Supplementary Information

Organic Molecular Tessellations and Intertwined Double Helices Assembled by Halogen Bonding

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Content

¹H NMR spectrum of L1

L1

2.000

9   8   7   6   5   4   3   2   1   ppm

— 8.6874

— 3.3332

⌐ 2.5071
├ 2.5036
┤ 2.5000
├ 2.4964
└ 2.4930

¹³C NMR spectrum of L1

— 147.5058

— 137.3152

— 90.2827

⌈ 40.0211
⌈ 39.8542
⌈ 39.6871
— 39.5200
⌊ 39.3530
⌊ 39.1860
⌊ 39.0192
⌊ 28.7041

Current Data Parameters
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P1   10.00 usec
PLW1   61.00000000 W
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NUC2   1H
CPDPRG[2   waltz16
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F2 - Processing parameters
SI   32768
SF   125.7578466 MHz
WDW   EM
SSB   0
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PC   1.40

ESI mass spectrum of L1

380.83862

381.84239

383.20460

m/z

¹H NMR spectrum of L2

2.000

9 8 7 6 5 4 3 2 1 ppm

L2

— 8.6909

— 3.3351

— 2.5000
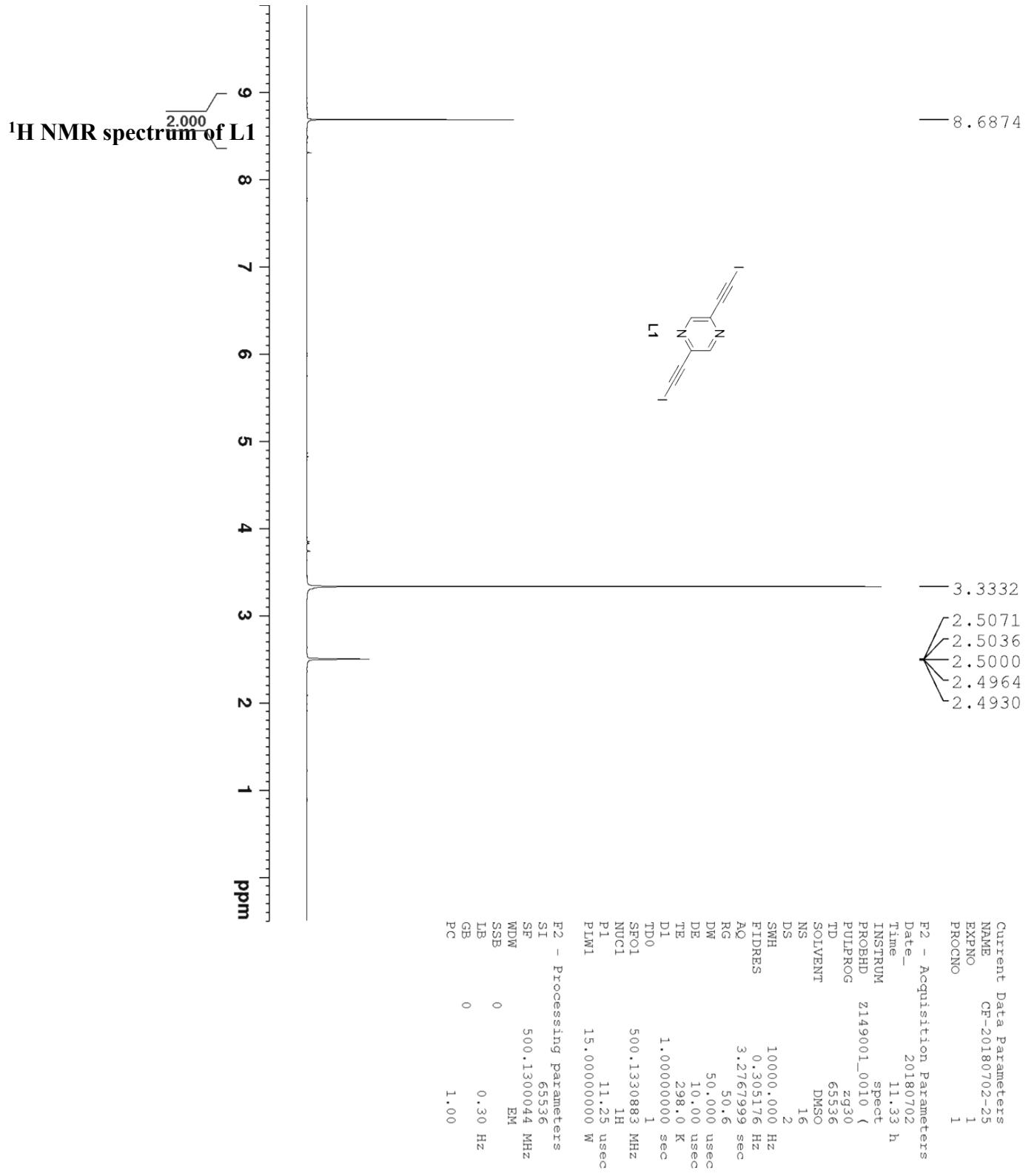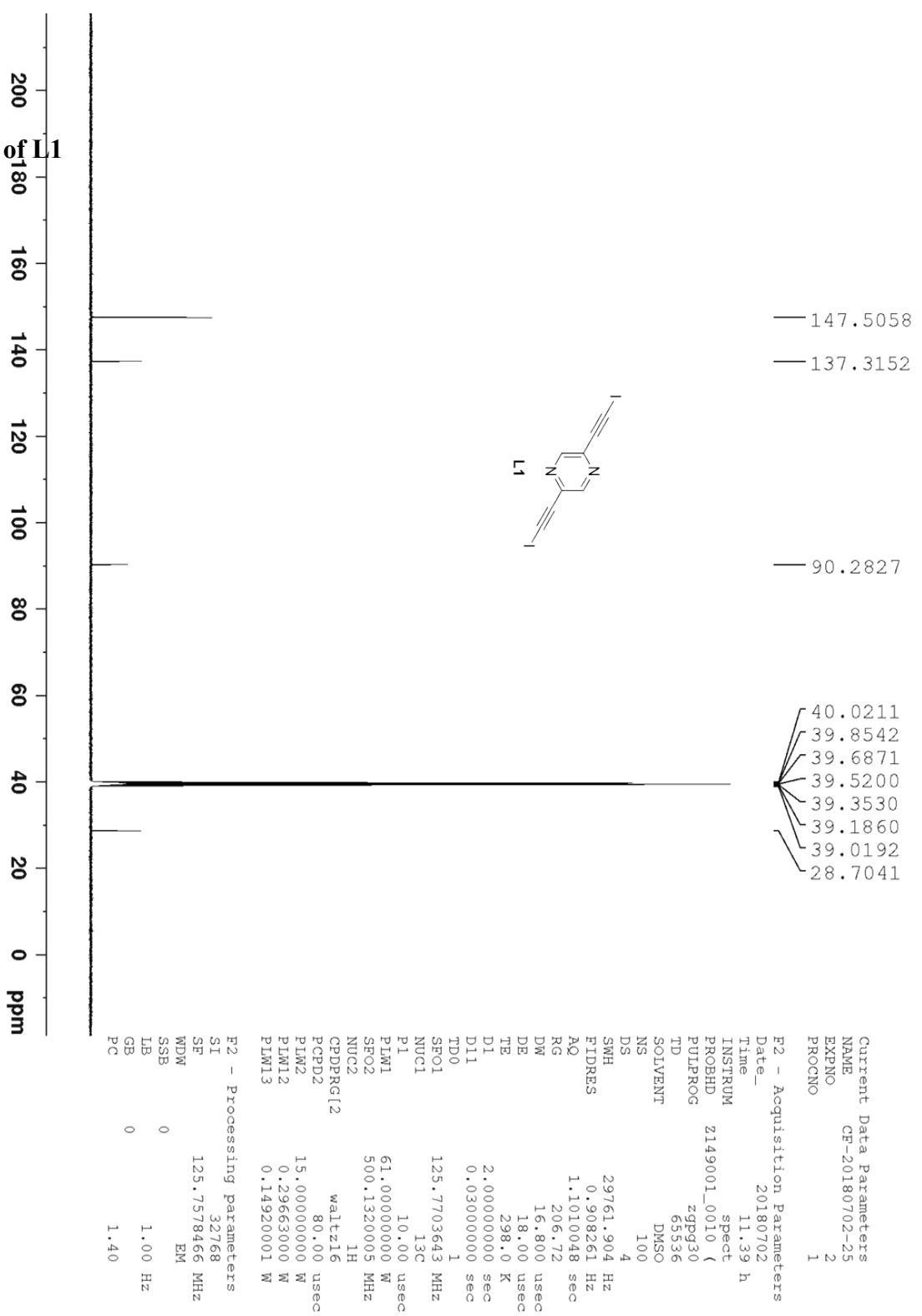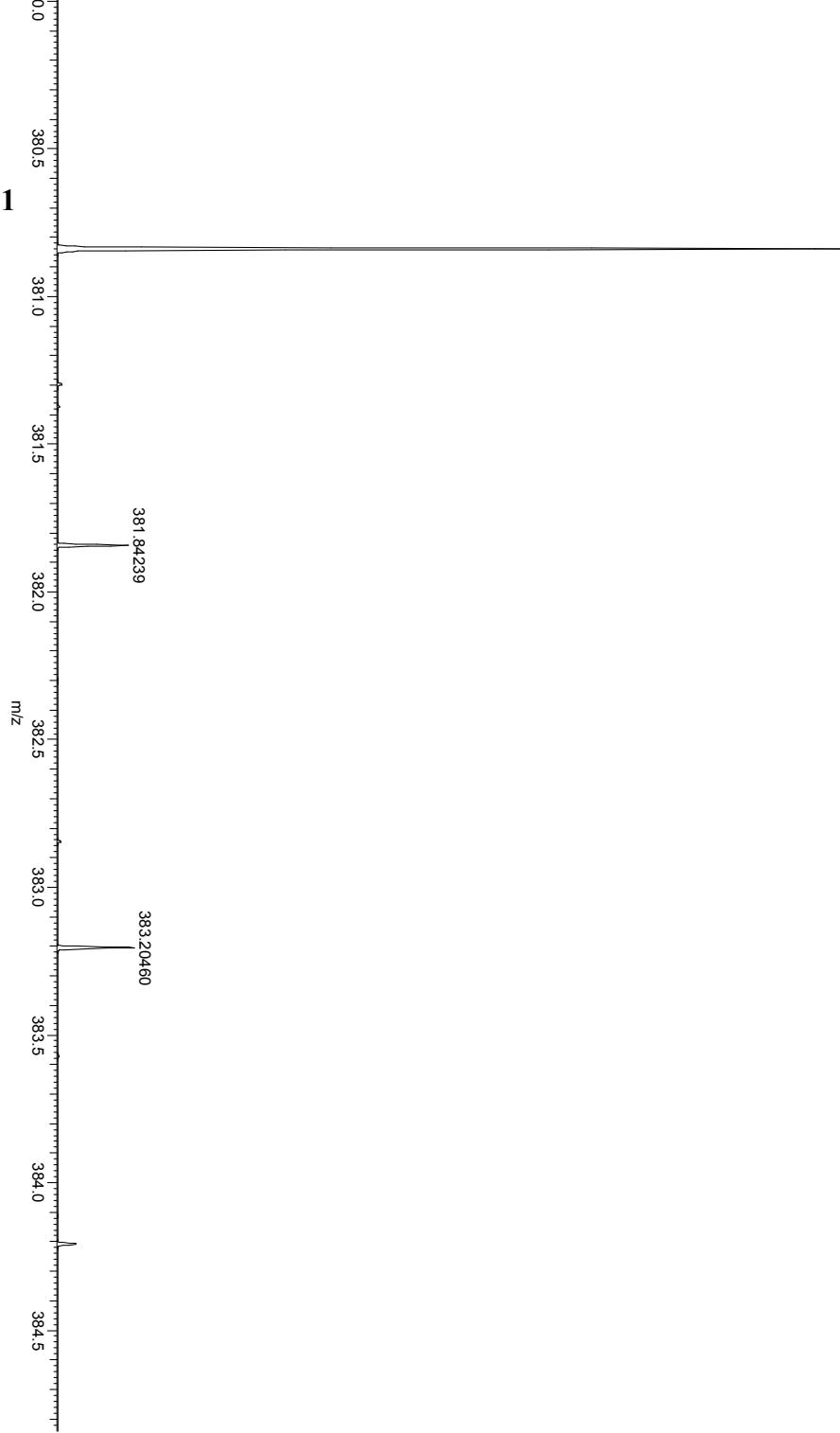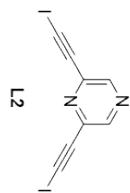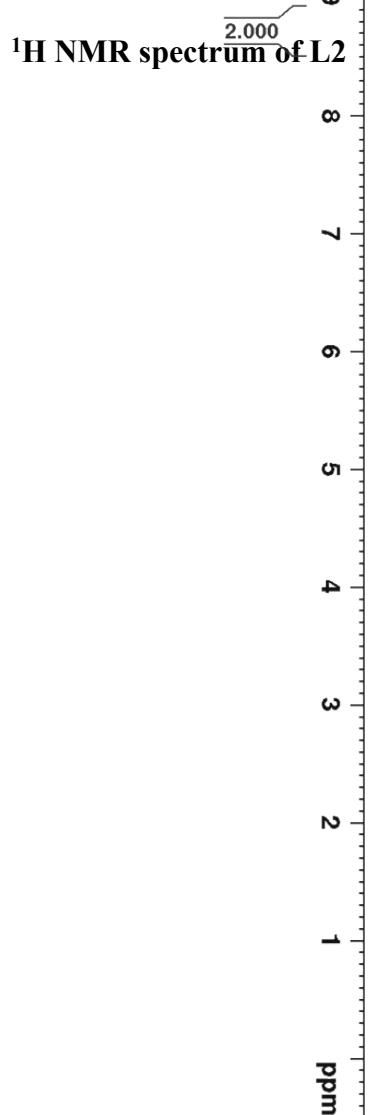
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RG   30.85
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NUC1   1H
P1   11.25 usec
PLW1   15.00000000 W

