Supporting Information

Visible-Light-Enabled Reduction of Arylsulfonyl and Benzoyl Azides

to Access Primary Arylsulfonamides and Benzoyl Amides

Qing Li, Xiaoxu Ma, Yue Bi, Mengxue Ma, Ping Zhang* and Xianwei Sui*

Key Laboratory of Green and Precise Synthetic Chemistry and Applications, Ministry of Education, School of Chemistry and Materials Science, Huaibei Normal University, Huaibei, Anhui 235000, P. R. China.

Email: suixw@chnu.edu.cn; zhangping@chnu.edu.cn.

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1. General Information

Nuclear magnetic resonance spectra were recorded on Bruker 600 MHz instruments internally referenced to TMS (0.0 ppm) or residue of CDCl₃ (7.26 ppm for ¹H, and 77.00 ppm for ¹³C) or DMSO- d_6 (2.50 ppm for ¹H, and 39.60 ppm for ¹³C) signal. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were conducted at Micromass Q-Tof instrument (ESI). All reagents were purchased at the highest commercial quality and used without further purification unless otherwise stated. All reactions were performed under an inert atmosphere of dry argon in dried glassware, unless otherwise stated. Solvents were distilled using standard techniques. Acetonitrile and dichloromethane were distilled over calcium hydride under an atmosphere of argon. Room temperature reactions were performed between 25-28 °C. The reactions that require heating were put in an oil bath with a temperature monitor. Kessil lamp (440 nm) was used as the light source. The sulfonyl and acyl azides were synthesized according to the literature.¹

2. Experimental Section

2.1. Optimization Studies

Me N ₃		MesAcr ⁺ (5 mol%) Base, CH ₃ CN, Ar, 18 h Blue LEDs		NH ₂	
Entries	1a Base	Temn	Time	2a 	
1	Me ₃ N	r.t	18 h	50%	
2	Et ₃ N	r.t	18 h	29%	
3	Bn ₃ N	r.t	18 h	94%	
4	DABCO	r.t	18 h	14%	
5	DMAP	r.t	18 h	10%	
6	DBU	r.t	18 h	5%	

Table S1. Screening of Bases *a,b*

^{*a*} Reaction conditions: **1a** (0.2 mmol), Base (1.5 equiv.), 9-Mesityl-10-methylacridinium perchlorate (**MesAcr**⁺) (5 mol%) in CH₃CN (2.0 mL) at room temperature under Ar and Blue LEDs irradiation for 18 h. ^{*b*} The yield was determined by ¹H NMR analysis of crude product using

1,3,5-trimethoxybenzene as an internal standard.

	O S ⁼⁰	MesAcr ⁺ (5 mol%)			
Me N ₃		Bn ₃ N, Solvent, Ar, 18 h Blue LEDs		NH ₂	
	1a			2a	
Entries	Solvent	Temp.	Time	Yield (%) ^b	
1	DCM	r.t	18 h	70%	
2	Dioxane	r.t	18 h	87%	
3	DMF	r.t	18 h	22%	
4	CH ₃ CN	r.t	18 h	94%	
5	DMSO	r.t	18 h	59%	
6	THF	r.t	18 h	77%	
7	toluene	r.t	18 h	43%	

Table S2. Screening of Solvents ^{a,b}

^{*a*} Reaction conditions: **1a** (0.2 mmol), Bn₃N (1.5 equiv.), 9-Mesityl-10-methylacridinium perchlorate (**MesAcr**+) (5 mol%) in solvent (2.0 mL) at room temperature under Ar and Blue LEDs irradiation for 18 h. ^{*b*} The yield was determined by ¹H NMR analysis of crude product using 1,3,5-trimethoxybenzene as an internal standard.

