

Supplementary material

Microwave-assisted synthesis and fluorescent properties of 4-phenyl-1,8-naphthalimide

Frank Betancourt, James Helmckay, Hongbin Yan*

Department of Chemistry, Brock University, 1812 Sir Isaac Brock Way, St. Catharines, L2S
3A1, ON Canada

Table of contents

Figure S1. ^1H NMR spectrum of compound **2** in DMSO- d_6 , 7.0-12.5 ppm.

Figure S2. ^1H NMR spectrum of compound **2** in DMSO- d_6 , 0-13 ppm.

Figure S3. ^{13}C NMR spectrum of compound **2** in DMSO- d_6 , 120-170 ppm.

Figure S4. ^{13}C NMR spectrum of compound **2** in DMSO- d_6 , 0-220 ppm.

Figure S5. ^1H NMR spectrum of compound **3** in DMSO- d_6 , 0-13 ppm.

Figure S6. COSY NMR spectrum of compound **3** in DMSO- d_6 .

Figure S7. ^{13}C NMR spectrum of compound **3** in DMSO- d_6 , 120-170 ppm.

Figure S8. ^{13}C NMR spectrum of compound **3** in DMSO- d_6 , 0-220 ppm.

Figure S9. HSQC NMR spectrum of compound **3** in DMSO- d_6 .

Figure S10. HMBC NMR spectrum of compound **3** in DMSO- d_6 .

Figure S11. Microwave reaction parameters used for the amidation reaction.

Figure S12. Microwave conditions used for the Suzuki coupling reactions.

Table S1. Preparation of 0.01 μM ss-DNA 21-mer (5'-d(CTTTAAGAAGGAGATATACCA)-3').

Table S2. Preparation of 0.01 μM ds-DNA 21-mer.

Table S3. Preparation of 0.01 μM d(AT)₁₅.

Table S4. Preparation of 0.01 μM d(CG)₉

Table S5. Preparation of solutions of dye **3** containing NaCl of different concentrations.

Table S6. Preparation of solutions of dye **3** containing MgCl₂ of different concentrations.

Table S7. Preparation of solutions of dye **3** containing KCl of different concentrations.

Table S8. Preparation of solutions of dye **3** containing CaCl₂ of different concentrations.

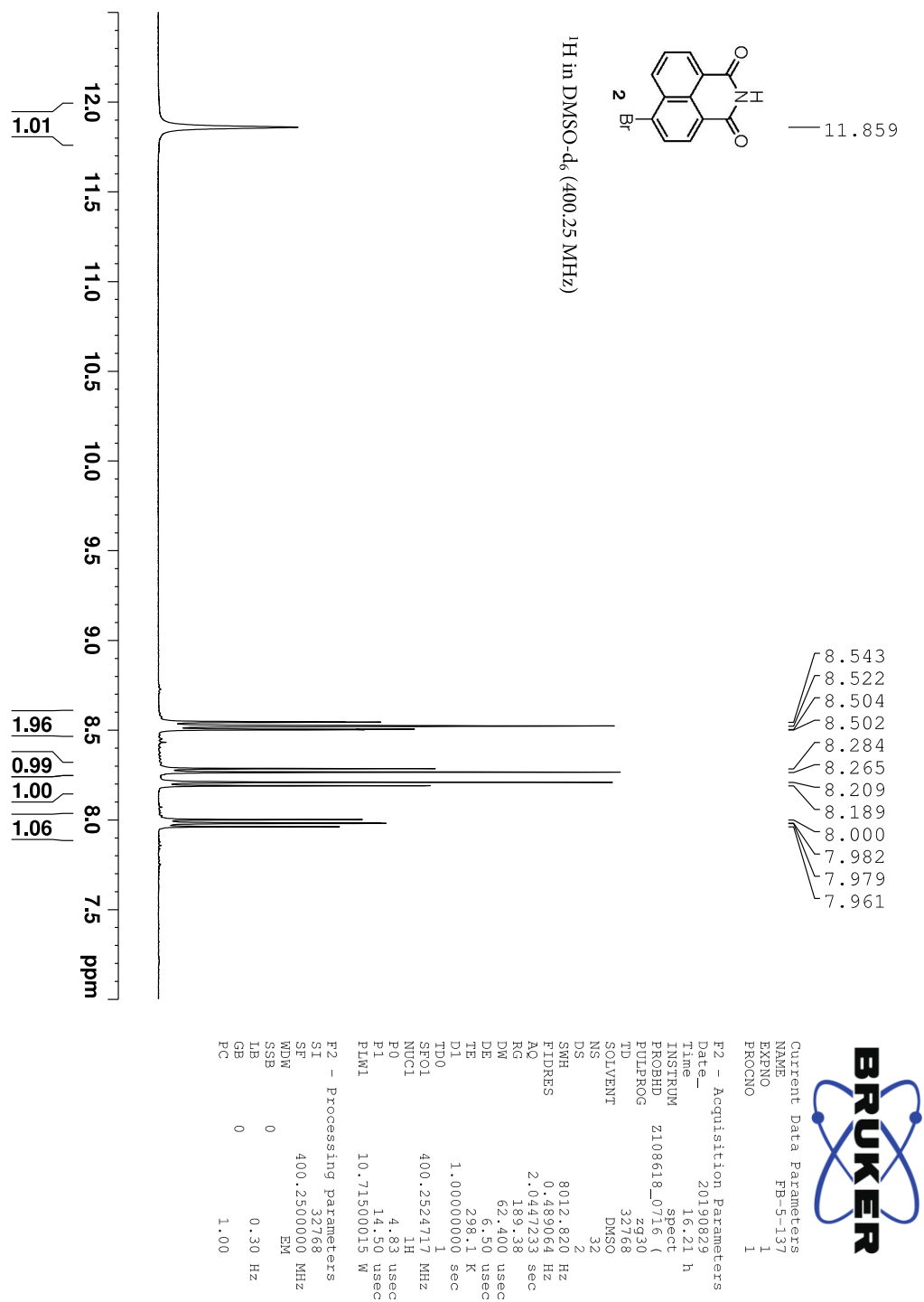


Figure S1. ¹H NMR spectrum of compound **2** in DMSO-d₆, 7.0-12.5 ppm.

