BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Halogen-bonding-induced diverse aggregation of 4,5-diiodo-1,2,3-triazolium salts with different anions

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Crystallographic data, computational details, copies of ^{1}H and ^{13}C NMR spectra

Contents

1. Crystallography data

Table S1 Crystal data and structure refinement for 2-I.

CCDC NO.	1923173
Identification code	mo_XXY2017120402_0m_a
Empirical formula	$C_{20}H_{22}I_3N_3$
Formula weight	685.10
Temperature/K	200.0
Crystal system	tetragonal
Space group	$P\ \bar{4}2_1c$
a/Å	23.9419(10)
b/Å	23.9419(10)
c/Å	10.1217(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	5801.9(5)
Z	8
ρ_{calc}g/cm^3	1.569
μ/mm^{-1}	3.239
F(000)	2576.0
Crystal size/mm^3	0.38 × 0.31 × 0.23
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.37 to 58.272
Index ranges	-32 ≤ h ≤ 24, -29 ≤ k ≤ 24, -11 ≤ l ≤ 13
Reflections collected	20762
Independent reflections	7767 [R_{int} = 0.0533, R_{sigma} = 0.0845]
Data/restraints/parameters	7767/9/217
Goodness-of-fit on F^2	1.120
Final R indexes [I>=2σ (I)]	R_1 = 0.0613, wR_2 = 0.1361
Final R indexes [all data]	R_1 = 0.1114, wR_2 = 0.1531
Largest diff. peak/hole / e Å^{-3}	1.38/-0.93
Flack parameter	-0.03(2)

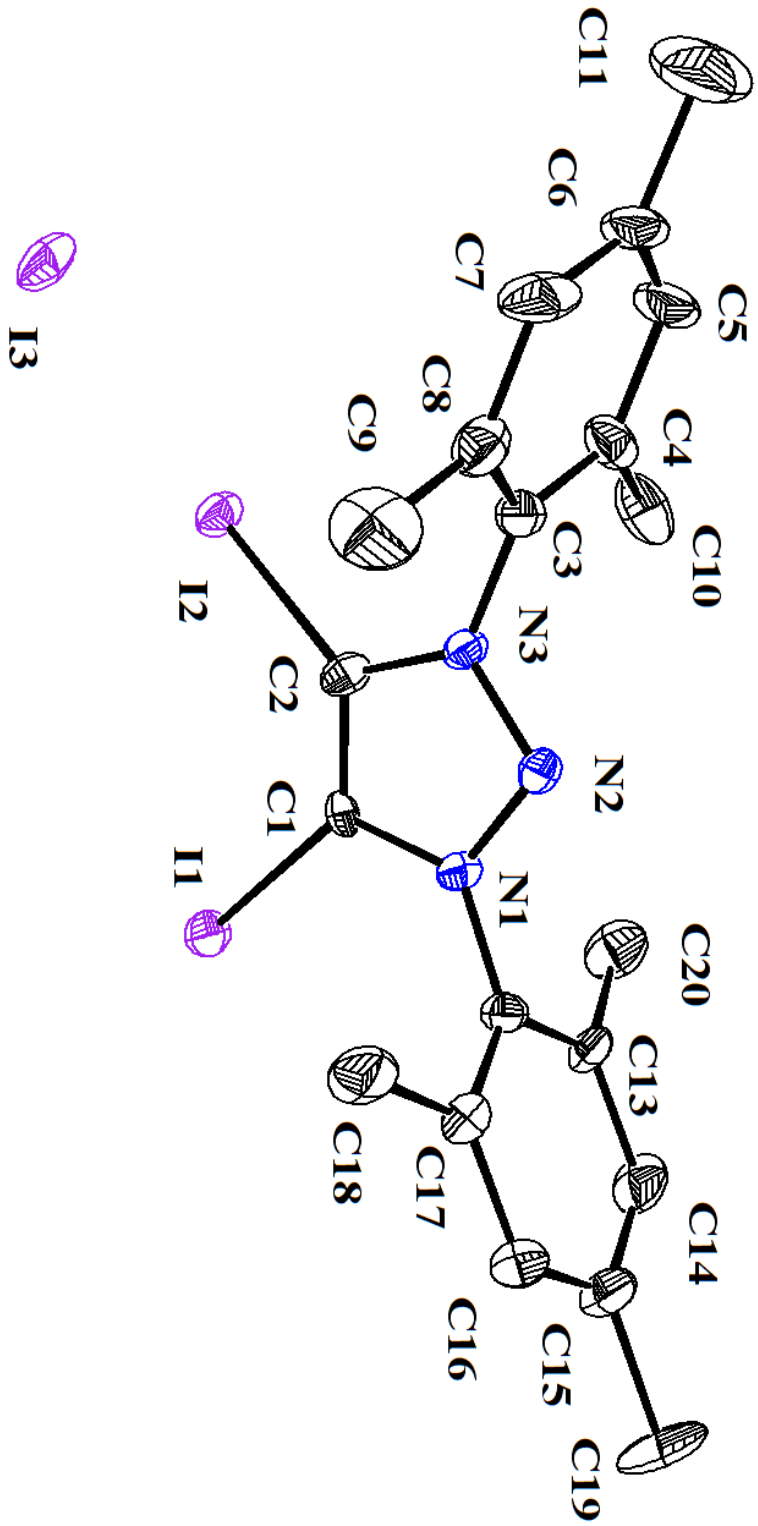


Figure S1 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-I**. Hydrogen atoms are omitted for clarity.

Table S2 Crystal data and structure refinement for 2-Br.

CCDC NO.	1923174
Identification code	mo_xxy201809201_0m
Empirical formula	$C_{20}H_{22}BrI_2N_3$
Formula weight	638.11
Temperature/K	200.0
Crystal system	tetragonal
Space group	$I\bar{4}$
a/Å	23.5982(13)
b/Å	23.5982(13)
c/Å	9.8455(6)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	5482.7(7)
Z	8
ρ_{calc}g/cm^3	1.546
μ/mm^{-1}	3.759
F(000)	2432.0
Crystal size/mm^3	0.37 × 0.36 × 0.29
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.482 to 52.978
Index ranges	-33 ≤ h ≤ 31, -34 ≤ k ≤ 34, -14 ≤ l ≤ 14
Reflections collected	20093
Independent reflections	8644 [R_{int} = 0.0536, R_{sigma} = 0.0824]
Data/restraints/parameters	8644/6/218
Goodness-of-fit on F^2	1.074
Final R indexes [I>=2σ (I)]	R_1 = 0.0542, wR_2 = 0.1425
Final R indexes [all data]	R_1 = 0.0790, wR_2 = 0.1553
Largest diff. peak/hole / e Å^{-3}	1.90/-1.27
Flack parameter	0.057(13)

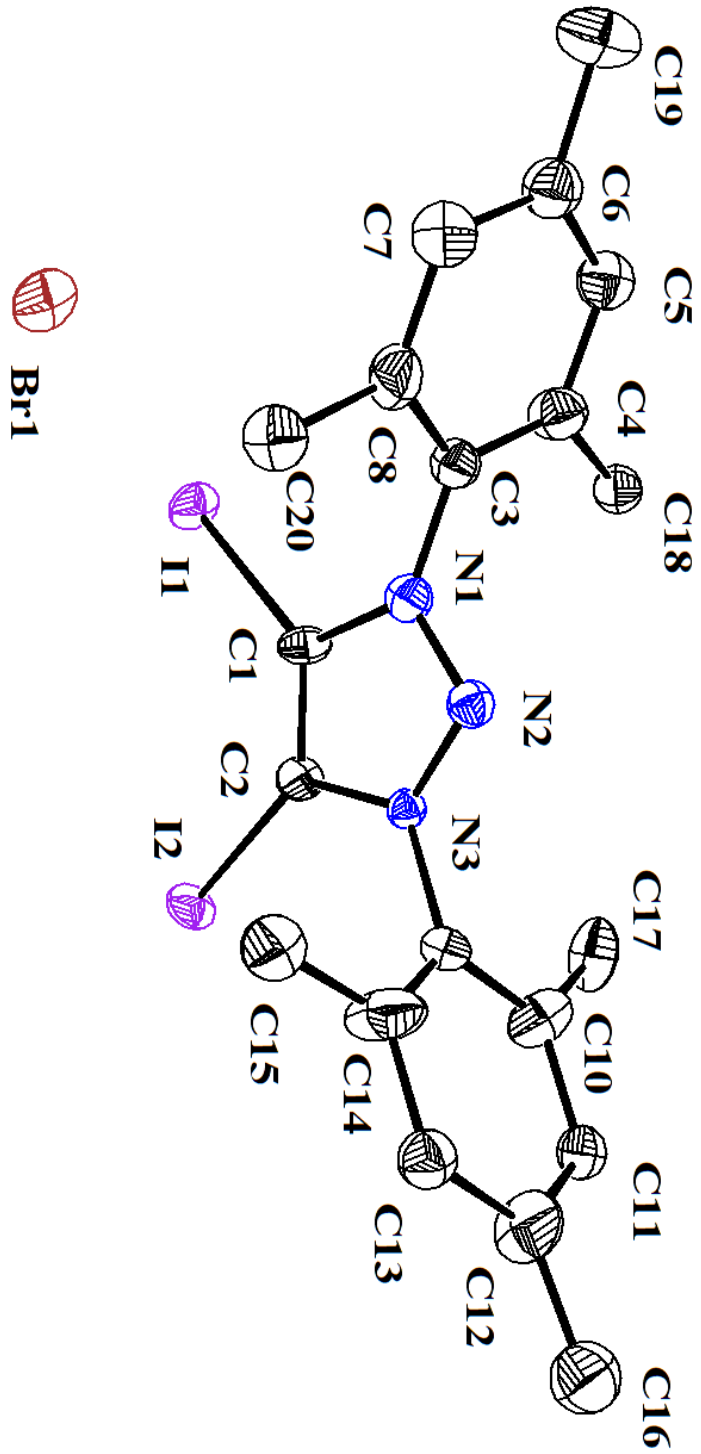


Figure S2 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-Br**. Hydrogen atoms are omitted for clarity.

Table S3 Crystal data and structure refinement for 2-Cl.

CCDC NO.	1923175
Identification code	mo_XXY318_0m_a
Empirical formula	$C_{20}H_{22}ClI_2N_3$
Formula weight	593.65
Temperature/K	200.0
Crystal system	monoclinic
Space group	*C*2
a/Å	32.770(4)
b/Å	9.7269(13)
c/Å	23.215(3)
α/°	90
β/°	134.483(3)
γ/°	90
Volume/Å^3	5279.3(12)
Z	8
ρ_{calc}g/cm^3	1.494
μ/mm^{-1}	2.491
F(000)	2288.0
Crystal size/mm^3	0.16 × 0.15 × 0.15
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.536 to 55.164
Index ranges	-35 ≤ h ≤ 42, -11 ≤ k ≤ 12, -30 ≤ l ≤ 27
Reflections collected	20706
Independent reflections	9877 [R_{int} = 0.0557, R_{sigma} = 0.0904]
Data/restraints/parameters	9877/301/430
Goodness-of-fit on F^2	1.054
Final R indexes [I>=2σ (I)]	R_1 = 0.0591, wR_2 = 0.1166
Final R indexes [all data]	R_1 = 0.0893, wR_2 = 0.1272
Largest diff. peak/hole / e Å^{-3}	1.62/-1.72
Flack parameter	0.009(19)

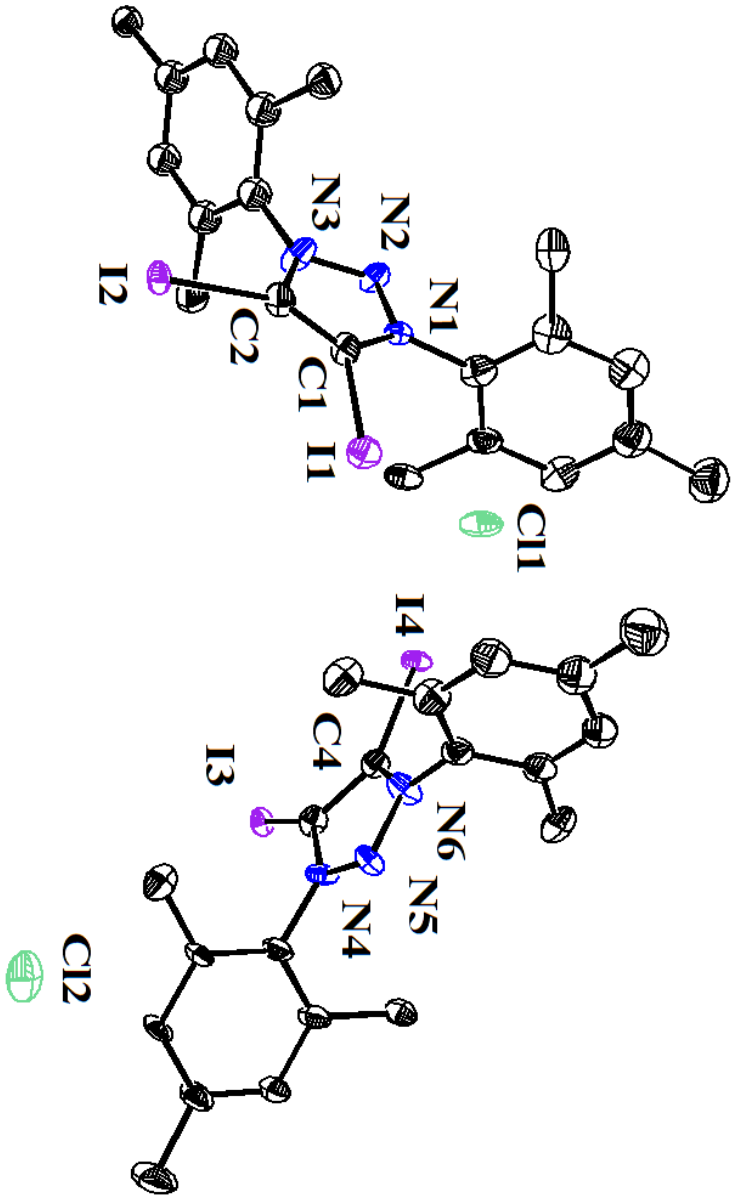


Figure S3 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-Cl.** Hydrogen atoms are omitted for clarity.