Table S3. Screening of Catalysts ^{a,b}

0 S=0	PC (5 mol%)	0 \$=0
Me N ₃	Bn ₃ N, CH ₃ CN, Ar, 18 h Blue LEDs	Me NH ₂
1a		2a

Entries	PC	Temp.	Time	Yield (%) ^b
1	$MesAcr^+$	r.t	18 h	94%
2	PTH	r.t	18 h	29%
3	<i>fac</i> -Ir(ppy) ₃	r.t	18 h	77%
4	4CzIPN	r.t	18 h	46%
5	Ru(bpy) ₃ Cl ₂	r.t	18 h	61%

^{*a*} Reaction conditions: **1a** (0.2 mmol), Bn₃N (1.5 equiv.), catalyst (5 mol%) in CH₃CN (2.0 mL) at room temperature under Ar and Blue LEDs irradiation for 18 h. ^{*b*} The yield was determined by ¹H NMR analysis of crude product using 1,3,5-trimethoxybenzene as an internal standard.

2.2. General Procedure for the Reduction of Arylsulfonyl and Benzoyl azides

9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.0100 mmol, 5 mol%), arylsulfonyl azide or benzoyl azide (0.200 mmol, 1.0 equiv.) and Bn₃N (0.300 mmol, 1.5 equiv.) were added in a Schlenk tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added and the mixture was stirred for 18 h irradiated by the LEDs at room temperature. After the reaction is complete, the reaction mixture was filtered through a pad of Celite. The filtrate was concentrated in vacuo to afford crude products, which were purified by flash column chromatography on silica gel to give the pure products.

2.3. Gram-Scale Synthesis for Product 2a



9-mesityl-10-methylacridinium perchlorate (102.5 mg, 0.250 mmol, 5 mol%), 4methylbenzenesulfonyl azide **1a** (985 mg, 5.0 mmol, 1.0 equiv.) and Bn₃N (7.5 mmol, 1.5 equiv.) were added to a 100 mL round bottom flask, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (50.0 mL) were added and the mixture was stirred for 18 h irradiated by the LEDs at room temperature. After the reaction is complete, the reaction mixture was filtered through a pad of Celite. The filtrate was concentrated in vacuo to afford crude products, which were purified by flash column chromatography on silica gel to give the pure product **2a** (670 mg, 78%).

2.4. Synthesis for Bioactive Molecule



The reaction of 4-ethoxy-3-(1-methyl-7-oxo-3-propyl-6,7-dihydro-1H-pyrazolo[4,3-d]pyrimidin-5yl)benzenesulfonyl azide **8** (83.4 mg, 0.200 mmol, 1.0 equiv.) which is afforded by reacting 4-ethoxy-3-(1methyl-7-oxo-3-propyl-6,7-dihydro-1H-pyrazolo[4,3-d]pyrimidin-5-yl)benzenesulfonyl chloride **7** with NaN₃ in acetone solution, Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **9** (65.7 mg, 84%) as a white solid after purification via silica gel column chromatography with 20% ethyl acetate/hexanes (Rf = 0.4, 20% ethyl acetate/hexanes). ¹H NMR (600 MHz, DMSO-d₆) δ 12.19 (s, 1H), 8.01 (d, *J* = 2.4 Hz, 1H), 7.90 (dd, *J* = 2.4, 9.0 Hz, 1H), 7.36 (s, 2H), 7.31 (d, *J* = 9.0 Hz, 1H), 4.21-4.15 (m, 5H), 2.78 (t, *J* = 7.8 Hz, 2H), 1.78-1.70 (m, 2H), 1.34 (t, *J* = 7.2Hz, 3H), 0.94 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (150 MHz, DMSO-d₆) δ 158.8, 153.8, 148.6, 150.0, 137.9, 136.0, 129.6, 128.3, 124.4, 123.0, 112.9, 64.8, 37.9, 27.2, 21.8, 14.4, 13.9; **HRMS (ESI)** Calcd for C₁₇H₂₁N₅O₄S [M+H]⁺ 392.1387, found 392.1386.

