

Supporting Information

Highly fluorescent solid-state benzothiadiazole derivatives as saturated red emitters for efficient solution-processed non-doped electroluminescent devices

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Materials Synthesis

General method for Suzuki cross coupling reaction: A mixture of aryl bromide (1.71 mmol), aryl borolane/aryl boronic acid, Pd(PPh₃)₄ (0.05 mmol) and 2 M Na₂CO₃ (15 ml) in THF (30 ml) was degassed with N₂ for 10 min. The mixture was stirred at reflux under N₂ for 24 h. After cooling, water (50 ml) was added, and the mixture was extracted with CH₂Cl₂ (3 x 50 ml). The combined organic phase was washed with water (50 ml), brine solution (50 ml), dried over anhydrous Na₂SO₄ and filtered. The solvent was removed to dryness and the crude product was purified by column chromatography on silica gel eluting with a mixture of CH₂Cl₂/hexane.

General method for bromination reaction: To a solution of the bis(hexylthiophenyl)-benzothiadiazoles (0.39 mmol) in THF (20 ml) was added with NBS in small portions. After completion, water was added and the mixture was extracted with CH₂Cl₂ (3 x 50 ml). The combined organic phase was washed with water (50 ml), brine solution (50 ml), dried over anhydrous Na₂SO₄ and filtered. The solvent was removed to dryness and the crude product was purified by column chromatography on silica gel eluting with a mixture of CH₂Cl₂/hexane.

4,7-Bis(3-hexylthiophen-2-yl)-2,1,3-benzothiadiazole (2)

Compound **2** was prepared from the reaction of **1** (0.50 g, 1.71 mmol) and 3-hexylthiophene-2-yl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.06 g, 3.61 mmol) and obtained as orange solids (0.78 g, 97%); m.p. 87-88 °C; ¹H-NMR (600 MHz, CDCl₃) δ = 7.65 (s, 2H), 7.44 (d, *J* = 5.4 Hz, 2H), 7.11 (d, *J* = 5.4 Hz, 2H), 2.67 (t, *J* = 7.8 Hz, 4H), 1.65-1.60 (m, 4H), 1.26-1.18 (m, 12H), 0.81 (t, *J* = 6.6 Hz, 6H); ¹³C-NMR (150 MHz, CDCl₃) δ = 153.28, 140.68, 131.18, 128.88, 128.20, 126.48, 124.82, 30.53, 29.64, 28.33, 28.07, 21.49, 12.99; HRMS (MALDI-TOF) *clacd.* for C₂₆H₃₂N₂S₃: 468.1728 (M⁺), found: 468.1335.

4,7-Bis(5-bromo-3-hexylthiophen-2-yl)-2,1,3-benzothiadiazole (3)

Compound **3** was prepared from bromination of **2** (0.18 g, 0.39 mmol) with NBS (0.15 g, 0.81 mmol) and obtained as orange solids (0.23 g, 95%); m.p. 90-91 °C; ¹H-NMR (600 MHz, CDCl₃) δ = 7.06 (s, 2H), 7.05 (s, 2H), 2.60 (t, *J* = 7.8 Hz, 4H), 1.60-1.58 (m, 4H), 1.25-1.24 (m, 12H), 0.89-0.80(m, 6H); ¹³C-NMR (150 MHz, CDCl₃) δ = 153.95, 142.46, 133.57, 132.01, 129.71, 126.66, 113.20, 31.94, 31.52, 30.50, 29.72, 29.42, 29.38, 29.03, 22.71, 22.51, 14.12, 14.01; HRMS (MALDI-TOF) clacd. for C₂₆H₃₀Br₂N₂S₃: 625.9917 (M⁺), found: 626.1159.

4,7-Bis(4,5-dibromo-3-hexylthiophen-2-yl)-2,1,3-benzothiadiazole (4)

Compound **4** was prepared from bromination of **3** (0.23 g, 0.38 mmol) with NBS (0.26 g, 1.47 mmol) and obtained as orange solids (0.21 g, 73%); m.p. 97-98 °C; ¹H-NMR (600 MHz, CDCl₃) δ = 7.64 (s, 2H), 2.69 (t, *J* = 7.8 Hz, 4H), 1.53-1.26 (m, 16H), 0.88-0.77(m, 6H); ¹³C-NMR (150 MHz, CDCl₃) δ = 153.93, 153.80, 153.75, 142.62, 142.45, 140.90, 138.72, 133.55, 133.20, 132.05, 132.00, 130.03, 129.86, 129.55, 127.03, 126.64, 117.66, 113.19, 112.51, 31.50, 31.24, 30.48, 29.70, 29.40, 29.34, 29.01, 28.95, 22.49, 22.45, 14.00, 13.96; HRMS (APCI) clacd. for C₂₆H₂₈Br₄N₂S₃: 783.8107 (M⁺ + 1), found: 784.8135.

4,7-Bis(4-hexylthiophen-2-yl)-2,1,3-benzothiadiazole (5)

Compound **5** was prepared from the reaction of **1** (0.70 g, 2.38 mmol) and 4-hexylthiophen-2-yl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.47g, 5.00 mmol) and obtained as orange solids (1.04 g, 93%); m.p. 93-94 °C; ¹H-NMR (500 MHz, CDCl₃) δ = 7.97 (s, 2H), 7.82 (s, 2H), 7.038 (s, 2H), 2.69 (t, *J* = 7.5 Hz, 4H), 1.73-1.67 (m, 4H), 1.40-1.32 (m, 12H), 0.90 (t, *J* = 6.5 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ = 152.65, 144.37, 139.01, 132.26, 129.62, 129.00, 126.03, 125.59, 125.54, 122.07, 121.53, 31.71, 30.67, 30.61, 30.49, 29.06, 22.64, 14.12; HRMS (MALDI-TOF) clacd. for C₂₆H₃₂N₂S₃: 468.1728 (M⁺), found: 468.1920.

4,7-Bis(4-hexyl-5-bromothiophen-2-yl)-2,1,3-benzothiadiazole (6)

Compound **6** was prepared from bromination of **5** (1.00 g, 1.39 mmol) with NBS (0.65 g, 2.91 mmol) and obtained as orange-red solids (1.51g, 98%); m.p. 98-99 °C; ¹H-NMR (500 MHz, CDCl₃) δ = 7.75 (s, 2H), 7.70 (s, 2H), 2.61 (t, *J* = 8 Hz, 4H), 1.67-1.64 (m, 4H), 1.55 (s, 6H), 1.42-1.33 (m, 14H), 0.90 (t, *J* = 6.5 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ = 152.25, 148.27, 143.61, 127.75, 125.44, 125.04, 32.25, 31.67, 30.06, 29.71, 28.96, 22.63, 14.12; HRMS (MALDI-TOF) clacd. for C₂₆H₃₀I₂N₂S₃: 719.9660 (M⁺), found: 719.9730.

4,7-Bis(3,5-dibromo-4-hexylthiophen-2-yl)-2,1,3-benzothiadiazole (7)

Compound **7** was prepared from bromination of **6** (0.29 g, 0.59 mmol) with NBS (0.42 g, 2.39 mmol) and obtained as orange solids (0.22 g, 78%); m.p. 97-98 °C; ¹H-NMR (600 MHz, CDCl₃) δ = 8.06 (s, 2H), 2.75 (t, *J* = 9.6 Hz, 4H), 1.63-1.58 (m, 4H), 1.45-1.44(m, 4H), 1.42 (s, 8H), 1.37-1.34 (m, 6H); ¹³C-NMR (150 MHz, CDCl₃) δ = 153.24, 150.06, 141.75, 132.84, 129.87, 125.97, 112.20, 111.53, 31.95, 31.59, 30.40, 29.68, 29.38, 28.67, 22.71, 22.65, 14.12; HRMS (MALDI-TOF) clacd. for C₂₆H₂₈Br₄N₂S₃: 783.8107 (M⁺), found: 783.8642.