Table S4 Crystal data and structure refinement for 2-OAc.

CCDC NO.	1923176
Identification code	mo_XXY201808311_0ma_a
Empirical formula	$C_{23}H_{27}Cl_2I_2N_3O_2$
Formula weight	702.17
Temperature/K	150.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	11.9179(10)
b/Å	27.124(2)
c/Å	8.6997(7)
α/°	90
β/°	107.515(3)
γ/°	90
Volume/Å^3	2681.9(4)
Z	4
ρ_{calc}g/cm^3	1.739
μ/mm^{-1}	2.568
F(000)	1368.0
Crystal size/mm^3	0.46 × 0.35 × 0.31
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.676 to 61.094
Index ranges	$-17 \le h \le 12$, $-38 \le k \le 38$, $-12 \le l \le 12$
Reflections collected	37189
Independent reflections	8206 [R_{int} = 0.0316, R_{sigma} = 0.0255]
Data/restraints/parameters	8206/0/296
Goodness-of-fit on F^2	1.117
Final R indexes [I>=2σ (I)]	R_1 = 0.0258, wR_2 = 0.0563
Final R indexes [all data]	R_1 = 0.0321, wR_2 = 0.0582
Largest diff. peak/hole / e Å^{-3}	1.16/-0.96

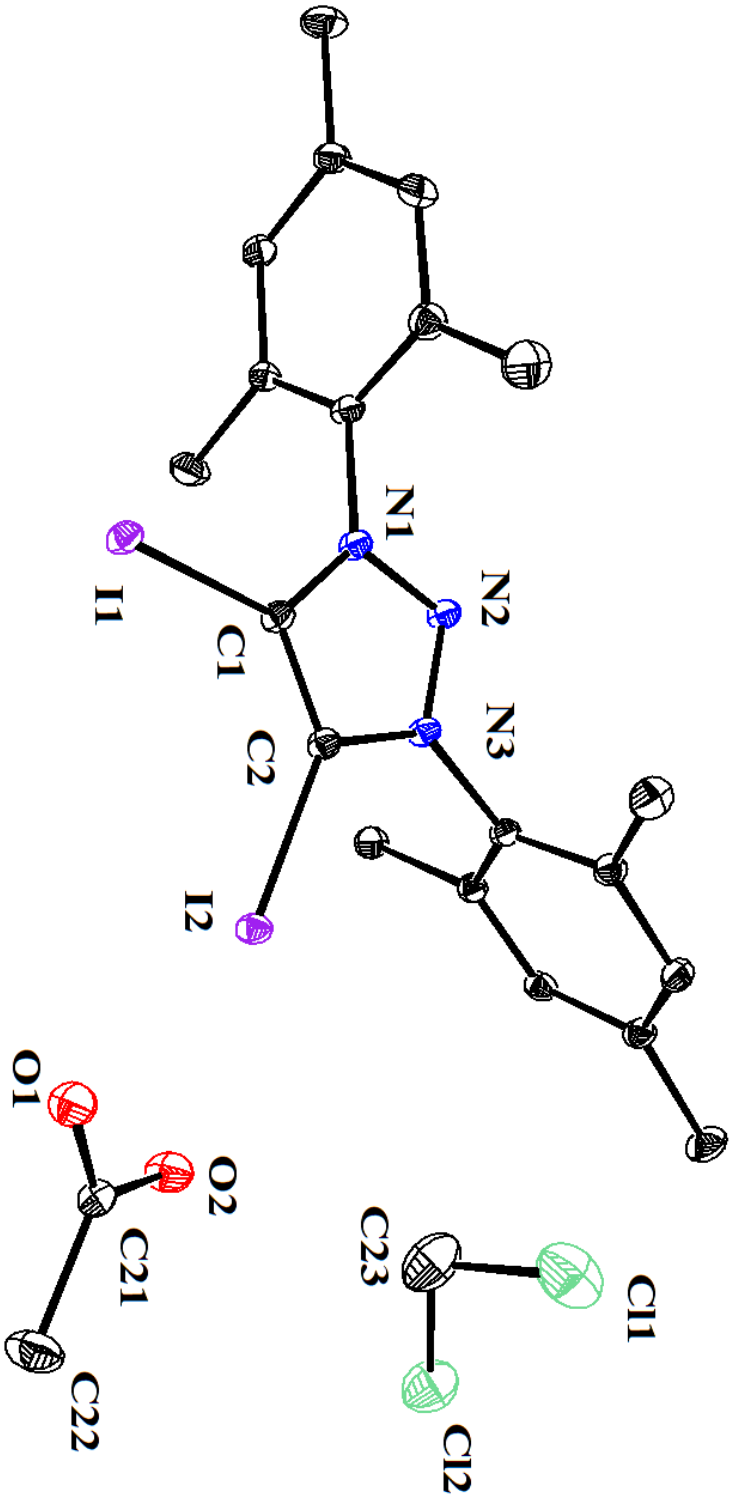


Figure S4 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-OAc.** Hydrogen atoms are omitted for clarity.

Table S5 Crystal data and structure refinement for 2-TFA.

CCDC NO.	1923177
Identification code	mo_XXY4251_0m_a
Empirical formula	$C_{22}H_{22}F_3I_2N_3O_2$
Formula weight	671.22
Temperature/K	150.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	22.3708(19)
b/Å	11.2189(12)
c/Å	22.842(2)
α/°	90
β/°	117.810(3)
γ/°	90
Volume/Å^3	5070.7(9)
Z	8
ρ_{calc}g/cm^3	1.758
μ/mm^{-1}	2.525
F(000)	2592.0
Crystal size/mm^3	0.23 × 0.19 × 0.15
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.036 to 55.072
Index ranges	$-29 \le h \le 26$, $-14 \le k \le 14$, $-29 \le l \le 29$
Reflections collected	49462
Independent reflections	11611 [R_{int} = 0.0535, R_{sigma} = 0.0501]
Data/restraints/parameters	11611/630/597
Goodness-of-fit on F^2	1.132
Final R indexes [I>=2σ (I)]	R_1 = 0.0914, wR_2 = 0.2130
Final R indexes [all data]	R_1 = 0.1111, wR_2 = 0.2216
Largest diff. peak/hole / e Å^{-3}	3.58/-1.87

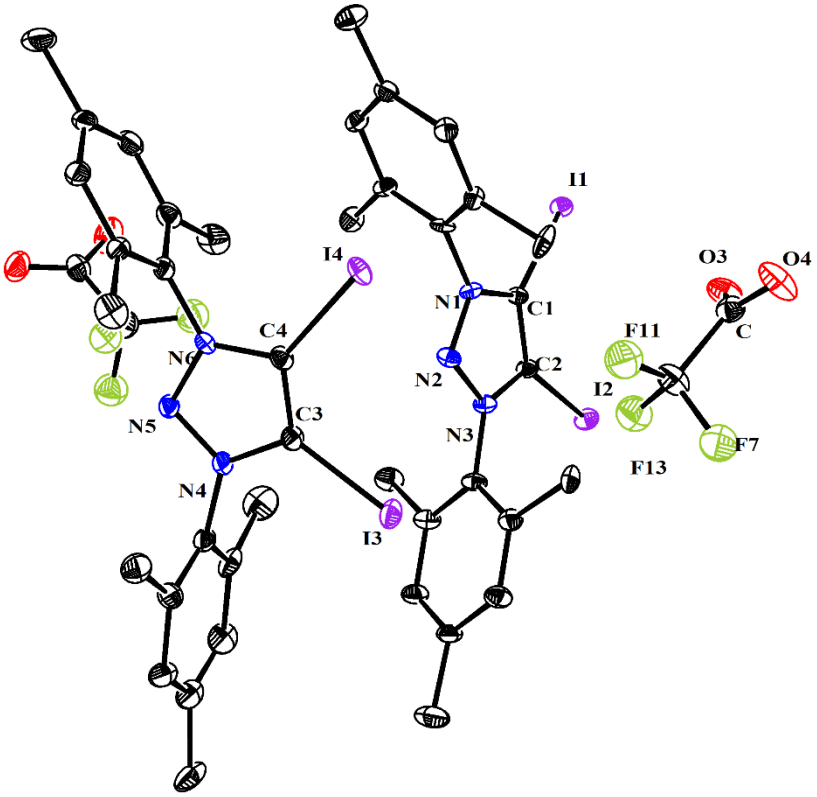


Figure S5 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-TFA.** Hydrogen atoms are omitted for clarity.

Table S6 Crystal data and structure refinement for 2-BF_4.

CCDC NO.	1923178
Identification code	mo_XXY2017111702_0m_a
Empirical formula	$C_{20}H_{22}BF_4I_2N_3$
Formula weight	645.01
Temperature/K	297.85
Crystal system	triclinic
Space group	$P\ \bar{1}$
a/Å	15.734(3)
b/Å	16.150(4)
c/Å	18.906(4)
α/°	109.625(6)
β/°	90.918(6)
γ/°	118.792(5)
Volume/Å^3	3872.4(15)
Z	6
ρ_{calc}g/cm^3	1.660
μ/mm^{-1}	2.475
F(000)	1860.0
Crystal size/mm^3	0.26 × 0.23 × 0.21
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.498 to 49.998
Index ranges	$-18 \le h \le 18$, $-17 \le k \le 19$, $-22 \le l \le 21$
Reflections collected	40817
Independent reflections	13582 [R_{int} = 0.0987, R_{sigma} = 0.1191]
Data/restraints/parameters	13582/79/829
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0921, wR_2 = 0.2475
Final R indexes [all data]	R_1 = 0.1692, wR_2 = 0.2991
Largest diff. peak/hole / e Å^{-3}	1.71/-1.19

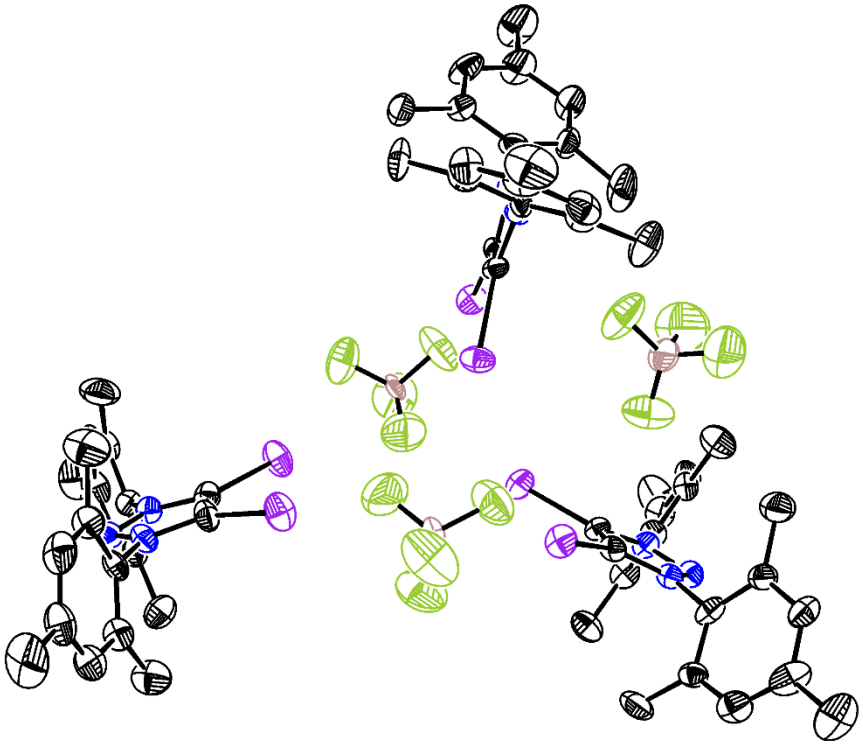


Figure S6 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-BF_4.** Hydrogen atoms are omitted for clarity.