2.5. Mechanism Experiments

2.5.1 Radical- trapping with DMPO



9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.0100 mmol, 5 mol%), 4-methylbenzenesulfonyl azide **1a** or **3a** (0.200 mmol, 1.0 equiv.), Bn_3N (0.300 mmol, 1.5 equiv.) and 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) (3.0 equiv.) were added to a Schlenk tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added and the mixture was stirred for 12 h irradiated by the LEDs at room temperature. After the reaction is complete, the reaction mixture was filtered through a pad of Celite. The diluted filtrate was analyzed by HRMS, and products **2a** and **4a** were not detected, but possible radical intermediates **1a' and 3a'** were detected.

2.5.2 Radical trapping with 1,1-diphenylethene



9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.0100 mmol, 5 mol%), 4-methylbenzenesulfonyl azide **1a** or **3a** (0.200 mmol, 1.0 equiv.), Bn_3N (0.300 mmol, 1.5 equiv.) and 1,1diphenylethene (3.0 equiv.) were added to a Schlenk tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added and the mixture was stirred for 12 h irradiated by the LEDs at room temperature. After the reaction is complete, the reaction mixture was filtered through a pad of Celite. The diluted filtrate was analyzed by HRMS, and products **2a** and **4a** were detected, meanwhile, radical intermediates **10** and **11** also were detected.

2.5.3 Radical clock experiments



9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.0100 mmol, 5 mol%), 4-methylbenzenesulfonyl azide 1a or 3a (0.200 mmol, 1.0 equiv.), Bn_3N (0.300 mmol, 1.5 equiv.) and 12 (3.0 equiv.) were added to a Schlenk tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added and the mixture was stirred for 12 h irradiated by the LEDs at room temperature. After the reaction is complete, the reaction mixture was filtered through a pad of Celite. The diluted filtrate was analyzed by HRMS, and the ring-opened products 13 and 14 were detected.

2.5.4 Quantum yield measurement



9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.0100 mmol, 5 mol%), arylsulfonyl azide **1a** (0.200 mmol, 1.0 equiv.) and Bn₃N (0.300 mmol, 1.5 equiv.) were added in a Schlenk tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added and the mixture was irradiated ($\lambda = 440$ nm, with intensity of 7.65 mW/cm²) by the LEDs for 7200s at room temperature. After irradiated, the yield of product **2a** was determined by ¹HNMR based on 1,3,5-trimethoxybenzene standard. The quantum yield Φ was determined as follows.

$$\phi = \frac{n_{2a}N_A / t}{fP\lambda / hc}$$

Notes: n_{2a} : the mole number(3.4×10^{-5} mol) of the product **2a**; t: reaction time (7200 s); N_A: 6.02×10^{23} /mol; f: 1-10^{-A} (440 nm, A =0.31); P: P = E*S (E = 7.65 mW/cm², S: the area that irradiated S = 3.10 cm²); λ : wavelength (4.4×10^{-7} m); h: planck constant (6.626×10^{-34} J*s); c: speed of light (3×10^{8} m/s).

$\Phi = 0.11$

2.5.5 Light on-off experiment

Light on-off experiments were carried out by following the procedure below. 4-methylbenzenesulfonyl azide **1a** (39.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) were added in a Schlenk

tube, and the mixture was degassed under vacuum and purged with argon for three times at room temperature. CH₃CN (2.0 mL) were added. The tube was stirred at room temperature alternating intervals of irradiation by blue LEDs and two-hour dark, during a total of 14 hours. The GC-MS yield of the product was monitored with dodecane as internal standard after each interval.



2.6. Characterization of Products



Compound $2a^2$ was synthesized according to the General Procedure. The reaction of 4methylbenzenesulfonyl azide 1a (39.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2a (30.8 mg, 90%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.81 (d, *J* = 7.8 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 4.94 (s, 2H), 2.43 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 143.6, 139.0, 129.7, 126.4, 21.5.