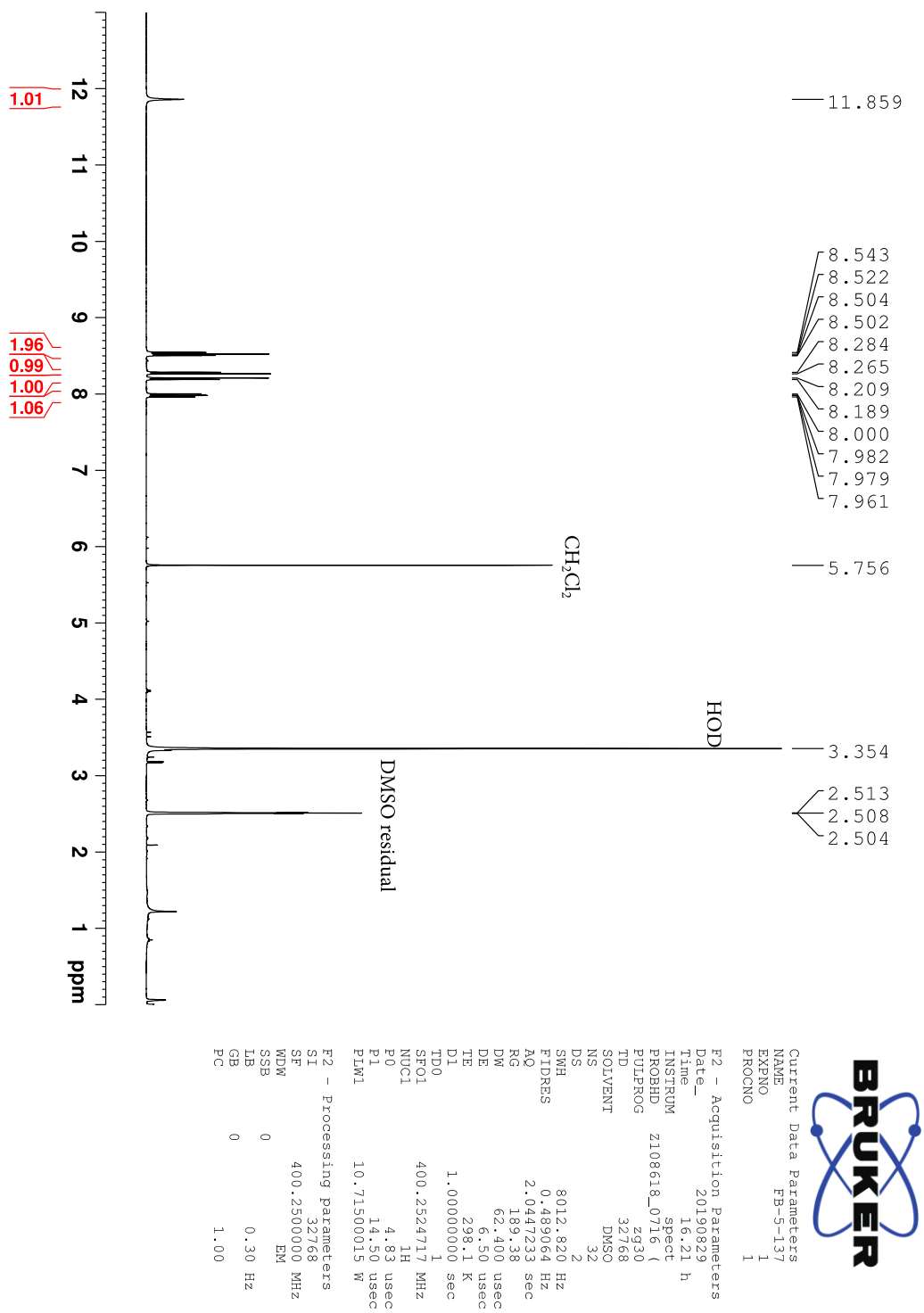


Figure S2. ¹H NMR spectrum of compound **2** in DMSO-d₆, 0-13 ppm.