Table S7 Crystal data and structure refinement for 2-I·1.5I_2.

CCDC NO.	1923179
Identification code	mo_XXY18922_0m_a
Empirical formula	$C_{40}H_{44}I_{12}N_6$
Formula weight	2131.61
Temperature/K	150.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	16.791(6)
b/Å	28.467(11)
c/Å	12.617(5)
α/°	90
β/°	107.753(15)
γ/°	90
Volume/Å^3	5744(4)
Z	4
ρ_{calc}g/cm^3	2.465
μ/mm^{-1}	6.502
F(000)	3848.0
Crystal size/mm^3	0.45 × 0.39 × 0.35
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.436 to 55.084
Index ranges	$-21 \le h \le 21$, $-36 \le k \le 36$, $-16 \le l \le 16$
Reflections collected	95836
Independent reflections	13122 [R_{int} = 0.0547, R_{sigma} = 0.0322]
Data/restraints/parameters	13122/0/555
Goodness-of-fit on F^2	1.088
Final R indexes [I>=2σ (I)]	R_1 = 0.0310, wR_2 = 0.0645
Final R indexes [all data]	R_1 = 0.0394, wR_2 = 0.0673
Largest diff. peak/hole / e Å^{-3}	2.14/-2.36

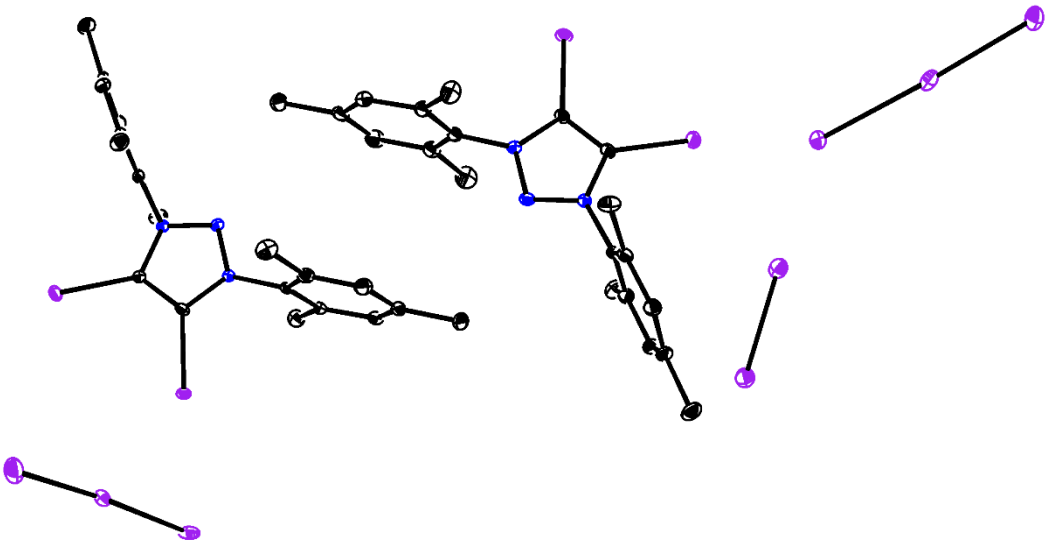


Figure S7 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-I·1.5I$_2$**. Hydrogen atoms are omitted for clarity.

Table S8 Crystal data and structure refinement for 2-I·3.5I_2.

CCDC NO.	1923180
Identification code	mo_XXY18934_0m_a
Empirical formula	$C_{40}H_{44}I_{20}N_6$
Formula weight	3146.81
Temperature/K	149.98
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.2054(5)
b/Å	28.5826(15)
c/Å	20.7771(11)
α/°	90
β/°	99.232(2)
γ/°	90
Volume/$Å^3$	7154.5(6)
Z	4
$\rho_{calc}g/cm^3$	2.921
μ/mm^{-1}	8.677
F(000)	5544.0
Crystal size/mm^3	0.53 × 0.43 × 0.42
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.422 to 55.058
Index ranges	$-15 \le h \le 15$, $-37 \le k \le 37$, $-26 \le l \le 22$
Reflections collected	56334
Independent reflections	16405 [R_{int} = 0.0579, R_{sigma} = 0.0614]
Data/restraints/parameters	16405/0/626
Goodness-of-fit on F^2	1.012
Final R indexes [I>=2σ (I)]	R_1 = 0.0430, wR_2 = 0.0900
Final R indexes [all data]	R_1 = 0.0673, wR_2 = 0.0997
Largest diff. peak/hole / e $Å^{-3}$	2.02/-2.68

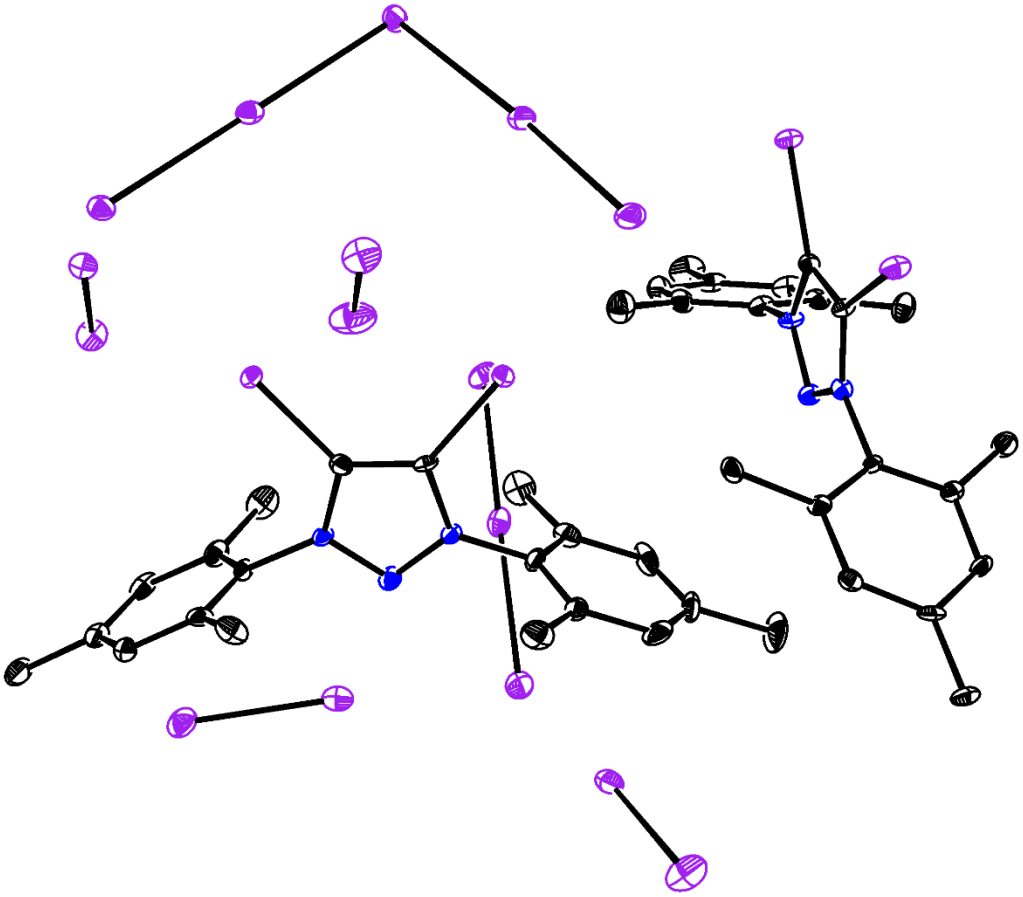


Figure S8 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-I·3.5I$_2$**. Hydrogen atoms are omitted for clarity.

Table S9 Crystal data and structure refinement for 2-BF_4.0.5 bpy

CCDC NO.	1923181
Identification code	mo_XXY1891_0m_a
Empirical formula	$C_{54}H_{60}B_2Cl_8F_8I_4N_8$
Formula weight	1785.92
Temperature/K	149.97
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	18.1509(13)
b/Å	18.7643(13)
c/Å	20.4941(16)
α/°	90
β/°	92.369(2)
γ/°	90
Volume/Å^3	6974.1(9)
Z	4
ρ_{calc}g/cm^3	1.701
μ/mm^{-1}	2.156
F(000)	3480.0
Crystal size/mm^3	0.42 × 0.35 × 0.31
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.488 to 55.088
Index ranges	$-23 \le h \le 23$, $-24 \le k \le 24$, $-25 \le l \le 26$
Reflections collected	67328
Independent reflections	15951 [R_{int} = 0.0439, R_{sigma} = 0.0404]
Data/restraints/parameters	15951/54/809
Goodness-of-fit on F^2	1.152
Final R indexes [I>=2σ (I)]	R_1 = 0.0965, wR_2 = 0.2020
Final R indexes [all data]	R_1 = 0.1121, wR_2 = 0.2088
Largest diff. peak/hole / e Å^{-3}	3.01/-2.19

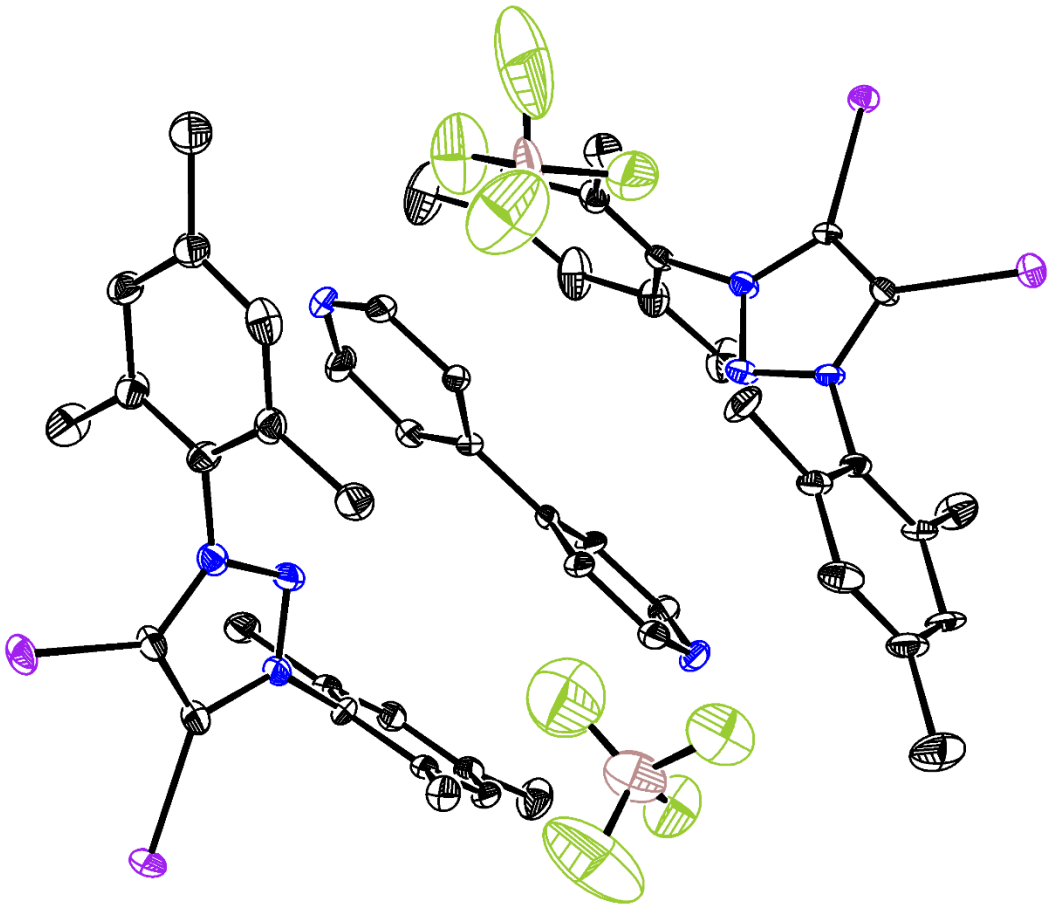


Figure S9 Thermal ellipsoid plot (ellipsoid contour 30%) for **2-BF$_4$.0.5 bpy**. Hydrogen atoms and solvents CH_2Cl_2 are omitted for clarity.