Compound $2b^2$ was synthesized according to the General Procedure. The reaction of 4methoxybenzenesulfonyl azide 1b (42.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2b (34.0 mg, 91%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.86 (d, *J* = 9.0 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 4.84 (s, 2H), 3.87 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 163.0, 133.6, 128.6, 114.2, 55.6.



Compound $2c^5$ was synthesized according to the General Procedure. The reaction of 4acetamidobenzenesulfonyl azide 1c (48.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2c (35.9 mg, 84%) as a white solid after purification via silica gel column chromatography (Rf = 0.3, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, DMSO-d₆) δ 10.26 (s, 1H), 7.81-7.68 (m, 4H), 7.22 (s, 2H), 2.08 (s, 3H); ¹³C NMR (150 MHz, DMSO-d₆) δ 169.0, 142.3, 138.2, 126.8, 118.5, 24.2.



Compound **2d** was synthesized according to the General Procedure. The reaction of 4-(trifluoromethoxy)benzenesulfonyl azide **1d** (53.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2d** (41.0 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.48, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.95 (d, *J* = 7.8 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.50 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 150.4, 143.2, 128.3, 121.5, 120.0 (q, *J* = 256.5 Hz); ¹⁹F NMR (565 MHz, DMSO-d₆) δ -56.8; HRMS (ESI) Calcd for C₇H₆F₃NO₃S [M+H]⁺ 242.0093, found 242.0095.



Compound $2e^2$ was synthesized according to the General Procedure. The reaction of 4-fluorobenzenesulfonyl azide 1e (40.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2e (29.8 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.92-7.87 (m, 2H), 7.44-7.38 (m, 4H); ¹³C NMR (150 MHz, DMSO-d₆) δ 164.6, 163.0, 140.7 (d, *J* = 3.0 Hz), 128.6 (d, *J* = 9.0 Hz), 116.1 (d, *J* = 21.0 Hz); ¹⁹F NMR (565 MHz, DMSO-d₆) δ -108.1.



Compound $2f^3$ was synthesized according to the General Procedure. The reaction of 4chlorobenzenesulfonyl azide 1f (43.3 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL)

delivered **2f** (33.2 mg, 87%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.85-7.82 (m, 2H), 7.68-7.64 (m, 2H), 7.47 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 143.1, 136.7, 129.2, 127.7.



Compound $2g^2$ was synthesized according to the General Procedure. The reaction of 4-(trifluoromethyl)benzenesulfonyl azide 1g (50.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH3CN (2.0 mL) delivered 2g (34.2 mg, 76%) as a white solid after purification via silica gel column chromatography (Rf = 0.4, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.04 (d, J = 7.8 Hz, 2H), 7.97 (d, J = 7.8 Hz, 2H), 7.62 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 147.9, 131.8 (q, J = 33.0 Hz), 126.7, 126.4 (q, J = 3.0 Hz), 124.6, 122.8, 121.0; ¹⁹F NMR (565 MHz, DMSO-d₆) δ -61.6.



Compound $2h^4$ was synthesized according to the General Procedure. The reaction of 4cyanobenzenesulfonyl azide 1h (41.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2h (29.0 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.51, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.07 (d, *J* = 8.4 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.65 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 148.1, 133.4, 126.5, 118.0, 114.4.



Compound $2i^2$ was synthesized according to the General Procedure. The reaction of 4nitrobenzenesulfonyl azide 1i (45.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2i (34.3 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.6, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.42 (d, *J* = 8.4 Hz, 2H), 8.07 (d, *J* = 9.0 Hz, 2H), 7.73 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 149.5, 149.3, 127.3, 124.5.



Compound $2j^6$ was synthesized according to the General Procedure. The reaction of 2chlorobenzenesulfonyl azide 1j (43.3 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2j (32.4 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.4, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.98 (dd, *J* = 1.2, 7.8 Hz, 1H), 7.64 (dd, *J* = 1.2, 7.8 Hz, 1H), 7.62-7.58 (m, 3H), 7.52 (td, *J* = 1.2, 7.8 Hz, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 141.1, 133.5, 131.6, 130.4, 129.1, 127.5.