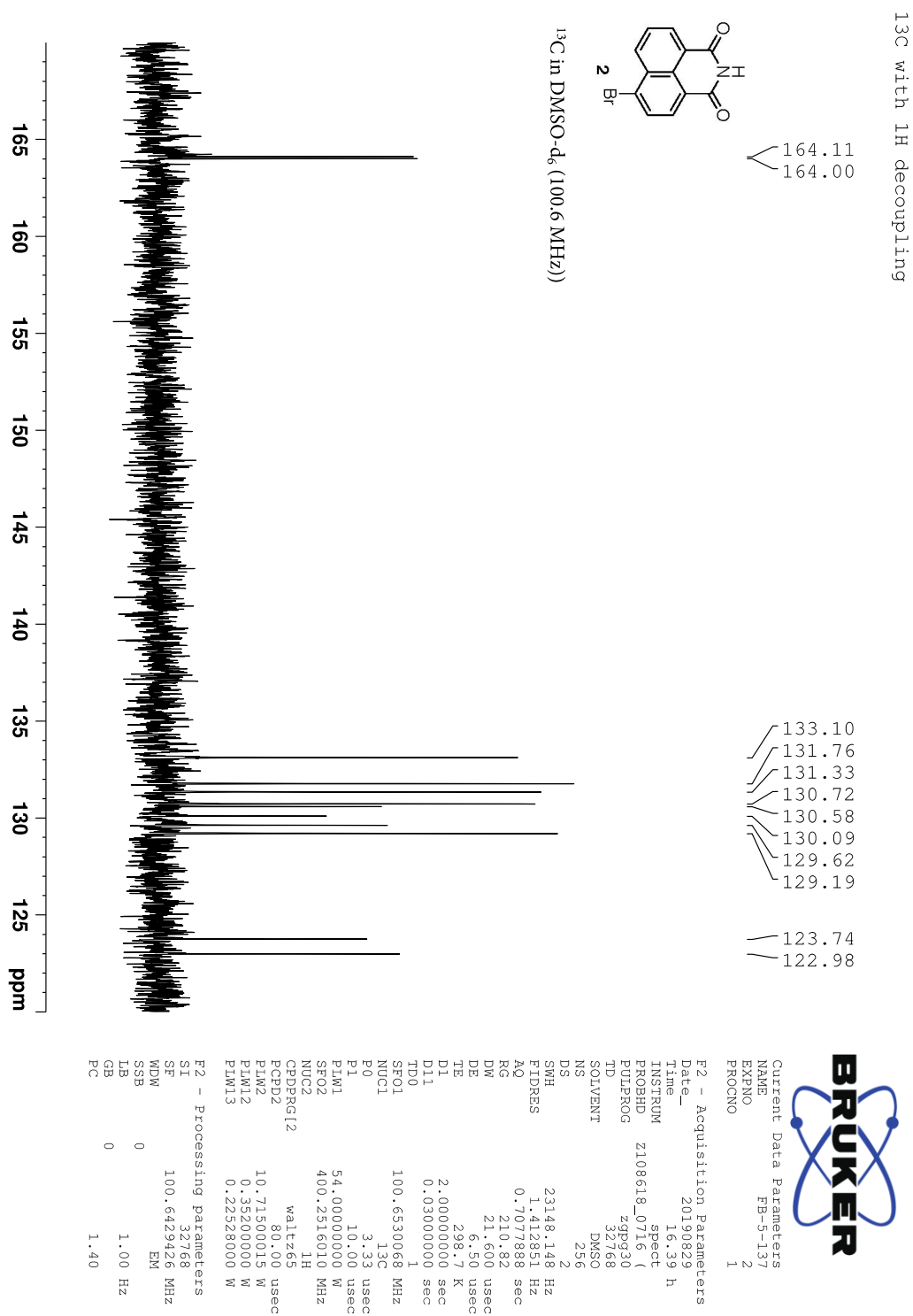
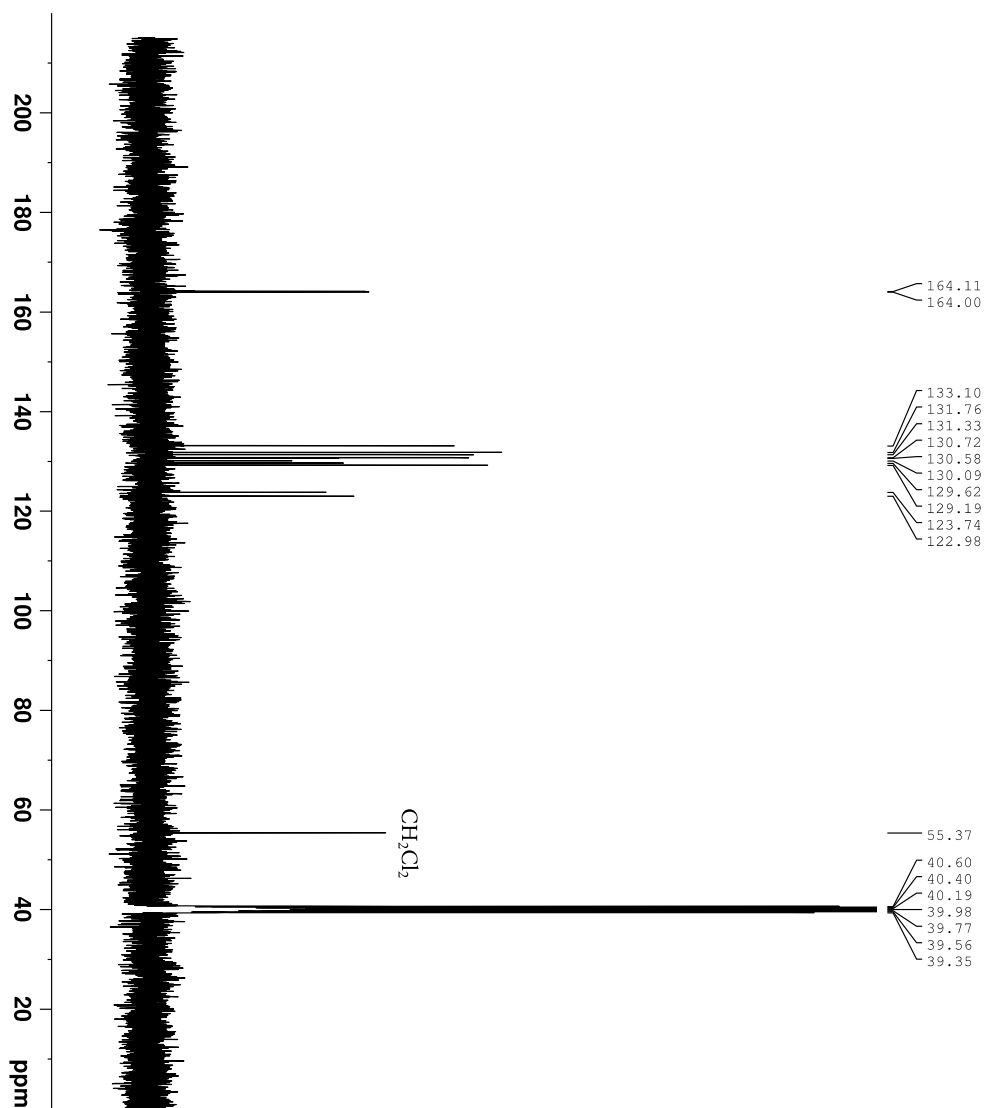


Figure S3. ¹³C NMR spectrum of compound **2** in DMSO-d₆, 120-170 ppm.