2. Computational details

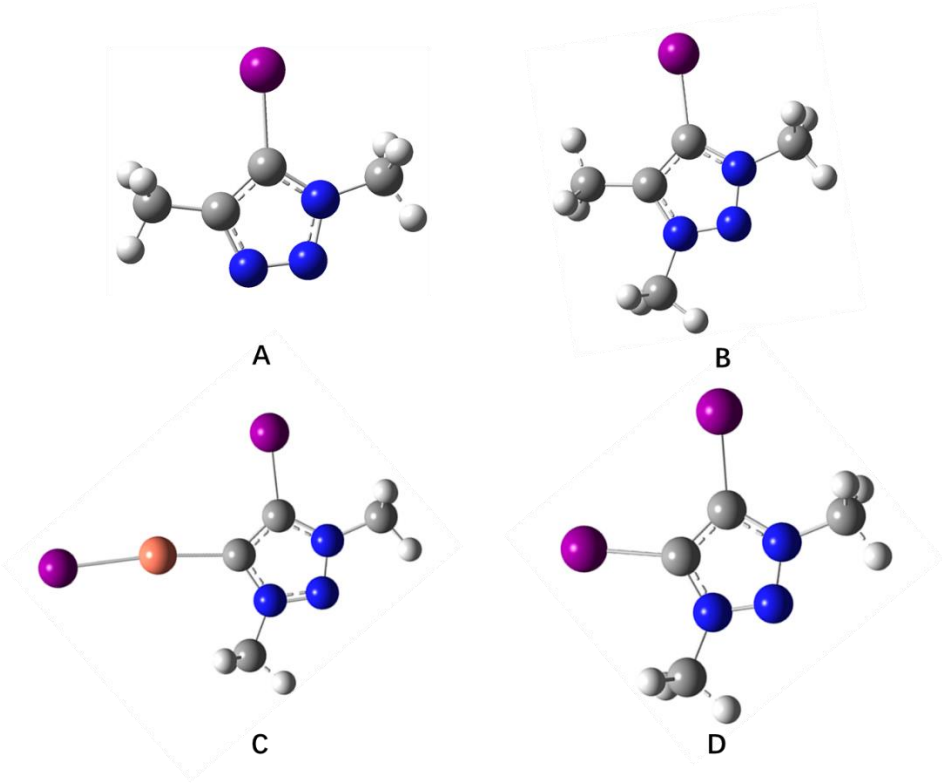


Figure 1. The optimized structures of **A**, **B**, **C-CuI**, and **D** at m06-2x level with basis of 6-311G (d, p) for H, C, N atoms, LanL2DZ for Cu atom and MWB46 for I atoms.

Table 1. Thermodynamic data of **A**, **B**, **C**, and **D**.

Compouds	E(Thermal) KCal/Mol	CV Cal/Mol-Kelvin	S Cal/Mol-Kelvin
A	71.378	27.800	92.641
B	98.483	34.422	102.835
C-CuI	75.209	36.805	118.913
D	74.261	32.711	103.736

Cartesian coordinates

A

C	-1.49248200	1.07009900	-0.00003200
C	-0.59459600	0.02853000	-0.00024400
N	-2.64391500	-0.76996200	0.00035500
N	-1.34920800	-1.09362300	0.00001000
N	-2.73438400	0.52153200	0.00032400
I	1.48869000	0.04139300	0.00006000
C	-0.93881800	-2.48398100	-0.00034300
H	-1.85134100	-3.07477000	-0.00027500
H	-0.35060500	-2.70242100	-0.89199200

H	-0.35023400	-2.70275200	0.89098300
C	-1.25172600	2.54017200	-0.00030300
H	-0.69314600	2.85070700	0.88534600
H	-0.68406400	2.84873900	-0.88082300
H	-2.21288600	3.05213100	-0.00572500

B

C	-1.29520300	-0.80273300	0.00006900
C	-0.25223900	0.09754800	0.00011200
N	-2.13437700	1.25782700	-0.00006800
N	-0.83296500	1.32830900	0.00006400
I	1.79771500	-0.23137600	-0.00005300
C	-0.17901100	2.64044100	0.00018000
H	0.43511000	2.72768000	-0.89460100
H	-0.96706200	3.38811800	0.00051900
H	0.43553600	2.72730600	0.89470200
N	-2.40717000	-0.01808200	-0.00006800
C	-3.80350800	-0.46097100	-0.00020500
H	-3.98790500	-1.05293000	-0.89520700
H	-3.98804800	-1.05317100	0.89463000
H	-4.42458200	0.43047000	-0.00016800
C	-1.33011000	-2.28708100	0.00026000
H	-1.84330900	-2.66391400	-0.88685500
H	-0.31353100	-2.67657500	0.00016300
H	-1.84307500	-2.66364300	0.88763100

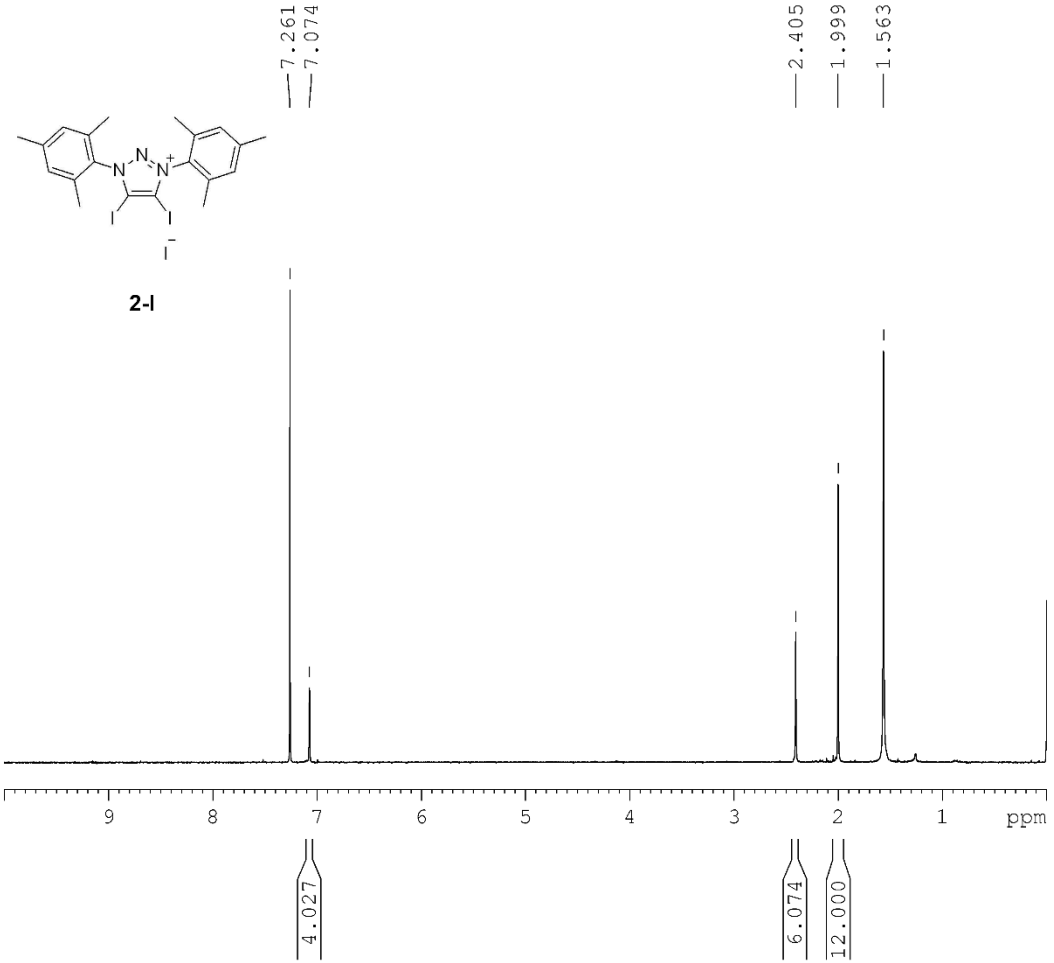
C-CuI

C	0.66821400	0.96996100	0.00119800
C	1.93910500	0.42249500	0.00000700
N	2.24901900	2.60245500	-0.00304700
N	2.84462600	1.43946100	-0.00264100
I	2.48592900	-1.58579900	0.00044900
C	4.30092700	1.37421600	-0.00698300
H	4.66891500	2.39616300	-0.00730500
H	4.63873300	0.84591500	0.88337600
H	4.63360400	0.84719500	-0.90005300
N	0.97211000	2.30357000	-0.00106700
C	-0.00292600	3.39164600	0.00560300
H	-0.65851200	3.27570700	-0.85534400
H	-0.58930000	3.33094500	0.92068400
H	0.53926100	4.33227700	-0.04419200
Cu	-1.22083700	0.24249300	0.00292300
I	-3.65046900	-0.36568800	-0.00108300

D

C	-0.68945400	0.63199900	-0.00004300
N	-0.00012900	2.73617400	0.00004200
N	-1.03851700	1.94633600	-0.00001800
I	-2.03188800	-0.94604300	0.00003500
C	-2.38641000	2.52520400	-0.00016500
H	-2.26742300	3.60506300	-0.00008000
H	-2.91035000	2.19406000	-0.89518900
H	-2.91056500	2.19398100	0.89470100
N	1.03839900	1.94648600	0.00003100
C	2.38620000	2.52556100	0.00012700
H	2.91020900	2.19441600	0.89510700
H	2.91035500	2.19445700	-0.89477500
H	2.26709800	3.60542500	0.00014400
C	0.68955400	0.63211600	0.00001400
I	2.03194600	-0.94602600	-0.00003300

3. Copies of ^{1}H and ^{13}C NMR Spectra



2-I

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PROCNO                  1
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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
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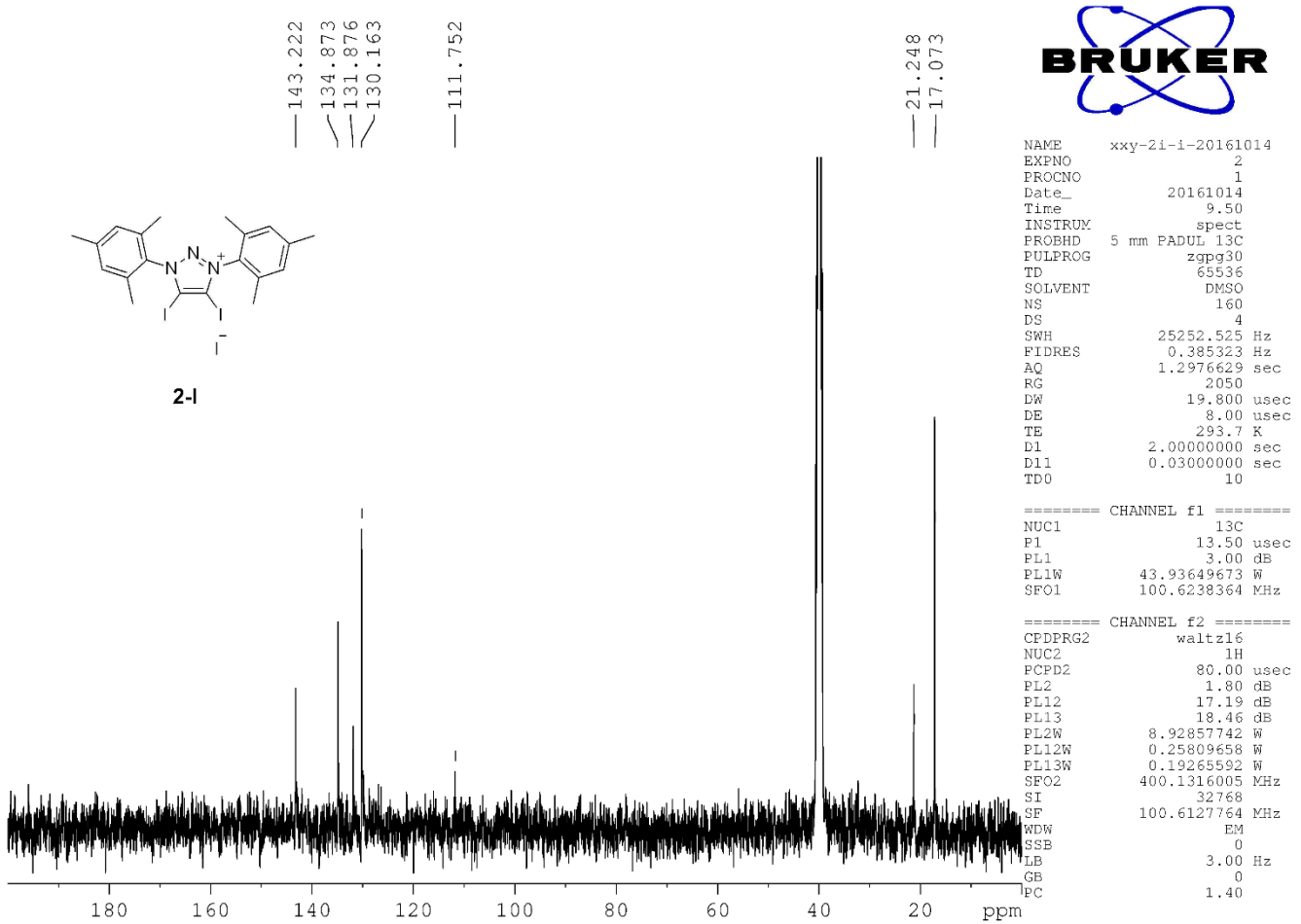
FIDRES           0.195125 Hz
AQ               2.5625076 sec
RG                    575
DW                 78.200 usec
DE                  6.50 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.10 usec
PL1                 1.80 dB
PL1W          8.92857742 W
SFO1         400.1326008 MHz
SI                  32768
SF           400.1300096 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