Device Fabrication and Performance

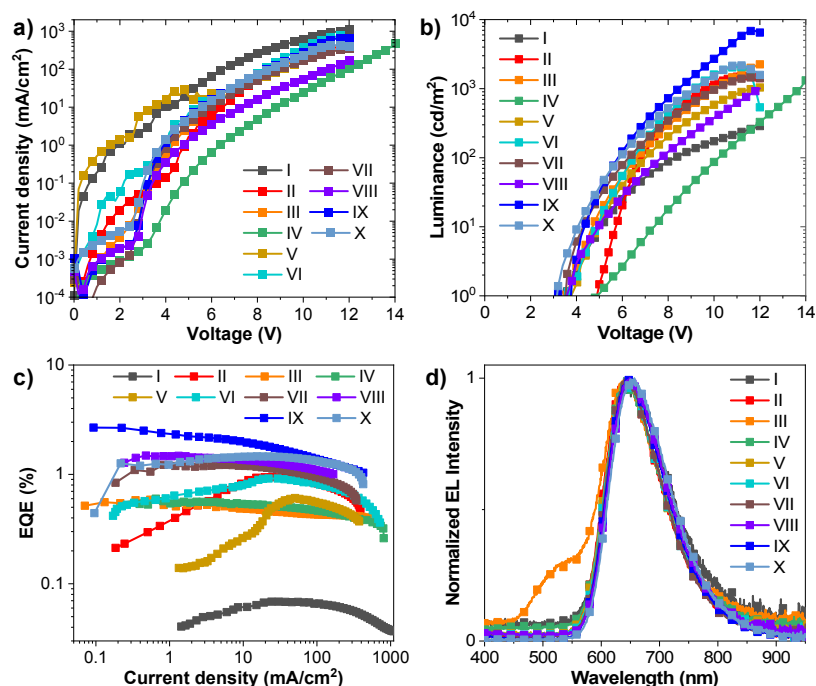


Fig. S1. a) Current density-voltage (J - V) characteristics. b) Luminance-voltage (L - V) characteristics. c) External quantum efficiency-current density (EQE - J) characteristics. d) Compared electroluminescence spectra of devices I to IV using **BTZ4** as EML.

Table S1. Electroluminescent data of non-doped OLEDs fabricated with **BTZ4** as EML

Device ^{a)}	ETL	V_{on} (V)	L_{max}/J_{max} (cd/m^2)/ (mA/cm^2)	λ_{EL} (nm)	% EQE_{max} at V	CE (cd/A) ^{b)}	CIE (x,y)
I	-	3.4	287/	652	0.07/5.2	0.03	(0.66, 0.34)
II	BCP(40 nm)	4.9	1518/	643	0.94/7.2	0.66	(0.65, 0.35)
III	BCP(10 nm)/Alq3(40 nm)	3.5	2247/	643	0.94/3.4	0.48	(0.54, 0.42)
IV	BCP(20 nm)/Alq3(30 nm)	5.1	1934/	644	0.57/7.0	0.34	(0.65, 0.34)
V	TAZ(40nm)	3.8	1040/	644	0.61/8.0	0.32	(0.66, 0.34)
VI	TAZ(30nm)/TPBi(10 nm)	3.8	2073/	644	0.95/6.6	0.61	(0.66, 0.34)
VII	TPBi(10 nm)/TAZ(30nm)	3.5	1449/	645	1.22/4.0	0.73	(0.66, 0.34)
VIII	TPBi(40nm)/TAZ(10nm)	3.6	1007/	649	1.49/4.2	0.75	(0.66, 0.33)
IX	TPBi(50nm)	3.3	2334/636	650	2.66/3.5	0.71	(0.66, 0.34)
X	TmPyPB(60nm)	3.1	2193/	657	1.46/6.0	0.72	(0.67, 0.33)

^{a)} ITO/PEDOT:PSS(40 nm)/**BTZ4** (60 nm)/ETL/LiF(0.5 nm):Al(150 nm). ^{b)} Luminous efficiency at 100 cd/m^2

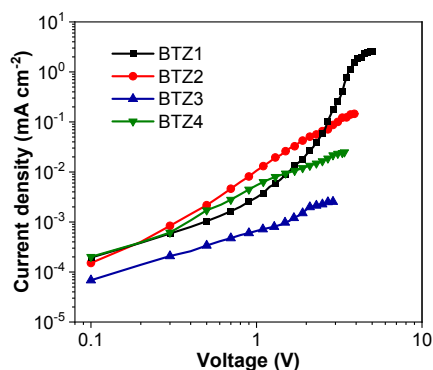
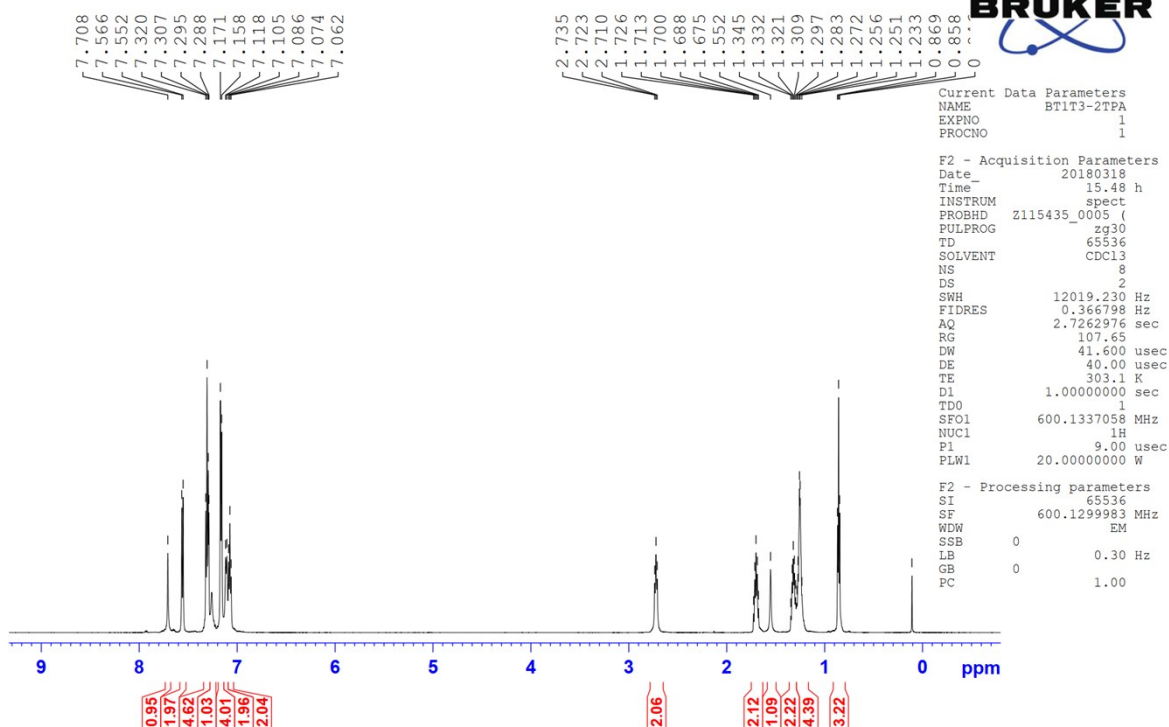


Fig. S2 Current density-voltage (J - V) plots of electron only device (ITO/ SnO_2 /BTZn (120 nm)/LiF/Al).

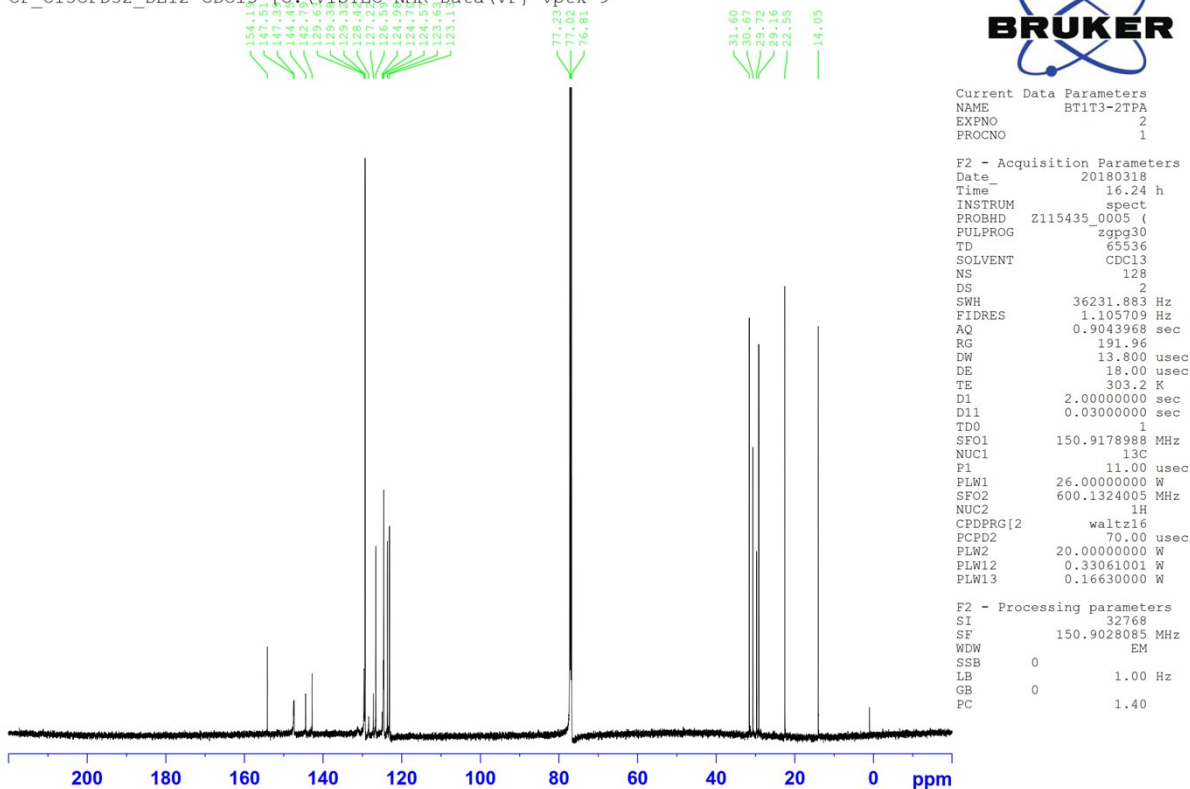
Materials Characterization Data (¹H-NMR, ¹³C-NMR and HRMS MALDI-TOF)