Current Data Parameters
NAME FB-5-137
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190829
Time 16:39 h
INSTRUM spect
PROBHD Z108618_0716 (zgp930
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 256
DS 2
SWH 23148.148 Hz
FIDRES 1.412851 Hz
AQ 0.7077888 sec
RG 210.82
DM 21.600 usec
DE 6.50 usec
TE 298.7 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
SF01 100.6530068 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 54.0000000 W
SFO2 400.2516010 MHz
NUC2 1H
CPOBPG12 waltz65
PCPD2 80.00 usec
PLW2 10.71500015 W
PLW12 0.3520000 W
PLW13 0.22528000 W

F2 - Processing Parameters
SI 32768
SF 100.6429426 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S4. ¹³C NMR spectrum of compound 2 in DMSO-d₆, 0-220 ppm.

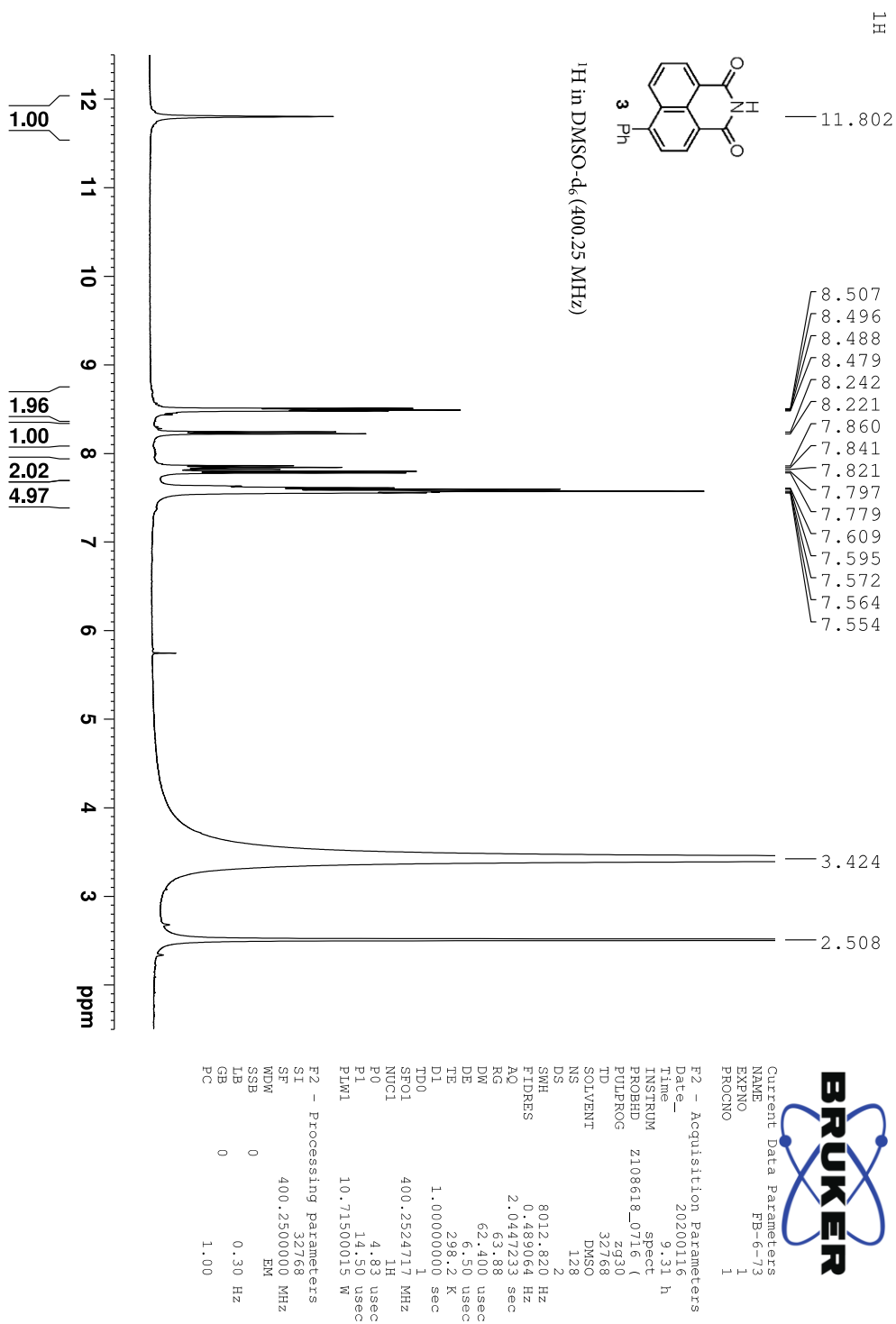


Figure S5. ¹H NMR spectrum of compound **3** in DMSO-d₆, 0-13 ppm.