Compound $2k^6$ was synthesized according to the General Procedure. The reaction of methyl 2-(azidosulfonyl)benzoate 1k (48.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2k (37.4 mg, 87%) as a white solid after purification via silica gel column chromatography (Rf = 0.65, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, CDCl₃) δ 8.17-8.11 (m, 1H), 7.92-7.81 (m, 1H), 7.70-7.58 (m, 2H), 5.76 (s, 2H), 3.99 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 168.0, 141.5, 132.4, 132.0, 130.7, 129.9, 128.4, 53.4.



Compound $2l^3$ was synthesized according to the General Procedure. The reaction of 3chlorobenzenesulfonyl azide 11 (43.3 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 21 (31.7 mg, 83%) as a white solid after purification via silica gel column chromatography (Rf = 0.4, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.85 (t, J = 1.8 Hz, 1H), 7.80-7.77 (m, 1H), 7.71-7.66 (m, 1H), 7.62 (t, J = 7.8 Hz, 1H), 7.52 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 146.0, 133.6, 131.9, 131.2, 125.5, 124.4.



Compound $2m^6$ was synthesized according to the General Procedure. The reaction of 3bromobenzenesulfonyl azide 1m (52.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2m (35.2 mg, 75%) as a white solid after purification via silica gel column chromatography (Rf = 0.58, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.98 (t, J = 1.8 Hz, 1H), 7.85-7.80 (m, 2H), 7.55 (t, J = 7.8 Hz, 1H), 7.51 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 146.2, 134.7, 131.4, 128.3, 124.7, 121.9.



Compound $2n^2$ was synthesized according to the General Procedure. The reaction of benzenesulfonyl

azide **1n** (36.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2n** (27.6 mg, 88%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.94 (d, *J* = 7.2 Hz, 2H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.53 (t, *J* = 7.8 Hz, 2H), 4.85 (s, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 141.9, 132.8, 129.2, 126.4.



Compound 20^2 was synthesized according to the General Procedure. The reaction of naphthalene-2-sulfonyl azide 10 (46.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 20 (31.9 mg, 77%) as a white solid after purification via silica gel column chromatography (Rf = 0.48, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.43 (d, *J* = 1.2 Hz, 1H), 8.16-8.10 (m, 2H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.89 (dd, *J* = 2.4, 9.0 Hz, 1H), 7.72-7.63 (m, 2H), 7.45 (s, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 141.3, 134.0, 131.8, 129.1, 128.5, 127.9, 127.6, 125.8, 122.2.



Compound **2p** was synthesized according to the General Procedure. The reaction of 2-oxo-*2H*-chromene-6-sulfonyl azide **1p** (50.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2p** (39.1 mg, 87%) as a white solid after purification via silica gel column chromatography (Rf = 0.66, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.23-8.20 (m, 2H), 8.01 (dd, *J* = 2.4, 8.4 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.49 (s, 2H), 6.62 (d, *J* = 9.6 Hz, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 159.4, 155.3, 143.8, 140.3, 129.1, 126.3, 118.7, 117.7, 117.4; HRMS (ESI) Calcd for C₉H₇NO₄S [M+H]⁺ 226.0169, found 226.0170.



Compound **2q** was synthesized according to the General Procedure. The reaction of 2,3dihydrobenzofuran-5-sulfonyl azide **1q** (45.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2q** (33.8 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.58, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.67 (s, 1H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.14 (s, 2H), 6.89 (d, *J* = 8.4 Hz, 1H), 4.62 (t, *J* = 9.0 Hz, 2H), 3.24 (t, *J* = 8.4 Hz, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 162.4, 136.3, 128.4, 126.8, 123.1, 108.8, 72.1, 28.6; HRMS (ESI) Calcd for C₈H₉NO₃S [M+H]⁺ 200.0376, found 200.0376.