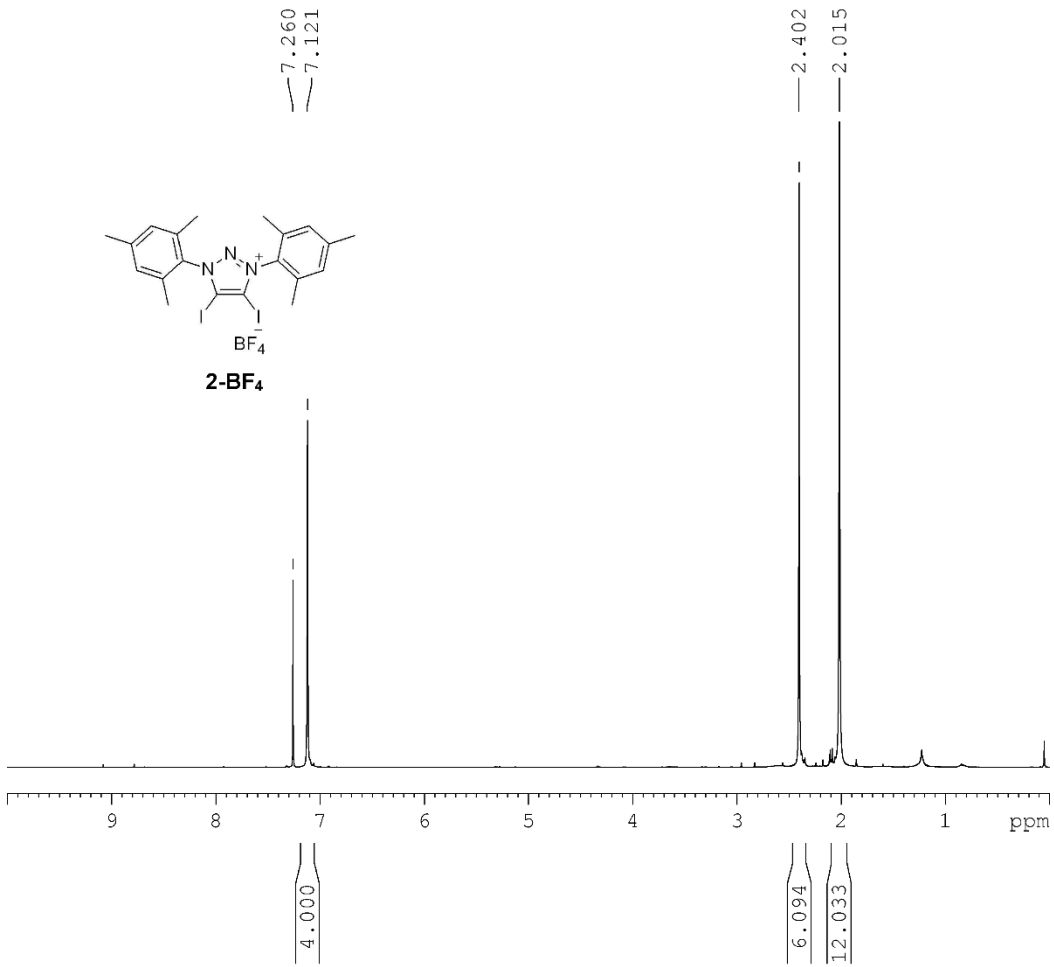


2-I

```
NAME      xxy-2i-i-20161014
EXPNO                    2
PROCNO                   1
Date_             20161014
Time                 9.50
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                65536
SOLVENT             DMSO
NS                    160
DS                      4
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 sec
RG                 2050
DW                19.800 usec
DE                 8.00 usec
TE                293.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   10

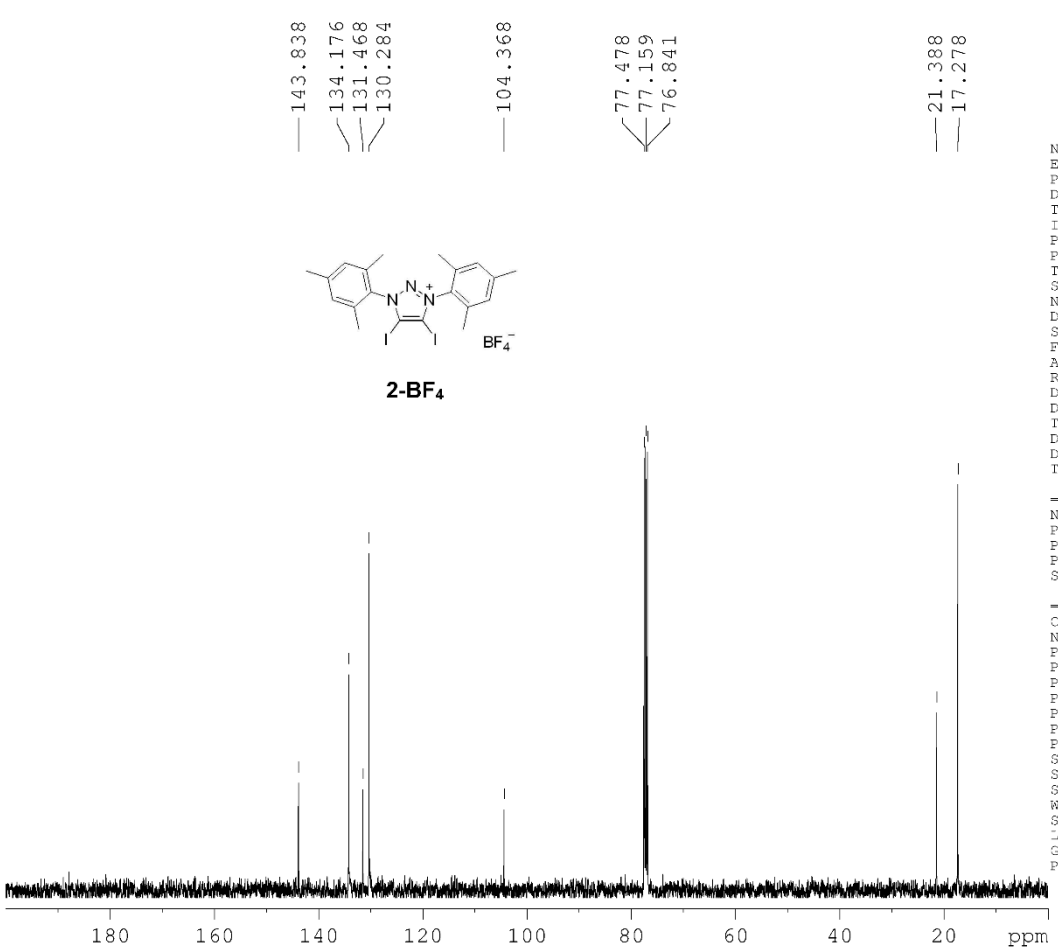
======== CHANNEL f1 ========
NUC1                 13C
P1                 13.50 usec
PL1                 3.00 dB
PL1W         43.93649673 W
SFO1        100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 1.80 dB
PL12               17.19 dB
PL13               18.46 dB
PL2W          8.92857742 W
PL12W         0.25809658 W
PL13W         0.19265592 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127764 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```




```
NAME        xxy-2i-agbf4-20161013
EXPNO                           2
PROCNO                          1
Date_                    20161013
Time                        17.09
INSTRUM                     spect
PROBHD   5 mm PADUL 13C
PULPROG                      zg30
TD                          32768
SOLVENT                     CDCl3
NS                              8
DS                              0
SWH                      6393.862 Hz
FIDRES                   0.195125 Hz
AQ                       2.5625076 sec
RG                            128
DW                         78.200 usec
DE                          6.50 usec
TE                          293.2 K
D1                    1.00000000 sec
TD0                             1