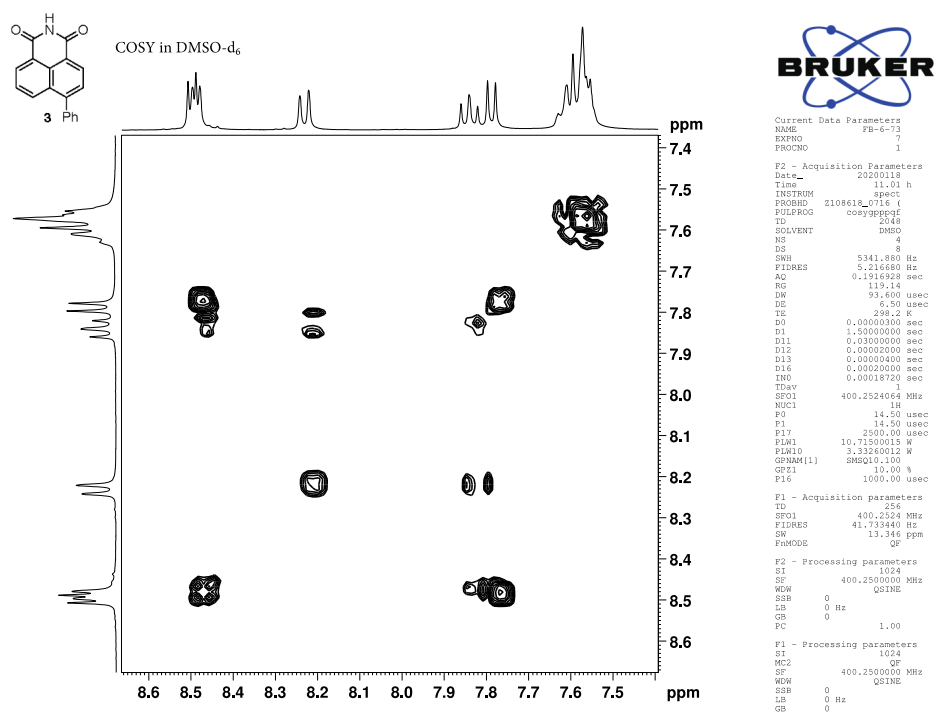


Figure S6. COSY NMR spectrum of compound **3** in DMSO-d₆.

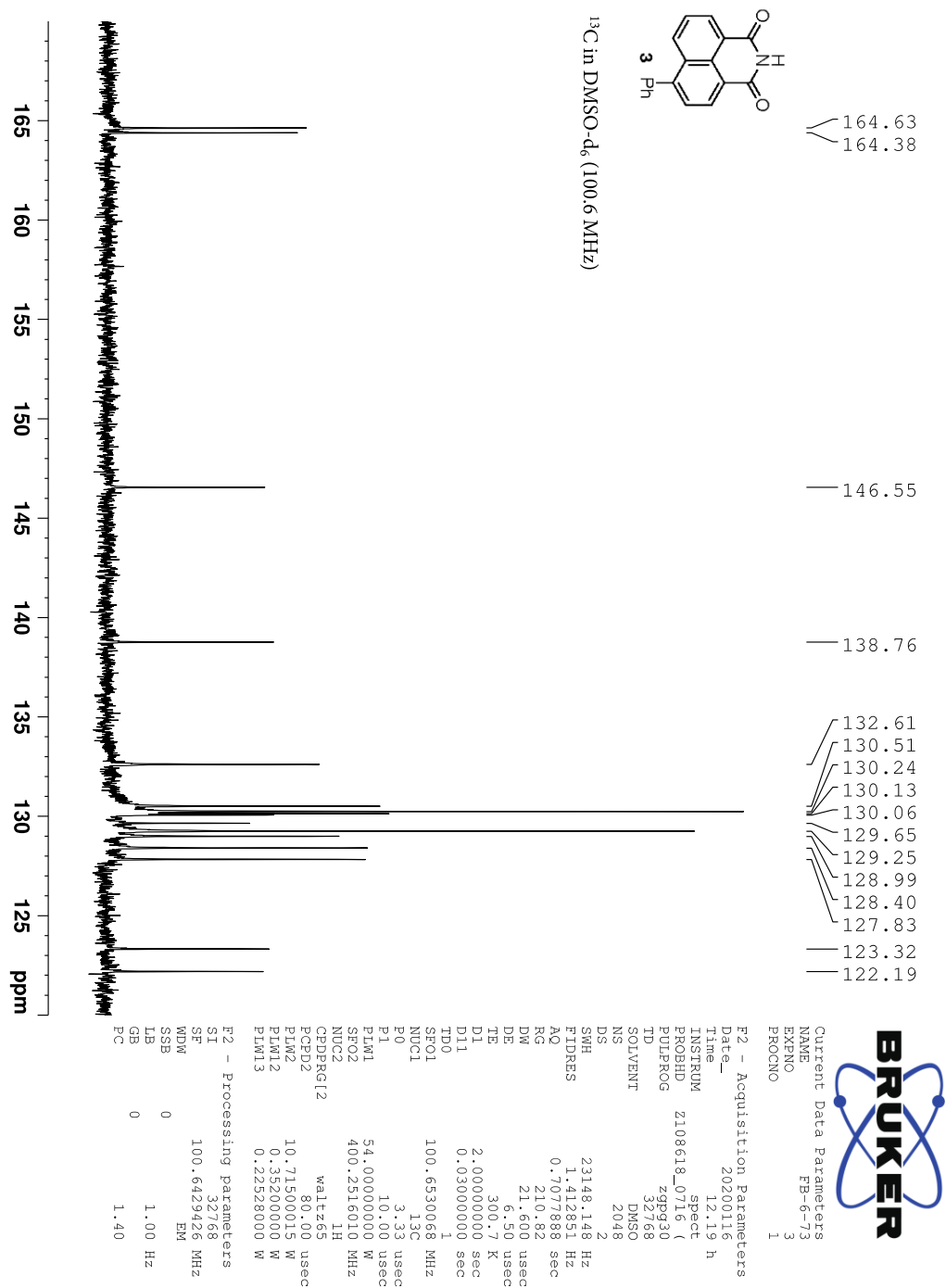
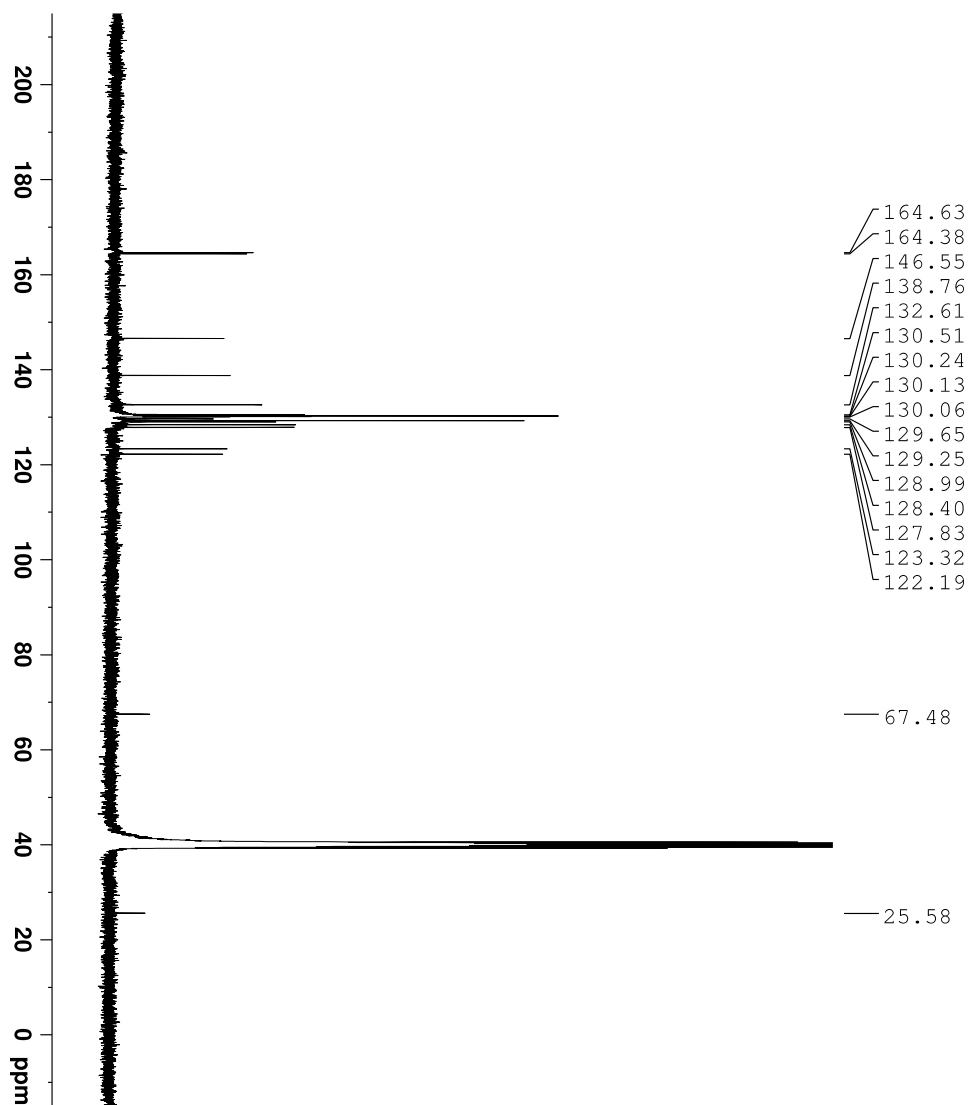