Compound **2r** was synthesized according to the General Procedure. The reaction of 5-chloro-2methoxybenzenesulfonyl azide **1r** (49.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2r** (38.0 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.45, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.89 (d, *J* = 2.4 Hz, 1H), 7.50 (dd, *J* = 3.0, 9.0 Hz, 1H), 7.00 (d, *J* = 9.0 Hz, 1H), 5.08 (s, 2H), 4.01 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 154.5, 134.0, 131.2, 128.3, 126.0, 113.6, 56.8; HRMS (ESI) Calcd for C₇H₈ClNO₃S [M+H]⁺ 221.9986, found 221.9985.



Compound $2s^4$ was synthesized according to the General Procedure. The reaction of 2,4,6-trimethylbenzenesulfonyl azide 1s (45.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 2s (25.1 mg, 63%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.18 (s, 2H), 6.99 (s, 2H), 2.55 (s, 6 H), 2.24 (s, 3H); ¹³C NMR (150 MHz, DMSO-d₆) δ 140.6, 138.3, 137.2, 131.4, 22.7, 20.4.



Compound **2t** was synthesized according to the General Procedure. The reaction of 4,4'oxydibenzenesulfonyl azide **1t** (76.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **2t** (56.4 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.6, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.87 (d, *J* = 7.8 Hz, 4H), 7.37 (s, 4H), 7.24 (d, *J* = 8.4 Hz, 4H); ¹³C NMR (150 MHz, DMSO-d₆) δ 158.4, 139.8, 128.4, 119.1; HRMS (ESI) Calcd for C₁₂H₁₂N₂O₅S₂ [M+H]⁺ 329.0260, found 329.0258.



Compound $4a^7$ was synthesized according to the General Procedure. The reaction of benzoyl azide 3a (29.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 4a (20.8 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/

petroleum ether =2:1). ¹**H NMR** (600 MHz, CDCl₃) δ 7.82 (d, *J* = 7.8 Hz, 2H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 2H), 6.27 (s, 2H); ¹³**C NMR** (150 MHz, CDCl₃) δ 169.5, 133.4, 132.0, 128.6, 127.3.



Compound $4b^7$ was synthesized according to the General Procedure. The reaction of 4-methylbenzoyl azide **3b** (32.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4b** (24.0 mg, 89%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.71 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 7.8 Hz, 2H), 6.21-5.81 (br, 2H), 2.40 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 169.4, 142.5, 130.5, 129.2, 127.4, 21.5.



Compound $4c^7$ was synthesized according to the General Procedure. The reaction of 4-methoxybenzoyl azide 3c (35.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered 4c (26.9 mg, 89%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.78 (d, J = 9.0 Hz, 2H), 6.94 (d, J = 8.4 Hz, 2H), 6.23-5.51 (br, 2H), 3.86 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 162.6, 129.3, 125.6, 113.8, 55.4.



Compound **4d** was synthesized according to the General Procedure. The reaction of methyl [1,1'biphenyl]-4-carbonyl azide **3d** (44.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4d** (34.7 mg, 88%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.02 (s, 1H), 7.97 (d, *J* = 7.2 Hz, 2H), 7.79-7.69 (m, 4H), 7.49 (t, *J* = 6.6 Hz, 2H), 7.43-7.35 (m, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 167.6, 142.8, 139.3, 133.2, 129.1, 128.2, 128.1, 127.0, 126.5; HRMS (ESI) Calcd for C₁₃H₁₁NO [M+H]⁺ 198.0913, found 198.0915.