F2 - Processing parameters
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WDW   EM
SSB   0
LB   0.30 Hz
GB   0
PC   1.00

¹³C NMR spectrum of L2

— 146.5571
— 138.5036

— 89.6864

— 40.0205
— 39.8537
— 39.6868
— 39.5200
— 39.3531
— 39.1862
— 39.0192
— 27.0196
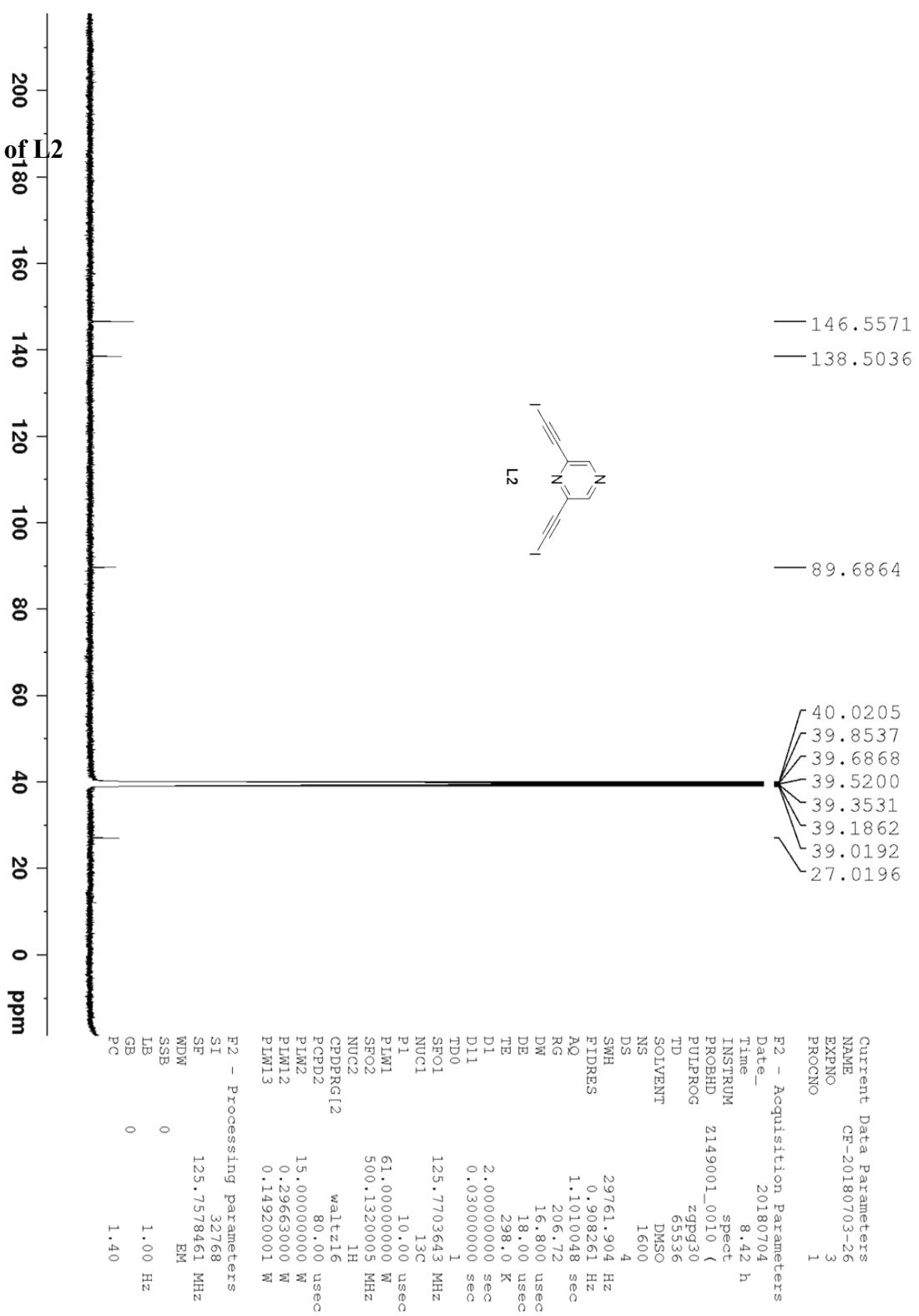
ESI mass spectrum of L2