Figure S7. ¹³C NMR spectrum of compound **3** in DMSO-d₆, 120-170 ppm.



BRUKER

Current Data Parameters		F2 - Acquisition Parameters	
NAME	FB-6-73	Date_	20200116
EXPNO	3	Time	12.19 h
PROCNO	1	INSTRUM	spect
		PROBHD	Z108618-0716 (
		PULPROG	zgpg30
		TD	32768
		SOLVENT	DMSO
		NS	2048
		DS	2
		SWH	23148.148 Hz
		FIDRES	1.412851 Hz
		AQ	0.7077888 sec
		RG	210.82
		DM	21.600 usec
		DE	6.50 usec
		TE	300.7 K
		D1	2.00000000 sec
		D11	0.03000000 sec
		TD0	1
		SFO1	100.6530068 MHz
		NUC1	¹³ C
		P0	3.33 usec
		P1	10.00 usec
		PLM1	54.00000000 W
		SFO2	400.2516010 MHz
		NUC2	¹ H
		CPDPRG12	waltz65
		PCPD2	80.00 usec
		PLM2	10.71500015 W
		PLM12	0.35200000 W
		PLM13	0.22528000 W
F2 - Processing parameters			
		SI	32768
		SF	100.6429426 MHz
		WDW	EM
		SSB	0
		LB	1.00 Hz
		GB	0
		PC	1.40

Figure S8. ¹³C NMR spectrum of compound **3** in DMSO-d₆, 0-220 ppm.