Compound $4e^8$ was synthesized according to the General Procedure. The reaction of 4-fluorobenzoyl

azide **3e** (33.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4e** (22.0 mg, 79%) as a white solid after purification via silica gel column chromatography (Rf = 0.46, ethyl acetate/ petroleum ether =2:1)). ¹H NMR (600 MHz, CDCl₃) δ 7.83 (s, 2H), 7.13 (s, 2H), 6.19-5.66 (br, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 168.2, 165.9, 164.2, 129.8 (d, *J* = 9.0 Hz), 129.5 (d, *J* = 3.0 Hz), 115.7 (d, *J* = 22.5 Hz); ¹⁹F NMR (565 MHz, CDCl₃) δ -107.3.



Compound **4f**⁷ was synthesized according to the General Procedure. The reaction of 4-chlorobenzoyl azide **3f** (36.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4f** (26.7 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.46, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.04 (s, 1H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.46 (s, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 166.9, 136.2, 133.1, 129.5, 128.4.



Compound **4g** was synthesized according to the General Procedure. The reaction of methyl 4-(azidocarbonyl)benzoate **3g** (41.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4g** (30.4 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =1:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.15 (s, 1H), 8.05-7.95 (m, 4H), 7.57 (s, 1H), 3.87 (s, 3H); ¹³C NMR (150 MHz, DMSO-d₆) δ 167.1, 165.8, 138.5, 131.9, 129.1, 127.9, 52.4; HRMS (ESI) Calcd for C₉H₉NO₃ [M+H]⁺ 180.0655, found 234.0538.



Compound **4h**⁸ was synthesized according to the General Procedure. The reaction of 4-(trifluoromethyl)benzoyl azide **3h** (43.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4h** (30.2 mg, 80%) as a white solid after purification via silica gel column chromatography (Rf = 0.45, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.19 (s, 1H), 8.06 (d, *J* = 7.8 Hz, 2H), 7.83 (d, *J* = 7.8 Hz, 2H), 7.62 (s, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 166.8, 138.2, 131.3 (q, *J* = 31.5 Hz), 128.4, 124.1 (q, *J* = 271.5 Hz), 125.3 (q, *J* = 3.0 Hz); ¹⁹F NMR (565 MHz, DMSO-d₆) δ -61.4.



Compound **4i** was synthesized according to the General Procedure. The reaction of methyl 4-(trifluoromethoxy)benzoyl azide **3i** (46.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4i** (33.6 mg, 82%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.07 (s, 1H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.49 (s, 1H), 7.44 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (150 MHz, DMSO-d₆) δ 166.7, 150.4, 133.5, 129.9, 120.9, 120.6, 119.2; ¹⁹F NMR (565 MHz, CDCl₃) δ -56.7; HRMS (ESI) Calcd for C₈H₆F₃NO₂ [M+H]⁺ 206.0423, found 206.0428.



Compound **4j** was synthesized according to the General Procedure. The reaction of 3-methylbenzoyl azide **3j** (32.2 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4j** (23.2 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.64 (s, 1H), 7.58 (d, *J* = 6.6 Hz, 1H), 7.34-7.28 (m, 2H), 6.47-6.23 (br, 2H), 2.38 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 170.0, 138.4, 133.3, 132.6, 128.4, 128.0, 124.3, 21.2; HRMS (ESI) Calcd for C₈H₉NO [M+H]⁺ 136.0757, found 136.0757.



Compound **4k** was synthesized according to the General Procedure. The reaction of 3-bromobenzoyl azide **3k** (45.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4k** (33.8 mg, 85%) as a white solid after purification via silica gel column chromatography (Rf = 0.42, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.09 (s, 1H), 8.05 (t, *J* = 1.8 Hz, 1H), 7.90-7.86 (m, 1H), 7.75-7.69 (m, 1H), 7.43 (s, 1H), 7.42 (t, *J* = 7.8 Hz, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 166.4, 136.6, 134.1, 130.6, 130.3, 126.6, 121.7; HRMS (ESI) Calcd for C₇H₆BrNO[M+H]⁺ 199.9706, found 199.9708.