BTZ1

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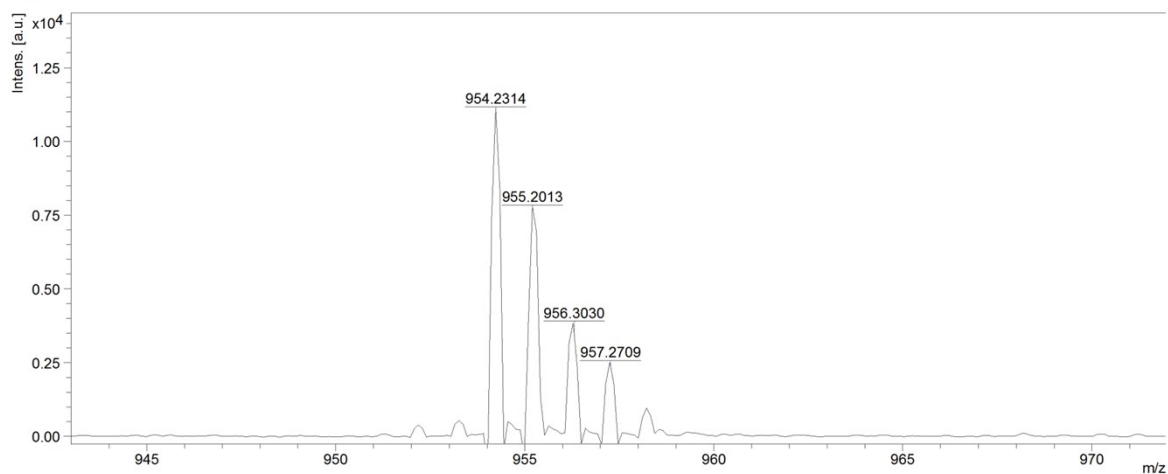


MALDI-TOF-MS Report

Frontier Research Center, Vidyasirimedhi Institute of Science and Technology

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Comment 2



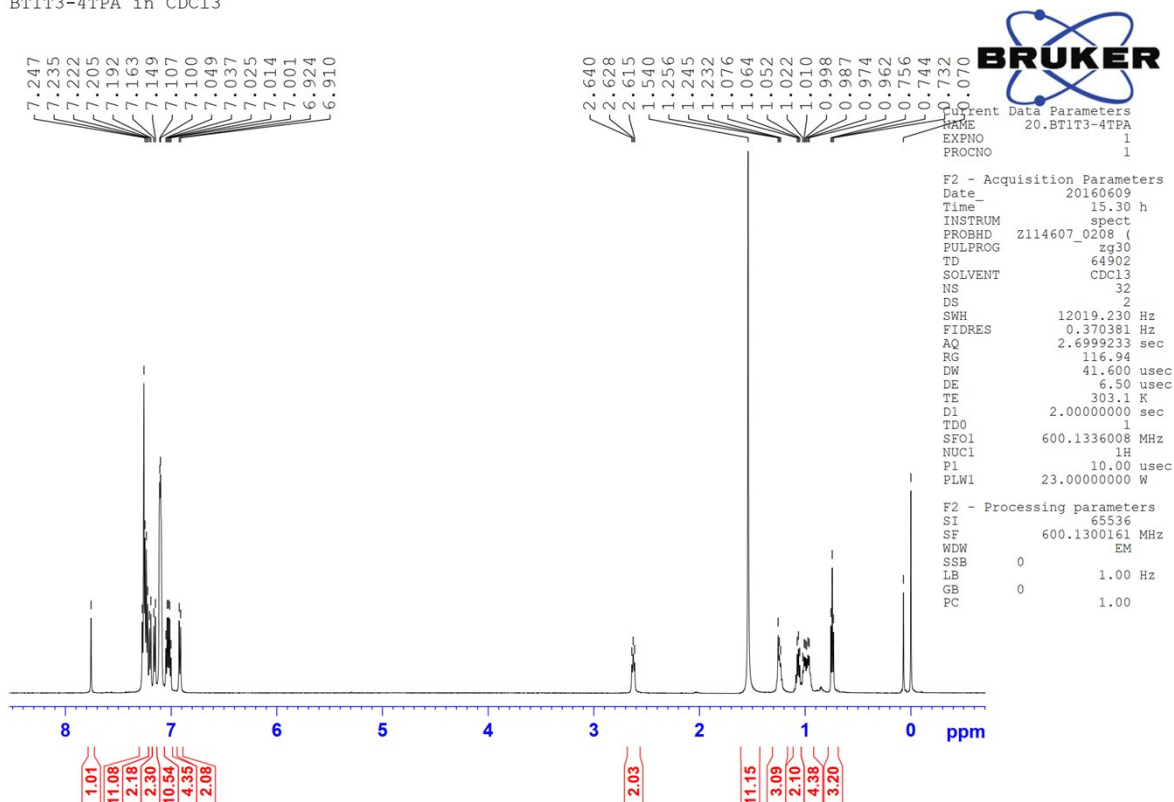
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Bruker Autoflex Speed

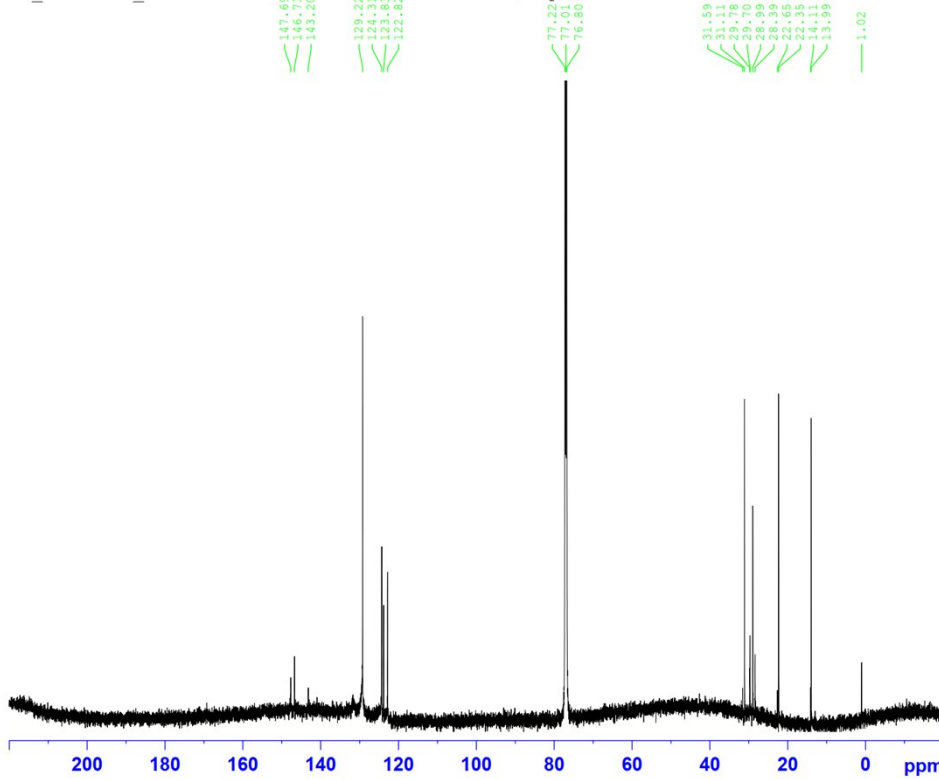
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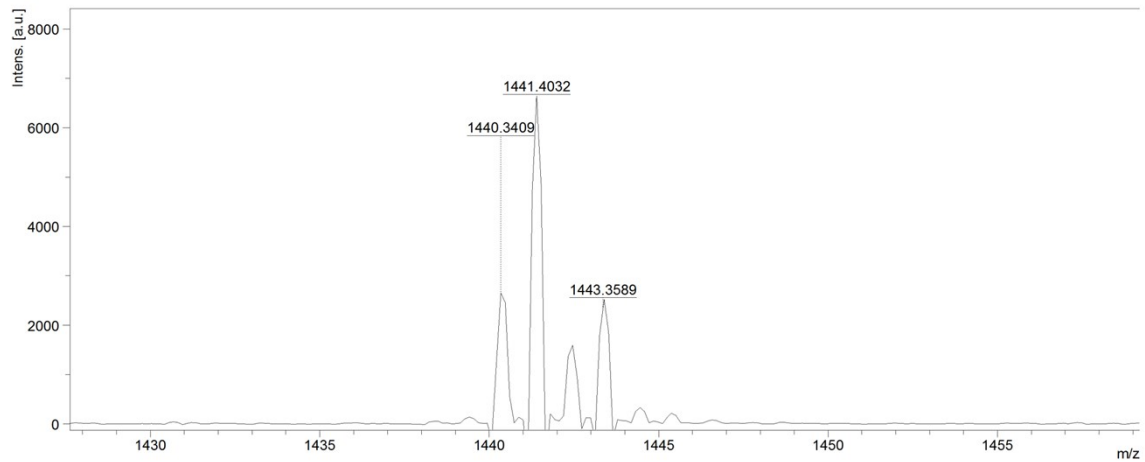
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MALDI-TOF-MS Report