380.83867

381.84207

m/z
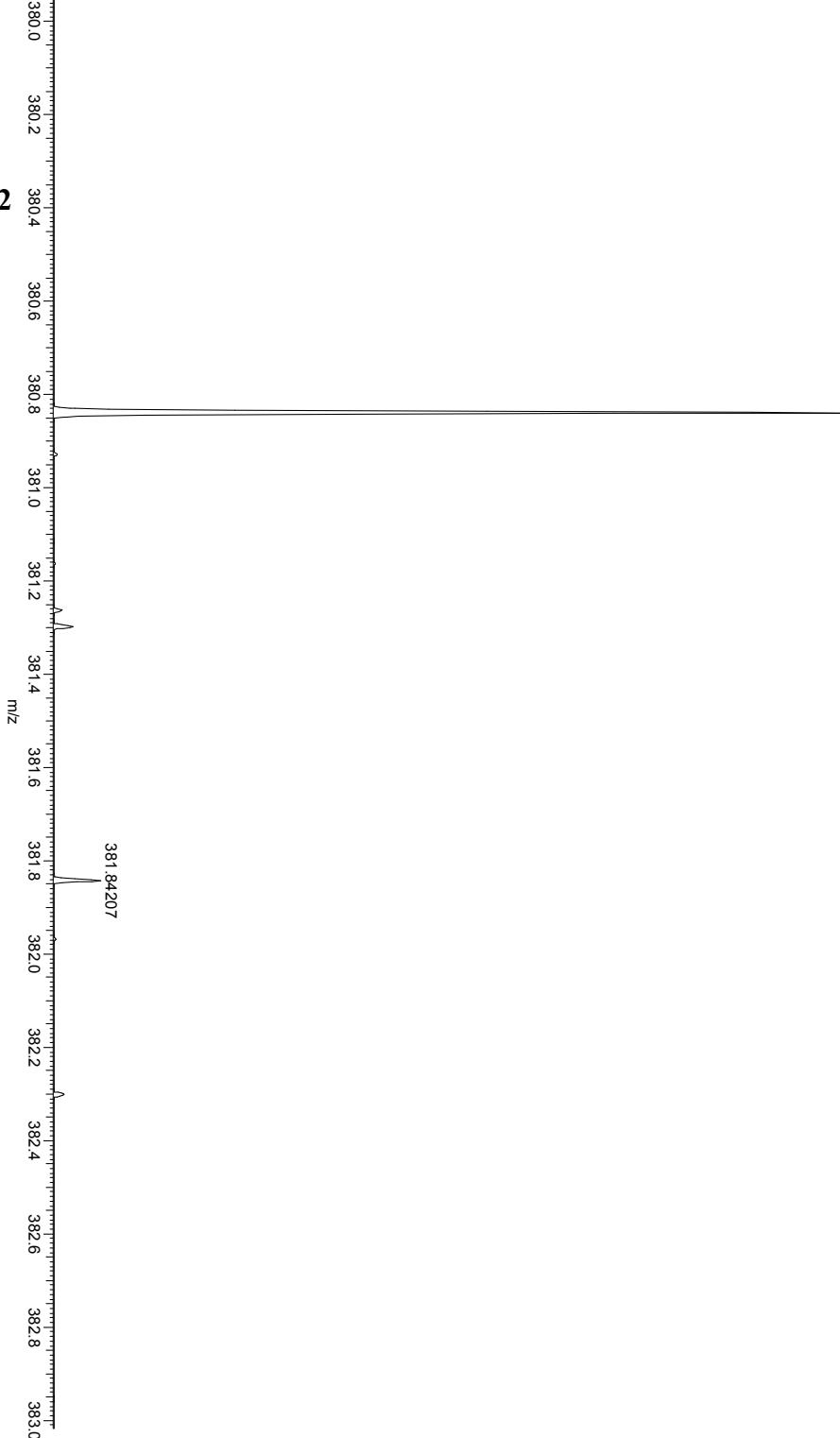
X-ray Crystallography

Crystallographic data and structure refinement parameters for crystalline phases **L1a−L3d** are given in Table 1 below:

Table 1. Crystallographic data and least-squares refinement parameters for crystals **L1a−L2c**.

	L1a	L1b	L2a	L2b	L2c
Molecular formula	$C_8H_2I_2N_2 \cdot 2.6(CH_3CN)$	$C_8H_2I_2N_2 \cdot CHCl_3$	$C_8H_2I_2N_2 \cdot 0.6(CH_3CN)$	$C_8H_2I_2N_2 \cdot C_6H_5CH_3$	$C_8H_2I_2N_2$
Molecular weight	486.66	499.28	404.55	420.97	379.92
Solvent for crystallization	Acetonitrile	Chloroform	Acetonitrile	Toluene	Chloroform
Crystal system	Orthorhombic	Monoclinic	Triclinic	Triclinic	Monoclinic
Space Group	$Pca2_1$(No. 29)[S1]	$P2_1/c$ (No. 14)	P-1 (No. 2)	P-1 (No. 2)	$P2_1/c$ (No. 14)
Temperature (K)	298	298	298	298	173
a / Å	17.5338(8)	4.338(1)	4.3229(14)	4.2425(6)	13.9441(6)