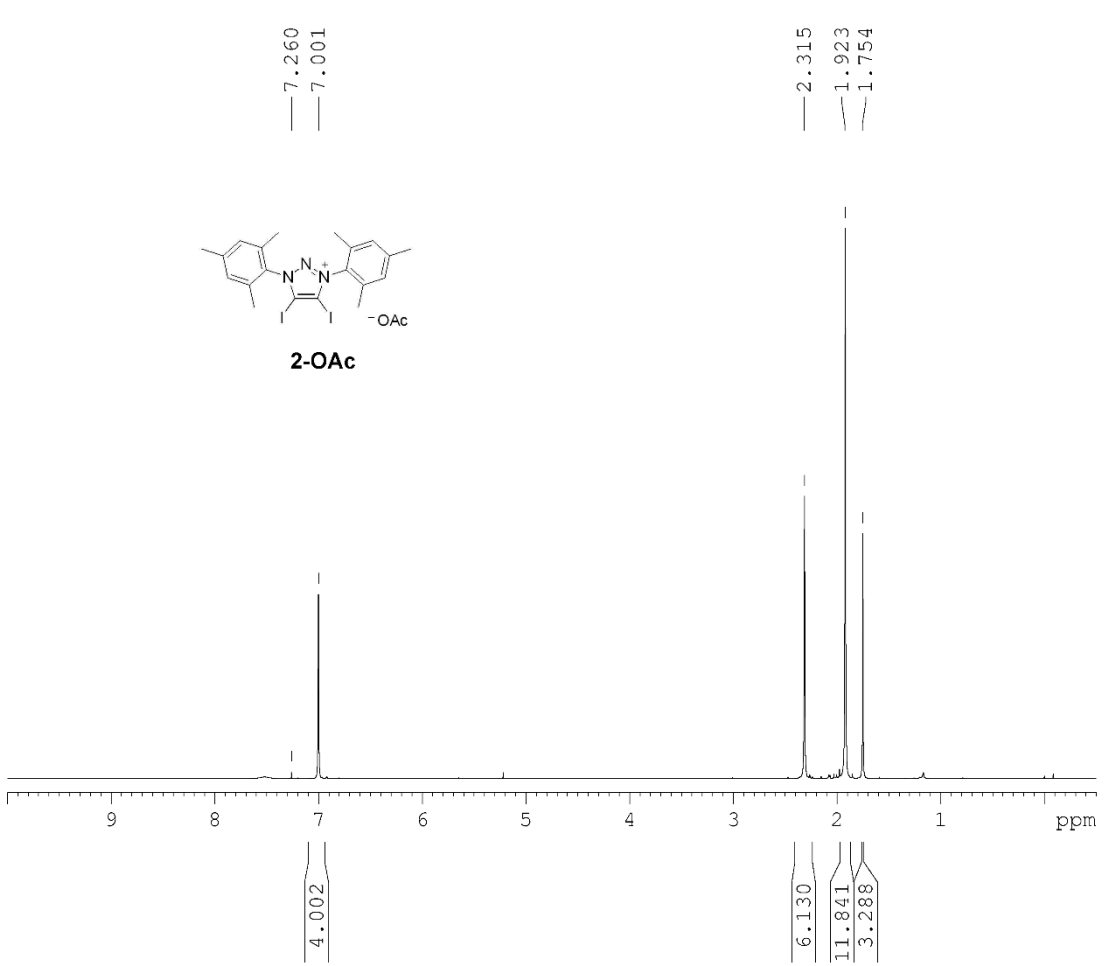
======== CHANNEL f1 ========
NUC1                          1H
P1                         13.10 usec
PL1                         1.80 dB
PL1W                 8.92857742 W
SFO1                400.1326008 MHz
SI                          32768
SF                  400.1300096 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```




```
NAME          xxy-2i-agbf4-20161013
EXPNO                             2
PROCNO                            1
Date_                      20161013
Time                           17.14
INSTRUM                        spect
PROBHD     5 mm PADUL 13C
PULPROG                     zgpg30
TD                             65536
SOLVENT                      CDCl3
NS                                24
DS                                 4
SWH                       25252.525 Hz
FIDRES                     0.385323 Hz
AQ                         1.2976629 sec
RG                              2050
DW                            19.800 usec
DE                             8.00 usec
TE                            293.8 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                               10

======== CHANNEL f1 ========
NUC1                             13C
P1                             13.50 usec
PL1                             3.00 dB
PL1W                    43.93649673 W
SFO1                   100.6238364 MHz

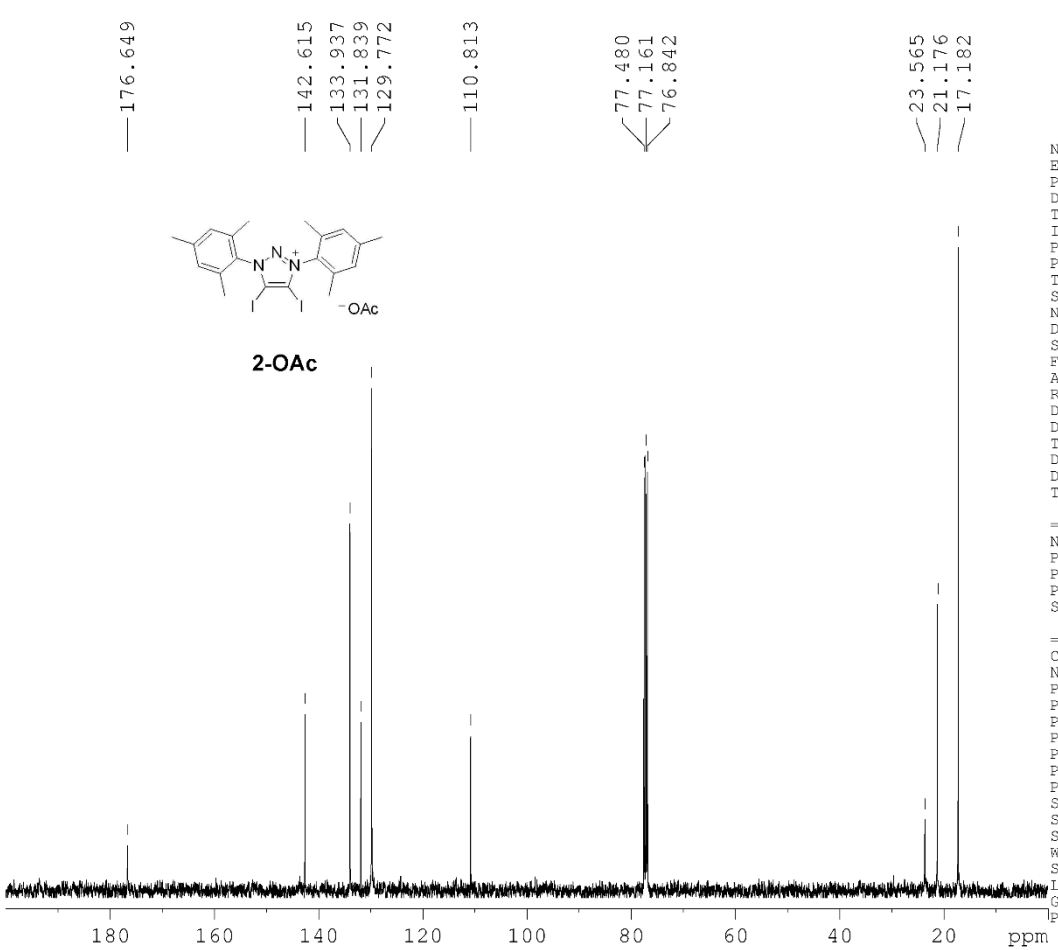
======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                          80.00 usec
PL2                             1.80 dB
PL12                           17.19 dB
PL13                           18.46 dB
PL2W                     8.92857742 W
PL12W                    0.25809658 W
PL13W                    0.19265592 W
SFO2                   400.1316005 MHz
SI                            32768
SF                     100.6127681 MHz
WDW                              EM
SSB                               0
LB                             3.00 Hz
GB                                0
PC                             1.40
```



2-OAc

```
NAME        xxy-828ac-20180829
EXPNO                        1
PROCNO                       1
Date_                 20180829
Time                     14.07
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                   2.5625076 sec
RG                         64
DW                      78.200 usec
DE                       6.50 usec
TE                      292.7 K
D1                 1.00000000 sec
TD0                         1

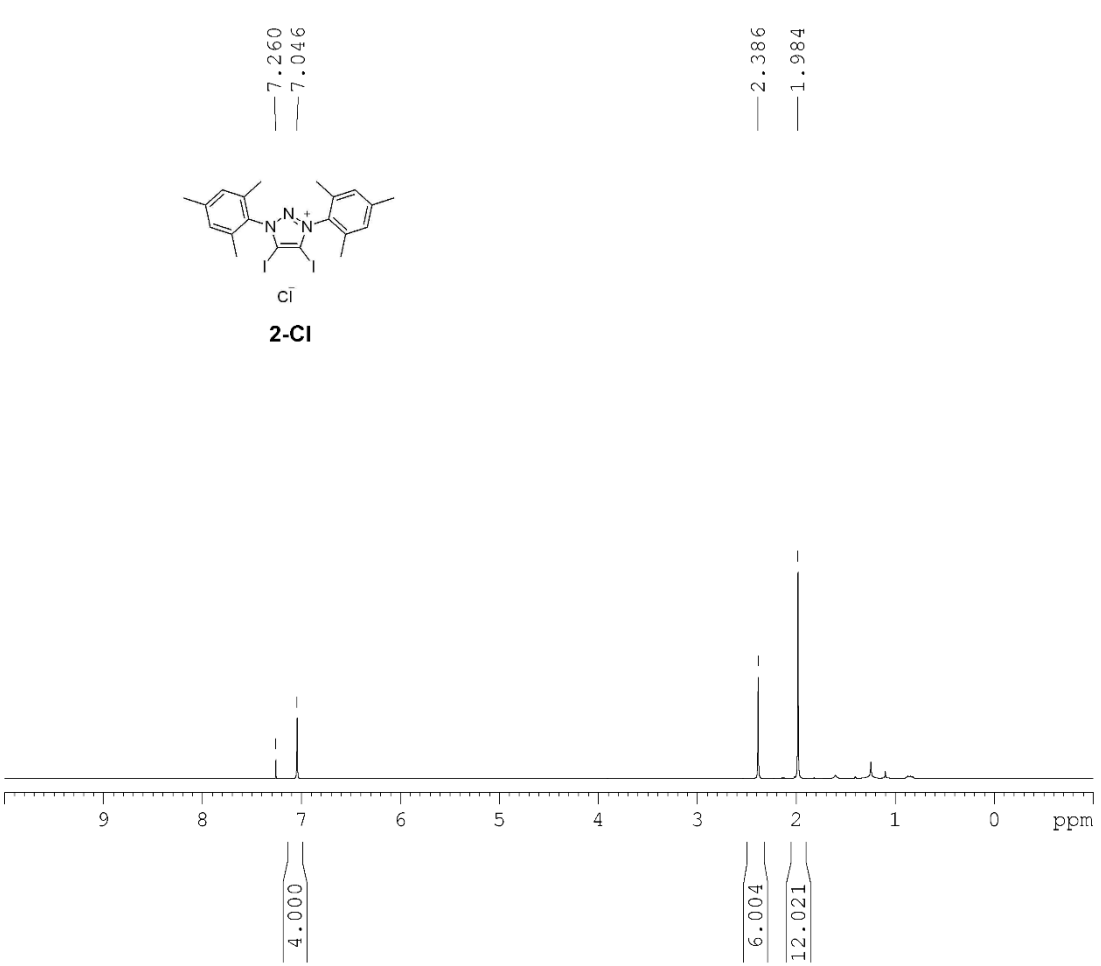
======== CHANNEL f1 ========
NUC1                        1H
P1                       10.40 usec
PL1                      -1.00 dB
PL1W               17.01305389 W
SFO1               400.1326008 MHz
SI                      32768
SF                 400.1300119 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```




```
NAME          xxy-828ac-20180829
EXPNO                          2
PROCNO                         1
Date_                   20180829
Time                       14.12
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            4
SWH                   25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                          181
DW                       19.800 usec
DE                        6.50 usec
TE                       293.4 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           3

======== CHANNEL f1 ========
NUC1                        13C
P1                       15.00 usec
PL1                       2.00 dB
PL1W                55.31277084 W
SFO1               100.6238364 MHz