Frontier Research Center, Vidyasirimedhi Institute of Science and Technology

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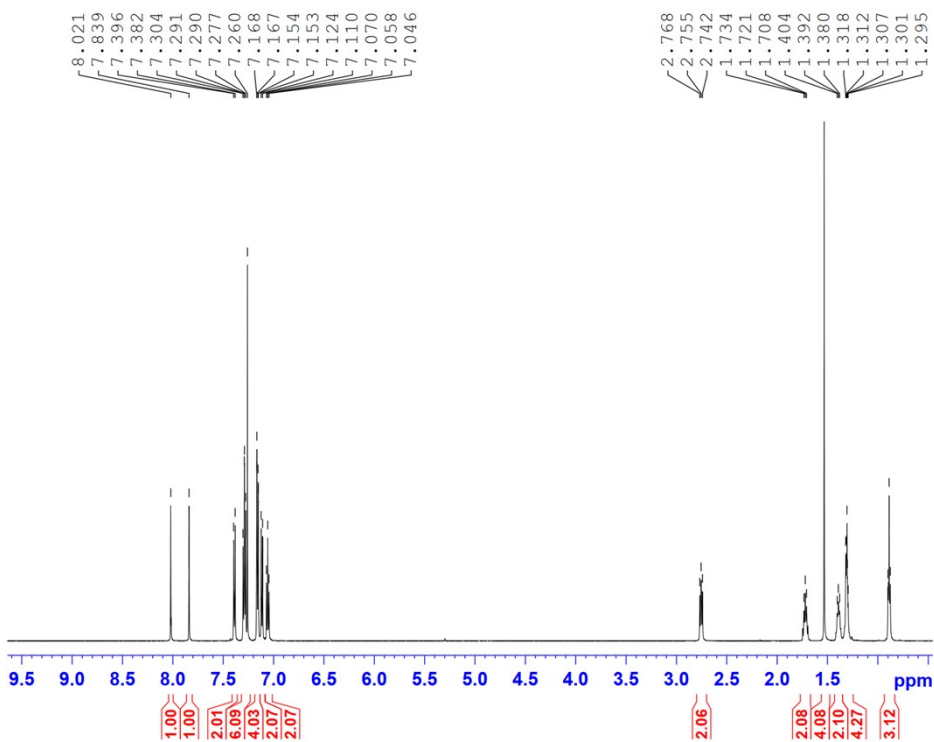
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Bruker Autoflex Speed

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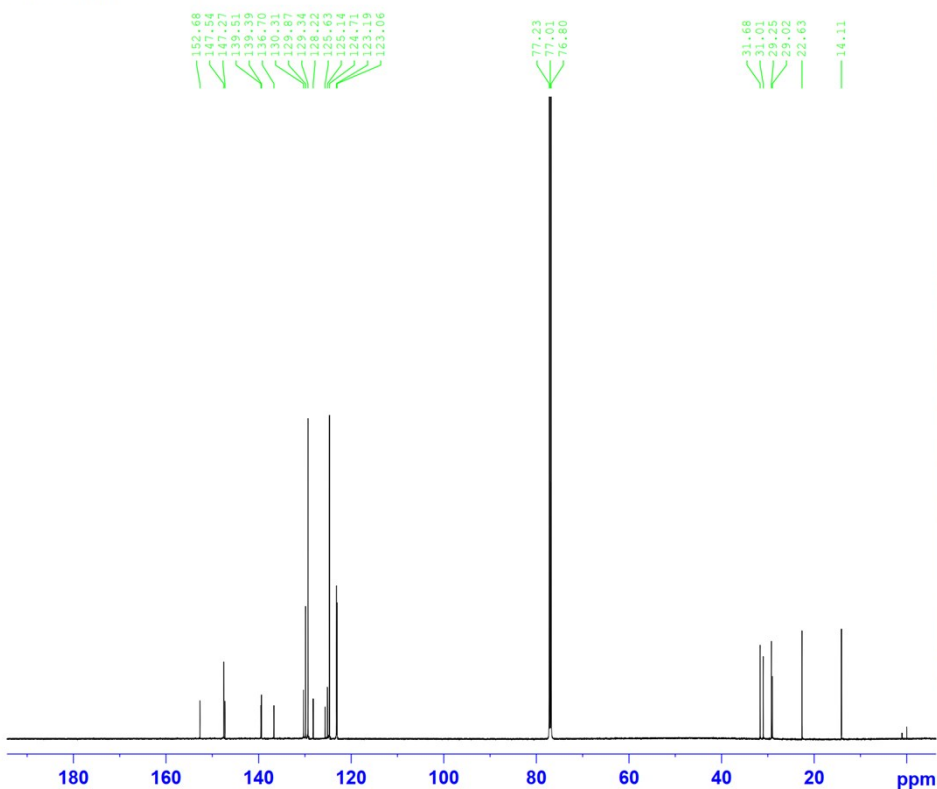


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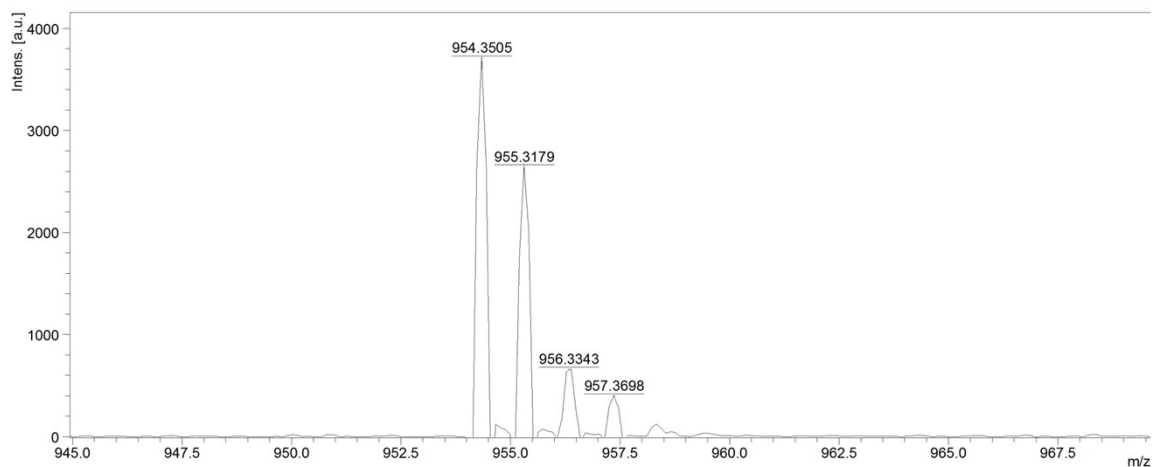
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MALDI-TOF-MS Report