b/ Å	10.4983(5)	18.327(4)	13.047(4)	23.537(4)	35.2209(15)
c/ Å	28.7697(11)	18.018(4)	29.586(9)	24.567(4)	8.2172(3)
α / °	90	90	90.190(8)	111.725(5)	90
β / °	90	91.705(7)	91.770(8)	94.131(5)	90.331(1)
γ / °	90	90	95.024(8)	93.861(5)	90
V / Å^3	5295.8(4)	1431.9(6)	1661.4(9)	2261.3(6)	4035.6(3)
Z	16	4	6	6	16
D_c (g/cm^3)	2.442	2.316	2.426	1.855	2.501
F(000)	3635.0	912.0	1099.0	1152.0	2720.0
R_{int}	0.0389	0.0676	0.0703	0.0699	0.0319
GOF	1.098	1.294	1.207	1.045	1.107
Measured reflection	108372	15630	39069	74965	79182
Independent reflection	9023	2584	5978	8134	7304
R_1 ($I > 4\sigma(I)$)[a]	0.0424	0.1024	0.0976	0.1002	0.0354
R_1 (all data)	0.0552	0.1155	0.1134	0.1182	0.0401
wR_2[b]	0.1122	0.2683	0.1958	0.2690	0.0856
CCDC number	1861306	1861307	1861308	1861309	1861310

a: $R_1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$, b: $wR_2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma w(F_o^2)^2]^{1/2}$

