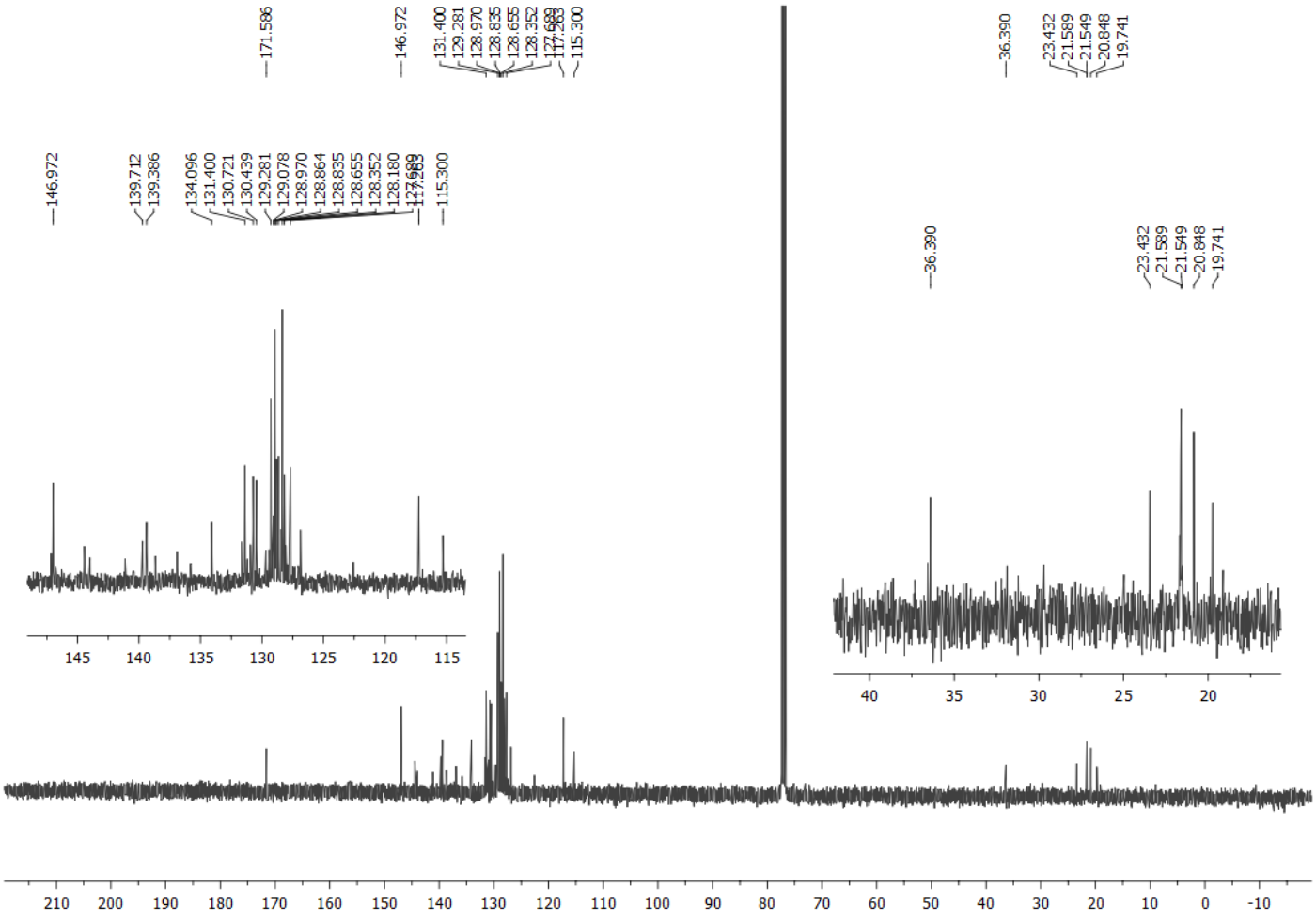
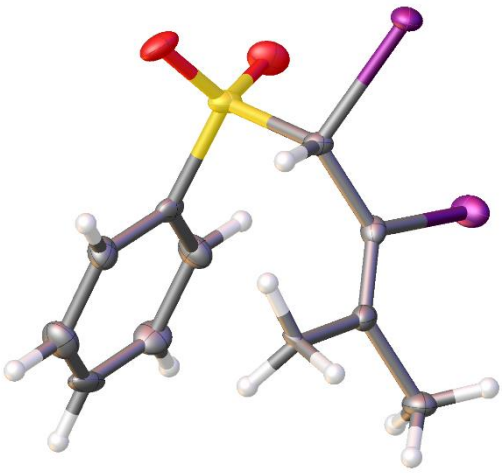
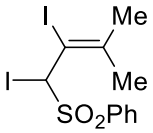


Fig. S34. ^{13}C NMR spectrum of the mixture of compounds *E*-**3b** and *Z*-**3b** (100 MHz, $CDCl_3$).