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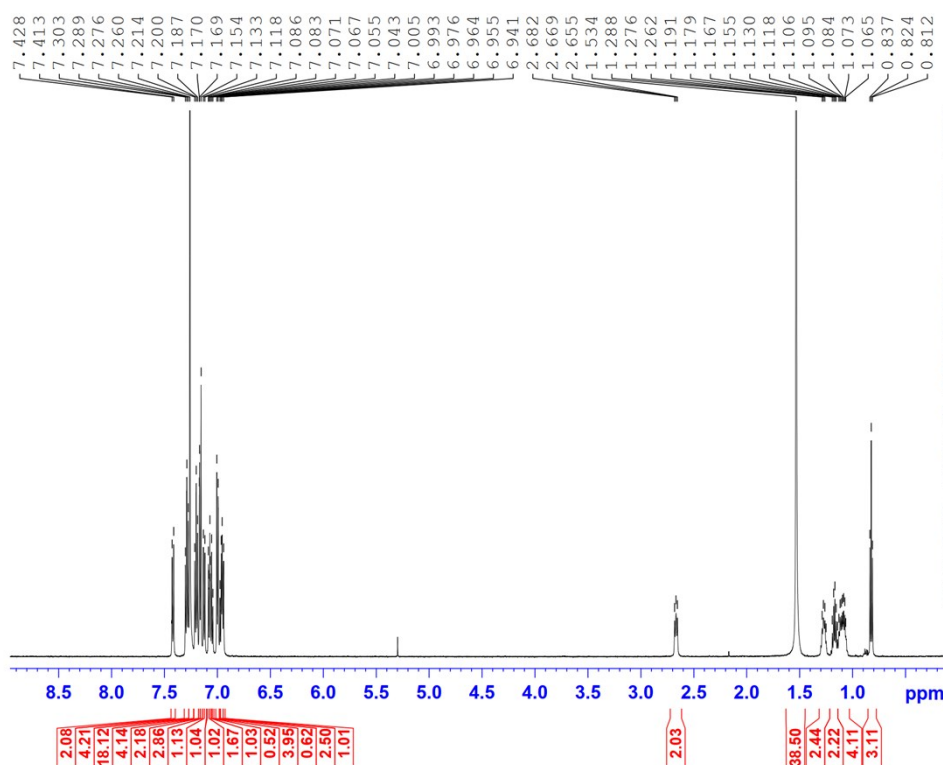
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Bruker Autoflex Speed

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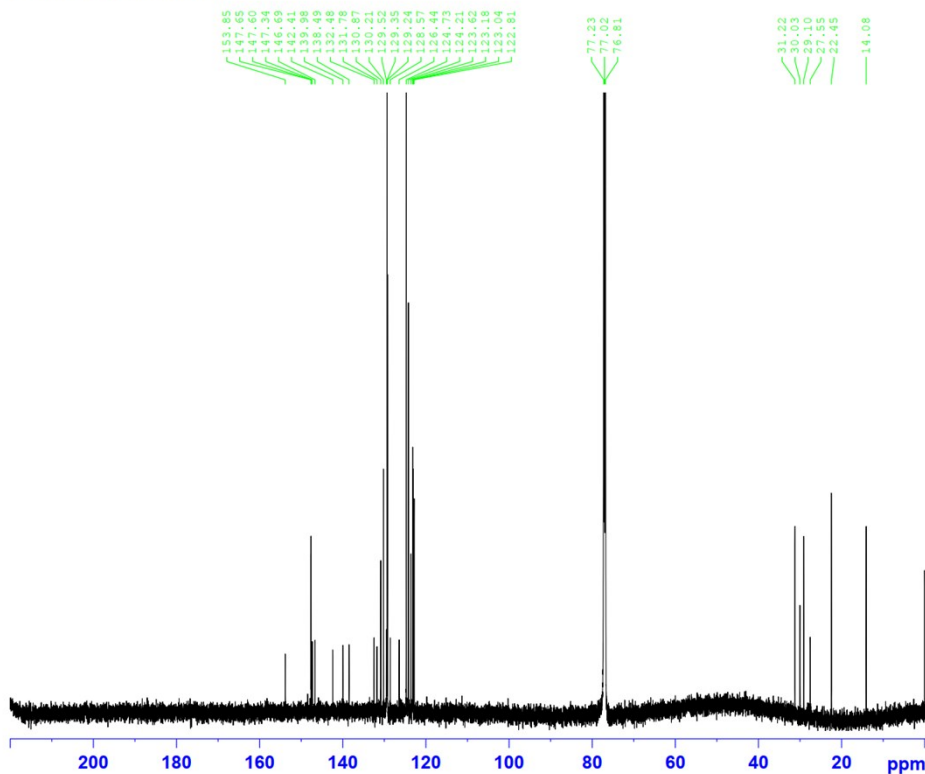


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BT1T4-4TPA in CDCl3



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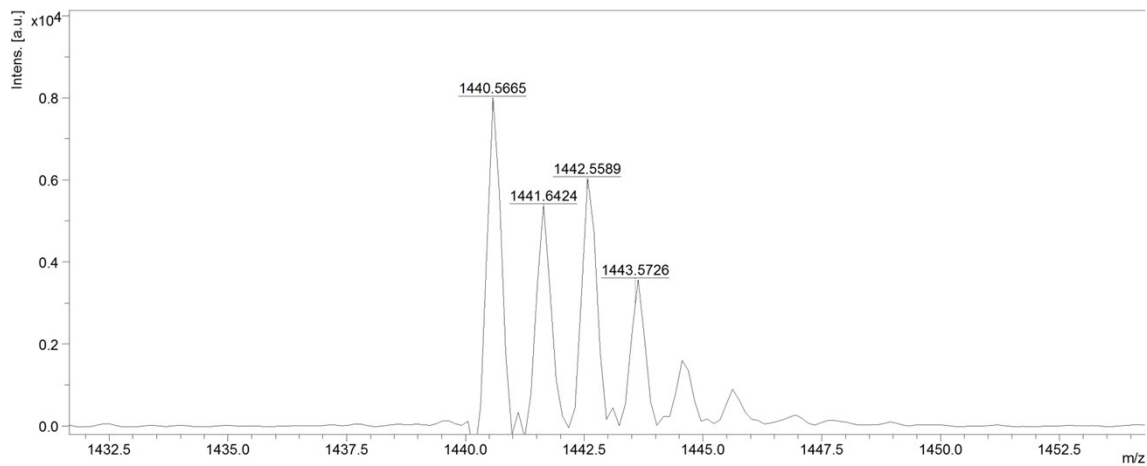
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MALDI-TOF-MS Report

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Comment 1 (MW=1441.5953)
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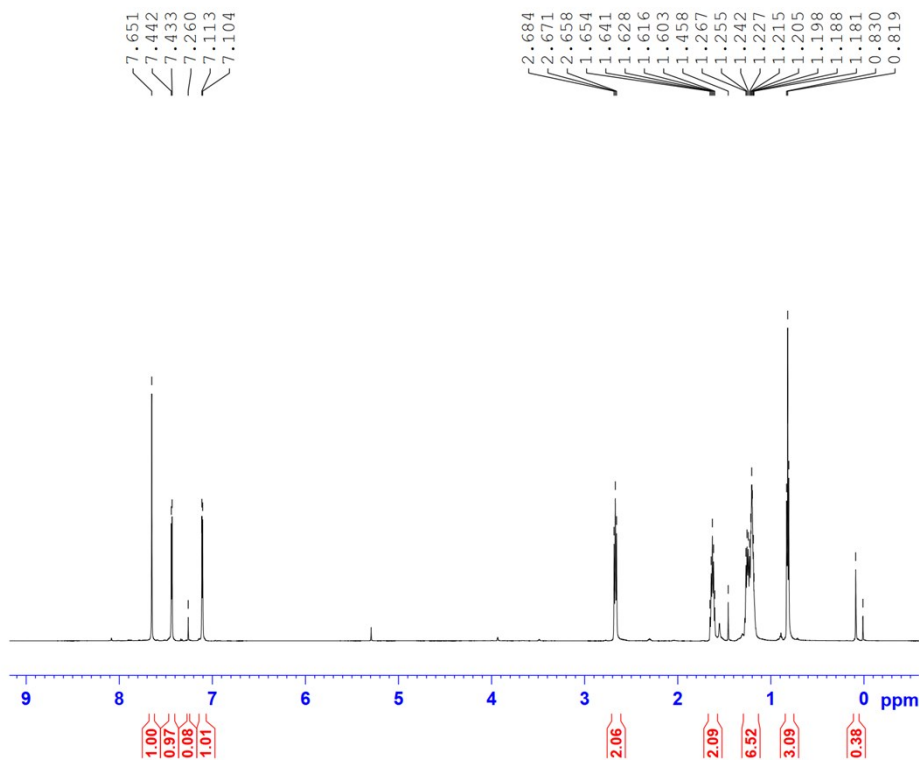
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Bruker Autoflex Speed