Geometric Parameter of Crystals

Crystal	Bond[i]	Distance I····N d (Å)	Angle C—I····N (deg)	Relative distance[ii,iii] R_{IN}
L1a	C24a—I6a···N5b	2.971(12)	178.46(61)	0.84
	C17c—I5c···N6a	2.948(13)	174.74(54)	0.84
	C16a—I4a···N4c	2.944(13)	177.34(49)	0.83
	C32a—I7a···N7b	2.916(12)	175.39(55)	0.83
	C8d—I2d···N1b	2.888(11)	173.94(56)	0.82
	C9b—I3b···N3a	2.880(14)	177.21(67)	0.82
	C1c—I1c···N2d	2.862(14)	177.92(55)	0.81
	C25c—I8c···N8a	2.853(12)	174.11(48)	0.81
L1b	C5e—I1e···N1f	2.948(20)	167.54(72)	0.84
	C1g—I2g···N2e	2.875(17)	174.89(78)	0.81
L2a	C24i—I6i···N2h	2.986(15)	176.21(73)	0.85
	C16h—I4h···N5j	2.957(16)	176.26(62)	0.84
	C1k—I1k···N4h	2.939(14)	177.21(60)	0.83
	C9h—I3h···N1f	2.860(16)	179.48(62)	0.81
L2b	C16h—I4h···N5j	2.993(12)	179.07(50)	0.85
	C24l—I6l···N2h	2.979(14)	177.42(60)	0.84
	C8h—I2h···N3m	2.956(10)	177.65(53)	0.84
	C17e—I5e···N4h	2.920(12)	177.64(60)	0.83
	C1h—I1h···N6n	2.868(14)	177.81(63)	0.81
	C9j—I3j···N1h	2.867(14)	178.08(60)	0.81
L2c	C18o—I11o···N8h	3.001(51)	172.67(23)	0.85
	C65h—I1h···N5h	2.979(48)	177.74(22)	0.84
	C40h—I6h···N4g	2.922(56)	173.86(22)	0.83
	C55p—I3p···N7h	2.865(54)	171.71(22)	0.81
	C1q—I7q···N1h	2.823(54)	177.11(23)	0.80
	C67h—I8h···N3r	2.820(57)	169.31(23)	0.80

i: Symmetry code:

a: x, 1+y, 1+z

b: -1/2+x, -y, 1+z

c: -1/2+x, 1-y, 1+z

d: x, y, 1+z

e: 1-x, 1-y, 1-z

f: -x, 1-y, 1-z

g: -1+x, 1/2-y, 1/2+z

h: x, y, z

i: x, -1+y, z

j: -1+x, y, z

k: -1+x, 1+y, z

l: 3-x, -y, 1-z

m: 2-x, -y, 1-z

n: 1+x, y, 1+z

o: 1+x, y, z

p: 2-x, -y, 2-z

q: 1-x, -1/2+y, 3/2-z

r: 1-x, -y, 1-z

ii: Relative distance $R_{IN} = d/(r_I + r_N)$, see reference S2.
iii: Van der Waals radii $r_I = 1.98$ $r_N = 1.55$ Å, see reference S3.

References

S1. Attempt of structural refinement in the centrosymmetric space group *Pbcn* (No. 60) did not give meaningful solution while the space group *Pca*2_1 (No. 29) give reasonable structure.

S2. J. P. M. Lommerse, A. J. Stone, R. Taylor, F. H. Allen, *J. Am. Chem. Soc.* 1996, **118**, 3108–3116.

S3. A. Bondi, *J. Phys. Chem.* 1964, **68**, 441–451.