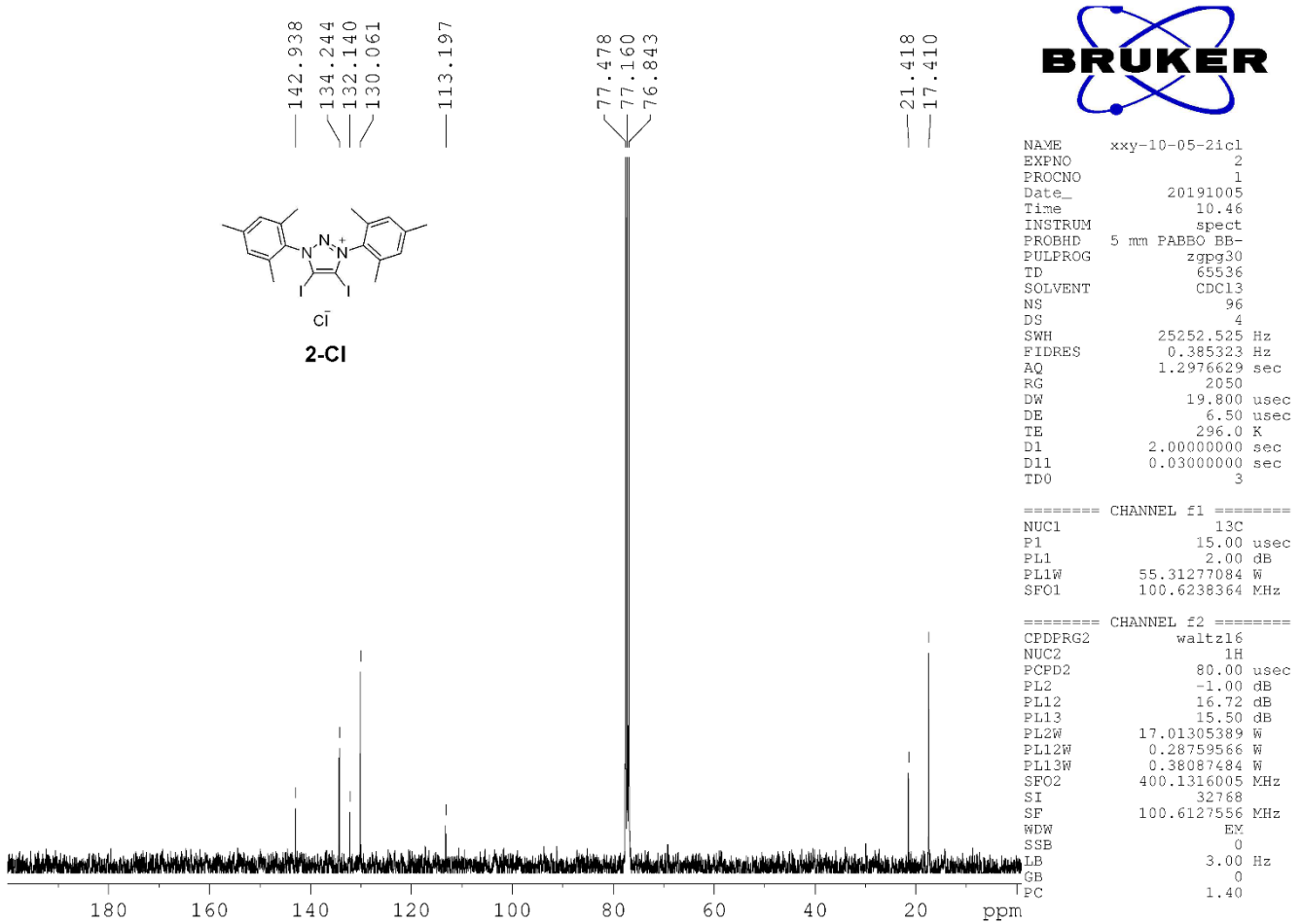
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      -1.00 dB
PL12                     16.72 dB
PL13                     15.50 dB
PL2W                17.01305389 W
PL12W                0.28759566 W
PL13W                0.38087484 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127797 MHz
WDW                          EM
SSB                           0
LB                        3.00 Hz
GB                            0
PC                        1.40
```



2-Cl

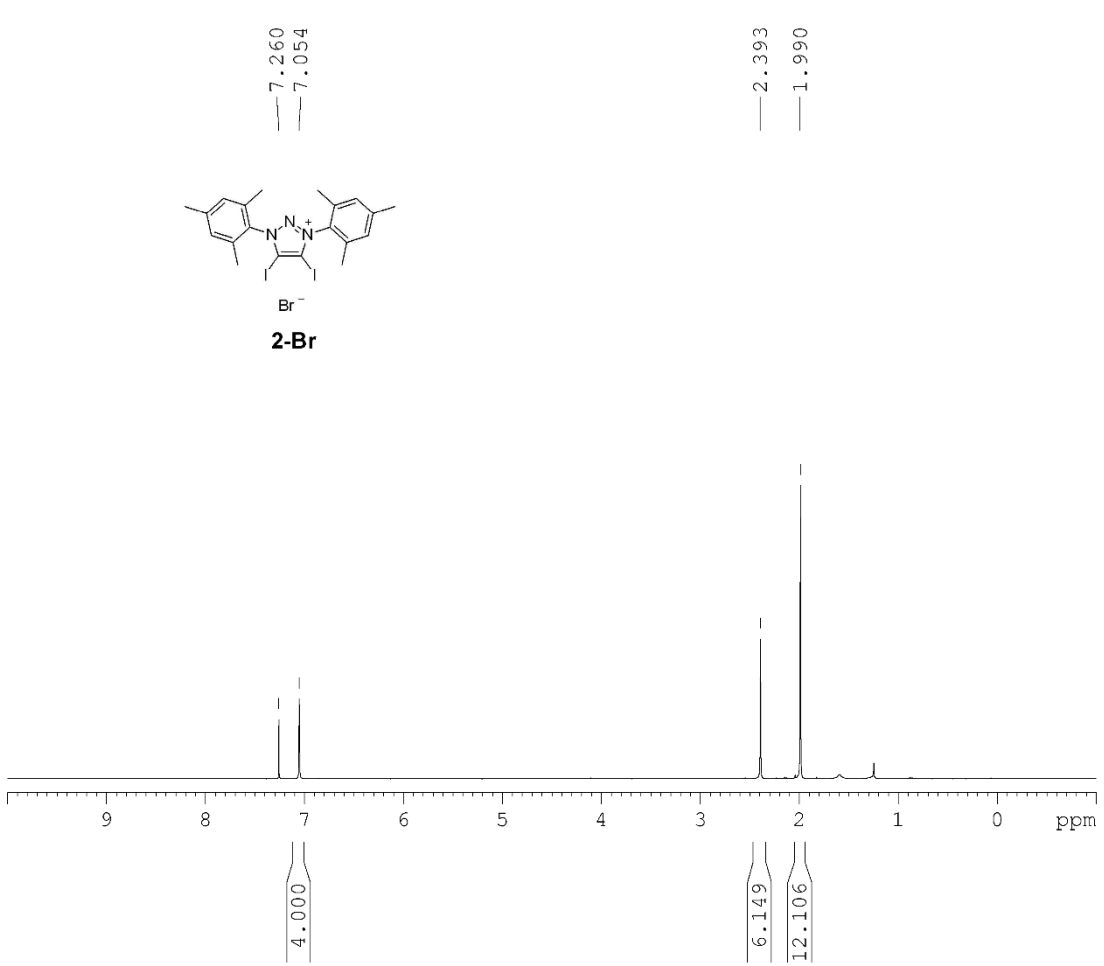
```
NAME      xxy-10-05-2icl
EXPNO                  1
PROCNO                 1
Date_           20191005
Time               10.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ              2.5625076 sec
RG                   322
DW                78.200 usec
DE                 6.50 usec
TE                295.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.40 usec
PL1                -1.00 dB
PL1W        17.01305389 W
SFO1        400.1326008 MHz
SI                 32768
SF          400.1300095 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



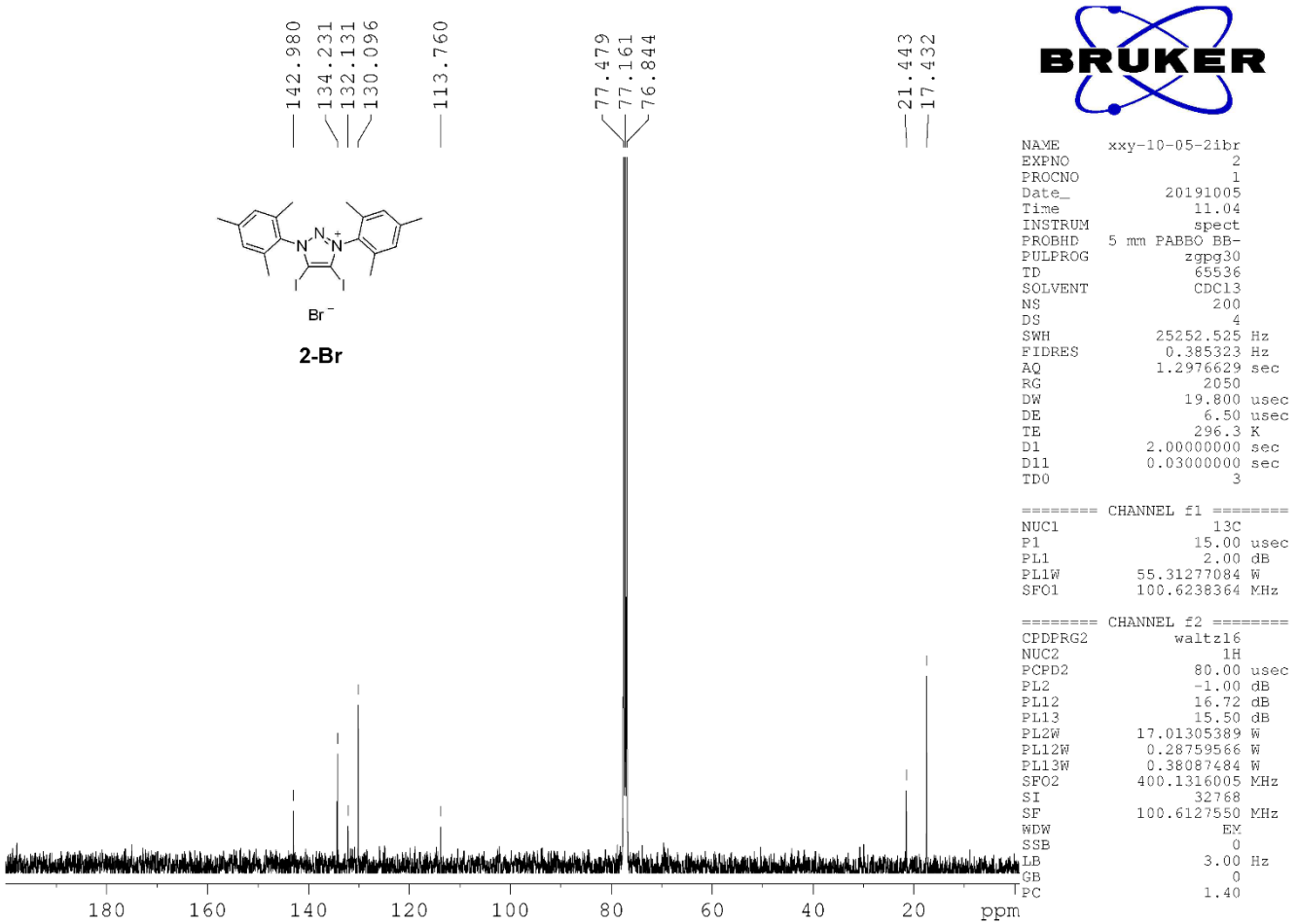
BRUKER

NAME	xxy-10-05-2icl
EXPNO	2
PROCNO	1
Date_	20191005
Time	10.46
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	96
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	296.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	3
======== CHANNEL f1 ========	
NUC1	13C
P1	15.00 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	16.72 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.28759566 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127556 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



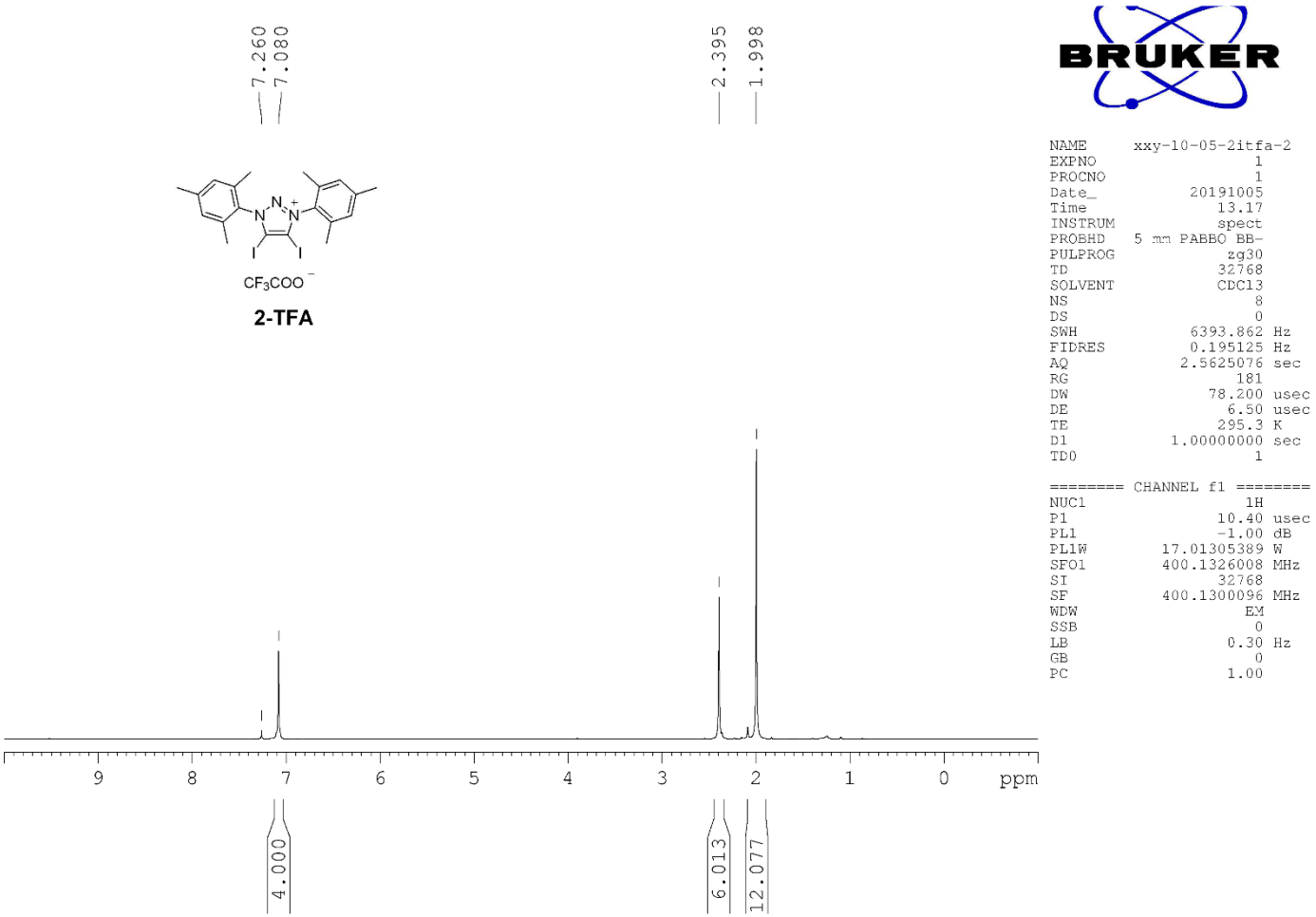

```
NAME      xxy-10-05-2ibr
EXPNO                  1
PROCNO                 1
Date_           20191005
Time               11.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ              2.5625076 sec
RG                   322
DW                78.200 usec
DE                 6.50 usec
TE                295.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.40 usec
PL1                -1.00 dB
PL1W        17.01305389 W
SFO1        400.1326008 MHz
SI                 32768
SF          400.1300095 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



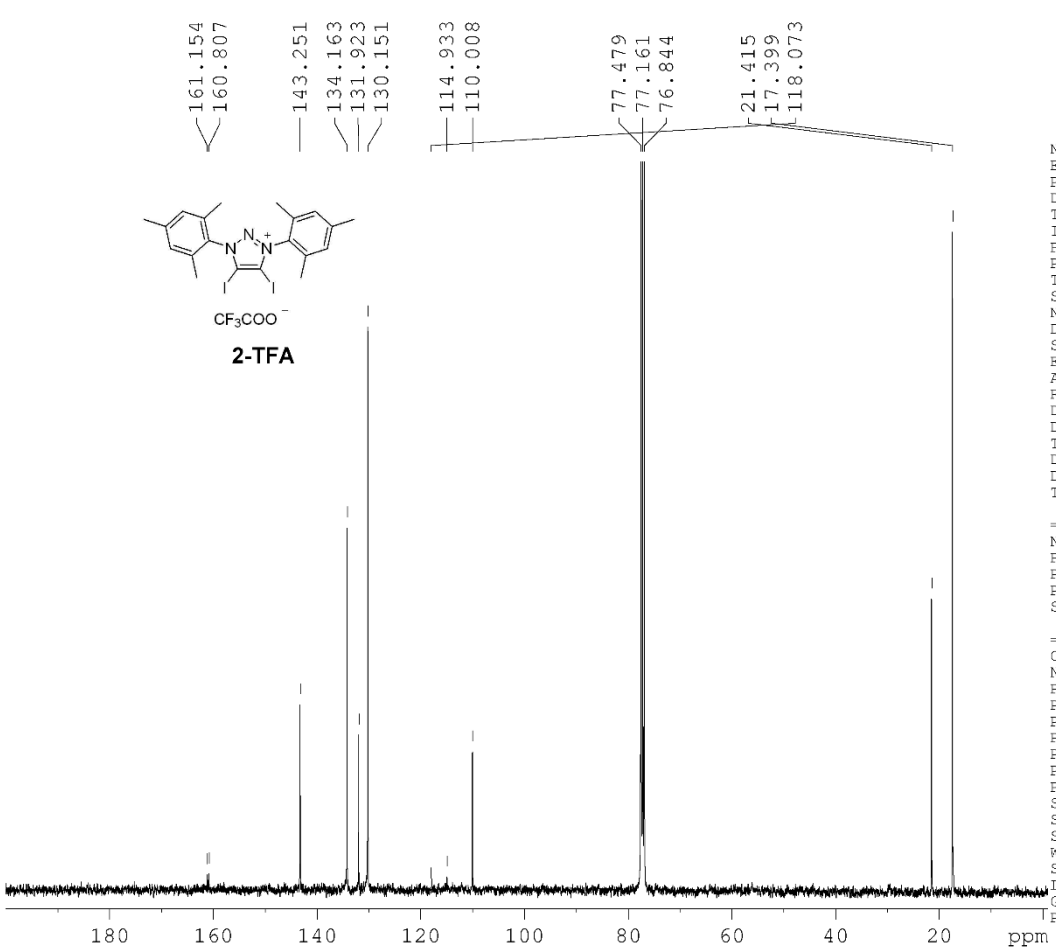
BRUKER

NAME	xxy-10-05-2ibr
EXPNO	2
PROCNO	1
Date_	20191005
Time	11.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	296.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	3
======== CHANNEL f1 ========	
NUC1	13C
P1	15.00 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	16.72 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.28759566 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127550 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40