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Compound 2

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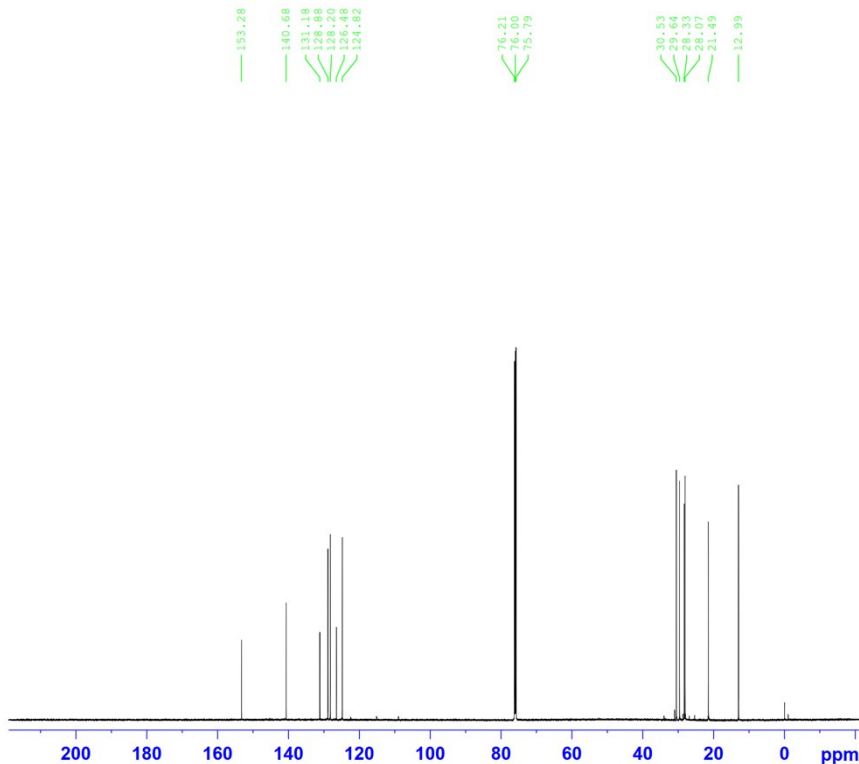


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BT1T3 in CDCl3



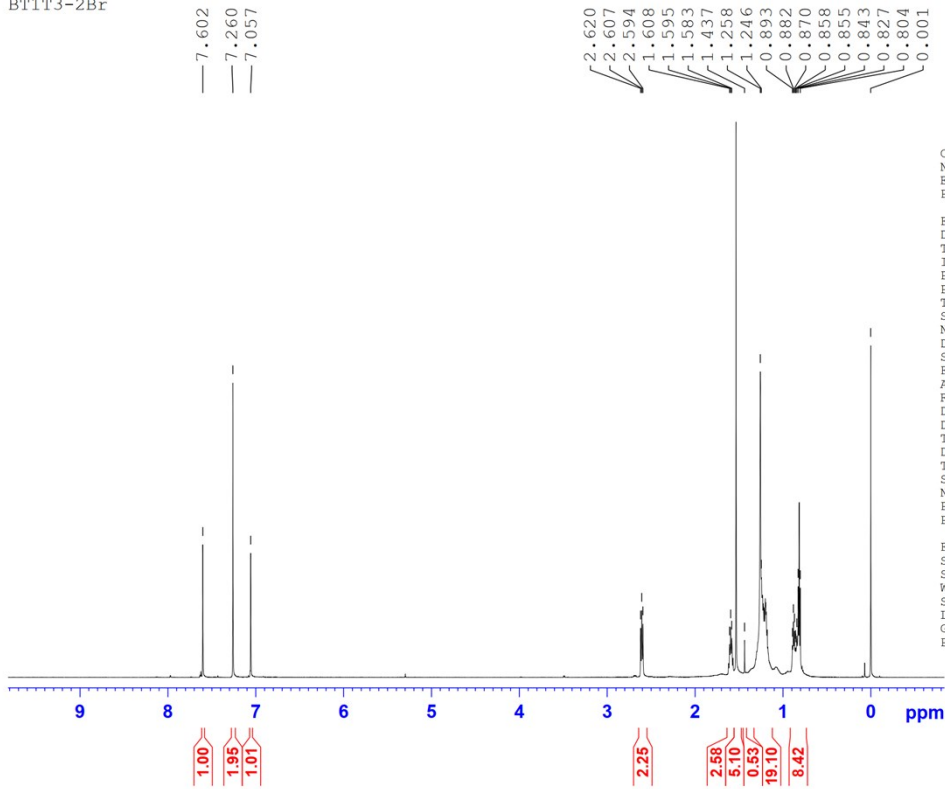
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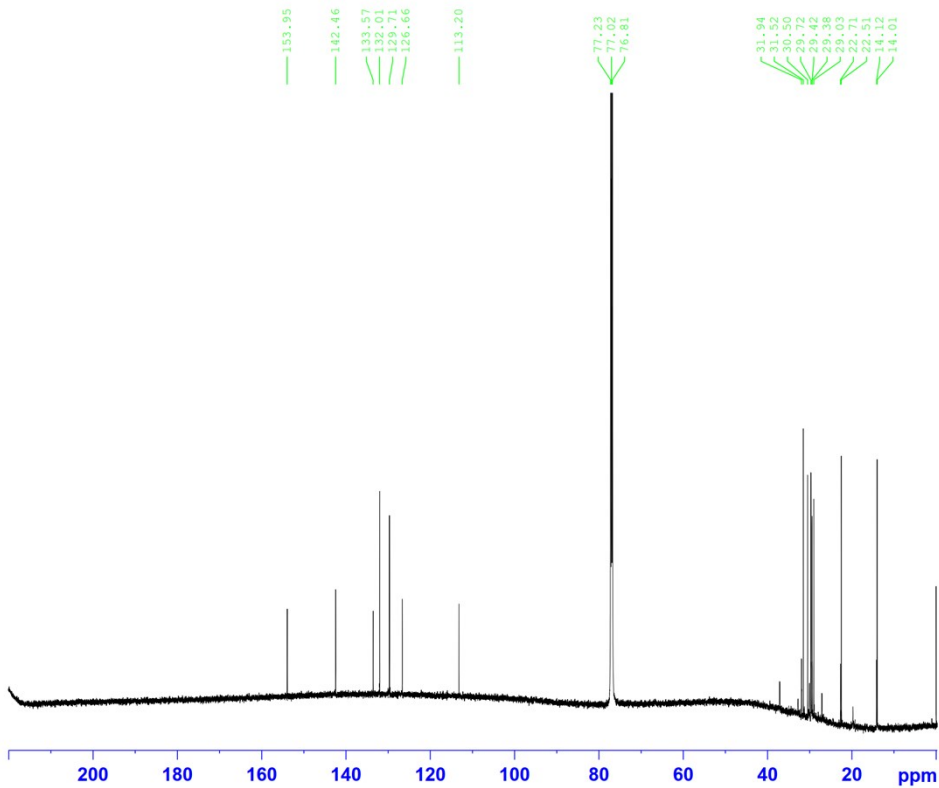
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F2 - Processing parameters
SI       65536
SF       600.1300148 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```