Compound 417 was synthesized according to the General Procedure. The reaction of methyl 2-naphthoyl

azide **31** (39.4 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mol, 1.5 equiv.) and 9-mesityl-10methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mmol%) in CH₃CN (2.0 mL) delivered **41** (27.7 mg, 81%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.49 (s, 1H), 8.14 (s, 1H), 8.02-7.95 (m, 4H), 7.63-7.56 (m, 2H), 7.47 (s, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 168.0, 134.2, 132.2, 131.7, 128.9, 127.9, 127.8, 127.7, 127.6, 126.7, 124.5; HRMS (ESI) Calcd for C₁₁H₉NO [M+H]⁺ 172.0757, found 172.0755.



Compound **4m** was synthesized according to the General Procedure. The reaction of 3,5-dimethylbenzoyl azide **3m** (35.0 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4m** (23.6 mg, 86%) as a white solid after purification via silica gel column chromatography (Rf = 0.5, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, CDCl₃) δ 7.42 (s, 2H), 7.15 (s, 1H), 6.26-5.96 (br, 2H), 2.35 (s, 6H); ¹³C NMR (150 MHz, CDCl₃) δ 170.0, 138.3, 133.6, 133.3, 125.1, 21.2; HRMS (ESI) Calcd for C₉H₁₁NO [M+H]⁺ 150.0913, found 150.0910.



Compound **4n** was synthesized according to the General Procedure. The reaction of 3-bromo-4chlorobenzoyl azide **3n** (51.8 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **4n** (39.1 mg, 84%) as a white solid after purification via silica gel column chromatography (Rf = 0.45, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 8.23 (d, *J* = 1.8 Hz, 1H), 8.14 (s, 1H), 7.88 (dd, *J* = 1.8, 8.4 Hz, 1H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.59 (s, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 165.6, 136.2, 134.7, 132.8, 130.6, 128.4, 121.6; HRMS (ESI) Calcd for C₇H₃BrCINO [M+H]⁺ 233.9316, found 233.9317.

Compound 40^7 was synthesized according to the General Procedure. The reaction of thiophene-2carbonyl azidez **30** (30.6 mg, 0.200 mmol, 1.0 equiv.), Bn₃N (86.1 mg, 0.300 mmol, 1.5 equiv.) and 9mesityl-10-methylacridinium perchlorate (4.1 mg, 0.010 mmol, 5 mol%) in CH₃CN (2.0 mL) delivered **40** (20.8 mg, 82%) as a white solid after purification via silica gel column chromatography (Rf = 0.48, ethyl acetate/ petroleum ether =2:1). ¹H NMR (600 MHz, DMSO-d₆) δ 7.95 (s, 1H), 7.76-7.70 (m, 2H), 7.37 (s, 1H), 7.12 (t, *J* = 4.2 Hz, 1H); ¹³C NMR (150 MHz, DMSO-d₆) δ 163.0, 140.4, 131.1, 128.8, 128.0.

2.7. References

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. NMR Spectra for Compounds













100 90 fl (ppm)





0 -100 -110 -120 -130 -140 f1 (ppm) -10 -20 -30 -40 -50 -60 -70 -90 -80 -150 -160 -170 -180 -190 -200





2g ¹H NMR, DMSO-d₆, 600 MHz



--2.500































 $$\rm 2s$ $\rm ^{13}C$ NMR, DMSO-d_6, 150 MHz















10 0 -10 -20 -30 -40 -50 -50 -70 -80 -90 -110 -120 -120 -140 -150 -160 -170 -180 -190 -200 -210 f1 (pps)











4h ¹H NMR, DMSO-d₆, 600 MHz



-2.500







10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -150 -150 -190 -200 -210 f1 (ppn)



fl (ppm) 130 120



0.0



10 0 100 90 fl (ppm)



4I ¹H NMR, DMSO-d₆, 600 MHz





100 90 fl (ppm) . .





190 180 160 150 100 90 fl (ppm)



100 90 fl (ppm)







¹³C NMR DMSO-d₆, 150 MHz



100 90 fl (ppm)