III. X-Ray data

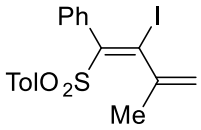




2a CCDC 1580907

Table S1. Crystal data and structure refinement for 2a

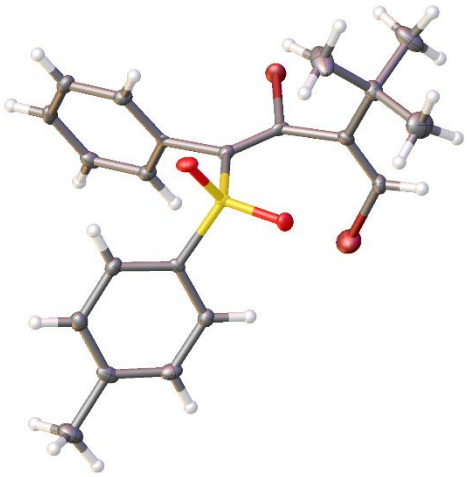
Identification code	AVV308
Empirical formula	$C_{11}H_{12}I_2O_2S$
Formula weight	462.07
Temperature/K	100(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.0871(7)
b/Å	8.4818(6)
c/Å	11.6632(9)
α/°	98.836(6)
β/°	97.451(7)
γ/°	117.945(8)
Volume/Å^3	679.45(11)
Z	2
ρ_{calc}g/cm^3	2.259
μ/mm^{-1}	4.766
F(000)	432.0
Crystal size/mm^3	0.1 × 0.1 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.142 to 54.994
Index ranges	$-10 \leq h \leq 10$, $-7 \leq k \leq 11$, $-15 \leq l \leq 13$
Reflections collected	6246
Independent reflections	3119 [R_{int} = 0.0448, R_{sigma} = 0.0653]
Data/restraints/parameters	3119/0/147
Goodness-of-fit on F^2	1.086
Final R indexes [I>=2σ (I)]	R_1 = 0.0363, wR_2 = 0.0712
Final R indexes [all data]	R_1 = 0.0497, wR_2 = 0.0806
Largest diff. peak/hole / e Å^{-3}	1.02/-1.06

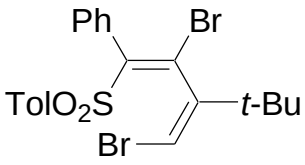


3b CCDC 1843278

Table S2. Crystal data and structure refinement for 3b

Identification code	iols
Empirical formula	$C_{18.25}H_{16.5}IO_2S$
Formula weight	426.77
Temperature/K	100(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	16.4248(4)
b/Å	5.65370(10)
c/Å	19.5043(5)
α/°	90
β/°	111.012(3)
γ/°	90
Volume/Å^3	1690.75(7)
Z	4
ρ_{calc}g/cm^3	1.677
μ/mm^{-1}	16.071
F(000)	844.0
Crystal size/mm^3	0.26 × 0.18 × 0.12
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.06 to 139.934
Index ranges	$-16 \le h \le 20$, $-6 \le k \le 6$, $-23 \le l \le 23$
Reflections collected	14807
Independent reflections	3199 [R_{int} = 0.0471, R_{sigma} = 0.0334]
Data/restraints/parameters	3199/1/210
Goodness-of-fit on F^2	1.063
Final R indexes [I>=2σ (I)]	R_1 = 0.0438, wR_2 = 0.1232
Final R indexes [all data]	R_1 = 0.0462, wR_2 = 0.1257
Largest diff. peak/hole / e Å^{-3}	3.17/-1.15





5 CCDC 1843308

Table S3. Crystal data and structure refinement for 5

Identification code	LZA091
Empirical formula	$C_{21}H_{22}Br_2O_2S$
Formula weight	498.26
Temperature/K	100(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	7.83654(12)
b/Å	17.8958(3)
c/Å	14.6788(2)
α/°	90
β/°	93.0497(16)
γ/°	90
Volume/Å^3	2055.66(6)
Z	4
ρ_{calc}g/cm^3	1.610
μ/mm^{-1}	6.048
F(000)	1000.0
Crystal size/mm^3	0.18 × 0.18 × 0.15
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.796 to 139.888
Index ranges	$-6 \le h \le 9$, $-15 \le k \le 21$, $-17 \le l \le 16$
Reflections collected	8963
Independent reflections	3895 [R_{int} = 0.0264, R_{sigma} = 0.0294]
Data/restraints/parameters	3895/0/239
Goodness-of-fit on F^2	1.107
Final R indexes [I>=2σ (I)]	R_1 = 0.0528, wR_2 = 0.1583
Final R indexes [all data]	R_1 = 0.0563, wR_2 = 0.1614
Largest diff. peak/hole / e Å^{-3}	1.03/-1.90

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