BT1T3-2Br in CDCl3



```

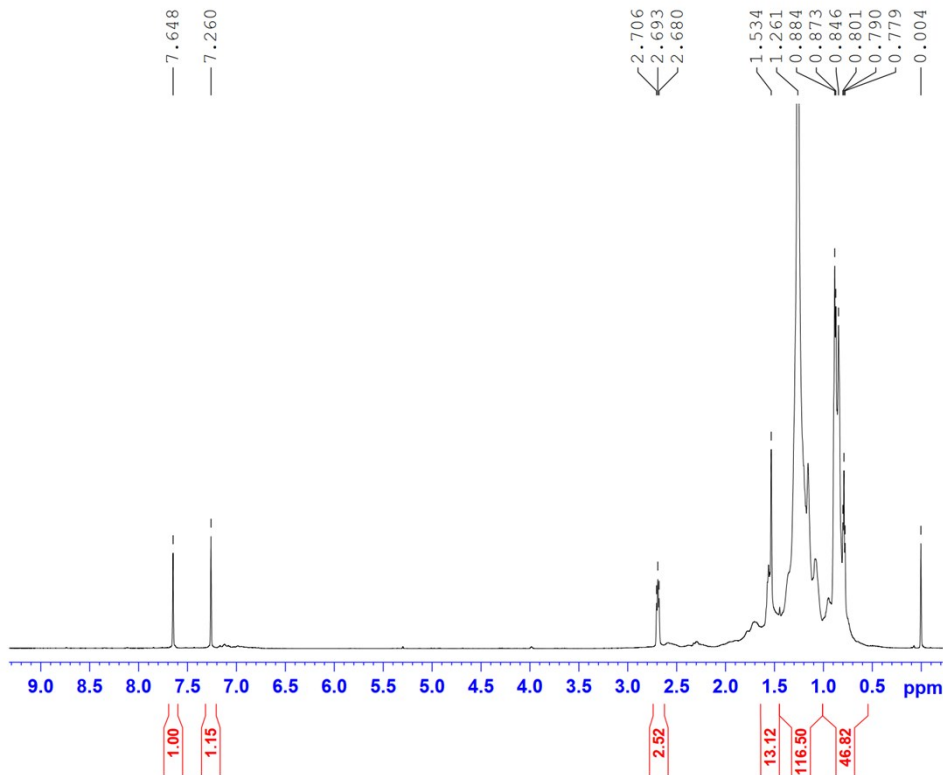
Current Data Parameters
NAME      BT1T3-2Br
EXPNO    3
PROCNO   1

F2 - Acquisition Parameters
Date_    20160308
Time     19.58 h
INSTRUM spect
PROBHD   Z115435_0005 (
PULPROG zgpg30
TD       65536
SOLVENT  CDCl3
NS       1024
DS       2
SWH      36231.883 Hz
FIDRES   1.105709 Hz
AQ       0.9043968 sec
RG       191.96
DW       13.800 usec
DE       18.00 usec
TE       303.2 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
SF01     150.9178988 MHz
NUC1     13C
P1       10.50 usec
PLW1     26.00000000 W
SF02     600.1324005 MHz
NUC2     1H
CPDPRG2 waltz16
PCPD2    70.00 usec
PLW2     20.00000000 W
PLW12    0.29490000 W
PLW13    0.14833000 W

F2 - Processing parameters
SI       32768
SF       150.9028058 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Compound 4

BT1T3-4Br in CDCl3

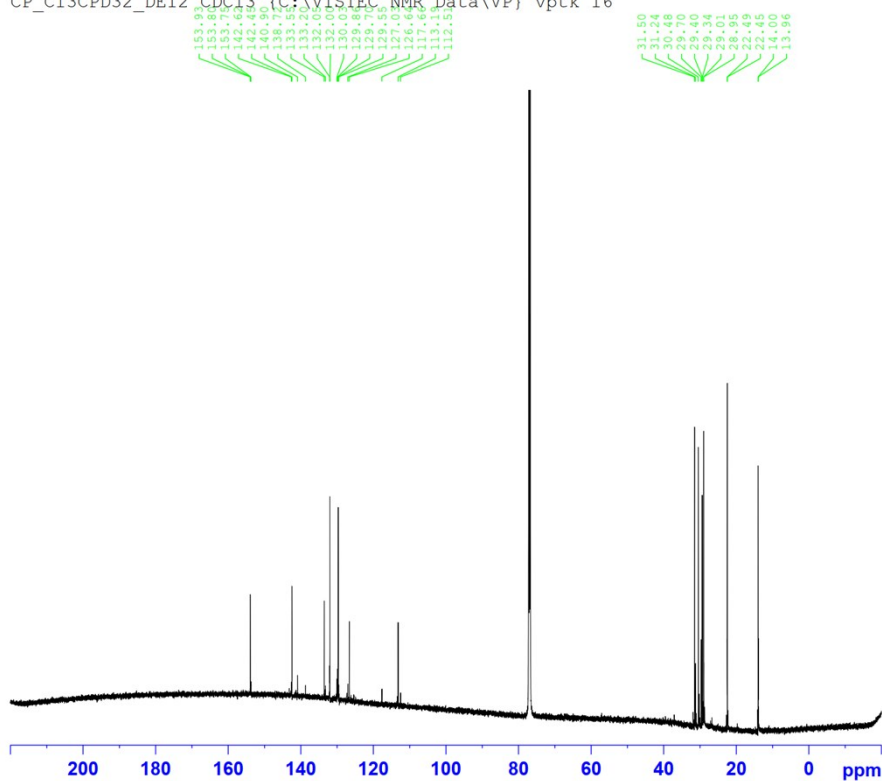


```
Current Data Parameters
NAME      BT1T3-4Br
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20160317
Time     21.37 h
INSTRUM  spect
PROBHD   Z115435_0005 (
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       32
DS       2
SWH      12019.230 Hz
FIDRES   0.366798 Hz
AQ       2.7262976 sec
RG       59.53
DW       41.600 usec
DE       40.00 usec
TE       303.2 K
D1       1.00000000 sec
TD0      1
SFO1     600.1337058 MHz
NUC1     1H
P1       8.50 usec
PLW1     20.00000000 W

F2 - Processing parameters
SI       65536
SF       600.1300143 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```