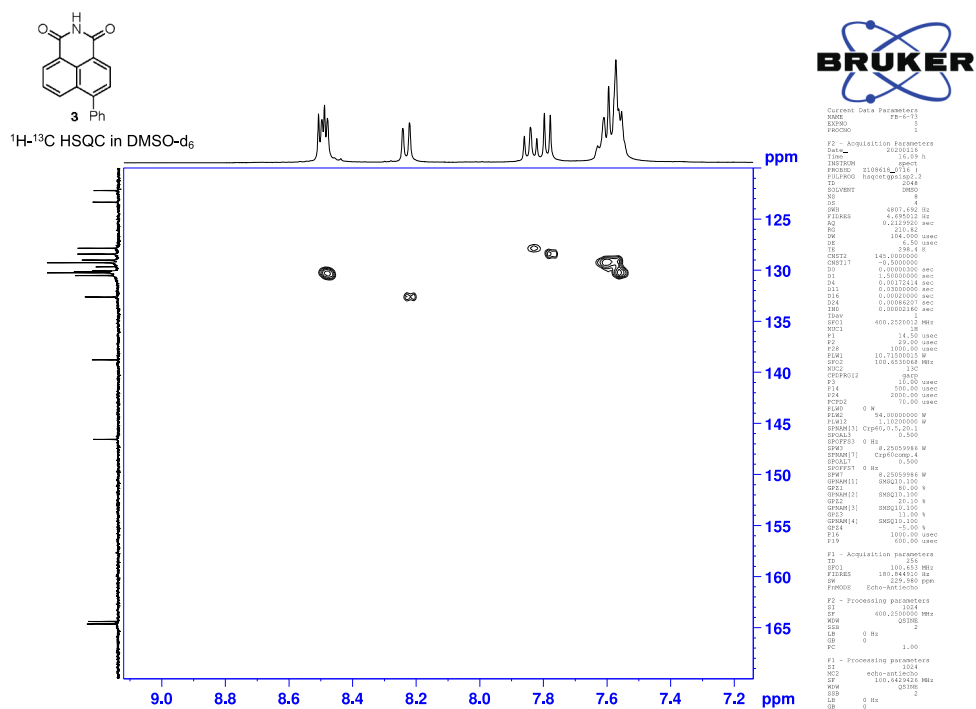


Figure S9. HSQC NMR spectrum of compound **3** in DMSO-d₆.

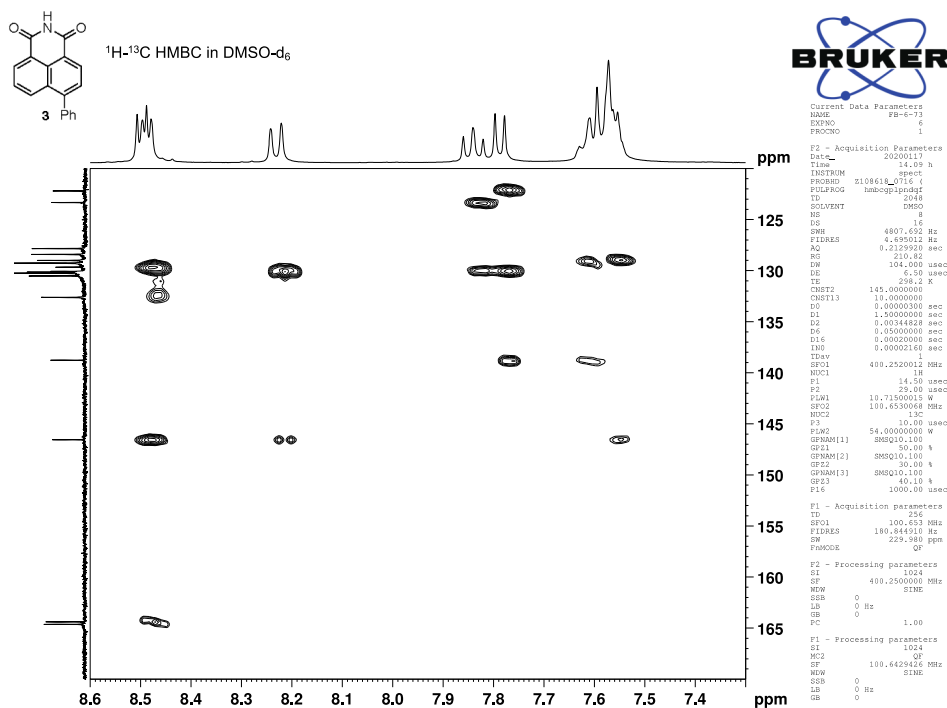
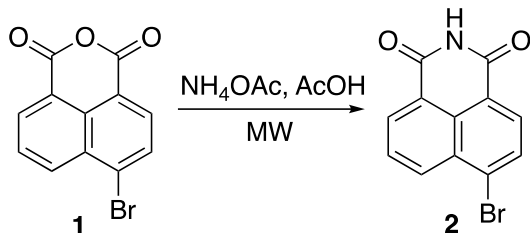


Figure S10. HMBC NMR spectrum of compound **3** in DMSO-d₆.



Reaction

Suz fb 5 159 10/18/2019 8:25:23 AM User: Tony Yan 10mL Vessel Snap Cap

Method Parameters

Name: Suz fb 5 159

Prestirring(mm:ss): 00:00

Type: Dynamic

Stage	Temp(C)	Time(mm:ss)	Pressure(Psi)	Power(W)	PowerMAX	Stirring
1	70	30:00	60	100	No	High

Graphs

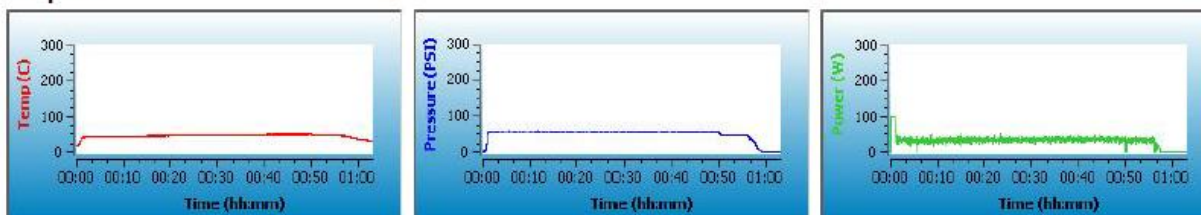
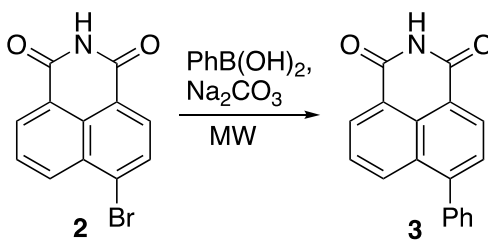


Figure S11. Microwave reaction parameters used for the amidation reaction.