```
NAME      xxy-10-05-2itfa-2
EXPNO                    1
PROCNO                   1
Date_             20191005
Time                 13.17
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               6393.862 Hz
FIDRES            0.195125 Hz
AQ                2.5625076 sec
RG                     181
DW                  78.200 usec
DE                   6.50 usec
TE                  295.3 K
D1             1.00000000 sec
TD0                      1

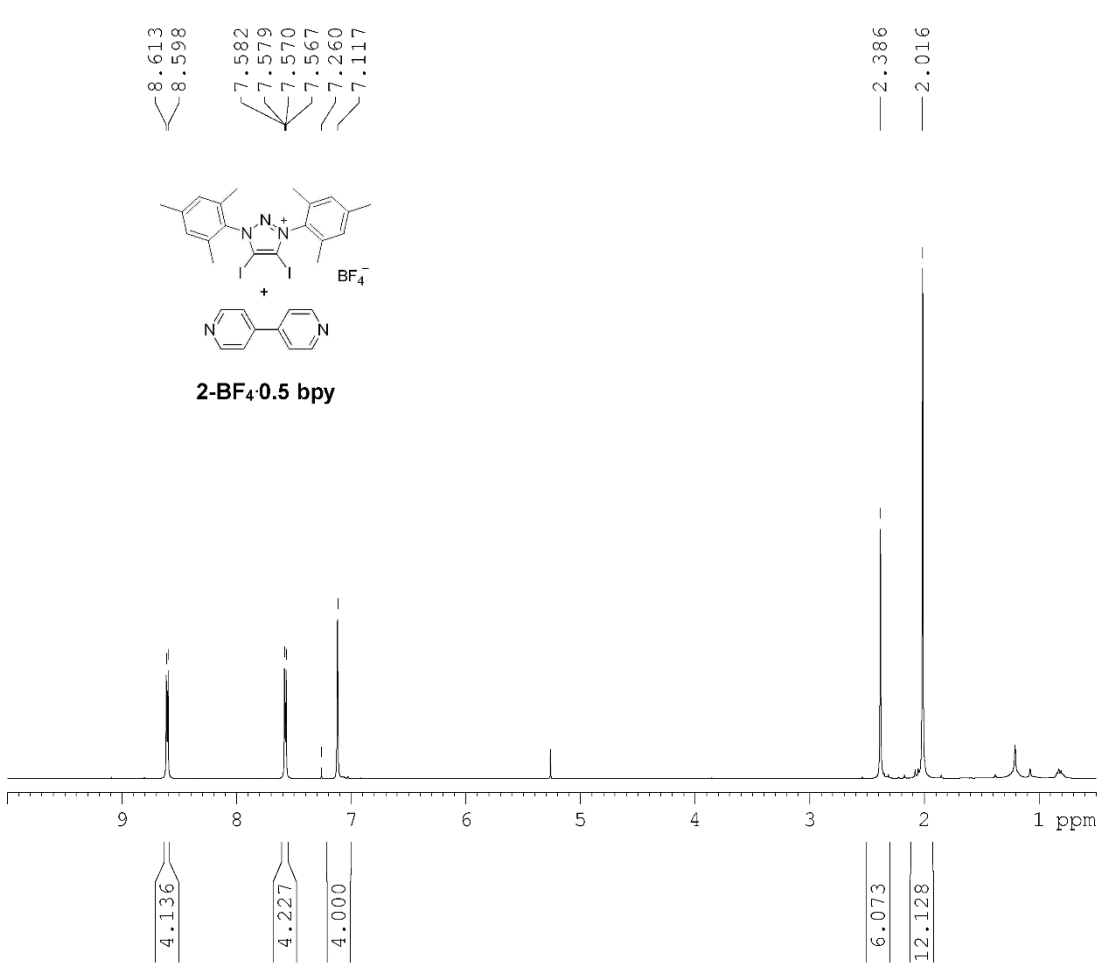
======== CHANNEL f1 ========
NUC1                    1H
P1                   10.40 usec
PL1                  -1.00 dB
PL1W          17.01305389 W
SFO1          400.1326008 MHz
SI                   32768
SF            400.1300096 MHz
WDW                    EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
NAME          xxy-10-05-2itfa-2
EXPNO                         2
PROCNO                        1
Date_                  20191005
Time                      13.21
INSTRUM                   spect
PROBHD       5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1032
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                   1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       6.50 usec
TE                      295.9 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          3

======== CHANNEL f1 ========
NUC1                        13C
P1                        15.00 usec
PL1                        2.00 dB
PL1W                55.31277084 W
SFO1               100.6238364 MHz

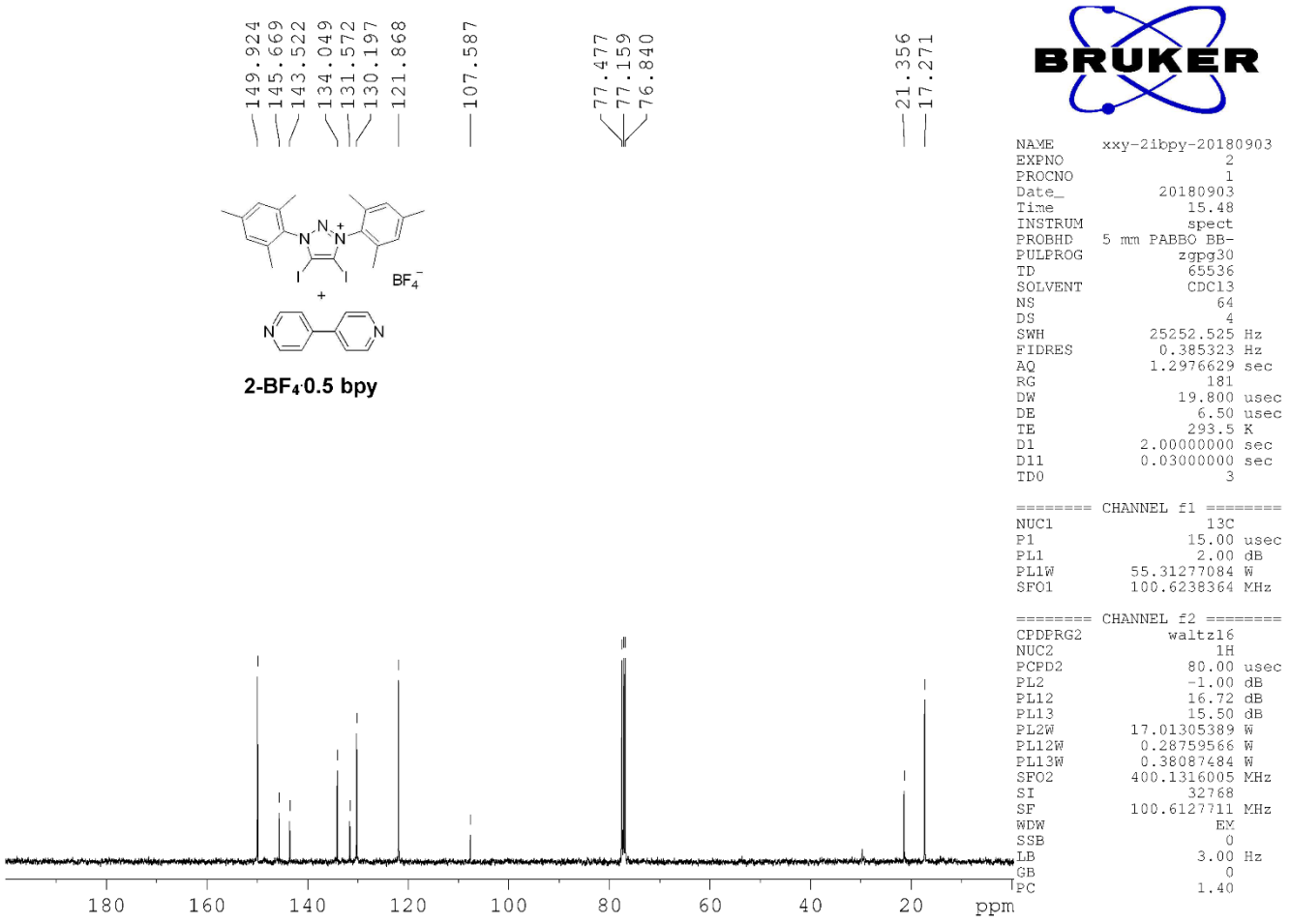
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                       -1.00 dB
PL12                      16.72 dB
PL13                      15.50 dB
PL2W                17.01305389 W
PL12W                0.28759566 W
PL13W                0.38087484 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127577 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40
```



2-BF_4·0.5 bpy

```
NAME          xxy-2ibpy-20180903
EXPNO                          1
PROCNO                         1
Date_                   20180903
Time                       15.58
INSTRUM                    spect
PROBHD   5 mm PABBO BB-
PULPROG                     zg30
TD                         32768
SOLVENT                    CDCl3
NS                             8
DS                             0
SWH                     6393.862 Hz
FIDRES                  0.195125 Hz
AQ                     2.5625076 sec
RG                          80.6
DW                        78.200 usec
DE                         6.50 usec
TE                        292.6 K
D1                   1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        10.40 usec
PL1                       -1.00 dB
PL1W                17.01305389 W
SFO1                400.1326008 MHz
SI                         32768
SF                  400.1300109 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```



BRUKER

NAME	xxy-2ibpy-20180903
EXPNO	2
PROCNO	1
Date_	20180903
Time	15.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	181
DW	19.800 usec
DE	6.50 usec
TE	293.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	3
======== CHANNEL f1 ========	
NUC1	13C
P1	15.00 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	16.72 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.28759566 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127711 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40