CP_C13CPD32_DE12 CDCl3 (C:\VISTEC NMR Data\VP} vptk 16

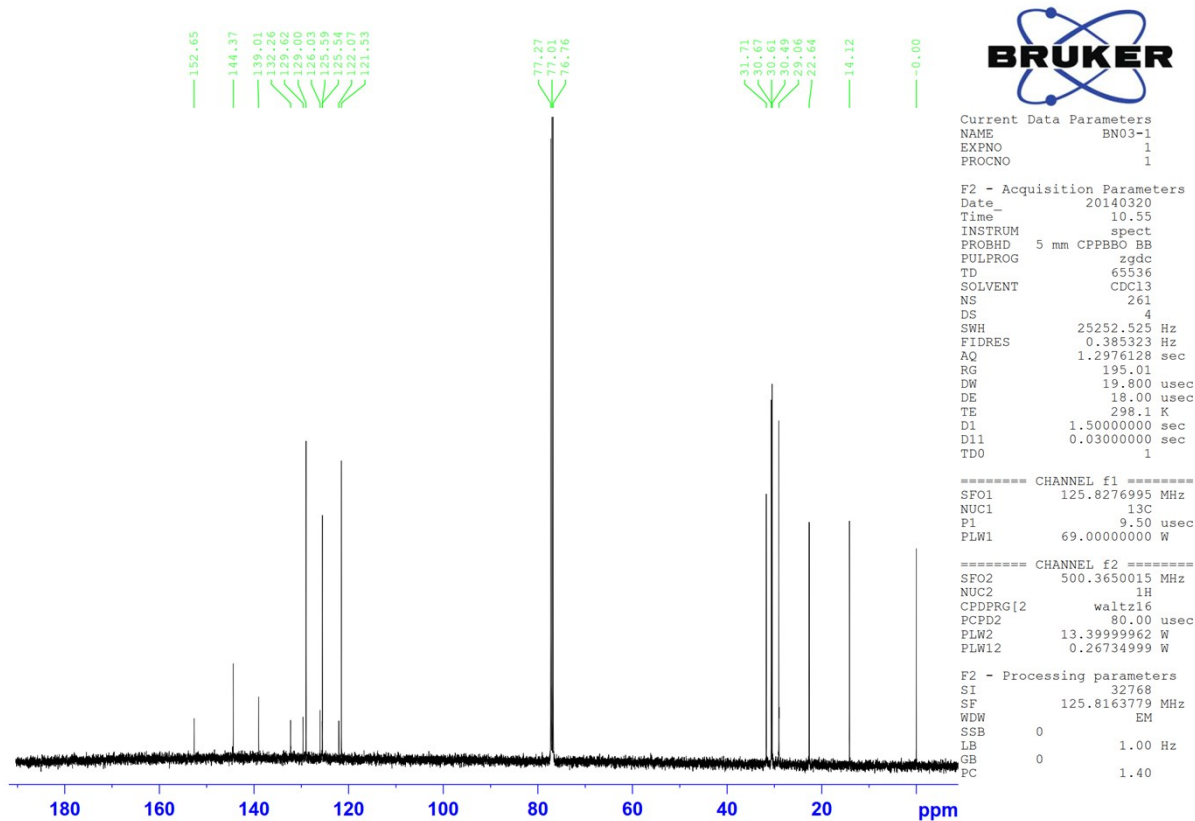
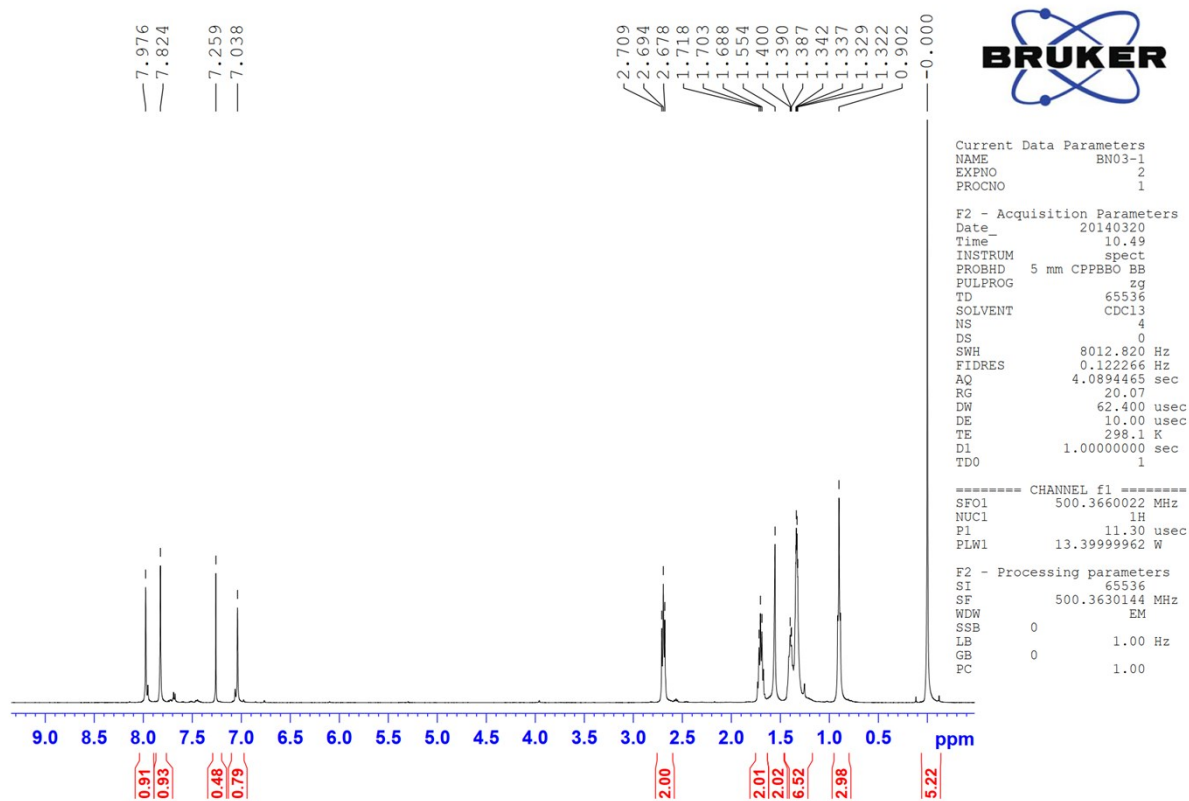


```
Current Data Parameters
NAME      BT1T3-4Br
EXPNO    10
PROCNO   1

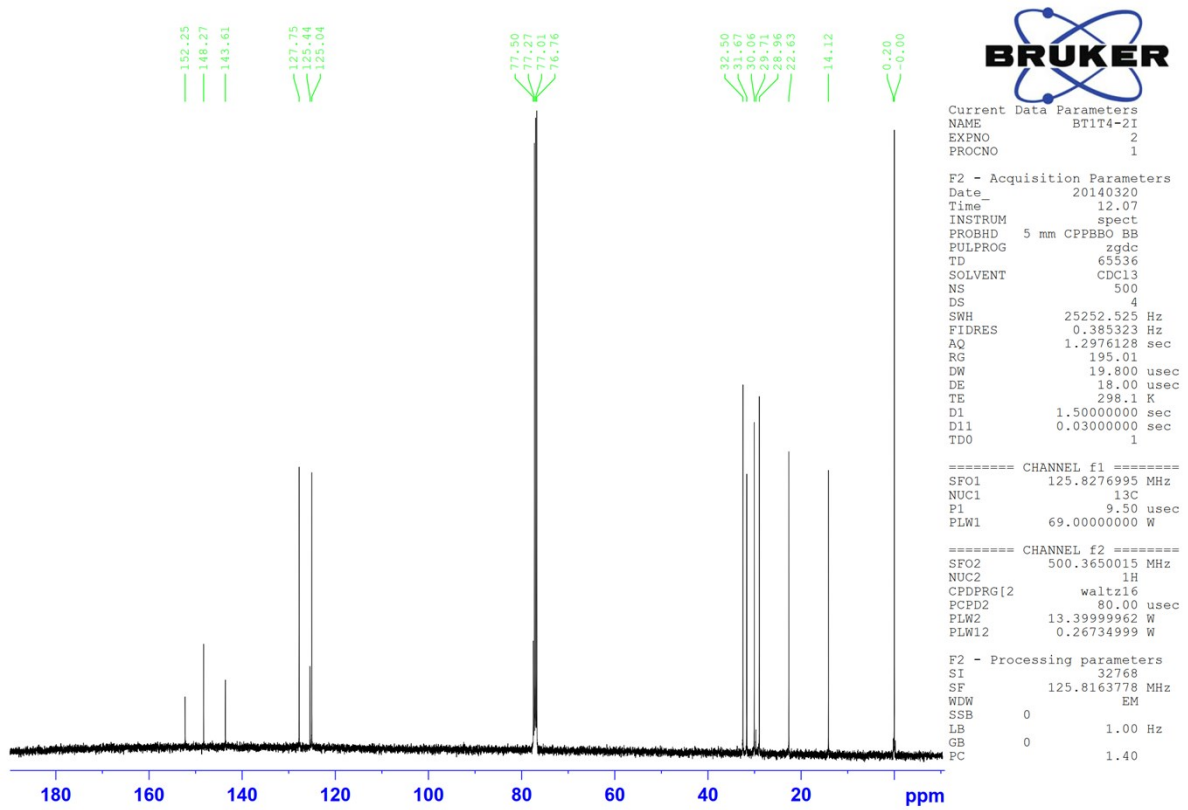
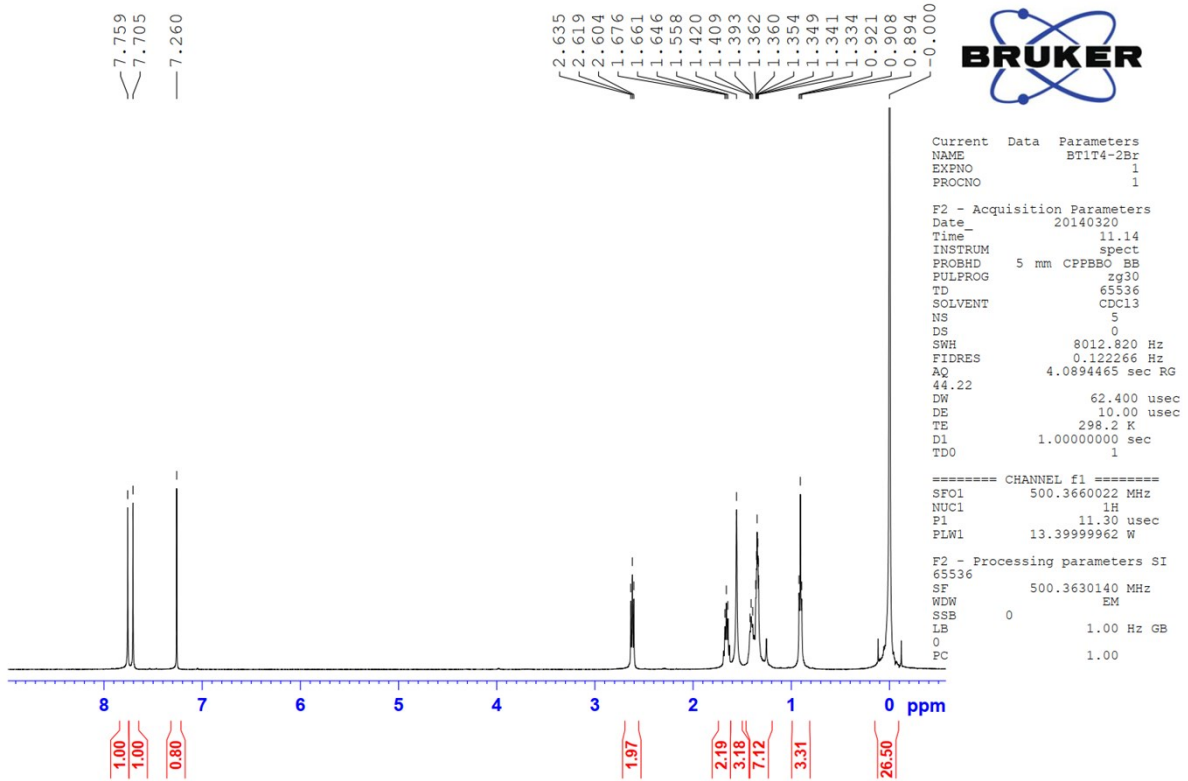
F2 - Acquisition Parameters
Date_    20180321
Time     17.08 h
INSTRUM  spect
PROBHD   Z115435_0005 (
PULPROG  zpgp30
TD       65536
SOLVENT  CDCl3
NS       512
DS       2
SWH      36231.883 Hz
FIDRES   1.105709 Hz
AQ       0.9043968 sec
RG       191.96
DW       13.800 usec
DE       18.00 usec
TE       303.2 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
SFO1     150.9178988 MHz
NUC1     13C
P1       11.00 usec
PLW1     26.00000000 W
SFO2     600.1324005 MHz
NUC2     1H
CPDPRG[2] waltz16
PCPD2    70.00 usec
PLW2     20.00000000 W
PLW12    0.33061001 W
PLW13    0.16630000 W

F2 - Processing parameters
SI       32768
SF       150.9028085 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

Compound 5



Compound 6



Compound 7