Type: Dynamic

Stage	Temp(C)	Time(mm:ss)	Pressure(Psi)	Power(W)	PowerMAX	Stirring
1	70	30:00	60	100	No	High

Graphs

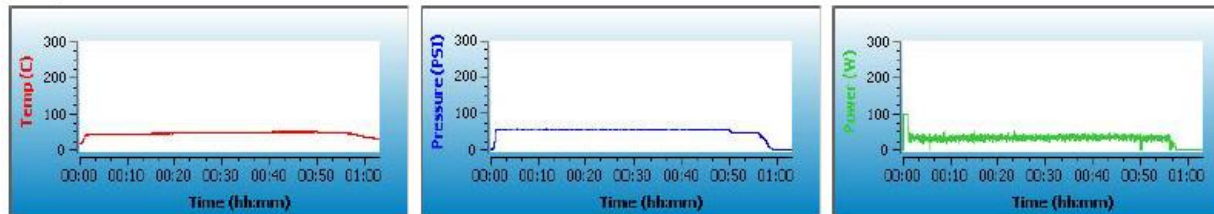


Figure S12. Microwave conditions used for the Suzuki coupling reactions.

Table S1. Preparation of 0.01 μM ss-DNA 21-mer (5'-d(CTTTAAGAAGGAGATATACCA)-3').

Mol. equiv. of dye	Dye stock solution (μL)	ssDNA stock solution (μL)	Buffer (μL)	1%(v/v) DMSO (μL)
0.1	21	10	872	97
0.5	105	10	796	89
1.0	210	10	701	79
1.5	315	10	607	68
2.0	420	10	512	58
2.5	525	10	418	47
3.0	630	10	323	37
3.5	735	10	229	26

Table S2. Preparation of 0.01 μM ds-DNA 21-mer.

Mol. equiv. of dye	Dye stock solution (μL)	ssDNA stock solution (μL)	Buffer (μL)	1%(v/v) DMSO (μL)
0.1	21	10	872	97
0.5	105	10	796	89
1.0	210	10	701	79
1.5	315	10	607	68
2.0	420	10	512	58
2.5	525	10	418	47
3.0	630	10	323	37
3.5	735	10	229	26

Table S3. Preparation of 0.01 μM d(CG)₉.

Mol. equiv. of dye	Dye stock solution (μL)	ssDNA stock solution (μL)	Buffer (μL)	1%(v/v) DMSO (μL)
0.1	18	10	874	98
0.2	36	10	858	96
0.3	54	10	842	95
0.4	72	10	826	93
0.5	90	10	809	91
0.7	126	10	777	88
0.9	162	10	745	84
1	180	10	729	82
1.5	270	10	648	73
2	360	10	567	64
2.5	450	10	485	55
3	540	10	404	46

Table S4. Preparation of 0.01 μM d(AT)₁₅.

Mol. equiv. of dye	Dye stock solution (μL)	ssDNA stock solution (μL)	Buffer (μL)	1%(v/v) DMSO (μL)
0.1	30	10	863	97
0.2	60	10	836	94
0.3	90	10	809	91
0.4	120	10	782	88
0.5	150	10	755	85
0.7	210	10	701	79
0.9	270	10	647	73
1	300	10	620	70
1.5	450	10	485	55
2	600	10	350	40
2.5	750	10	215	25
3	900	10	80	10

Table S5. Preparation of solutions of dye **3** containing NaCl of different concentrations.

Volume (μL)	100 mM	1 M	2 M	3 M	4 M
6 M NaCl	17	167	334	500	667
TRIS buffer (50 mM TRIS, 0.1 M EDTA, pH 7.5)	200	200	200	200	200
Dye 3 (10 μM in 10% (v/v) aqueous DMSO)	100	100	100	100	100
milliQ water	683	533	366	200	33

Table S6. Preparation of solutions of dye **3** containing MgCl₂ of different concentrations.

	100 mM	1 M	2 M	3 M	4 M
5.7 M MgCl ₂	17	175	350	527	700
TRIS buffer (50 mM TRIS, 0.1 M EDTA, pH 7.5)	200	200	200	200	200
Dye 3 (10 μM in 10% (v/v) aqueous DMSO)	100	100	100	100	100
milliQ water	683	525	350	173	0

Table S7. Preparation of solutions of dye **3** containing KCl of different concentrations.

	100 mM	1 M	2 M	3 M	4 M
4.76 M KCl	21	210	420	630	840
TRIS buffer (50 mM TRIS, 0.1 M EDTA, pH 7.5)	200	200	200	200	200
Dye 3 (10 μ M in 10% (v/v) aqueous DMSO)	100	100	100	100	100
milliQ water	679	490	280	70	0

Table S8. Preparation of solutions of dye **3** containing CaCl₂ of different concentrations.

	100 mM	1 M	2 M	3 M	4 M
6.7 M CaCl ₂	15	150	299	448	596
TRIS buffer (50 mM TRIS, 0.1 M EDTA, pH 7.5)	200	200	200	200	200
Dye 3 (10 μ M in 10% (v/v) aqueous DMSO)	100	100	100	100	100
milliQ water	685	550	401	252	104